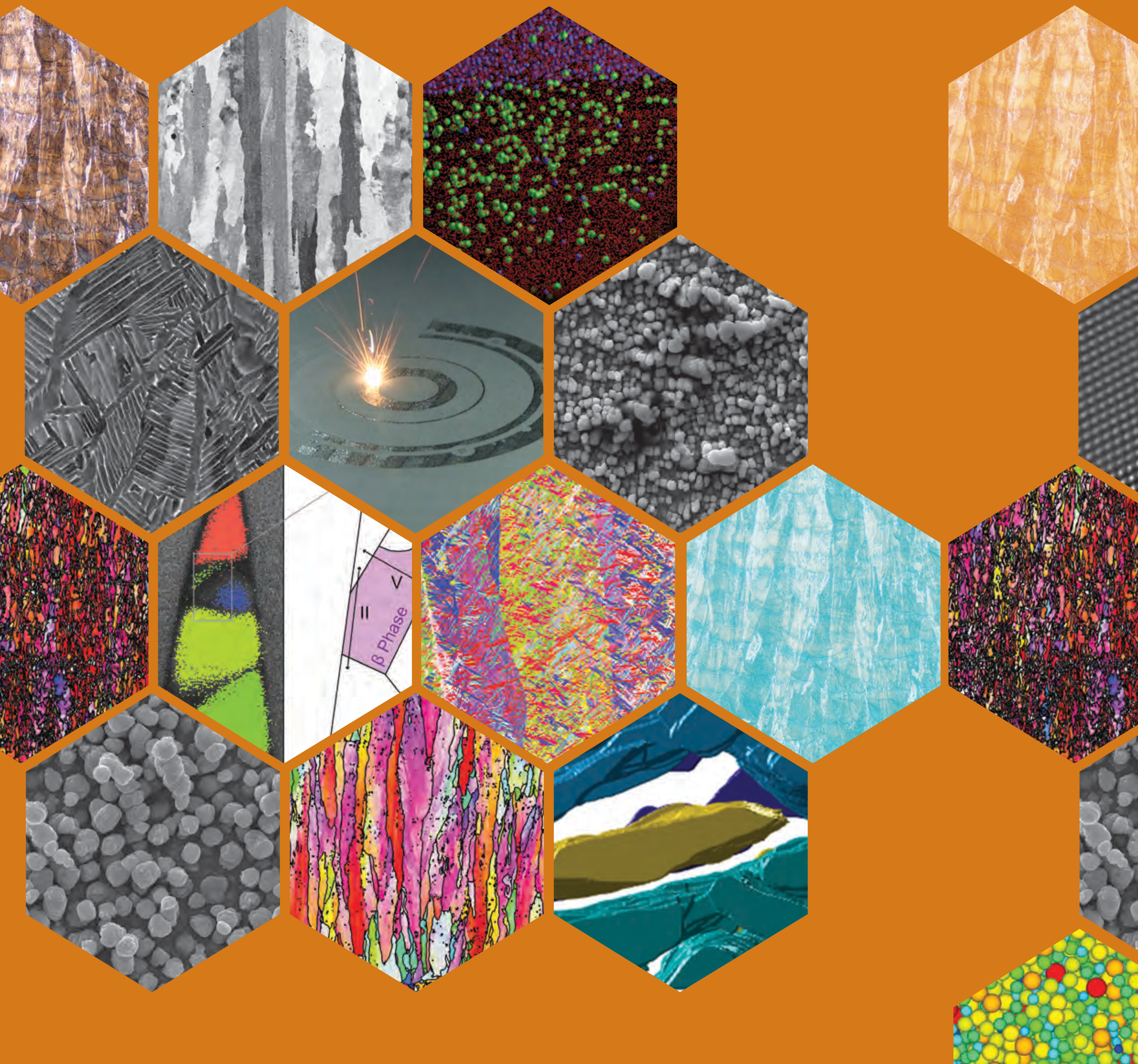
FRONTIERS IN ADDITIVE MANUFACTURING

MURI-AUSMURI PROJECT
FINAL REPORT (2018-2024)



MURI
AUSMURI

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MURI-AUSMURI PROJECT
FINAL REPORT (2018-2024)

THE UNIVERSITY OF TENNESSEE KNOXVILLE

THE UNIVERSITY OF SYDNEY

IOWA STATE UNIVERSITY OF SCIENCE AND TECHNOLOGY

THE OHIO STATE UNIVERSITY

VIRGINIA TECH

UNSW THE UNIVERSITY OF NEW SOUTH WALES

COLORADO SCHOOL OF MINES

UC SANTA BARBARA

Australian Government
Department of Defence
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Office of Naval Research Science & Technology

Australian Government
Department of Industry, Innovation and Science

We recognise and pay respect to the Elders and communities – past, present, and emerging – of the lands Australian University campuses contributing to this research stand on. For thousands of years they have shared and exchanged knowledges across innumerable generations for the benefit of all.

Table of Contents

1. Executive Summary

We are delighted to present this final report on the research efforts of an outstanding global research team focussed on rationalising liquid/solid and solid/solid interphase instabilities during the thermo-mechanical transients of metal additive manufacturing (AM). This document has been prepared as a penultimate milestone for our final review meeting to be held in USA in March 2024. The structure of the MURI+AUSMURI program and the project details are articulated below.

One of the most exciting aspects of this program is that it represents a rare opportunity for researchers to work on a globally structured R&D initiative. More precisely, we were selected and entrusted to pioneer this global program and sustain in the last six years! Within days of the excitement surrounding the initial MURI funding announcement, in what is an enormously competitive selection process, the teams were gearing up to travel to Washington D.C. for planning meetings, kick-off discussions and the various follow-ups and breakout meetings. To the great credit of this team, we immediately struck a strong rhythm in terms of our fortnightly meetings and other satellite meetings. Establishing a forum for team troubleshooting as issues presented, and a safe-space for critiquing each other's work was critical to the success of the program. Because AM has taken the field of materials science "off road", operating so very far from equilibrium and in conditions previously impossible to generate, this research team have had cause to visit some contentious spaces in the field. Therefore, the team dynamic has been key. In essence, the PIs worked together to coach their teams to generate a culture that was inclusive, collegial, fun, and yet one whereby each has held each other accountable.

Pioneering a global R&D program amidst the breakout of a global pandemic was a daunting challenge. Fortunately, at the onset of the COVID-19 pandemic in 2020, we had already established a strong momentum, research rhythm and research culture. To the further great credit of the team, the research collaborations continued largely as planned, with skilful pivots as logistics demanded. For us, this is a great testimony to the power of making a purposeful and strong start from the very outset of the program.

These pages summarise the project details, reflect highlights and contain summaries and links to the more substantive findings. All of this is mapped against the key questions posed in the program during Phase 1 and Phase 2. The highly significant networking arising from this program is also detailed, and we have chosen to summarise this in terms of industry networks and capability networks. Based on the quality and quantity of these research outputs and the networks generated from the program, we trust that you will agree this initiative is set to deliver important scientific, economic, and defense research outcomes. The success of this MURI and AUSMURI collaborative research can be understood by the study of emerging career trajectories of researchers moving back and forth between Australia and USA, as well as the launch of companies that is building on the fundamental science developed throughout the program.

On behalf of the entire global team of researchers, we want to acknowledge very gratefully the advice, support, and guidance from Dr. Jennifer Wolk (US Office of Naval Research), and Zoran Sterjoski (Australian Defence Science and Technology). We are especially grateful for the way that you challenged us as we navigated the frontiers of this exciting research field!

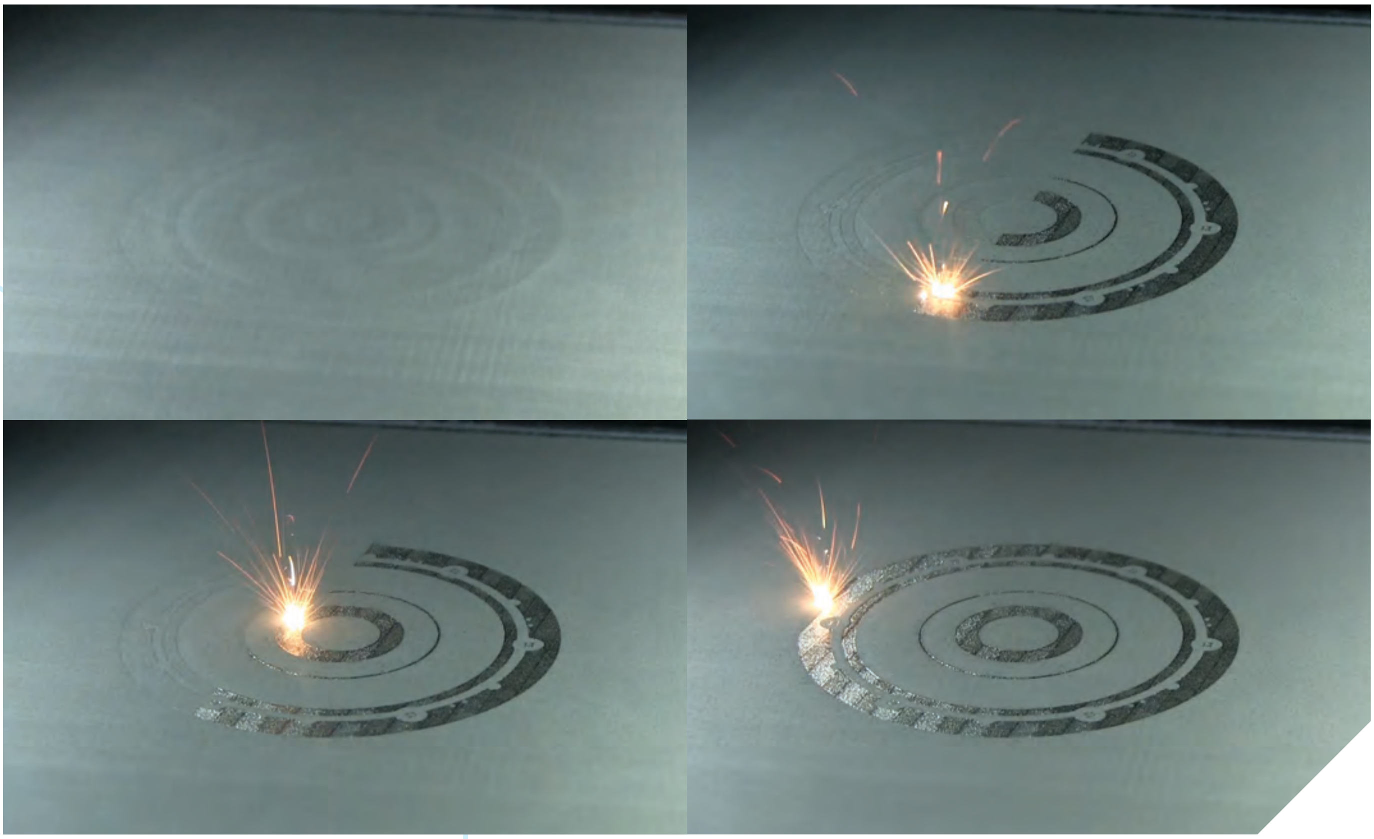
Please contact sbabu@utk.edu and simon.ringer@sydney.edu.au for any further information related to the contents of this report and thank you for your interest in this MURI+AUSMURI project.

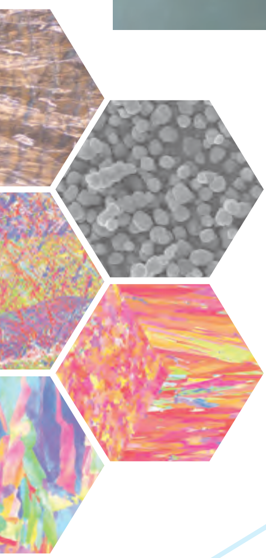
Sudarsanam Suresh Babu
Fellow of ASM, AAAS and AWS

Governors Chair of Advanced Manufacturing, University of Tennessee-Knoxville, and Oak Ridge National Laboratory USA

Simon P. Ringer,
FIEAust, FRSN, FTSE

Professor of Materials Science and Engineering The University of Sydney Australia.





2. Brief Overview- MURI-AUSMURI Project

Project details

AUSMURI is an agreement between the US Department of Defense (DoD) and the Australian Department of Defence (Defence), wherein the Australian Government offers grant funding to Australian higher education providers (universities) to collaborate with US universities in the US Multidisciplinary University Research Initiative (MURI). Australian grant funding is offered through the US–Australia International MURI project for topics determined by Defence as having high potential for significant future Defence capability. The MURI grant funds for the US universities and the AUSMURI grant funds for the Australian universities. MURI is sponsored by the Office of Naval Research (ONR). The initiative supports university research involving mixed disciplines in science and engineering, within a range of topics with high potential for future defence capability.

AUSMURI supports the Australian Government's commitment to

- strengthening Australian university research capacity, skills and global networks in research topics of priority to Defence future capabilities
- building international collaborative links in key research topics of mutual defence interest for improved efficiency and burden sharing.

The program forms part of the Next Generation Technologies Fund that was announced as part of the 2016 Defence White Paper.

The program's intended outcomes are as follows:

✓ Produce transformational research and innovation that leads to significant advancement of capabilities and knowledge that have potential to be game-changing for Defence capability

✓ Develop relationships and link US and Australian research strengths to build critical mass with new capacity for inter-disciplinary, collaborative approaches in fields of high potential for future Defence needs

✓ Build networks with US universities to strengthen research and achieve global competitiveness in fields of high potential for future Defence capabilities

✓ Build Australia's human capacity in a range of research areas of priority to Defence future capabilities by attracting and retaining, from within Australia and abroad, researchers of high international standing as well as the most promising research students which aligns with similar goals of US-MURI program

✓ Provide high quality postgraduate and postdoctoral training environments for the next generation of researchers to support the future capabilities of Defence.

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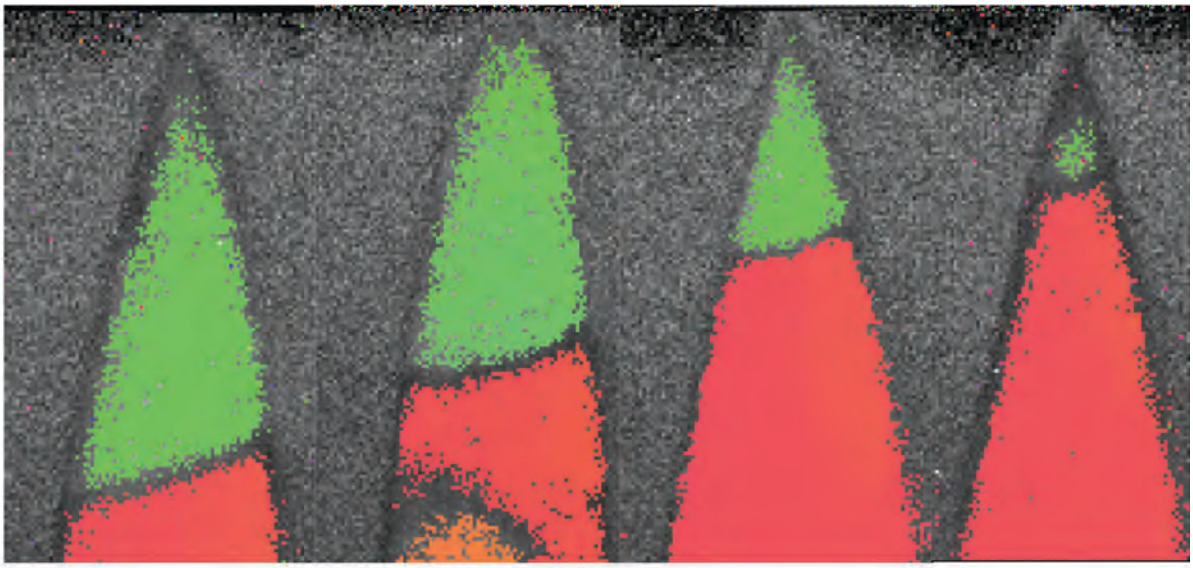
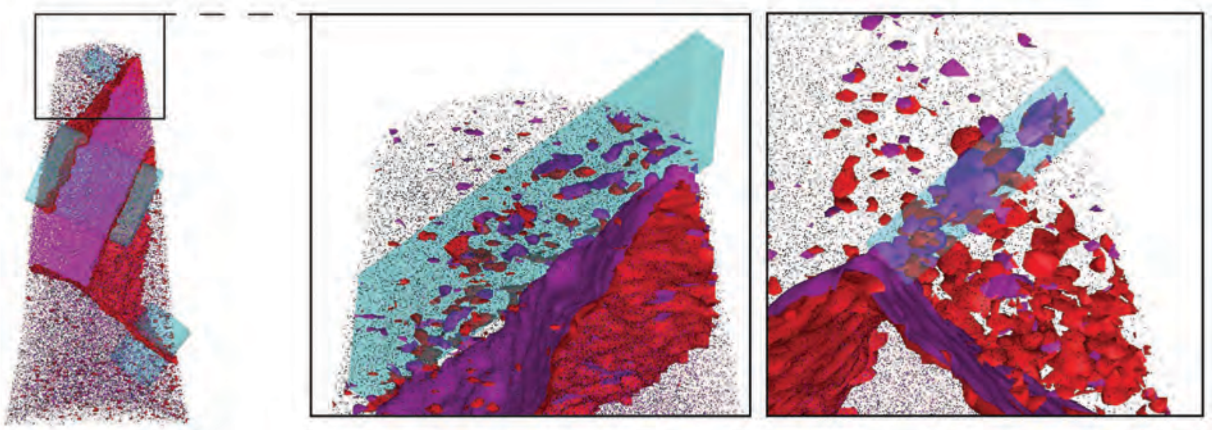
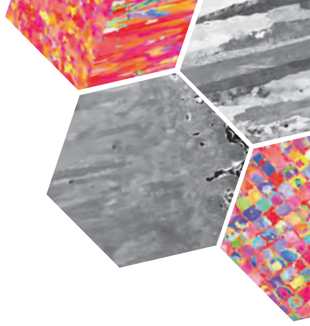
[1] https://business.gov.au/grants-and-programs/australia-us-international-multidisciplinary-university-research-initiative-ausmuri







3. MURI-AUSMURI Project

Project Background

Rationalization of Liquid/Solid and Solid/Solid Interphase Instabilities During Thermal-Mechanical Transients of Metal Additive Manufacturing, in short '3D Additive'

This grant was awarded as part of the successful MURI-AUSMURI competition in 2018. The project is administered under the MURI grant program by the US Department of Defense, Office of Naval Research and the Australian Department of Industry, Innovation and Science (now known as the Department of Industry, Science, Energy and Resources).

The project was selected based on a peer review process of a proposal submitted under MURI Topic #22, titled In-Situ Microstructural and Defect Evolution Below the Micron Scale in As Deposited Metal Alloys with the Award Number: N00014-18-1-2794. The companion AUSMURI PROJECT 00005 with the title "Understanding Additive Manufacturing of Advanced Metallurgical Alloys," was also approved in 2018. The project consortium comprises of eight Universities across the USA and Australia, as follows:

The University of Tennessee, Knoxville (UTK)

Colorado School of Mines (CSM)

Iowa State University (ISU)

The Ohio State University (OSU)

University of California, Santa Barbara (UCSB)

Virginia Polytechnic Institute and State University (VT)

The University of Sydney (USyd) and

The University of New South Wales (UNSW).

The MURI Lead Investigator (USA) is Professor Sudarsanam Suresh Babu (UTK), and the AUSMURI Lead Investigator (Australia) is Professor Simon P. Ringer (USyd).

The primary objective of this collaborative project was to perform fundamental research on additive manufacturing (AM). The impact of this fundamental research was sought to cut across all metal AM processes, including large-scale processes involving arc, plasma and laser, laser powder bed fusion, electron powder bed fusion, binder jet and metal jet processes. The research started with four basic overarching questions related to physical metallurgy of metals and alloys (Fig. 1). Our hypothesis was that some of the theories that were developed with zero thermal gradients may not be applicable for the physical processes that occur during additive manufacturing with large swings in thermal gyrations.

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Based on the research so far, we believe that physical metallurgy principles need to be modified for cyclic thermo-mechanical-chemical transients typical to that of additive manufacturing in different length - and time - scales. Evidence for the departure from traditional physical metallurgy principles were obtained in multiple alloy systems using tasks ranging from advanced processing, characterisation (ex-situ, in-situ) and computational modelling performed by a multi-disciplinary team (Fig. 1). The research has spanned various metallic alloys including Ti-6Al-4V, Inconel 738 superalloy, Cr-Co-Ni-Fe-Mn high entropy alloys, stainless steels and aluminium alloys and advanced ceramics all of which have potential applications in air, land, and maritime applications.

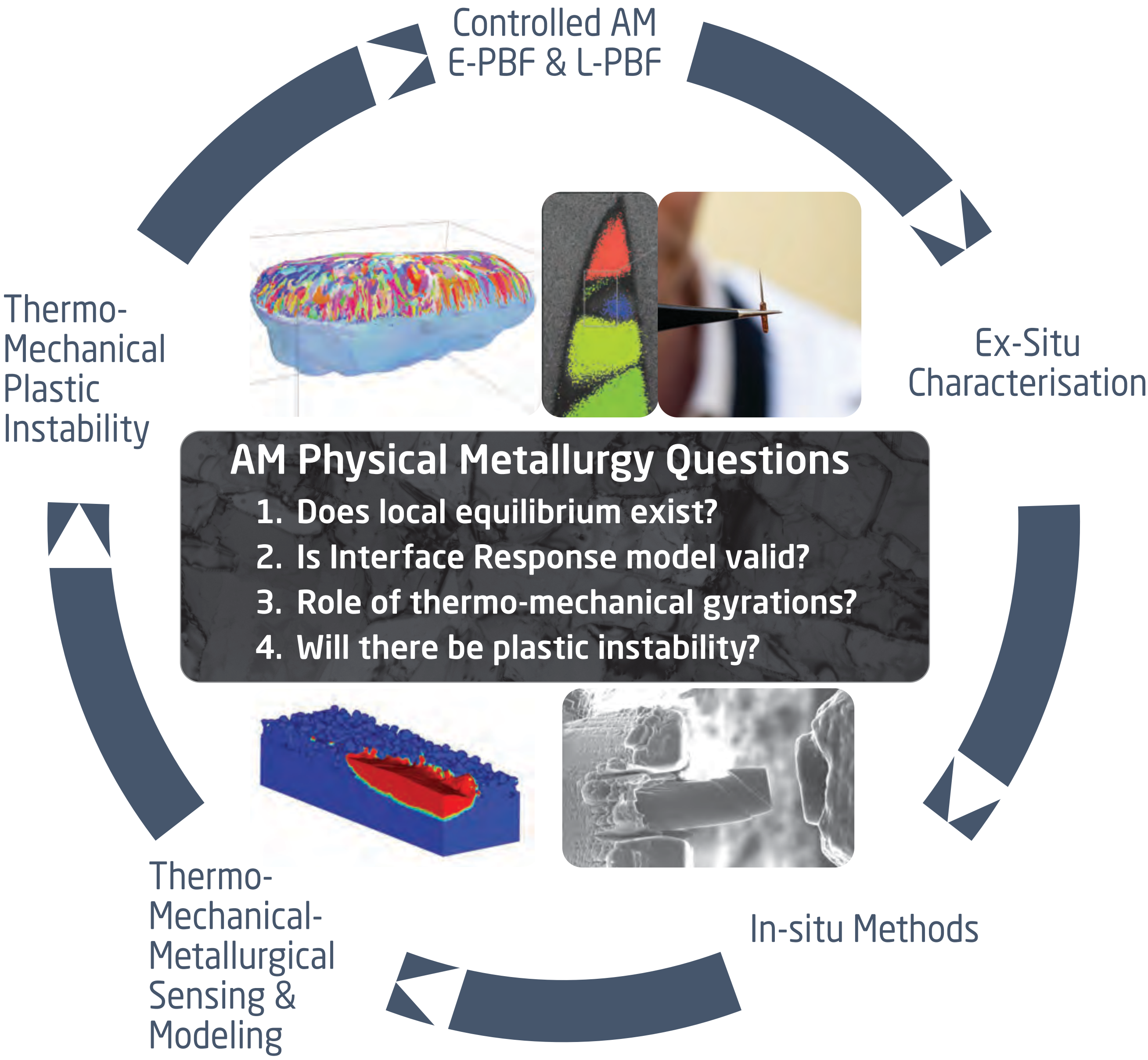


Figure 1: Overview of the approach used by the MURI and AUSMURI teams to answer the research questions

The MURI-AUSMURI team have fostered collaborative research across the world including universities in USA, Australia, and various, Research labs, DOE-User facilities, Australian Nuclear Science and Technology, Air Force Research Laboratory & industries. Knowledge dissemination activities are comprehensive and expanding rapidly. They are relevant to quantifying design strategies based on geometry-process-materials-properties-performance relationships. The team members and students on this project have received many fellowships and awards for demonstrating leadership and have been invited to serve in national level advisory roles, a testament to excellent and high-quality research outputs and service to community. The MURI and AUSMURI projects were granted a two-year extension in June 2021 to continue outstanding AM research in 2022-2023. The highly collaborative nature of the team has allowed us to endure the challenges faced by COVID-19. More details are presented in the following sections of the report.

The relevance of the AM to defence application is based on the following expectations. AM is set to produce transformational changes to the way that defence componentry is manufactured, both in the context of conventional production, and when deployed in the field. Dramatic enhancements to defence capability are anticipated with increased flexibility and dynamism in the management of component run/repair/replace decision-making, as well as the potential to deliver improved component performance and functionality. The potential to exploit complexity in the design of both the component and microstructure is significant. The ultimate benefit and relevancy of this project is to build knowledge-readiness in the form of case-studies that can be tapped for creation for critical materials and components for the defense needs of US and Australia.

PHASE 1

The Overarching Scientific Questions - phase 1

Additive Manufacturing discoveries suggest that there are many commonalities related to metallurgical phenomena observed during the traditional processes like casting, welding, powder metallurgy etc. However the steady state conditions for these processes are not valid for AM because of the cyclical nature of the thermal gradients in the energy delivery modes. Metal AM involves various phase transitions including powder to liquid, liquid to gas, gas to plasma, plasma to gas, gas to liquid, liquid to solid, and solid to solid. Due to the reasons previously mentioned, many scientific questions still remain unanswered with reference to ***liquid/solid (l/s)*** *and* ***solid/solid (s/s)*** interphases and plastic instabilities. This information is required to describe the residual stress, distortion and cracking during additive manufacturing of structural alloys. This work looks to address the deficiencies in the physical metallurgy of AM through various *in-situ* and *ex-situ* characterisation techniques, high end computational tools and integrating diverse expertise and infrastructure spread across the universities enabled through this highly collaborative funded project (see Fig.1). As a result, the project has attempted to address four overarching scientific questions:

Will there be a local-equilibrium at the interfaces with large thermal gradients?

How does interface motion respond to rapid reversals of thermal gradients?

Are phenomenological interface response functions valid for multi-component alloys?

Can we evaluate plastic instabilities within phases under complex thermal gyrations?

Figure. 3 Four key fundamental questions relevant to additive manufacturing that formed the background to the collaborative project

The impact of this fundamental research will be crosscutting across all the metal AM processes including large scale directed energy deposition (DED) processes involving arc, plasma and laser, laser powder bed fusion, and electron powder bed fusion and wire melting. Further details and the related publications against each of these questions are provided in the subsequent sections of this report.

The Overarching Scientific Questions - phase 2

Based on the 4 questions in Phase 1, the following 8 questions have been identified as the motivation for the research for the 2022-2024 period of research. Some are completed and many are ongoing.

5 How do we map the observed l/s and s/s interface instabilities on to high performance computational models?

6 What are the scientific limits of sensors and data fusion to describe the AM boundary conditions?

7 What are the roles of steady to transient thermal gradients in different length scale on s/s interface stability?

8 What is the role of cascading s/s interface stability due to sequential interaction of defects?

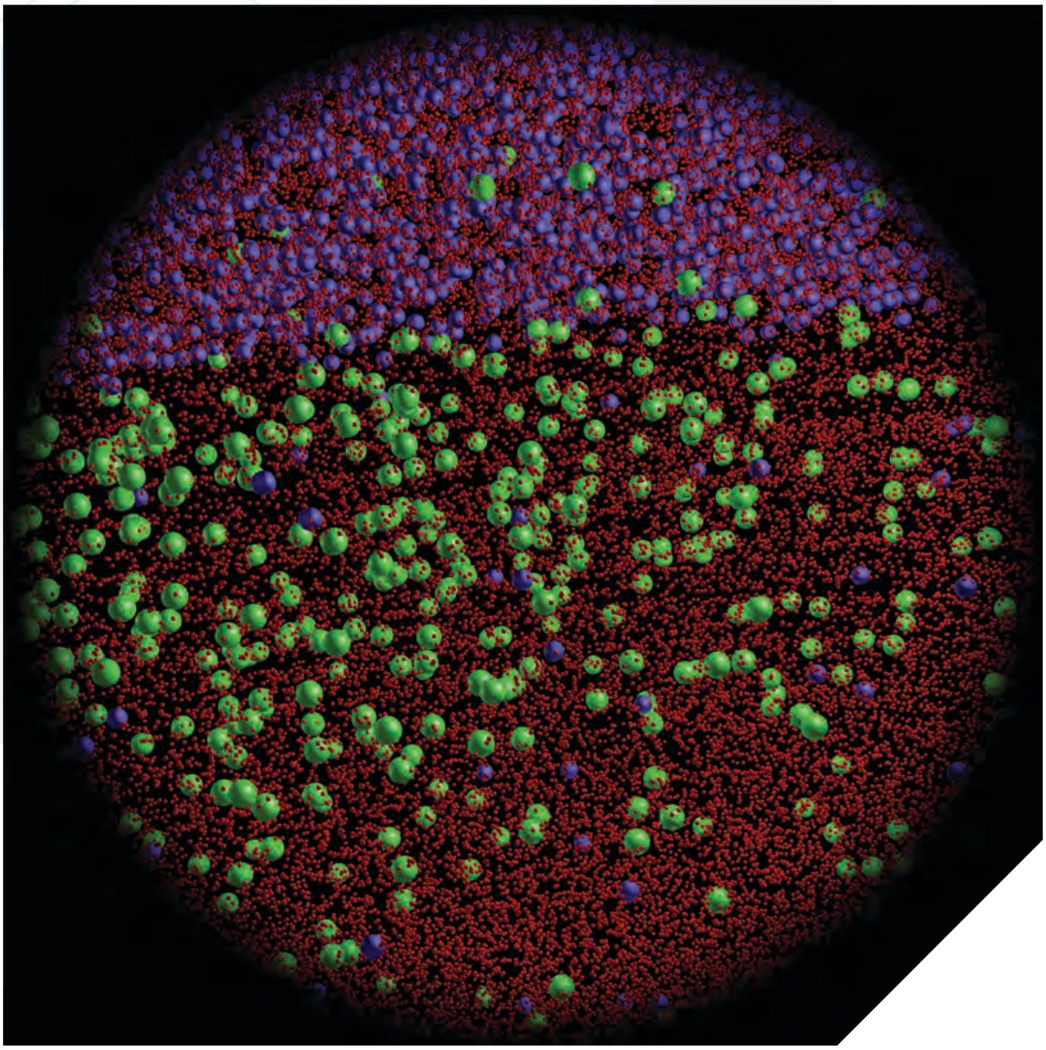
9 Is it possible to tailor the microstructures in different (nm to micron) length scales based on the new physical metallurgy principles?

10 How do we metallurgically control grain structure and stored energy during AM to trigger on-demand properties?

11 What is the role of O, N and H in the titanium alloys under AM conditions?

12 What are the roles of alloying / phase heterogeneity on deformations?

This phase of the project addresses critical knowledge gaps on the phenomenology of microstructural interface instabilities that occur during AM. In the medium term, it will develop a talent-pipeline for defence and to strengthen our capabilities at harnessing AM-based process-microstructure-property relationships. In the long-term, the program purpose is to strengthen Australian university AM research capacity, skills and global networks, orienting these towards the Australian Defence research enterprise.



4. 3D Additive MURI-AUSMURI- Leadership Team

MURI Team

Chief Investigators

THE UNIVERSITY OF TENNESSEE KNOXVILLE

University of Tennessee, Knoxville

Prof. Sudarsanam Suresh Babu

Sudarsanam Suresh Babu received his PhD in Materials Science from the University of Cambridge, Cambridge, UK in 1992. He is currently serving as UT/ORNL Governor's chair professor of advanced manufacturing in the Department of Mechanical, Aerospace and Biomedical engineering, at University of Tennessee, Knoxville, USA and Oak Ridge National Laboratory. His research interests include phase transformations, welding metallurgy, additive manufacturing and computational thermodynamics and kinetics. During this project period, Dr. Babu has also been appointed as the member of the national science board by the President of the United States of America.

Prof. Hahn Choo

Hahn Choo received the Ph.D. degree in Metallurgical and Materials Engineering from the Illinois Institute of Technology, Chicago, IL, USA in 1998. He is currently a Professor at the Department of Materials Science and Engineering, University of Tennessee, Knoxville, USA. His research interests include physical and mechanical behaviour studies of structural alloys using x-ray and neutron diffraction and imaging techniques.

Prof. Amy. J. Clarke

Colorado School of Mines

Amy J. Clarke is a Professor and John Henry Moore Endowed Chair of Metallurgy, Co-Director of the Center for Advanced Non-Ferrous Structural Alloys, and a faculty member with the Advanced Steel Processing and Products Research Center in the George S. Ansell Department of Metallurgical and Materials Engineering at the Colorado School of Mines (Mines). She holds joint appointments with Pacific Northwest National Laboratory in the Nuclear Sciences Division and Los Alamos National Laboratory (LANL) in the Materials Science and Technology Division and is a Guest Scientist in Sigma Division at LANL. She received her MS and PhD degrees from Mines and her BS degree at Michigan Technological University in Metallurgical and Materials Engineering. Her research focuses on physical metallurgy and making, measuring, and modelling metallic alloys during processing to realise advanced manufacturing. Amy serves on The Minerals, Metals & Materials Society (TMS) Foundation Board of Trustees, has served on the TMS and Association for Iron & Steel Technology Boards of Directors, and is an editor for Metallurgical and Materials Transactions A. She is a recipient of the Presidential Early Career Award for Scientists and Engineers (nominated by the U.S. Department of Energy and the National Nuclear Security Administration's Defense Programs), an Office of Naval Research Young Investigator Program, and U.S. Department of Energy Office of Science Early Career Research Program, is a TMS Brimacombe Medalist and a Fellow of ASM International.

Prof. Peter C Collins

Iowa State University

Peter. C. Collins is a Professor, Associate Chair of Undergraduate Education, Stanley Chair in Interdisciplinary Engineering, and Entrepreneurial Fellow within the Department of Materials Science and Engineering at Iowa State University and an affiliated faculty in Aerospace Engineering. He received his PhD from The Ohio State University in Materials Science and Engineering. Prior to starting in his first university role, he set up a not-for-profit advanced manufacturing facility embedded at Rock Island Arsenal-Joint Manufacturing and Technology Center. Dr. Collins is actively involved in two NSF Industry/University Cooperative Research Centers, serving as the co-director for the Center for Advanced Non-Ferrous Structural Alloys, and as past director for the Center for Nondestructive Evaluation. His primary research interests involve: the physical metallurgy of advanced non-ferrous materials; advanced characterisation techniques including various electron microscopies and emergent spectroscopic methods; quantification of defects and crystal orientation across length scales; combinatorial materials science; advanced materials processing with special interest in additive manufacturing; and the mechanical behaviour of non-ferrous materials, including establishing composition-microstructure-property relationships. He has conducted basic and applied research on metal-based additive manufacturing for over 20 years, and most recently has worked to demonstrate new methods to fully characterise the materials state of additively manufactured metallic systems.

Prof. Zhenyu (James) Kong

Zhenyu (James) Kong is currently a Professor with the Grado Department of Industrial and Systems Engineering at Virginia Tech. He received his Ph.D. from the Department of Industrial and System Engineering at the University of Wisconsin-Madison in 2004 and the B.S. and M.S. in Mechanical Engineering from the Harbin Institute of Technology, China, in 1993 and 1995, respectively. His research focuses on sensing and analytics for smart manufacturing and modelling/synthesis/diagnosis for large and complex manufacturing systems.

Virginia Polytechnic Institute and State University (VT)

Dr. Carolin Fink

Carolin Fink is Assistant Professor in the Department of Materials Science and Engineering at the Ohio State University (OSU). Her research interests include materials degradation and cracking phenomena from non-equilibrium processing and solidification. Dr. Fink research has received funding from NSF, ONR and the Electric Power Research Institute. She is Co-PI on one of the most successful NSF-IUCRC awards in the U.S., the Manufacturing and Materials Joining Innovation Center (Ma2JIC). Dr. Fink completed her post-doctoral studies in the Department of Materials Science and Engineering at OSU. She received her doctorate (Dr.-Ing., equiv. to Ph.D.) in welding engineering from the Otto-von-Guericke University Magdeburg in Germany. She was awarded the Henry Granjon Prize, Category B: Materials Behaviour and Weldability of the International Institute of Welding (IIW) in recognition of her Ph.D. research on ductility-dip cracking in solid-solution strengthened nickel base alloys.

The Ohio State University

The Ohio State University

Prof. Joerg R Jinschek

Joerg R Jinschek received his Master of Science (Diplom) in Physics ('97) and his doctorate (Dr. rer. nat. with "magna cum laude") in Solid State Physics ('01), respectively, from the Friedrich-Schiller-University in Jena, Germany. He was awarded with a prestigious Feodor-Lynen-Fellowship of the Alexander-von-Humboldt Foundation, and from 2001 to 2005 performed his post-doctoral research at the world-leading National Center for Electron Microscopy (NCEM) at Lawrence Berkeley National Laboratory (LBNL) in Berkeley, CA. From 2005 to 2007 he established a TEM lab as a Research Assistant Professor at Virgina Tech, Blacksburg, VA. In 2008, he joined FEI Company (now part of Thermo Fisher Scientific), the world-leading electron microscope (EM) provider as a Senior Research Scientist to extend his expertise in state-of-the-art EM into technology & product development. From 2012 to 2016, as the responsible Sr. Product Marketing Manager in FEI's Materials Science Business Unit, he introduced new in-situ EM products (eg, ETEM, NanoEx) and initiated collaborative research with key customers to scientifically market these new solutions. Following his heart and passion for academic research, in 2017 Joerg joined The Ohio State University (OSU) as an Associate Professor in Materials Science and Engineering (MSE), supporting efforts to make a global impact on basic research in energy, environment, and materials' sustainability under the 'Materials and Manufacturing for Sustainability' Discovery Theme. In September 2021, Joerg joined DTU Nanolab as a full professor - attracted by the unique combination of facilities at DTU and by the unique collaborative research environment in Denmark. He continues to focus on studying dynamic processes in materials at the atomic level by developing methods and technologies beyond the state of the art to expand the current research portfolio of DTU Nanolab.

University of California Santa Barbara

Prof. Tresa Pollock

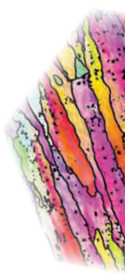
Tresa Pollock is the Alcoa Distinguished Professor of Materials and Associate Dean of Engineering at the University of California, Santa Barbara. Pollock's research focuses on the mechanical and environmental performance of materials in extreme environments, unique high temperature materials processing paths, ultrafast laser-material interactions, alloy design and 3-D materials Characterisation. Pollock graduated with a B.S. from Purdue University in 1984, and a Ph.D. from MIT in 1989. She was employed at General Electric Aircraft Engines from 1989 to 1991, where she conducted research and development on high temperature alloys for aircraft turbine engines and co-developed the single crystal alloy René N6 (now in service). Pollock was a professor in the Department of Materials Science and Engineering at Carnegie Mellon University from 1991 to 1999 and the University of Michigan from 2000 - 2010. Professor Pollock was elected to the U.S. National Academy of Engineering in 2005, the German Academy of Sciences Leopoldina in 2015, and is a DOD Vannevar Bush Fellow and Fellow of TMS and ASM International. She serves as Editor in Chief of the Metallurgical and Materials Transactions family of journals and was the 2005-2006 President of The Minerals, Metals and Materials Society.

Key Activity of Focus and Project Objectives

University of Tennessee, Knoxville

Prof. Babu's group focusses on the in-situ process monitoring and phase transformations in metals and alloys during additive manufacturing. As a part of MURI, students in his group focus on the phase transitions that occur in titanium and nickel base superalloys during repeated heating and cooling conditions typical to that of additive manufacturing. The above phase transitions are rationalized using multi-length scale characterisation including Gleeble® thermo-mechanical simulations, in-situ neutron, and synchrotron diffraction characterisation. Furthermore, the details of the interface between phases are probed using atom probe tomography and electron microscopy. The above results are rationalized based on the thermodynamic stability of phases with strain accumulation during cyclic thermomechanical gyrations.

Prof. Choo's group at UTK is working on the experimental investigation of melt-pool dynamics and solidification behaviour of structural alloys during additive manufacturing processes with a focus on powder bed fusion processing techniques. In particular, the laser melting process, melt-pool dimensional evolutions, melt-pool flow, and subsequent solidification kinetics of various alloys, including nickel, titanium, and ferrous alloys, have been studied using a high-energy, high-resolution synchrotron x-ray dynamic radiography technique. The experimental results are currently being used to help develop and validate a novel Exascale[2] computational modelling scheme (material point method, MPM) in collaboration with the Oak Ridge National Laboratory.



Colorado School of Mines

Large temperature gradients, high solidification velocities, and repeated cycles of heating and cooling are typically experienced during additive manufacturing (AM). Combinations of thermal gradient and solid/liquid interface velocity are known to impact microstructure development, including potential grain refinement produced by the columnar to equiaxed transition. A deeper understanding of solidification (and solid-state phase transformations) under AM conditions is needed. State-of-the-art, multiscale characterisation of solidification dynamics and resulting microstructures in the context of the local conditions experienced during AM is needed to achieve this aim. We are performing in-situ/ex-situ characterisation of microstructure development in conventional and model Ni and Ti alloys under AM conditions, including with in-situ synchrotron x-ray imaging, neutron diffraction, and multiscale electron microscopy. Comparisons are made to process and solidification modelling and theory to enable to microstructure prediction and control.

Iowa State University

The length and time scales in additive manufacturing (AM), as compared to other manufacturing techniques such as welding, casting, powder metallurgy, etc., create unique relationships between the resulting thermal gradients, microstructure, and properties. The goal of this project is to understand the science behind the relation between thermal gradients in AM builds, as a function of different scan strategies, and the microstructure and texture evolution, using tools across lengths scales when analysing samples of the following alloys: Ti-6Al-4V, and Inconel 738, and Haynes 282.

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[2] Exascale computing refers to computing systems capable of calculating at least 1018 floating point operations per second (1 exaFLOPS) (ref: Wikipedia)

University of California Santa Barbara

Deploy tomography techniques for acquisition of 3D multimodal information on structure development in powder bed additive manufacturing processes

Develop new automated workflows and analysis techniques for Characterisation of microstructure and defects in printed materials

Develop fundamental insights and strategies for control of structure along liquid/solid and solid/solid transformation paths

Virginia Polytechnic Institute and State University

Develop new optical sensing capabilities with ultra-high spatial resolution and integrate them with advanced data analytics and machine learning models to enable a deep understanding of the relationships between process parameters, process signature, and print quality.

Ohio State University

The motivation of group is aimed at understanding the role of process conditions on microstructural evolution in AM builds using a combination of *ex situ* and *in situ* electron microscopy. The activities are currently focused on two core areas

- Development of high throughput electron microscopy characterisation strategies to quantify the variations in microstructural heterogeneities within AM builds fabricated using electron beam melted powder bed fusion process. The focus here to understand the impact of AM process conditions on local variations in microstructure within the build.
- Development of a micro electromechanical systems (MEMS) -based in situ heating device that can replicate AM conditions inside the TEM. The focus here is to observe dynamic processes that govern the microstructural evolution within the build under AM operating conditions.

3D Additive AUSMURI Team

Chief Investigators

Prof. Simon Ringer

Simon Ringer is a Professor of Materials Science and Engineering in the School of Aerospace, Mechanical & Mechatronic Engineering, and an academic member of the Australian Centre for Microscopy & Microanalysis at the University of Sydney. He obtained his PhD from the University of New South Wales (Australia) in 1991 and has worked in Australia, Sweden, Japan and the USA in a variety of academic and industry roles. He has published extensively and holds patents in the design of steels and nanomaterials. He is a leading expert in electron microscopy, atom probe microscopy, density functional theory methods and, more recently, in AM process simulation. His interests in microstructure-property relationships for materials design span fundamental research and successful industry collaborations. He is presently the Pro-Vice-Chancellor (Research Infrastructure) at The University of Sydney. Prof. Ringer was elected to the Australian Academy of Technology and Engineering in 2020.

Prof. Sophie Primig

Sophie Primig is Professor at the School of Materials Science and Engineering at the University of New South Wales (UNSW), Sydney. Her research contributions are in processing-structure-property relationships of structural alloys. She was awarded her PhD from Montanuniversität Leoben (Austria) in 2012. After a short period of postdoctoral research and a role as leader of a group with strong industry linkages at the same university, she moved to UNSW in 2015. She holds two UNSW Grad Certs in Education and Management. She is a passionate student-focused teacher, editor of Journal of Materials Science, current TMS Phase Transformation Committee Vice Chair and active Materials Australia member.

Prof. Xiaozhou Liao

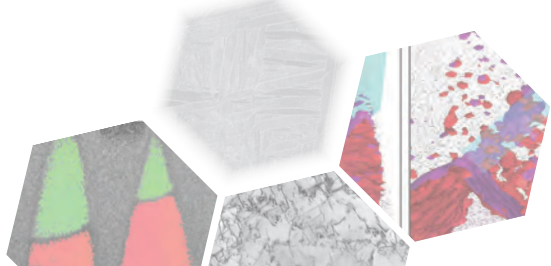
Xiaozhou Liao is a Professor in the School of Aerospace, Mechanical and Mechatronic Engineering and an academic member of the Australian Centre for Microscopy & Microanalysis at the University of Sydney. He received his PhD degree from the University of Sydney in 2000. In 2001, he relocated to the United States taking up a Director Funded Postdoctoral Fellowship in Los Alamos National Laboratory. He took up a research scientist role at the University of Chicago from 2004 to 2006. He established the first Australian group to work on the in situ deformation electron microscopy of advanced materials and this remains a strong focus of his research. His research interests include defect engineering in alloys and how additive manufacturing can generate novel defect structures that impart new materials properties.

Key Activity of Focus and Project Objectives

The AUSMURI project was setup to leverage world-class Australian expertise and facilities in physical metallurgy to provide key contributions to the delivery of a unified phenomenological framework for the AM of metals. Leveraging relationships with our American partners in the US MURI, the 3D Additive AUSMURI project is establishing world-class Australian expertise and facilities to underpin a fundamental knowledge capability in AM. With a strong emphasis on physical metallurgy, this TRL1-2 research addresses the issue that the steady-state conditions assumed during traditional manufacturing processes are clearly not valid in AM, giving rise to new, as yet unexplored metallurgical phenomena. The essence of this project is that this unexplored metallurgical phenomenon must be understood before AM can be harnessed as a qualified production method that exploits its potential around cost, design-flexibility and design-complexity. The project aims to dig deeper into the understanding of the differences in AM materials due to the spatial and temporal transients imposed by abrupt cyclical changes in energy delivery when compared to traditional manufacturing processes. The AUSMURI program is focused on delivering detailed insights from the in-situ microstructural and defect evolution below the micron scale, via advanced microscopy and microanalysis, X-ray and neutron scattering, heat flow modelling and thermo-mechanical simulations. The specific work delegations for the two participating universities is laid out in Table 1.

Table 1. Participating Universities and task lead for each "key activity" (KA)

<table>
<tr><th>Key Activity (KA) / Lead PI*</th><th>Lead University (With Relevant Organisational Units)</th></tr>
<tr><td>KA1. ex-situ & in-situ Scanning Electron Microscopy / (XZL)</td><td rowspan="4">The University of Sydney
• Australian Centre for Microscopy & Microanalysis
• Sydney Informatics Hub
• Formal agreements with ANSTO around access to the Australian Synchrotron and Australian Centre for Neutron Scattering
• Formal agreements with National Computational Infrastructure for access to high performance computing resources</td></tr>
<tr><td>KA2. ex-situ & in-situ Transmission Electron Microscopy / (XZL)</td></tr>
<tr><td>KA3. Atom Probe Tomography / (SPR)</td></tr>
<tr><td>KA4. Computational Simulations / (SPR)</td></tr>
<tr><td>KA5. Ex-situ & in-situ X-ray and Neutron Scattering / (SP)</td><td rowspan="2">The University of New South Wales
• Mark Wainwright Analytical Centre
• School of Materials Science & Engineering
• Agreements with ANSTO around access to the Australian Synchrotron and Australian Centre for Neutron Scattering</td></tr>
<tr><td>KA6. Thermo-mechanical Simulations / (SP)</td></tr>
</table>



Advisory Board Members

The MURI and AUSMURI teams have established an Advisory Board panel to provide advice on program implementation, advocacy and maximising impact of the research on the Australian and US defence capability.

The MURI advisory board panel comprises of individuals from the US defense and industry and are as follows:

- Dr. Ayman Salem, CEO, Material Resources LLC.
- Mike Tims, Advisor Engineer at Concurrent Technologies Corporation
- Justin Gambone, Senior Engineer at GE Research
- Dr. Glynn Adams, Manager, Lockheed Martin
- Dr. Brandon McWilliams, Information Technology Specialist at US Army Research Laboratory
- Dr. Andelle Kudzal, Materials Engineer at US Army Research Laboratory
- Dr. Mark D. Benedict, AM lead, US Air Force Research Laboratory
- Dr. Cindy Waters, Senior Science and Technology Manager for AM and Materials, US Navy
- Caroline Vail, Mechanical Engineer, US Navy

The AUSMURI component of the panel comprises of Dr. Mark Hodge as chair (DMTC), Dr. Anita Hill (CSIRO), Dr Zoran Sterjovski (DSTG), Dr. Janis Cocking (Retd. Defence Scientist), and Dr. John Harvey (Retd. Air Marshall).

The Australian Advisory Board (AAB) meets bi-annually and these meetings are held at the University of Sydney or UNSW campus, although the meetings have been held online in the last year due to COVID.

Dr. Mark Hodge

Dr. Mark Hodge is the chair of the Advisory Board. He has served as Chief Executive Officer of DMTC since its inception in June 2008, overseeing the organisation's success in a range of activities centred on Australia's defence and national security. He has led the strategy for DMTC's transition to a sustainable industry capability partner as agencies across Government increasingly look to industry to support Australia's strategic national security objectives. He is a tireless advocate for science and technology and its applications in advancing Australia's national interests, and has worked in leadership and professional roles in the defence and aerospace fields for his entire professional career. Dr Hodge serves on a range of advisory boards, committees and panels, including the Department of Defence's Innovation Steering Group. He is a former Director and Deputy Chair of the Cooperative Research Centre (CRC) Association. An author of several research publications on advanced defence materials, Dr Hodge is the recipient of several industry and research-sector awards and is a fellow of The Australian Academy of Technology and Engineering.

Dr. Anita Hill

Dr. Anita Hill is a former Executive Director of the CSIRO (Commonwealth Industrial Scientific and Research Organisation) responsible for overseeing the strategic direction and investment across Manufacturing, Agriculture and Food, Health and Biosecurity, Mineral Resources, Digital Productivity, Materials Science and Engineering, Process Science and Engineering, and CSIRO Services which includes Education and Outreach, Publishing, Futures, SME Engagement, and Infrastructure Testing. Prior to that role she was Chief of Process Science and Engineering and served as an Office of the Chief Executive Science Leader. She is a former Director of AeHRC, NCEDA, VCSCM, and MSA. She is a graduate of the Australian Institute of Company Directors and a fellow of ATSE, AAS, and RACI. Her research is in materials and process engineering, structure/property relationships, and novel materials Characterisation technique development.

Dr Zoran Sterjovski

In 2006, **Dr Zoran. Sterjovski** joined the Defence Science and Technology Group (DSTG) as a Defence Scientist in Materials and Welding in the Maritime Division to work on structural life validation for the Collins class submarines. From 2016 to 2019, Dr Sterjovski was the DSTG lead for Submarine Platform Integrity in the Future Submarine Program covering areas such as pressure hull boundary materials, structural collapse, underwater shock, fatigue and environmentally-assisted cracking, and corrosion management. Currently, Dr Sterjovski is the Head of Computational Materials and Structures (CoMS) in the Aerospace Materials Branch within Aerospace Division at DSTG. CoMS provides expert S&T advice and innovative solutions to Defence to ensure that its aerospace platforms are operationally effective, structurally safe and sustainable.

Dr. Janis Cocking

Dr. Janis Cocking, PSM, FTSE has over forty years experience leading and undertaking science and technology, particularly in undersea technology, for which she is an acknowledged international expert. Her career started with research into the behaviour of Ni-based materials at high temperatures. In 2016 she led the creation of the framework and launch of the 10-year, $730 million Next Generation Technologies Fund for Defence. She was awarded the Public Service Medal in the 2018 Australian Honours for her outstanding contributions to Defence S&T. In 2019 the Australian Defence Science and Technology Group created the Janis Cocking Award for Excellence in Leadership in recognition of her impact on and for Defence.

John Harvey

Air Marshal (Ret) John Harvey's Australian Defence Force career spanned more than 30 years, with early emphasis on employment as a navigator and weapons officer in Canberra and F-111 aircraft and later in more diverse roles such as technical intelligence, military strategy, visiting Fellow Strategic and Defence Studies Centre at the Australian National University, instructor at the Australian Defence College and Defence Attaché Southern Europe. He has had extensive experience in the Defence capability development process in his appointments as Air Warfare Adviser within Force Development and Analysis, Director General Aerospace Development, Director General New Air Combat Capability, Program Manager New Air Combat Capability (NACC) and In October 2010, John was promoted to the rank of Air Marshal and appointed as Chief Capability Development Group (CCDG) with responsibility for requirements development and obtaining government approval for approximately 200 projects with a total value of approximately $200 billion. Since retiring from the RAAF in 2012, John has completed a PhD in Computer Science at the UNSW (Canberra), has aerospace and defence advice to the Department of Defence as a Reserve Officer and acted as NSW Defence Advocate from 2016 to 2021.

The 3D-Additive MURI-AUSMURI Projects Collaboration Summary

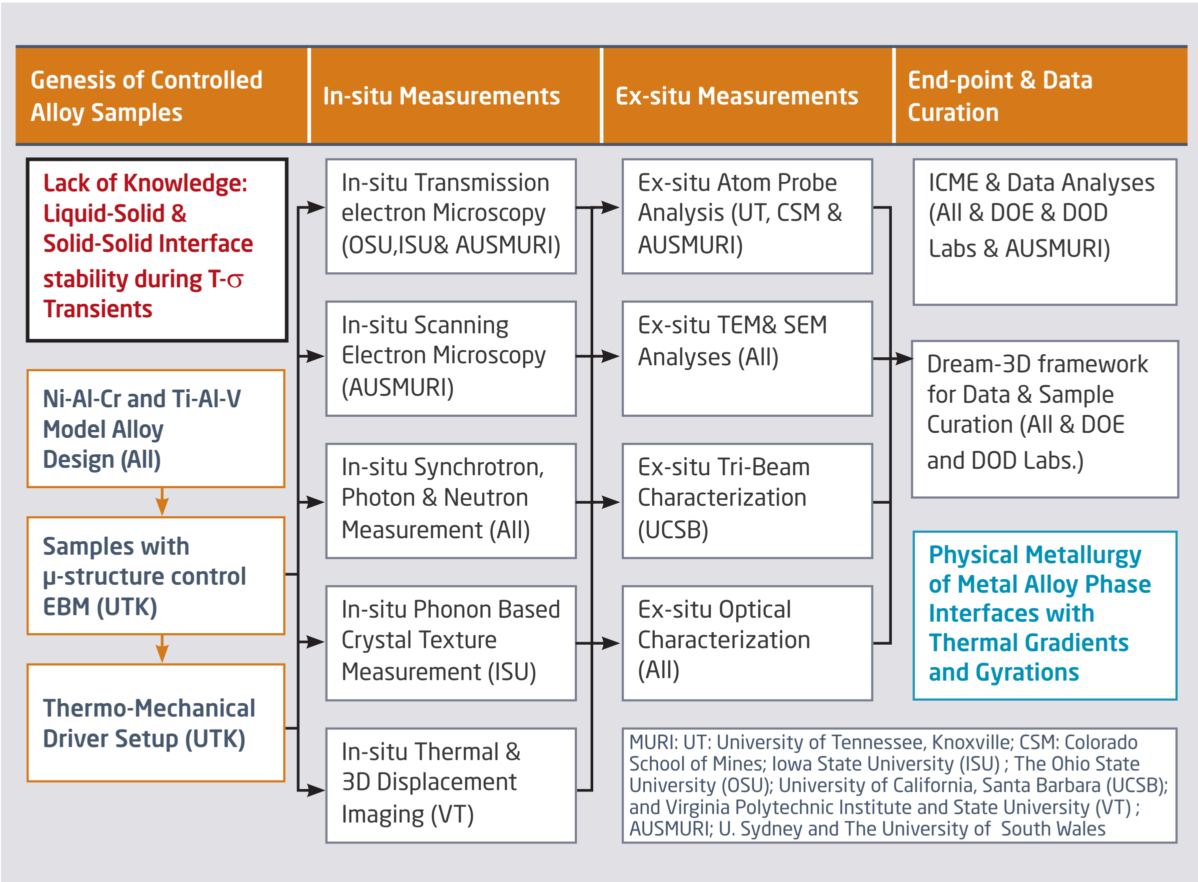
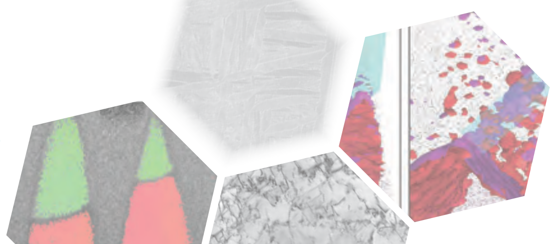


Figure 2. Overview of the research process flow across the collaborating universities, laboratories and the lead organisations.



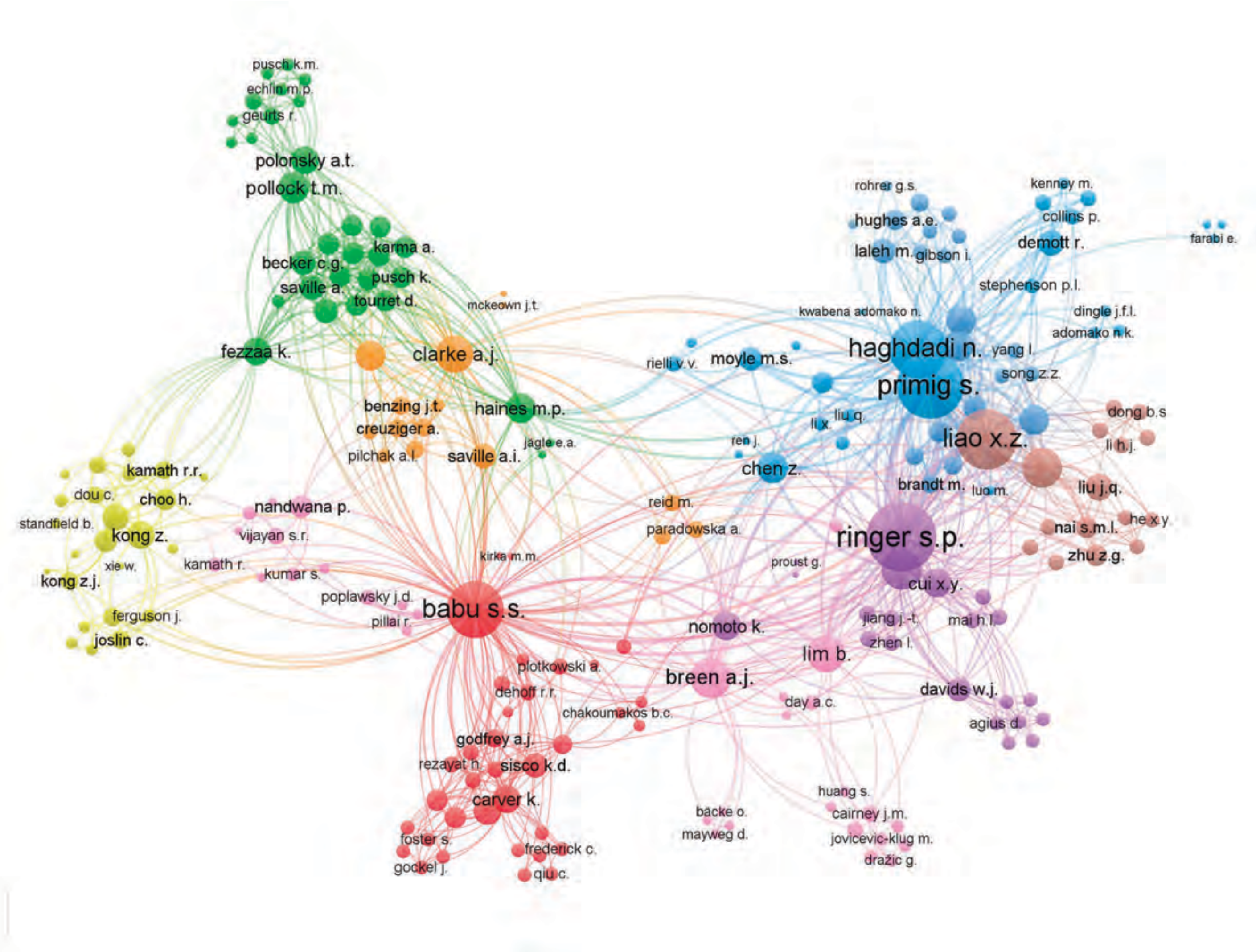
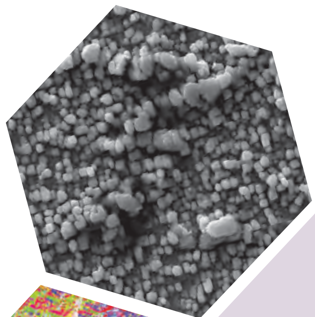
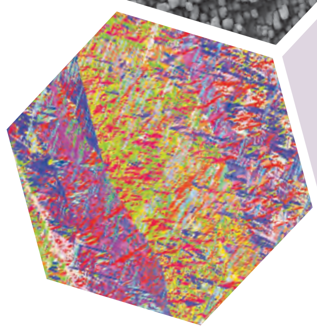


Figure 3. Network diagram showing the collaborating team members of the MURI-AUSMURI 3D additive project based on published papers listed in the Annexure.





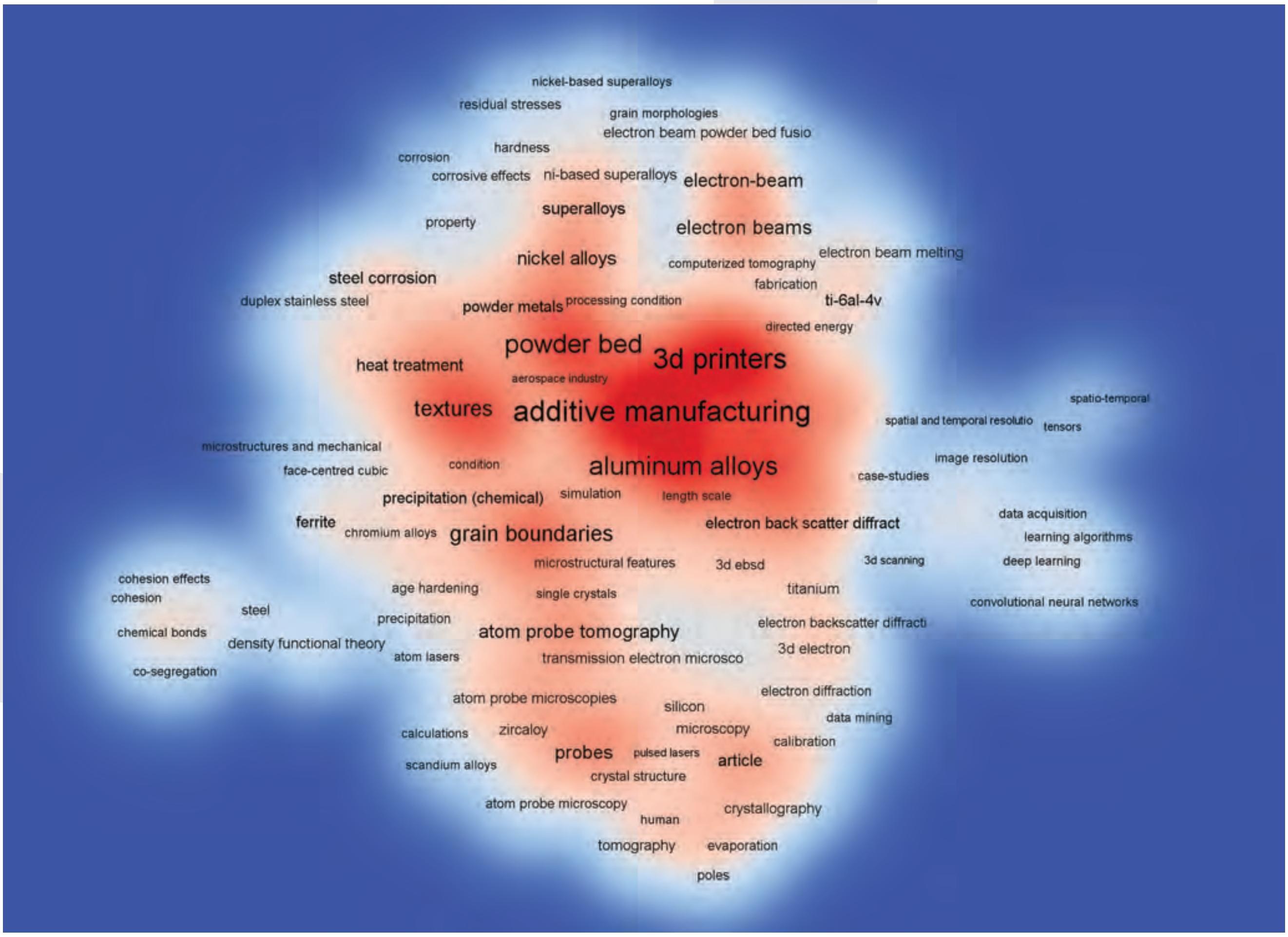


Figure 4. Density visualisation of the common keywords used in the publications of this collaborative project.

5. Research Highlights (2022 - 2024)

Q4: Will there be plastic instabilities during transient thermomechanical conditions of AM?

Q5: How do we map the observed l/s and s/s interface instabilities on to high performance computational models?

Answer: Some of the prior MURI/AUSMURI research has shown that these instabilities exist, and new set of questions have been raised on how to map these phenomena into the computational models at right length and timescales. The research highlight pertains to the role of computational mesh size and time resolution factors on the predicted magnitudes of distortion and stress. The research focussed on boundary conditions relevant to Wire Arc Additive Manufacturing (WAAM) of a large-curved wall geometry relevant to Navy applications. The results show while predicted nodal displacement was consistent at lower resolutions, the predicted residual stress state varied with change in mesh size.

Background and Motivation WAAM's potential to supplement manufacturing of large-scale metal geometries is inhibited by excessive residual stresses and deformation induced during the manufacturing process, which hinders the production of parts with geometrical conformity and consistent material properties. In order to deploy measures to accommodate for these defects, efficient and accurate modelling procedures are needed. However, the majority of work on modelling in the WAAM space does not incorporate geometrically complex, large scale shapes. As part scale increases, modelling the AM process on a layer-by-layer basis becomes computationally infeasible. There is therefore a need to observe and understand the effects of analysis resolution that is unable to fully capture the heat source scan strategy or layer-by-layer material deposition. This work aimed to close the gap in knowledge by incorporating finite element analysis of a large scale (495.3 x 527.3 x 20 mm) curved geometry that is asymmetrical across the Y axis.

Results: (i) While increment did have a large impact on the numerical temperature history, the influence on nodal displacement and residual stress was relatively low. Geometry and cooling rate could potentially be the largest factors in determining distortion of WAAM at this scale. (ii) Change in element size had a large influence on numerical residual stress. Lower resolution models exhibited the high stress distributions across the face of the part. As resolution increased, these peak stress distributions faded, resulting in a more homogenous stress state.

Significance: Similar distortion states can be resolved with large discrepancies in residual stress calculations, and further verification and validation steps should be established.

Reference: B. Solsbee, Y. Lee, S. Simunovic, and S.S. Babu, Role of computational parameters on predicting consistent residual stress and distortion during wire arc additive manufacturing, Unpublished Work, (2023).

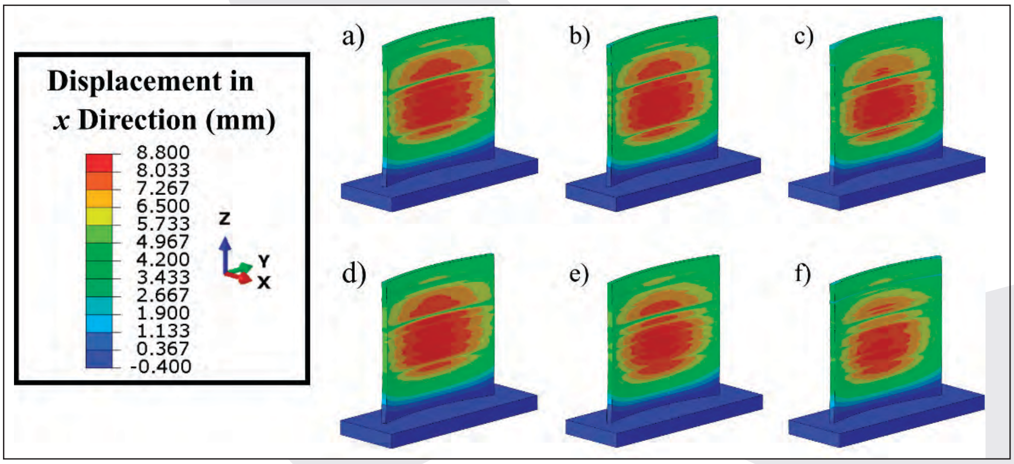


Figure: Predicted deformation in x direction across increment lengths (a-c): a) 25 sec, b) 100 sec, c) 400 sec; and element sizes (d-f): d) 8 mm, e) 4 mm, f) 2 mm

PHASE 2

Q1: Will there be local equilibrium at interfaces with large thermal gradients?
Q6: What are the scientific limits of sensors and data fusion to describe the AM boundary conditions?

Answer: A key piece of information necessary to better understand this question is the temperature (T) and thermal gradient (G) distribution at the interface and its vicinity. The focus of this work was to develop a methodology which uses a synchrotron x-ray radiography dataset to estimate T and G at and around the solid-liquid interface.

Background and Motivation Local equilibrium at interphase interfaces can be probed in-situ with sufficient spatial and temporal resolution under simulated AM conditions using techniques such as synchrotron x-ray radiography and transmission electron microscopy. While these advanced characterization techniques provide a resolved interface velocity, the corresponding thermal gradient information is not available readily. The corresponding temperature distributions either have to be calculated using thermal-fluid flow simulations or measured using simultaneous thermal imaging – both of which have their own shortcomings. This necessitates a novel methodology to extract temperature distributions from already available interface motion datasets. Through this work, we formulated and evaluated one such pathway using the dynamic synchrotron x-ray radiography data.

Results: (i) A novel methodology was developed to estimate the transient, sub-surface temperature distribution around the solid-liquid interface from dynamic x-ray radiography data of laser melts using Beer-Lambert's law as a physical basis. Additionally, the through-thickness temperature distribution was resolved for a spot melt case using the Gilbert-Deinert approach. Further, the process flow was evaluated for a laser spot melt performed on a Ti-6Al-4V plate.

(ii) The trends captured in T and G inside and around the melt pool for Ti-6Al-4V spot melt were similar to previous modelling studies. G was estimated to be 107 – 108 K/m depending on the location within the probed area. While the methodology was demonstrated successfully, future experiments with careful calibration are necessary to obtain a more accurate estimates.

Significance: This novel methodology could be pivotal in supplying more accurate estimates of T and G to better answer key questions (Q1, Q2 and Q3) posed in the initial proposal.

Reference: Rakesh R. Kamath, Hahn Choo, Kamel Fezzaa, and Sudarsanam Suresh Babu, Estimation of spatio-temporal temperature evolution during laser spot melting using in-situ dynamic x-ray radiography, Metallurgical and Materials Transactions A (Accepted 01/05/2024 – manuscript in production)

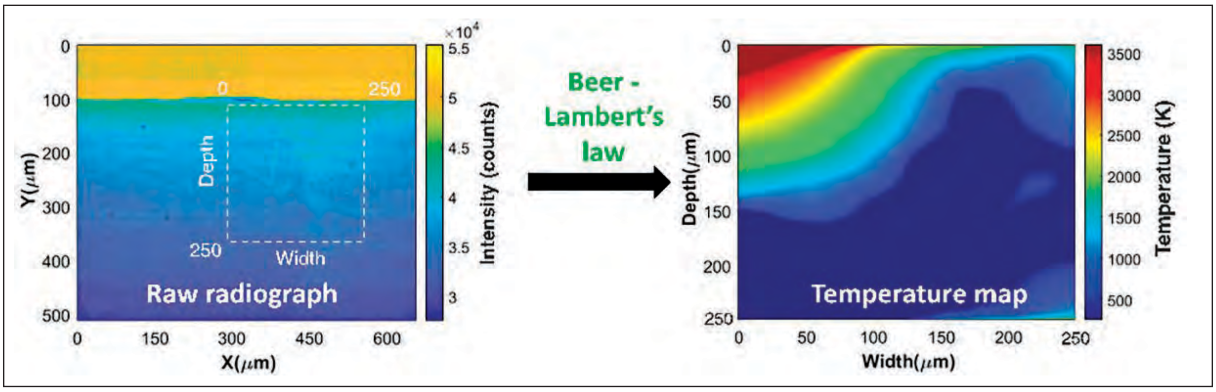


Figure: The start and end points of the methodology shown with the raw radiograph input and the corresponding temperature map for the dashed box in the radiograph.

Q6: What are the scientific limits of sensors and data fusion to describe the AM boundary conditions? and Can we improve the quality of built parts in L-PBF with advanced modeling and control?

Answer: Machine-learning-based modeling and real-time closed-loop control aid in designing process parameters that improve quality.

Background and Motivation: In a laser powder bed fusion (L-PBF) process, the processing conditions determine the quality of the build through complex thermal history. Modeling the influence of process parameters on the thermal history is necessary for optimising the process. The traditional numerical simulations for modeling the thermal history are time-consuming and typically inconsistent with the observations. This work provides a machine learning (ML)-based method for fast and accurate predictions of thermal history. Due to complex process dynamics, real-time process monitoring and control are also essential in an L-PBF machine for mitigating defects. This work demonstrates the benefits of closed-loop laser power control via real-time thermal monitoring. A custom L-PBF platform (Figure 1) enabled the deployment of closed-loop control.

Results: (i) Physics-informed neural networks (PINNs) directly learn the heat transfer model in the form of a partial differential equation (PDE) system to provide the thermal history for the L-PBF process. A novel activation function1 accelerates the learning process. The proposed method also combines the sensor data from the Infrared Camera to discover the inaccuracies in the PDE or the boundary conditions.

(ii) Closed-loop control of laser power profoundly reduces the dimensional error by effectively minimizing insufficient melting, denting, and swelling2. Verified by a set of Ti-64 parts, the controller successfully stabilized the melt pool size by regulating the laser power at 2 kHz.

Significance: : The developed methods improve the L-PBF process design for significant improvements in the quality of the built parts.

Reference: [1]R. Gnanasambandam, B. Shen, J. Chung, X. Yue, and Z. Kong, Self-Scalable Tanh (Stan): Multi-Scale Solutions for Physics-Informed Neural Networks, IEEE Transactions on Pattern Analysis and Machine Intelligence, Vol. 45, p. 15588-15603, (2023).

[2]R. Wang, B. Standfield, C. Dou, A. Law, and Z. Kong, Real-time process monitoring and closed-loop control on laser power via a customized laser powder bed fusion, Additive Manufacturing, Vol. 66, p. 103449, (2023)

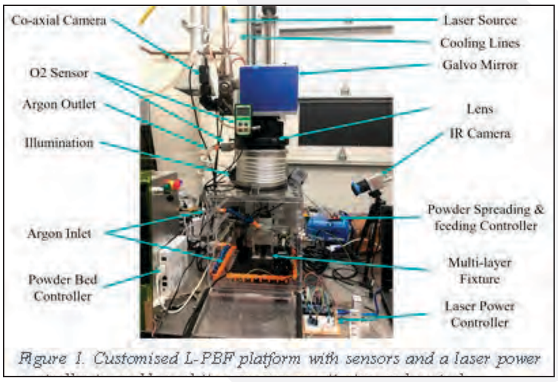


Figure: Customised L-PBF platform with sensors and a laser power controller to enable real-time process monitoring and control.

Q7: What are the roles of steady to transient thermal gradients in different length scale on s/s interface stability?

Answer: In AM INC738, complex thermal cycles control the solid-state phase transformation kinetics and the microstructural evolution at different locations within the build, and this can be evaluated using advanced computational tools.

Background and Motivation: The microstructures resulted from AM are complex and distinct from conventional processing due to intricate thermal cycles during layers deposition. Post-processing treatments are employed to attain desired microstructure and properties. However, this results in additional costs and the risk of undesired phase precipitation and grain coarsening. Therefore, advanced control of the AM process is desirable to realize the potential of as-built parts.

To achieve this goal, computational modelling methods are becoming increasingly popular and mature tools in AM research. However, much of the research and development in computational modelling for AM has been focused on predicting the thermal behaviour, identifying potential defects, and understanding larger scale microstructural features such as grain and dendrite structures. Efforts in the computational modelling of the solid-state phase transformations, which greatly impact the resulting properties remain limited.

Results: A new combined modelling approach to predict solid-state phase transformation kinetics and microstructural evolution during metal AM was put forward. We use a semi-analytical heat conduction model to simulate thermal history during AM builds, then feed thermal profiles to MatCalc software for a thermo-kinetic analysis. MatCalc predicts the microstructural evolution such as fractions, morphology, and composition of all individual phases of various layers during deposition. Electron beam powder bed fusion (E-PBF) of IN738 part is used as a case-study to provide insights into the dynamic evolution of γ′ during thermal cycling (Figure 1). Our simulations align well with our experimental results in predicting changes in the distribution of γ′ size along the build height, its multimodal size, and the evolution of MC carbides.

Significance: New tools to predict the microstructural evolution and properties during metal AM are important as they provide new insights into the complexities of AM. Our approach can be utilized for a variety of AM processes and alloys to predict and control their microstructures, towards advanced materials properties and performances.

Reference: N. K. Adomako, N. Haghdadi, J. F. L. Dingle, E. Kozeschnik, X. Z. Liao S. P. Ringer, S. Primig, Predicting solid-state phase transformations during metal additive manufacturing: A case study on electron-beam powder bed fusion of Inconel-738, Additive Manufacturing 76 (2023) 103771.

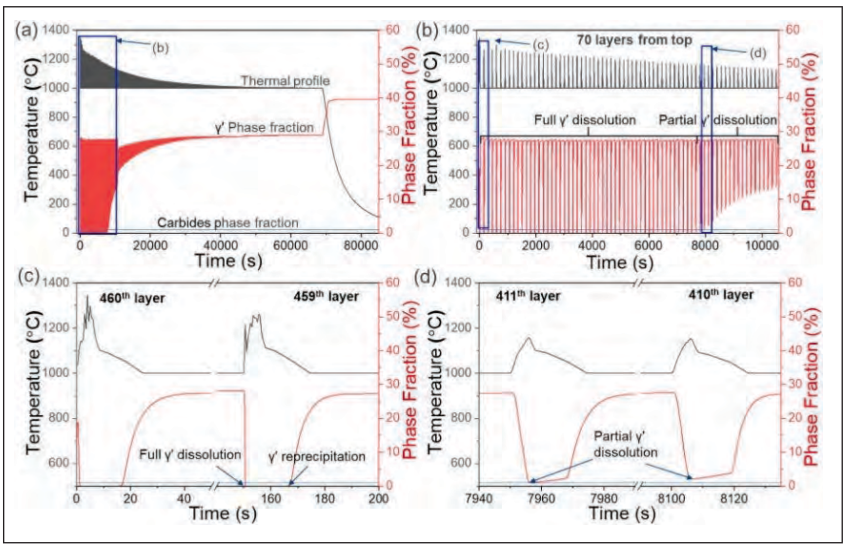


Figure: Simulated phase fractions of γ′ and MC carbide during thermal cycling of INC738 under E-PBF at 2 mm from the bottom. (a) Full cycle. (b) First 70 layers from top. (c) First 2 layers from the top (layer 460 and 459). (d) Layer 411 and 410.

Q1: Will there be a local equilibrium at the interfaces with large thermal gradients?

Q9: Is it possible to tailor the microstructure in different length scales based on the new physical metallurgy principles

Answer: The distribution of thermal gradient (G) and solidification velocity (V) values experienced in the melt pool during additive manufacturing (AM) is not the only relevant factor to consider when inoculation particles are added to promote grain refinement.

Background and Motivation: Previous work with tantalum (Ta)-inoculated aluminium (Al) powder reveals refined microstructures when subjected to laser powder bed fusion (LPBF), but heterogeneities are also observed. It is unclear whether these heterogeneities are due solely to the G/V conditions experienced in the melt pool, or if other factors also play a role.

Results: In-situ synchrotron x-ray radiography, ex-situ microstructural characterization, and computational fluid dynamics were used to study refinement mechanisms associated with Ta-(1 at.%) inoculated Al powders. Pure Al substrates were subjected to experimental LPBF simulations at the Advanced Photon Source at Argonne National Laboratory. Two sets of experiments with different powders were conducted. Some experiments included pure Al powder, whereas some included Ta-inoculated Al powder. The Al substrates were subjected to four raster track experiments (~430 W laser power, 0.3-1 m/s laser speed).

Figure 1(a, b) show EBSD mapping of different raster track conditions at different laser speeds for pure Al and Al+Ta powder. Addition of Ta to the powder results in refinement of the microstructure, especially at the melt pool top center, see Figure 1(b.4). Distributions of the solidification parameter G/V, calculated by Computational Fluid Dynamics (CFD), are shown in Figure 1(c), where lower G/V values mean there is a higher chance of crossing the columnar to equiaxed (CET) transition. It is expected that a more refined structure is found on the top right of the melt pool track, where the liquid solidified last, which disagrees with the location of the equiaxed grains in the melt pool top center, as detected by electron backscatter diffraction (EBSD). Therefore, it is suggested that other mechanisms like distribution and size of precipitates in the melt pool tracks that are dominated by flow convection are partially responsible for the observed heterogeneous grain refinement.

Significance: Local melt pool conditions impact the extent of grain refinement by inoculation, which must be better understood to produce uniform microstructures and properties produced by AM.

Reference: A. Eres-Castellanos, C. Becker, H. Martin, K. Fezzaa, A. Clarke, "Refinement mechanisms of tantalum-inoculated aluminum subjected to simulated laser powder bed fusion", 2024, in preparation.

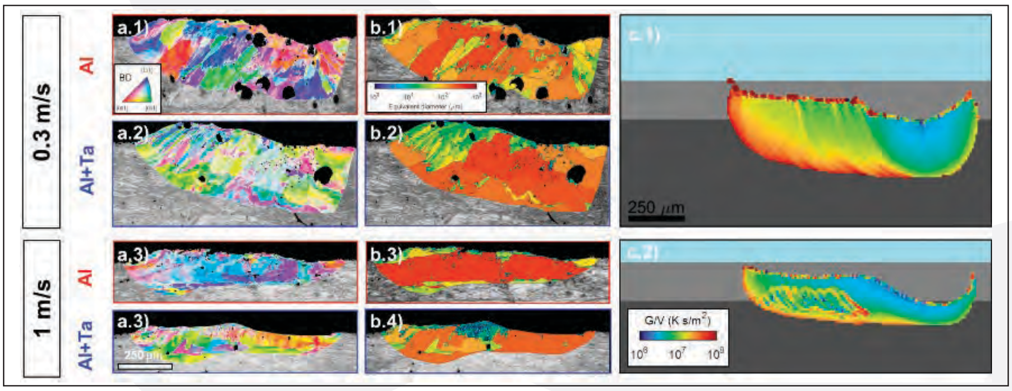


Figure: (a, b) EBSD results corresponding to different raster track conditions, conducted with pure Al and Al+Ta powder, at different laser speeds: (a) inverse pole figure maps (IPF, building direction - BD, vertical), and (b) grain equivalent diameter maps. Results are compared to G/V distributions obtained by CFD, as shown in (c). Laser moved from left to right.

Q9: Is it possible to tailor the microstructures in different (nm to micron) length scales based on the new physical metallurgy principles?

Answer: The formation of different phases in AM processed 17-4 PH depends on the thermal history and local composition. This can be used for targeted microstructural engineering.

Background and Motivation: Existing literature on 17-4 PH and similar steels has described the phase evolution to be heavily dependent on the composition and processing parameters with large variations on the reported phase. Both primary austenite and primary δ-ferrite solidification have been reported. The phase fraction of austenite is often linked to the cooling rate and processing parameters. Further, the formation of martensite during cooling is influenced by grain size, composition, and processing parameters, with works showing a mixture of martensite and retained austenite, or no martensite formation at all. However, comprehensive studies on the complex phase transformation pathways in 17-4 PH and their direct link to the complex thermal histories seen in L-PBF have yet to be established.

Results: Our work utilizes a combination of computational and experimental methods to describe the complex phase evolution in concentric 17-4 PH laser powder bed fusion (L-PBF) builds. The observed formation of δ-ferrite is explained through the use of interface response functions. In the outer regions of the sample, two distinct morphologies of austenite, Widmanstätten and allotriomorphic, are identified. Due to changes in thermal history, the center of the sample has a significantly higher phase fraction of austenite (Figure 1). Atom probe tomography reveals that austenite formation is controlled by the diffusion of interstitial elements C and N. We further report that a suppression of the martensite start temperature may occur due to local compositional variations during final cooling. The 17-4 PH phase transformation routes can be described as first solidifying as δ-ferrite that can exist down to room temperature or transform into allotriomorphic or Widmanstätten austenite. Subsequently, austenite can be retained or transform into either α-ferrite or martensite.

Significance: These possibilities for phase control via adjusting the thermal history and local composition can be harnessed for targeted microstructural engineering. The results presented in this manuscript are of great importance in the L-PBF production of steels owing to steels' particular sensitivity to processing parameters. The combination of experimental and computational tools demonstrates a process for methodically outlining the complex phase transformation pathways that can be applied to other alloys systems.

Reference: M. Haines, M. S. Moyle , V. V. Rielli, V. Luzin, S. Primig, Experimental and Computational Analysis of Site-Specific Formation of Phases in Laser Powder Bed Fusion 17-4 Precipitation Hardening Stainless Steel, Additive Manufacturing 73 (2023) 103686.

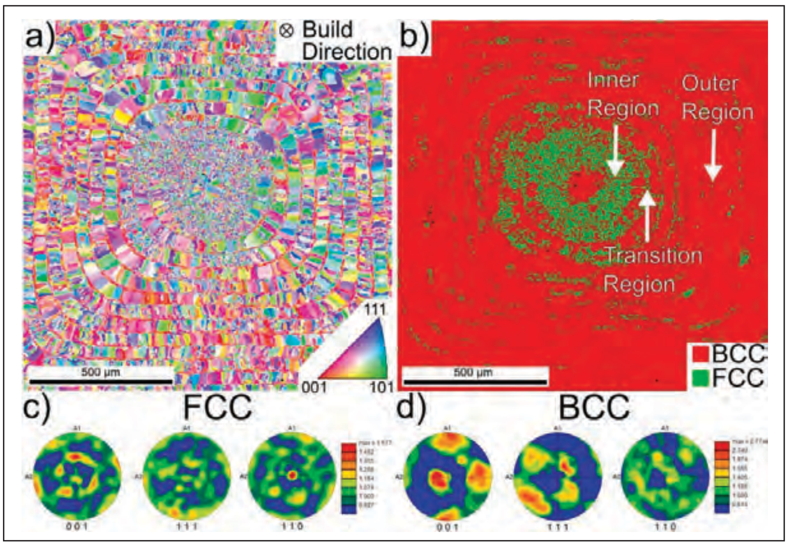


Figure: Electron backscatter diffraction of the centre of the 17-4 PH build with build direction (Z) pointed into the page. a) orientation, b) phase.

Q11: What's the role of O, N, and H in the titanium alloys under AM conditions?

Answer: The formation of different phases in AM processed 17-4 PH depends on the thermal history and local composition. This can be used for targeted microstructural engineering.

Background and Motivation: Titanium alloys, renowned for their lightweight and superior mechanical properties, are crucial in numerous high-stakes applications. The industry predominantly relies on α–β titanium alloys, tailored through the addition of elements that stabilize the α and β phases. Our research is dedicated to exploiting oxygen and iron, two potent stabilizers and enhancers for α–β titanium alloys, given their abundance. Despite their advantages, the embrittling effect of oxygen—often referred to as titanium's "kryptonite"—and the microsegregation issues posed by iron have limited their combined use in creating strong yet ductile α–β titanium-oxygen-iron alloys.

Results: By synergising alloy and additive manufacturing process design, we've developed a range of titanium-oxygen-iron compositions with exceptional tensile properties (Fig. 1a). We've elucidated the atomic-scale mechanisms underpinning these properties through comprehensive characterisation techniques (Fig. 1b). Given the plentiful availability of oxygen and iron, along with the streamlined process afforded by AM for producing net or near-net shapes, these α–β titanium-oxygen-iron alloys are poised for widespread application.

Significance: This advancement paves the way for the industrial-scale utilisation of lower-grade titanium resources or titanium-oxygen-iron sponge, currently considered an industrial by-product. The potential for these alloys to significantly reduce the carbon footprint associated with the production of sponge titanium, an energy-intensive process, represents a substantial economic and environmental benefit.

Reference: Song, T., Chen, Z., Cui, X., et al. (2023). Strong and ductile titanium–oxygen–iron alloys by additive manufacturing. Nature, 618(7963), 63-68.

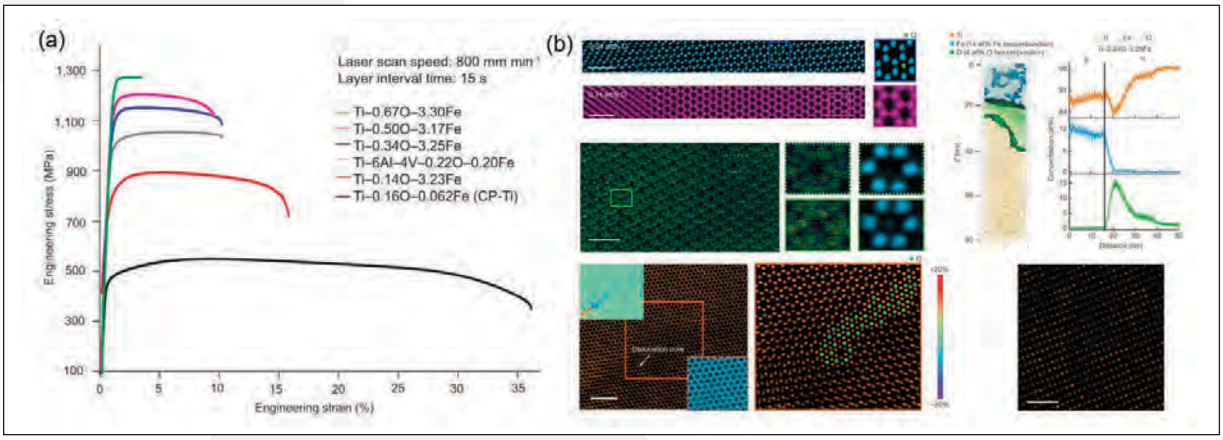


Figure: (a) Tensile properties of DED-printed Ti-O-Fe alloys at room temperature by focusing on varying alloy composition without changing the processing conditions. (b) Distribution of O and Fe atoms in DED-printed Ti-O-Fe alloys.

Q11: What is the role of O, N and H in the titanium alloys under AM conditions?

Answer: O performed as an interstitial solid solution to induce the formation of a new non-equilibrium FCC phase. This phase further strengthens the tensile and compression properties of AM Ti64, which can be applied to other AM Ti alloying systems.

Background and Motivation: Speculation that a face-centred cubic form of Ti can be stabilized at room temperature goes back several decades. Multiple controversies have emerged from these propositions, such that today, it is widely agreed that the FCC forms of Ti are either intermetallic compounds or highly localized and defect-stabilized metastable phase segments. Here, we explore the potential for interstitial oxygen to be purposefully integrated into the AM build to dramatically improve the mechanical properties through a novel phase transformation wherein an FCC form of Ti can be stabilized. This phase, accessible via LPF-AM, lays the groundwork for new mechanical property regimes in Ti-alloys.

Results: (i) An oxygen-rich FCC phase, designated as C phase, has been discovered in a Ti-6Al-4V alloy produced by L-PBF. The C phase exhibits a lattice parameter of 0.406 nm, an oxygen concentration of up to 33 at. % and an orientation relationship with the matrix ′ ′phase as follows: (0001)′ //{111}C, and ′ 12 ′ 10′ _α′//′ 11 ′ 0′ _C .

(ii) The significant thermal gradient, cyclic thermal loading, and incorporation of O during the L-PBF process are critical for the formation and stabilization of the C phase.

(iii) The introduction of this FCC phase significantly benefits the mechanical properties of the material. The compression strength of pillars comprising near equal volume fractions of the FCC and the HCP phases was more than 70% higher than that of pillars comprising only the HCP phase, yet no loss in plasticity was measured. High densities of dislocations and deformation twins were observed in the deformed C phase, which is typical of deformed FCC structures with low stacking fault energy. Moreover, the HCP/FCC phase boundaries contribute to improve the mechanical properties.

(iv) This research indicates that the mechanical properties of Ti-6Al-4V alloys prepared by AM and, more broadly other Ti alloys, may be significantly improved by the presence of the oxygen-stabilized FCC phase. We have demonstrated processing conditions whereby this phase can be deliberately introduced—the key parameters being the thermal gradient and the delivery of additional O via the feedstock materials to the additive manufacturing process.

Reference: H. Wang, Q. Chao, X.Y. Cui, Z.B. Chen, A.J. Breen, M. Cabral, N. Haghdadi, Q.W. Huang, R.M. Niu, H.S. Chen, S. Primig, M. Brandt, W. Xu, S.P. Ringer, X.Z. Liao, Introducing C Phase in additively manufactured Ti-6Al-4V: a new oxygen-stabilized face-centred cubic solid solution with improved mechanical properties, Materials Today, Vol. 61, p. 11-21, (2022)

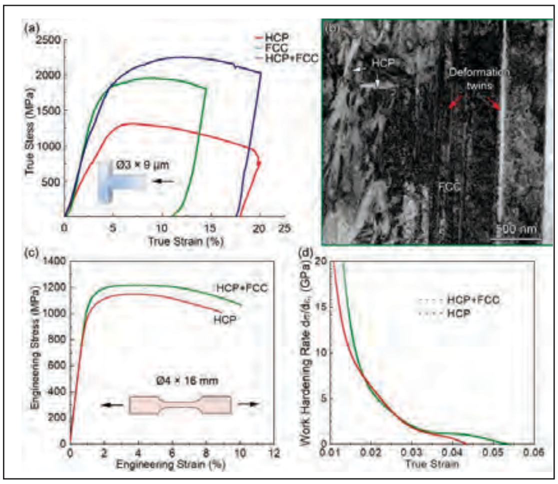


Figure: a) Typical true compression stress-strain curves recorded from micro-pillars prepared from each phase and a 50/50 mix of the two phases. (b) A bright-field image of a deformed (0.2 true strain) two-phase region. (c) Engineering tensile stress-strain curves of bulk samples printed as coupons for tensile testing with dimensions provided in the inset. (d) The corresponding strain hardening rates.

Q1: Will there be a local equilibrium at the interfaces with large thermal gradients?

Answer: Laser track melting of simulated additive manufacturing (AM) and complementary solidification modelling of two commercial refractory alloys (Nb-47Ti and C103) suggest existing models can be used to make defect and processing maps relevant to AM.

Background and Motivation: Refractory alloys, especially emerging refractory multi-principal element alloys (MPEAs), are difficult to process by conventional processes like thermomechanical processing. AM affords new opportunities to manufacture near net shape parts from refractory alloys. Yet, little is known about processing refractory alloys under AM conditions and whether existing models are applicable for predicting defects, microstructures, and processing conditions. In this work, two commercial refractory alloys (Nb-47Ti and C103) were subjected to laser track melting and characterization to correlate the formation of defects and microstructures with processing conditions and model predictions

Results: (i) Laser track melting at different powers and speeds were conducted in Nb-47Ti and C103. Defect maps enabled the processing parameter selection where keyholing and lack of fusion were expected. The predicted conduction/keyhole threshold for Nb-47Ti was found to be at higher laser powers than experimentally observed, due to an overestimated value of normalized enthalpy. The use of a more conservative value showed better matching to experiments.

(ii) The Ivantsov Marginal Stability (IMS) model was used to delimit planar, cellular, and dendritic growth regions, the Gaümann model was used to define columnar-to-equiaxed transition (CET), and the Rosenthal instantaneous moving point heat source model was used to determine solidification velocities V and thermal gradients G along the centerline of the melt pools. In general, the models matched the experiments.

Significance: Experimentally validated defect and processing maps will accelerate the prediction and control of microstructures produced by AM of refractory alloys and the optimization of processing conditions for a given AM technology

Reference: M. Le Corre, K. Mullins, T. Pollock, A. Clarke, "Implementation of solidification modelling for additive manufacturing of Nb-47Ti and C103", 2024, in preparation.

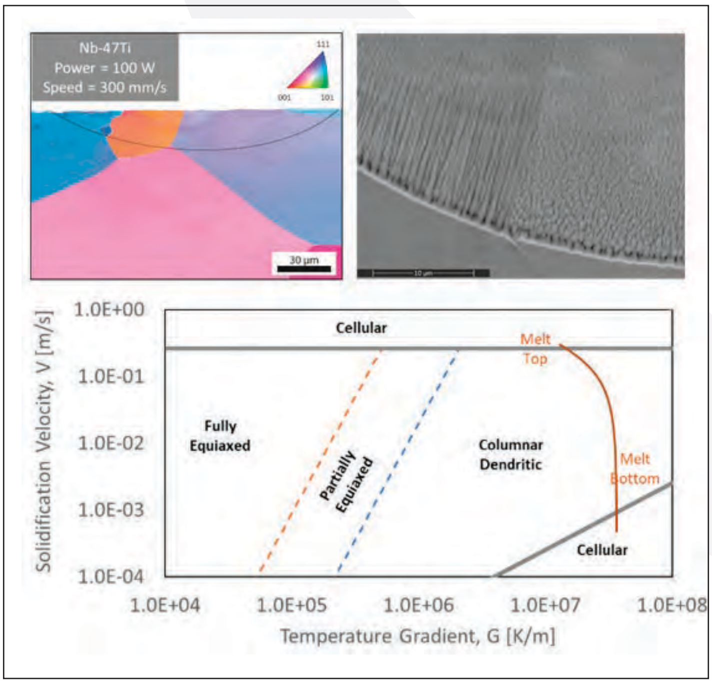


Figure: Electron back scatter diffraction of an Nb-47Ti melt pool track and scanning electron microscopy of the melt pool edge, showing the transition from cellular to columnar dendritic solidification structure. (Bottom) Processing map and G,V melt pool trace (solid orange line), predicting the transition from cellular to columnar dendritic structure at the edge of the melt pool. Maps such as these agreed with experimental observations.

Q1: Will there be a local equilibrium at the interfaces with large thermal gradients?

Answer: Measured solid-liquid interface velocities and microstructure characterisation of high pedigree, Ni-Mo-Al single crystals reveal discrepancies with classical theories regarding the onset velocity for absolute stability of a planar interface.

Background and Motivation: Ternary alloy single crystals with the chemistry Ni-22.2Mo-2.8Al (wt.%) were selected to study the absolute stability limit and validity of one of the most widely used models for solidification found in the literature by Kurz, Giovanola, and Trivedi (i.e., the KGT model). This model was used to determine the solidification modes corresponding to different temperature gradients (G) and growth velocities (V). Thermophysical parameters for the alloy were assessed from thermodynamic equilibrium calculations. Values for parameters like diffusivity, solid-liquid interfacial energy, and kinetic coefficient of the interface were based on literature values

Results: (i) Approximately 200 µm-thick, single crystal samples were prepared for simulated AM experiments, keeping the <100> direction along the sample thickness and either the <100> or <110> crystallographic direction perpendicular to the sample top surface. Samples were subjected to laser spot melting (1 ms dwell time) at powers ranging from 104 to 260 W during real-time synchrotron x-ray imaging at the Advanced Photon Source at Argonne National Laboratory. Solidification velocities were directly measured and found to increase from the bottom to the top of the melt pools.

(ii) The melt pools were characterised by electron microscopy. For both crystallographic orientations, an initial region of planar growth was seen at the bottom of the melt pools, followed by a transition to cellular and columnar dendritic patterns with increasing solidification velocity. The melt pools had the same crystallographic orientation as the substrate; the main growth direction of cells/dendrites clearly corresponded to <100> directions, as expected for dendrites, but not cells. No indication of re-stabilization of a planar interface at the melt pool tops was found, as predicted by the classical theoretical limit (KGT).

Significance: (i) Approximately 200 µm-thick, single crystal samples were prepared for simulated AM experiments, keeping the <100> direction along the sample thickness and either the <100> or <110> crystallographic direction perpendicular to the sample top surface. Samples were subjected to laser spot melting (1 ms dwell time) at powers ranging from 104 to 260 W during real-time synchrotron x-ray imaging at the Advanced Photon Source at Argonne National Laboratory. Solidification velocities were directly measured and found to increase from the bottom to the top of the melt pools.

Reference: D. Tourret, J. Klemm-Toole, A. Eres-Castellanos, B. Rodgers, G. Becker, A. Saville, B. Ellyson, C. Johnson, B. Milligan, J. Copley, R. Ochoa, A. Polonsky, K. Pusch, M. Haines, K. Fezzaa, T. Sun, K. Clarke, S. Babu, T. Pollock, A. Karma, A. Clarke, "Morphological stability of solid-liquid interfaces under additive manufacturing conditions", Acta Materialia, 2023, 250:118858

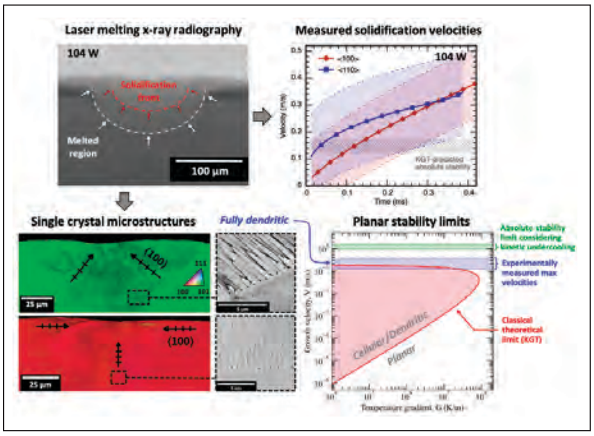


Figure: In-situ/ex-situ characterisation of Ni-Mo-Al single crystals during simulated AM and comparison to solidification theory/modeling

Q2: Are phenomenological interface response functions valid for multi-component alloys?

Answer: In a model Ni-1.9%Mo-6.6%Al (wt. %) alloy undergoing simulated additive manufacturing (AM), columnar to equiaxed transition (CET) was found to not be operational; new grains at the melt pool edge formed by recrystallization and grew epitaxially during solidification.

Background and Motivation: The reduction in Ni superalloy component lifetime is often associated with the existence of a polycrystalline structure with solidification cracks. For example, stray grain formation under repair-like solidification conditions has been associated with two different phenomena: CET that occurs when the thermal gradient (G) and solidification velocity (V) cross boundary conditions between these two modes; and recrystallization. Due to the complex thermal and temporal (e.g., millisecond) conditions experienced during laser melting, more work is needed to better understand new grain formation phenomena.

Results: Grain formation during laser spot melting experiments was assessed for a Ni-1.9%Mo-6.6%Al (wt. %) alloy subjected to different spot melting conditions: 108 W-1 ms, 108 W-2 ms and 260 W-1 ms. Simulated AM and real-time synchrotron x-ray imaging were conducted at the Advanced Photon Source at Argonne National Laboratory in thin, 200 µm-thick substrates.

(i) 3D TriBeam melt pool characterization (Figure 1a-c) shows equiaxed grains formed predominantly on the sample surface at the fusion line via recrystallization that later grew epitaxially during solidification. Higher Geometrically Necessary Dislocation (GNDs) densities are seen at the sample surface (Figure 1d), suggesting that plastic deformation due to sample preparation may have triggered recrystallization at these locations during simulated AM. Non-isothermal Avrami modelling (Figure 2a) shows that, for a fixed level of surface deformation, increasing laser power and/or decreasing laser spot dwell time decreases the fraction of recrystallized grains.

(ii) New grain formation via CET has been ruled out by modelling, using a pseudo-binary and a multicomponent approach, and utilizing G,V traces of melt pools obtained by accurately calibrated computational fluid dynamics modelling (Figure 2b).

Significance: More work is needed to determine the genesis (e.g., recrystallization and CET) of refined grains and to predict and control new grain formation under laser melting and AM conditions.

Reference: J. Lamb, R. Ochoa, A. Eres-Castellanos, J. Klemm-Toole, M.P. Echlin, T. Sun, K. Fezzaa, A. Clarke, T. Pollock, "Quantification of melt pool dynamics and microstructure during simulated additive manufacturing", Scripta Materialia, 2024, in review

A. Eres-Castellanos, R. Ochoa, J. Lamb, C.A. Hareland, C. Becker, B. Lim, J. Klemm-Toole, K. Fezzaa, S.P. Ringer, S. Primig, X. Liao, T. Pollock, P.W. Voorhees, A.J. Clarke, "Contributing factors to high-quality Ni-Mo-Al single crystal repairs by laser remelting: a multimodal approach", 2024, in preparation

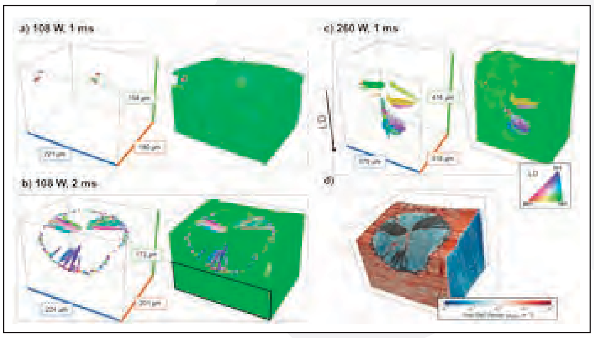


Figure: (a-c) 3D TriBeam datasets corresponding to three different laser melting conditions; (d) GNDs in (b).

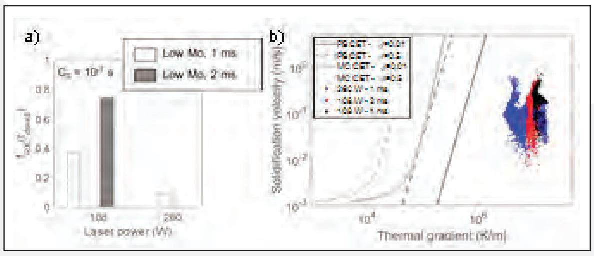


Figure 2. (a) Non isothermal Avrami modelling results and fraction of recrystallized grains at laser shutoff; (b) CET modelling via a pseudo-binary and a multicomponent approach, where G,V traces are superimposed.

Q4: Can we evaluate plastic instabilities within phases under complex thermal gyrations?

Answer: In simulated additive manufacturing (AM) of Ti-10V-2Fe-3Al (wt.%) (Ti-1023), residual strains from the rapid cooling rates experienced during the build process do impact the propensity for deformation-induced phase transformation of the metastable parent phase.

Background and Motivation: Metastable β-Ti alloys are an underutilised category of Ti alloys that can exhibit deformation induced phase transformations to modify mechanical response. One β-Ti alloy of particular interest is the transformation induced plasticity (TRIP)-susceptible Ti-1023, where the parent phase undergoes phase transformation to martensite with deformation. The presence of TRIP during the deformation of Ti alloys has been shown to impart unique mechanical properties for applications requiring increased damage tolerance, for example. However, how metastable β-Ti alloys respond to AM is not well understood. Do these alloys better accommodate potential residual stresses/strains that may develop and typically lead to cracking in traditional alloys like Ti-6Al-4V (wt.%)? A thorough understanding of how Ti-1023 responds to the transient conditions inherent to AM is needed.

Results: (i) Thermal strains, which develop from thermal contraction of melt-pools during cooling, trigger the β (parent phase) to α″ (martensite) transformation associated with TRIP. Formation of α″ occurred in the melt-pool and neighbouring areas (i.e., the heat affected zone HAZ). Increases in laser power created larger regions observed to exhibit TRIP.

(ii) Thermal cycling, introduced by successive laser hits, produced regions of gradual α″ reduction, unseen in single-hit experiments. Complementary thermomechanical modelling revealed these regions developed due to local strain relief, which enabled the reversion of the strain-induced α″ back into the parent β-phase.

(iii) Large regions of abrupt α″ suppression were also observed. These locations correlated to unfavourable grain orientations with respect to {112} 111 α″ shear. However, sufficiently large residual strains could result in transformation, regardless of grain orientation.

Significance: TRIP in AM-produced β-Ti alloys is influenced by processing and microstructural characteristics. These alloys may afford the accommodation of residual stresses/strains during a build process to produce high quality, crack-free parts with tuned microstructures and properties.

Reference: C. Jasien, A.I. Saville, K. Fezzaa, T. Sun, J. Foltz, K. Clarke, A. Clarke, "Effect of processing strategies on location and extent of TRIP product in simulated PBF-LB of Ti-1023, 2024, in preparation

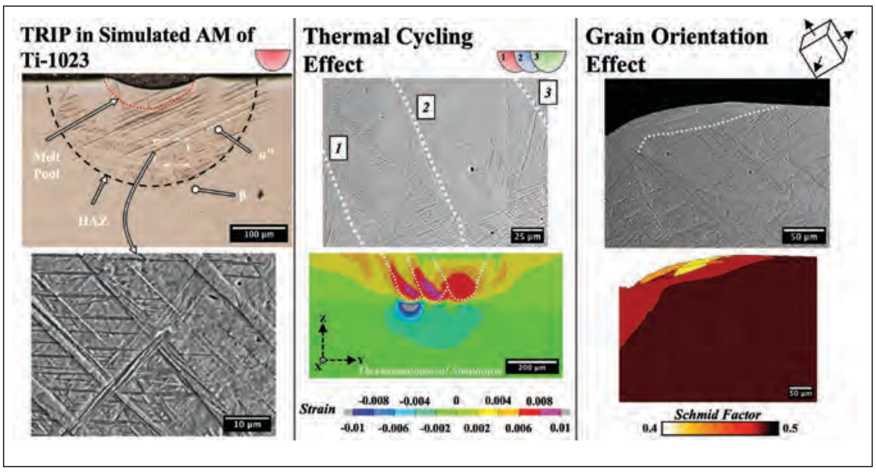


Figure: (Left) Formation of α″ after melt-pool solidification. (Middle) Reversion of α″ after the introduction of thermal cycling. (Right) Suppression of α″ based on grain orientation with respect to the α″ shear system.

Q4: Can we evaluate plastic instabilities within phases under complex thermal gyrations of Additive manufacturing (AM)?

Answer: In AM, thermal transients accompanied with mechanical constrains may lead to thermomechanical (TM) transients repeated numerous times over a single layer. Spatial and temporal changes in thermal gradients are expected to induce stresses above and below the yield stress of phases, leading to complex plastic instabilities.

Background and Motivation: Prior research has shown that metallurgical features in AM are primarily dictated by an overall interaction of thermal-mechanical-metallurgical signatures and local phase stability, due to the layer-by-layer build-up of part. However, macro level non-uniform plastic strains dictated by local changes in thermal expansion coefficients and micro-level plasticity due to crystallographic misfits between phases may also lead to complex phase transformations. To validate this hypothesis, we used Ti-6Al-4V as a model material and evaluated the role of accumulated plastic strains within β (hexagonal close-packed HCP) - β (body-centred cubic BCC) phases on alloying element partitioning.

Results: (1) In-situ behaviour of the $\alpha + \beta$ phases were monitored during imposed thermo-mechanical fluctuations between 400 °C and 700 °C using synchrotron radiation. An increase in the lattice strain of the β(110) peak was noticed along with an increase in the lattice parameter to α = 3.22 Å. The overall residual impact of the thermo-mechanical reversals also revealed an increase in the β phase fraction to around 3.5 Å ± 0.01%. (2) Atom probe tomography (Fig. 1) further confirmed the increase of vanadium (V) concentration in the β phase where the V concentration of 22.4 ± 0.19 at.% was noted in the centre region of the β phase. The changes in the V concentration profile along with the changes in the lattice parameters discussed earlier confirm that dynamic transformations do take place during TM cycling. (3) To confirm our hypothesis on the movement of the α/β interface in conjunction with solute re-distribution, a diffusion based kinetic model, i.e., DICTRA® was used. The results show the dynamic nature of the V concentration profile as the interface moves back and forth to maintain phase stability with relative differences in stored energy. The reduction of V concentrations within the β phase indicates a more stable β formation during TM cycling. With an addition of 400 J/mole and 500 J/mole to the HCP α phase, the α/β interface movement is much more significant supporting our experimental results.

Significance: Complex thermal gyrations associated with AM cause local variations in microstructure and the 'as-built' mechanical properties. This will be relevant to the validation of integrated computational modelling of AM processes.

Reference: Kumar et. al., Coupling Strain Partitioning and Phase Transformations in Ti6Al4V during Thermo-Mechanical Gyrations (unpublished research 2024).

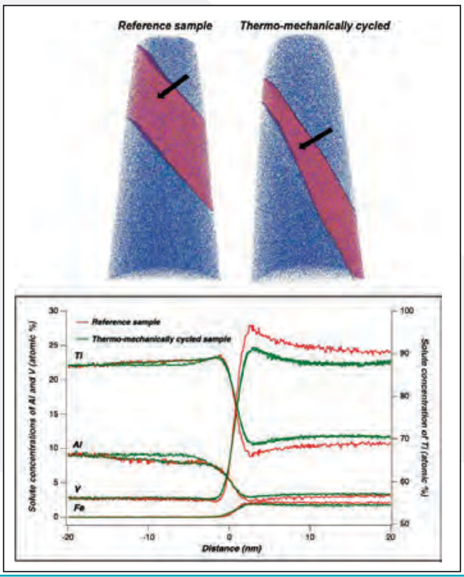


Figure: (top) atom images and (bottom) proxigram concentration profiles across the α/β interface measured by atom probe tomography.

PHASE 2

Q1: Will there be a local equilibrium at the interfaces with large thermal gradients?

Answer: To address this fundamental question we have developed a framework for the selection of the optimal high-throughput data collection workflow for microstructure characterization of AM Ti-6Al-4V using a deep-learning based image denoiser.

Background and Motivation: Autonomous experimentation (AE) systems have been used to greatly advance the Integrated Computational Materials Engineering paradigm. For AE systems' potential to be fully realized, their decision authority must be expanded in a practically implementable manner for experimenters. To achieve this, we designed a robust algorithmic framework for the selection of high-throughput workflows that can be completed by AE systems with minimal human intervention. In a case study, we used the framework to select the optimal high-throughput workflow for microstructure characterization on an AM Ti-6Al-4V sample using a deep-learning based image denoiser.

Results: The developed framework first searches for data collection workflows that generate high-quality information and then selects the workflow that generates the highest-value information as per a user-defined objective. For the typical scanning electron microscopy (SEM) characterization procedure in our case study, the selected workflow reduced the collection time of backscattered SEM images by a factor of 5 times as compared to the case study's benchmark (ground truth) workflow, and by a factor of 85 times as compared to the workflow used in a previously published study.

Significance: The developed framework enables optimal workflow selection for high-throughput microstructure characterization of metal AM components resulting in a considerable reduction in collection time of electron microscopy data. The steps of the SEM characterization procedure in our case study were well defined and understood. However, this framework can also be used to determine the optimal workflow for a scenario where numerous pieces of equipment, data processing pipelines, and types of models can be used in various combinations to arrive at the same kind of information, provided that the objective and ground-truth are still clearly defined by a human researcher. We envision that this framework will also assist with the growing problem of data continuity and reproducibility and enable the development of metal AM databases that are critically important to understand underlying process-structure-property relationships.

Reference: [1] Casukhela, R.; Vijayan, S.; Jinschek, J.R.; Niezgoda, S.R. (2022). A framework for the optimal selection of high-throughput data collection workflows by autonomous experimentation systems. Integrating Materials and Manufacturing Innovation. 11(4). 557-567. 10.1007/s40192-022-00280-5.

[2] Shao, M; Vijayan, S.; Nandwana, P.; Jinschek, J.R. (2020). The effect of beam scan strategies on microstructural variations in Ti-6Al-4V fabricated by electron beam powder bed fusion. Materials and Design. 196. 109165. 10.1016/j.matdes.2020.10916510.1016/j.matdes.2020.109165.

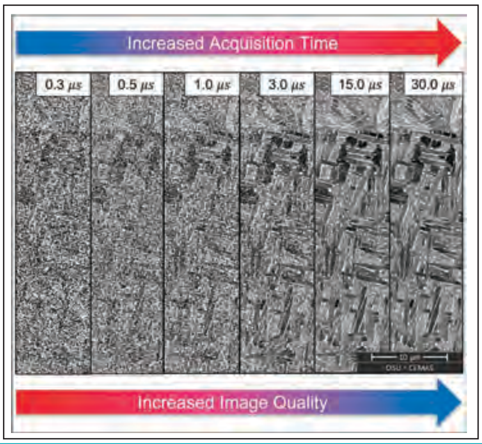


Figure: Backscattered electron (BSE) scanning electron microscopy (SEM) images taken with increasing dwell time visualizing one of the many trade-offs between acquisition time vs. image quality.

Q1: Will there be a local equilibrium at the interfaces with large thermal gradients?

Answer: In electron beam melted powder bed fusion (EBM-PBF) Haynes 282, changes in build geometry and associated scan velocity result in considerable local heterogeneity in microstructure and hardness.

Background and Motivation: Haynes 282 is a precipitation strengthened Ni-based superalloy with applications in industrial gas turbine engines due to high temperature creep resistance and thermal stability. Its improved performance at high temperatures has been attributed to the existence/appearance of the gamma prime (γ′) phase. The high weldability of Haynes 282 lends itself to fabrication by metal-based AM techniques, such as EBM-PBF. AM is advantageous for producing near net shapes of complex geometries with reduced material waste and potentially enhanced properties. To enable industrial qualification of EBM-PBF Haynes 282 components, it is critical to understand the evolution of solidification and solid-state microstructures in response to changes in build parameters and associated local variations in thermal history within the build. This work focuses on the effect of variations in build height, column thickness, and scan velocity on the size and morphology evolution of γ′ precipitates, γ grains and carbides in EBM-PBF Haynes 282. Site-specific microhardness was obtained to investigate the effect of microstructural heterogeneity on mechanical properties across the build.

Results: Considerable heterogeneity in microstructure was found across an EBM-PBF Haynes 282 build composed of thin columns of different thickness and similar height over a cuboidal base. γ′ precipitates were observed in the entire as-built microstructure but decreased in size along the build direction. This decrease in γ′ size correlated to an increase in Vickers microhardness. Significant changes in γ′ size and phase fraction were observed in response to drastic changes in scan velocity and column thickness in the top part of the build. Thermal modeling revealed associated changes in thermal history locally within the build that explain the observed variations in microstructure. It was also found that the presence of γ′ precipitates in as-built EBM-PBF Haynes 282 results in mean hardness that is comparable to what is reported for the conventionally processed alloy and does not significantly increase after a one-step or two-step post-process heat treatment.

Significance: Understanding local variations in as-built microstructure and mechanical properties as a result of changes to build parameters and associated local changes in thermal history are critical to improve reliability and consistency of metal AM components.

Reference: [1] Gupta, A.; Vijayan, S.; Schmid, O.; Jinschek, J.; Fink, C. "High Throughput Characterization to Quantify Microstructural Heterogeneities in Additively Manufactured Haynes 282", Microscopy and Microanalysis 28(S1), 2022, 2088-2090. 10.1017/S143192762200808X.

[2] Mourot, A., Gupta, A., Vijayan S., Jinschek J., Fink, C. "Gamma Prime Characterization in Additively Manufactured Haynes 282 after One-Step and Two-Step Post-Process Heat Treatments." Microscopy and Microanalysis 29(S1), 2023, 1421-1422. 10.1093/micmic/ozad067.731.

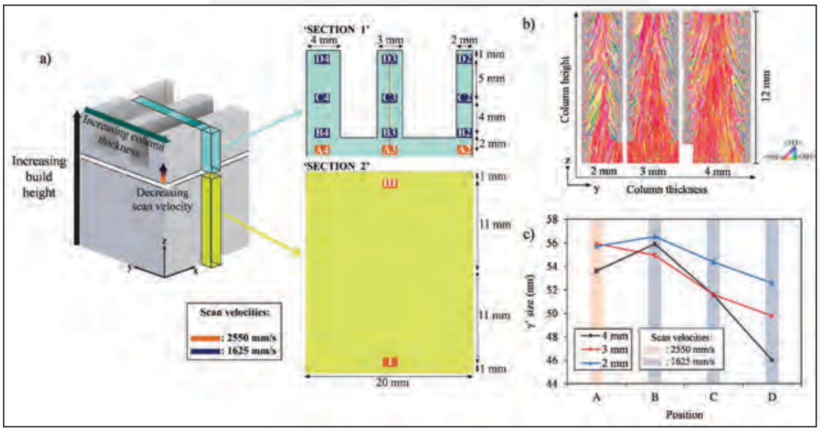


Figure: a) EBM-PBF Haynes 282 build geometry indicating variations in build geometry and scan velocity and locations of site-specific microstructure and microhardness characterization, b) grain structure in column of different thickness from EBSD, and c) variations in gamma prime (γ′) size in column section.

5. Research Highlights (2018 - 2022)

Q1: Will there be local equilibrium at the interfaces with large thermal gradients?

Answer: In electron beam melted (EBM) Ti-6Al-4V (Ti64), preferential vaporization of aluminum can be used to examine the local melt pool fluid dynamics, demonstrating that local equilibrium at the interfaces is not reached, and that solidification kinetics occur on a time-scale that prevents large scale mixing and homogenization.

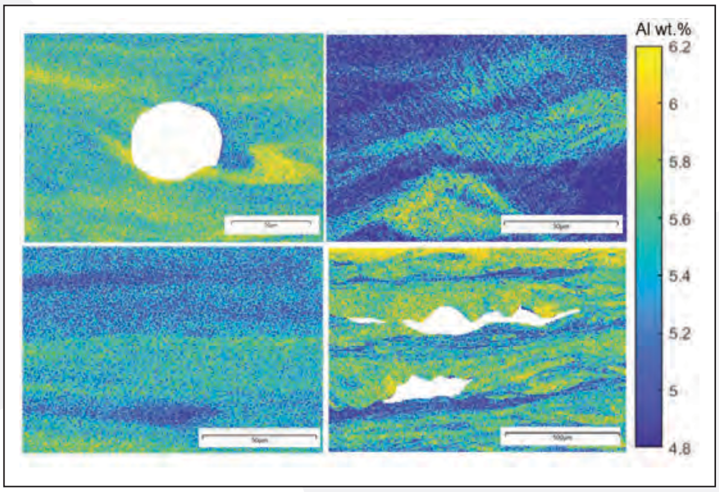
Background and Motivation: Preferential vaporization of select elements in metals processing, notably high temperature additive manufacturing processes, lends itself to poor compositional control. In titanium alloys, due to differences in the melting points of alloying elements, aluminum is known to have a much higher evaporation rate, leading to local and macro-scale undesirable variations in EBM built parts [1]. Understanding the interaction of these compositional variations with defects present in the build can help reveal key information about fluid flow dynamics in molten pools, the kinetics of solidification, and other physical phenomena in EBM.

Results: Observing the compositional variations that occur in the presence of defects, there are notable 'turbulent features' not present in compositional variations that occur in regions without defects. These turbulent features, structures or morphologies, for lack of a better term, show the way in which the presence of defects alter the fluid flow within melt pools, and also are evidence that the defects were present when the material was in a melted state. Figure 1 shows multiple EDS maps of local compositional variations, with one of these turbulent structures notably present to the left of the spherical pore in the top left quadrant of the image. In addition, the fact that these turbulent structures can be seen at all demonstrates that the kinetics of solidification in the EBM melt pools is occurring on a time scale that prevents any large-scale mixing and homogenization, and that future thermal cycles (from layers above the location of the compositional variation) does not affect the distribution of aluminum in the solid material, or, again, allow for homogenization. These results show the lack of a clear equilibrium state when it comes to the molten material and the resulting phase transition into solid-state, unaffected by the further solid-state phase transformations that follow.

Significance: These results show the non-equilibrium nature of melt pools in EBM build processes, revealing information about fluid dynamics before solidification and thus adding to a deeper understanding of the science involved in metal-based AM in general.

Reference: [1] M.J. Kenney, K. O'Donnell, M.J. Quintana, and P.C. Collins, *Spherical pores as 'microstructural informants': Understanding compositional, thermal, and mechanical gyrations in additively manufactured Ti-6Al-4V,* Scripta Materialia, Vol. 198, (2021).

Figure: Energy dispersive spectroscpy maps of local compositional variations in Ti64 samples, in regions both with and without defects.



Q1: Will there be local equilibrium at the interfaces with large thermal gradients?

Answer: In AM Ti-6Al-4V, a difference in melt strategies result in significant changes in thermal history which subsequently affect the build microstructure including phase fraction and texture.

Background and Motivation: One of the key processing variables to achieve microstructural changes in AM builds is the melt strategy. Scientists at the Oak Ridge National Laboratory have demonstrated that a spot-melt strategy can be used to effectively manipulate the grain size, grain orientation, and the grain morphology from columnar to equiaxed. Our study uses high-energy synchrotron x-ray diffraction technique and Rietveld refinement to understand the effect of spot melting on the build texture, orientation relationship, variant selection, phase fraction, and their distribution along the height of an E-PBF Ti-64 alloy build. From this study, we understand the relationships between the process parameters and build microstructure, the latter of which govern the mechanical properties of the final AM build.

Results: We found that: (i) The β phase fractions in both the linear and spot E-PBF Ti-64 builds were significantly higher than the feedstock powder. The linear-melt sample showed an overall higher β phase fraction and also a stronger trend of decreasing β fraction towards the substrate compared to the spot melt case likely due to a gradual increase in the heat accumulation towards the top of the build in the linear-melt case; (ii) Spot-melt strategy showed β<100> fiber texture, whereas the linear-melt strategy showed a cube texture about the build axis; (iii) The Burgers orientation relationship (BOR) between the parent β phase and the daughter α phase was clearly observed in both spot- and linear-melt samples across the entire build height despite the significant thermal excursions experienced during the build process; (iv) The daughter α texture intensities mostly inherited the trends set by the parent β texture. The α variant selection was more predominant in the spot-melt than in the linear-melt samples. In the case of spot-melt samples, the planar variant pair of V(2,8), corresponding to $(101)_\beta$ with $[222]_\beta$ and $[222]_{\beta'}$, was most consistently preferred across the build height.

Significance: In terms of both the phase fraction and texture, the novel spot-melt strategy produced a more homogeneous microstructure across the build height compared to the linear-melt case. This will result is a more homogenous mechanical property distribution across the build height.

Reference: [1] R.R. Kamath, P. Nandwana, Y. Ren, and H. Choo, *Solidification texture, variant selection, and phase fraction in a spot-melt electron-beam powder bed fusion processed Ti-6Al-4V,* Additive Manufacturing, Vol. 46, p. 102136, (2021)

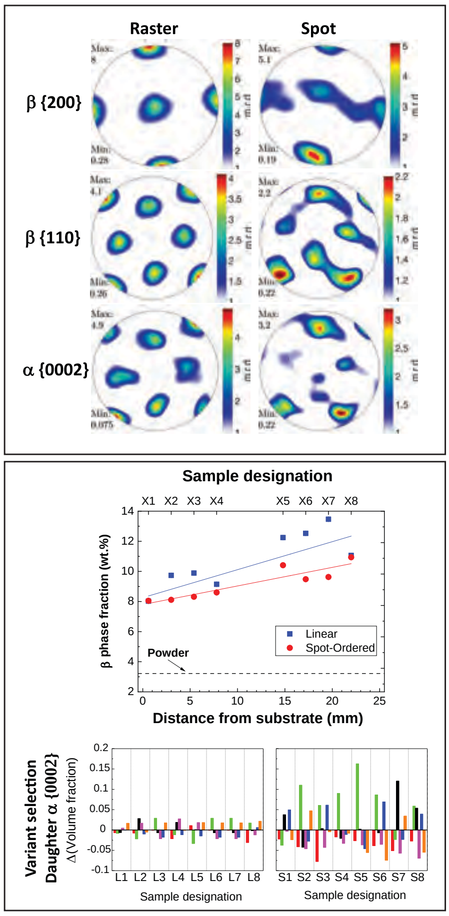


Figure: β {200} pole figures show raster exhibits cube texture and spot exhibits fiber texture. BOR is observed in the form of overlapping poles in parent β {110} and daughter β {0002}. Spot shows a higher degree of variant selection. β phase fraction increases with build height – more so for the raster than the spot.

Q1 & Q3: Will there be a local equilibrium at the interface under large thermal gradients? How does interface motion respond to rapid reversals of thermal gradients?

Answer: In an effort to address these fundamental questions we have developed a novel in situ heating device to observe dynamic processes under 'AM like' process conditions (large thermal gradients-106 K/m and rapid thermal reversals of upto106 K/s), inside the transmission electron microscope.

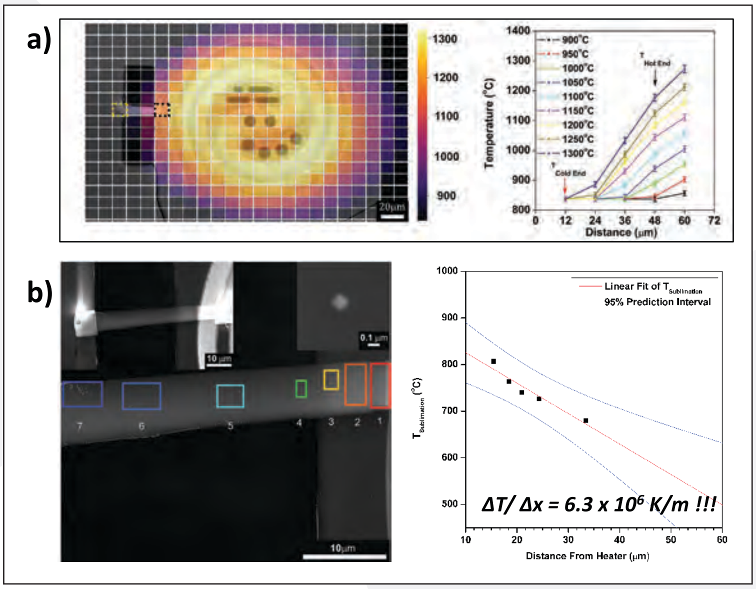
Background & Motivation: During electron beam melting powder bed fusion process, the electron beam (EB) interaction with powder particles creates varying spatio-temporal transient conditions, which affects the build microstructure. Although the phenomenological effects of these transients are understood, there is still a lack of understanding about metallurgical processes that occur within 'previously melted layers' during powder bed fusion (PBF). Currently, complex dynamic processes in the melt pool can be monitored using 'line of sight' techniques such as infra-red thermal imaging or observed directly via in situ synchrotron X-ray-based AM simulators. However, thus far, efforts to directly observe solid state dynamic processes (within previously melted layers) at high spatial resolution, under AM operating conditions, have been limited. Our goal is to address this grand challenge by developing an in-situ TEM based methodology to observe solid state dynamic processes, at high spatial resolution, under 'AM like' process conditions.

Results: A commercially available micro electro-mechanical system (MEMS) based in situ TEM heating device was modified, to generate extreme thermal gradients across a lamella. The heating device was modified by milling a large window adjacent to the heater using a focused ion beam (FIB) in a dual beam scanning electron microscope (SEM). A large FIB cut lamella was placed across the modified window to perform our in-situ gradient heating + rapid thermal reversal study. In order to test the validity of our concept device, the thermal gradient across the lamella was determined by measuring the temperature across the sample using two different approaches; infra-red thermography and the Ag-nanocube sublimation approach. This study confirmed that an extreme thermal gradient of ~ 6.3x106 K/m was generated across a lamella at a setpoint temperature of 1000˚C.

Significance: This in situ TEM characterization approach allows us to gain insight into dynamic processes that govern transformation pathways in novel alloy chemistries. The knowledge gained from these in situ studies will aid in AM process development and alloy design efforts.

Reference: [1] S. Vijayan, R. Wang, Z.Kong, J.R. Jinschek, *Quantification of extreme thermal gradients during in situ transimssion electron microscope heating experiments,* Microsc. Res. Techn. 85, 1527 (2022).

Figure: Temperature calibration of modified MEMS heating device via (a) infra-red thermal imaging and (b) Ag-nanocube sublimation approach. Figures replicated from publication [1].



Q2: Are phenomenological interface response functions valid for multicomponent alloys?

Answer: In high strength Ni-Co alloys mm3-scale 3D datasets reveal that the scan rotation strategy strongly influences the development of the columnar grain structure, with lower scan rotation angles inhibiting columnar grain growth through multiple print layers, in comparison to columnar grain growth with 90° scan rotations.

Background and Motivation: One of the challenges for additive manufacturing is the control of the grain structure and associated bulk mechanical properties given the complex scan strategies employed in laser and electron beam powder bed approaches. The TriBeam tomography technique developed at UCSB is ideally suited for understanding the response of grains to the complex scan strategies and remelting processes that occur during laser and electron beam powder bed printing processes. The TriBeam fs laser enabled FIB SEM is one of the only 3D serial sectioning methods available that can gather mm-scale multimodal datasets at sub-μm voxel resolutions; these length-scales are highly relevant to the scale of 3D printing. Multimodal chemical, crystallographic and morphological information can be gathered rapidly on a layer-by-layer basis and reconstructed in large (gigabyte to terabyte scale) 3D datasets. In this work, two selective laser melted prints of a Ni-Co alloy are reconstructed to examine how scan rotation and contouring control grain morphology and texture.

Results: The two datasets and their pole figures are shown in Figure 1. Both samples were printed utilizing a bi-directional raster scan strategy with one using a 90° scan rotation (Figure 1a) and one using a 16° rotation (Figure 1b). In both cases, the pole figures show heightened texturing along the build direction. The 90° dataset shows a secondary preference within the build plane along the scan directions due to the high frequency at which the raster direction repeats (every other build layer) during printing. The repetition also leads to large columnar grains occupying most of the dataset volume. In contrast, the 16° dataset shows a more uniform texture within the build plane, a consequence of the raster scan directions repeating every 45 build layers. Further, the decreased scan rotation between build layers and the resultant constant change in thermal gradient direction restricts columnar grain growth, as seen by both smaller columnar grains and a lower density of such grains.

Significance: The results show that unique thermal gradients induced with different scan strategies allow direct control of both the crystallographic texture and grain morphology of additively manufactured materials through fine tuning of printing parameters such as interlayer rotation.

Reference: [1] M.P. Echlin et al., "Recent Developments in Femtosecond Laser enabled TriBeam Systems", *JOM* 73, 4258, (2021) 10.1007/s11837-021-04919-0

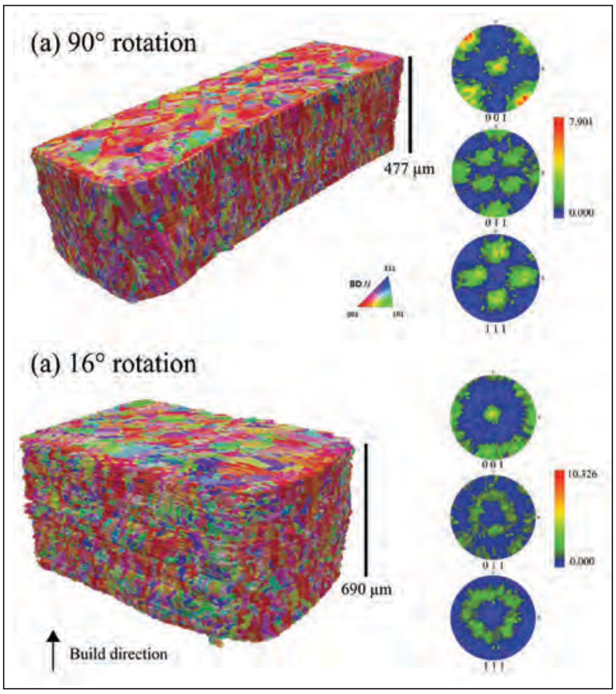


Figure: TriBeam 3D EBSD datasets and representative pole figures of SLM SB-CoNi-10 samples with (a) a 90° interlayer rotation and (b) a 16° interlayer rotation with a contour applied.

Q3: How does interface motion respond to rapid reversals of thermal gradients?

Answer: In simulated L-PBF of metastable beta-titanium, processing conditions have a large effect on resulting thermal gradients during solidification that lead to varying as-built microstructures, specifically grain morphology and phases present.

Background and motivation: Ti-6Al-4V has typically dominated in terms of use for metal AM processes, however beta-titanium alloys have begun to find increased use, due to their increased strength-to-density ratios, among other properties. Additionally, some beta-titanium alloys are known to exhibit Transformation-Induced Plasticity (TRIP) which can eliminate stress concentrations that typically result in cracking [1]. In this work, in-situ radiography experiments of simulated L-PBF of Ti-10V-2Fe-3Al reveals melt-pool solidification as a function of time and processing parameters. This information allows for calibration of computational fluid dynamics models using FLOW-3D®, which further informs of local solidification conditions such as thermal gradients. Combination of solidification velocities from experiments and thermal gradients from models allows for the development of solidification maps and subsequent prediction of as-built microstructures.

Results: (i) Increasing laser power results in decreasing solidification velocities as well as a transition into a keyholing regime (Figure 1a). Additionally, simulations predict a rapid increase in velocity at the final stages of solidification not observed in the experiments (Figure 1b). This increase has been previously reported during rapid solidification experiments like AM, suggesting simulations are able to reveal more details about solidification [2].

(ii) A solidification map for Ti-10V-2Fe-3Al was created to aid in the prediction of as-solidified grain morphology (Figure 1c). Thermal gradient and velocity predictions can accurately predict initial observations from top-down imaging. The only map that previously existed for titanium alloys was Ti-6Al-4V [3].

(iii) Post-mortem microstructural characterization reveals the presence of martensite plates throughout the solidified melt-pools and surrounding substrate (Figure 1d). This solid-state phase transformation occurs in response to the stresses that develop during solidification.

Significance: The combination of predicted thermal gradients and velocities during solidification provides a method for determining processing regimes that yield desirable microstructures.

Reference: [1] Y. J. Liu et al., *Materialia,* vol. 6, p. 100299, Jun. 2019. [2] J. T. McKeown *et al.*, JOM, vol. 68, no. 3, pp. 985–999, Mar. 2016. [3] P. A. Kobryn et al., *Journal of Materials Processing Technology*, vol. 135, no. 2–3, pp. 330–339, Apr. 2003.

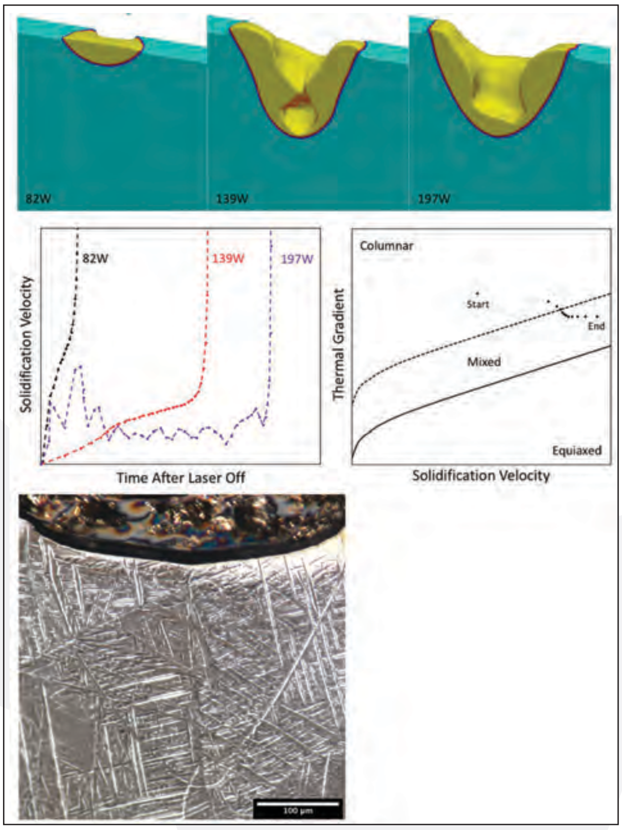


Figure: (a) Simulation results for three spot-melts and (b) Predicted velocities. (c) Solidification map with overlaid predictions. (d) Cross-section showing martensite filled melt-pool.

Q3: How does interface motion respond to rapid reversals of thermal gradients?

Answer: In duplex stainless steels, these conditions lead to formation of non-equilibrium phases in the as-printed condition and after post-AM heat treatments, with significant effects on the corrosion resistance.

Background and Motivation: Duplex stainless steels (DSSs) have a low thermal expansion coefficient and a relatively high thermal conductivity compared to austenitic stainless steels, making them fit for AM of complex and thin-walled geometries e.g., as heat exchangers [1]. The processing–microstructure–corrosion relationships in laser powder bed fusion (LPBF) DSSs are yet to be established in detail, especially at temperatures above room temperature, and after post-AM heat treatments [2]. Our study fills these important knowledge gaps by extending the current understanding of the pitting corrosion behaviour of DSSs processed by AM and after post-AM heat treatments using polarisation measurements, critical pitting temperature (CPT) experiments, and high-resolution correlative microscopy (Figure 1).

Results: We found that: (i) At room temperature, the polarisation curves do not show significant differences in the pitting corrosion resistance between as-built (AM), post-AM heat-treated, and hot-rolled 2205 DSS specimens in terms of pitting potentials and passive current densities despite significant differences in their microstructures. This is because no depletion of Cr occurs around intragranular Cr2N in AM DSS, as revealed by atom probe microscopy. A local depletion of Cr is observed adjacent to intergranular Cr2N particles, however, the Cr content in these regions remains above the critical values of 13 wt.%. (ii) The AM 2205 DSS shows a lower CPT compared to its hot-rolled counterpart (47°C vs. 61°C), while post-AM heat treatment results in a higher CPT, comparable to the hot-rolled specimen (60 °C vs. 61 °C). This is due to the non-equilibrium phase balance and composition of austenite and ferrite in the AM microstructure. The significantly lower pitting resistance equivalent number (PREN) value for the ferrite phase in the AM specimen is the main reason for the low CPT in this specimen, which results in the preferential dissolution of ferrite regions at temperatures above CPT. The post-AM heat treatment equalizes the PREN values for austenite and ferrite phases and, thus, enhances the CPT to values comparable to that of the hot-rolled counterpart [3].

Significance: There results show that unique thermal gradients of AM create in non-equilibrium phase balances and compositions, and this can significantly affect the in-service properties of materials. A thorough understanding of the relationship between AM processing – microstructure – corrosion properties is needed before duplex steel AM parts can be into service.

Reference: [1] Haghdadi et al., Materials Science Engineering A 835, 142695 (2022). [2] Laleh et al., International Materials Reviews 66, 563-599 (2021). [3] Haghdadi et al., Materials & Design 212 (2021) 110260.

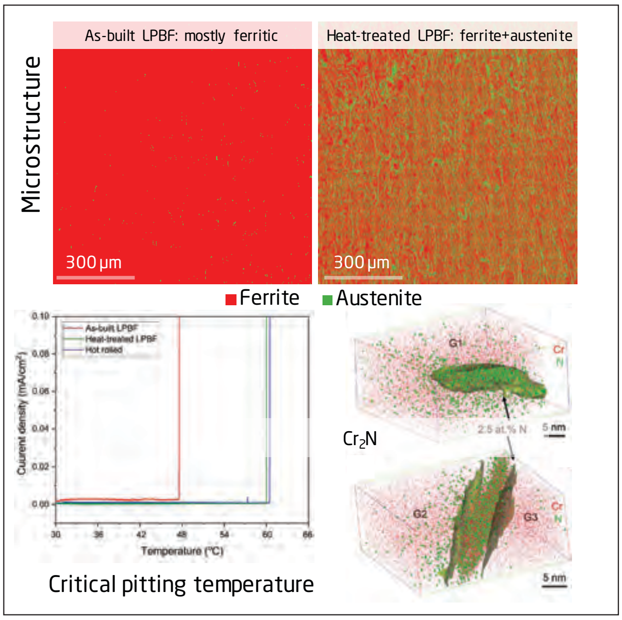


Figure. Electron back scatter diffraction phase map of the as-LPBF and heat treated 2205 DSS. (b) Current density versus temperature for different DSS specimens in 1.0 M NaCl solution. (c) and (d) D atom distribution maps of Cr and N adjacent to intragranular and intergranular Cr_2N, respectively.

Q3: How does interface motion respond to rapid reversals of thermal gradients?

Answer: In electron beam melted Ti-6Al-4V, rapid reversals of thermal gradients result in considerable microstructural variation when changing solely scan strategy.

Background and Motivation: Changes to build parameters in metallic additive manufacturing (AM) can drastically alter the thermal history of parts, even when only one parameter is changed. Altering scan strategy, the path an incident energy source takes to melt feedstock into an AM part is one such example. This work focuses on understanding the response of electron beam melted powder bed fusion (EBM-PBF) Ti-6Al-4V to changes in scan strategy. Alterations to thermal history can change both solidification and solid state microstructures, leading to considerably different material conditions after the build process has completed. Controlling this evolution is required to improve the implementation of metallic AM in structural or high-performance applications. Due to the present lack of microstructural control from this manufacturing technique, metallic AM has been prevented from realizing many of its design and engineering advantages.

Results: Implementing scan strategies where material is melted as a spot produce finer columnar/equiaxed dendritic microstructures during solidification and greater fractions of diffusional $\alpha+\beta$ colony solid state microstructures. Thermophysical modelling was able to predict the solidification scale and morphology, in agreement with reconstructed β-Ti microstructures. Increased presence of fiber textures in both reconstructed β-Ti and as-built α-Ti was found to indicate finer as-solidified and diffusional solid state microstructures. This texture ′ microstructure relationship was proposed as a diagnostic to ascertain microstructural condition, without extensive characterization/sample preparation, via techniques such as x-ray diffraction.

Significance: Understanding the systematic evolution of solidification and solid state microstructures in response to changes in build parameters is critical to improving consistency in the as-built condition of metallic AM parts. The insight generated here on texture, microstructure, and solidification phenomenon provide new diagnostics to evaluate as-built Ti-6Al-4V parts, while also developing fundamental knowledge on microstructural evolution under rapid thermal gradients in AM.

Reference: [1] A.I. Saville, S.C. Vogel, A. Creuziger, J.T. Benzing, A.L. Pilchak, P. Nandwana, J. Klemm-Toole, K.D. Clarke, S.L. Semiatin, and A.J. Clarke, *Texture evolution as a function of scan strategy and build height in electron beam melted Ti-6Al-4V, Additive Manufacturing,* Vol. 46, p. 102118, (2021)

[2] A.I. Saville, A. Creuziger, E.B. Mitchell, S.C. Vogel, J.T. Benzing, J. Klemm-Toole, K.D. Clarke, and A.J. Clarke, *MAUD Rietveld Refinement Software for Neutron Diffraction Texture Studies of Single- and Dual-Phase Materials, Integrating Materials and Manufacturing Innovation,* Vol. 10(3), pp. 461-487, (2021)

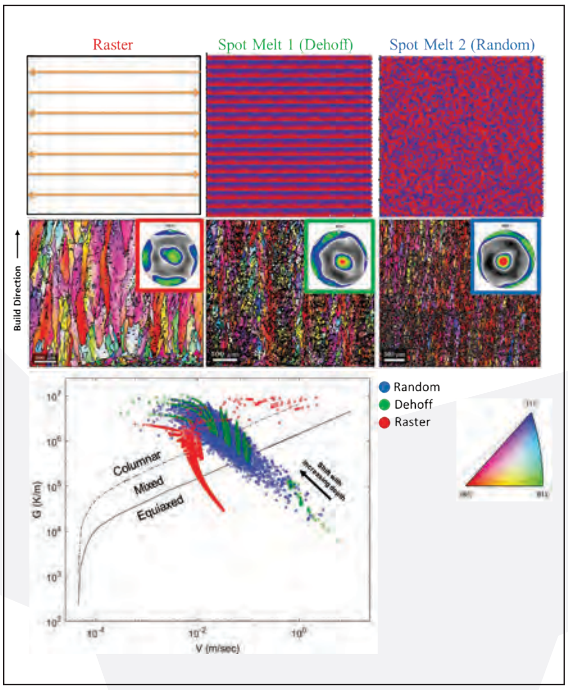


Figure. Illustration of the three different scan strategies implemented for this work. (Middle) Reconstructed β-Ti IPF maps of the three different scan strategies with corresponding textures plotted in pole figures. Build direction of pole figures is out of the page and up the page for reconstructed IPF maps (bottom) Predicted solidification conditions for the three scan strategies from thermophysical modelling.

Q4: Can we evaluate plastic instabilities within phases under complex thermal gyrations?

Answer: Phase instabilities significantly impact the microstructure and mechanical properties. The developed advanced Ex-situ and In-situ optical techniques help understand the role of spatial and temporal variations of thermal-mechanical signatures

Background and motivation: The 3D surface topological information can be obtained by 3D scanning, providing 3D point cloud data to evaluate the geometrical and dimensional quality of the printed parts, which includes critical quality information of the print, such as melt pool size, surface roughness, pores, other defects or unexpected process alterations, etc. But the current technology for AM in-situ monitoring is very limited. This is primarily due to the insufficient spatial resolution and the slow scan speed of current 3D scanning technologies. This work is to implement a new 3D scanner that can meet the in-process monitoring needs for metal AM.

Results: (i) In this study, a high spatial resolution 3D SLS is successfully built and calibrated (see Figure). The scanner can measure the surface with 5 µm spatial resolution so that it can resolve key surface features.

(ii) It achieves high accuracy results with a 0.056 µm average error. Furthermore, it takes as few as 4 seconds for measurement, which is sufficient for layer-by-layer characterization and uses 15-seconds of computational time to provide over 16 million data points.

(iii) The efficacy of the proposed scanner is demonstrated by an EBM manufactured part. The validation result concludes that the proposed 3D scanner has superior performance and can resolve more detailed melt pool features.

Significance: This high resolution and high-speed in-situ scanner can help understand the mechanical property of the print by capturing spatial and temporal variations of thermal-mechanical signatures.

Reference: [1] 0R. Wang, A.C. Law, D. Garcia, S. Yang, and Z.J. Kong, *Development of structured light 3D-scanner with high spatial resolution and its applications for additive manufacturing quality assurance, The International Journal of Advanced Manufacturing Technology,* Vol. 117, No. 3, pp.845-862, (2021).

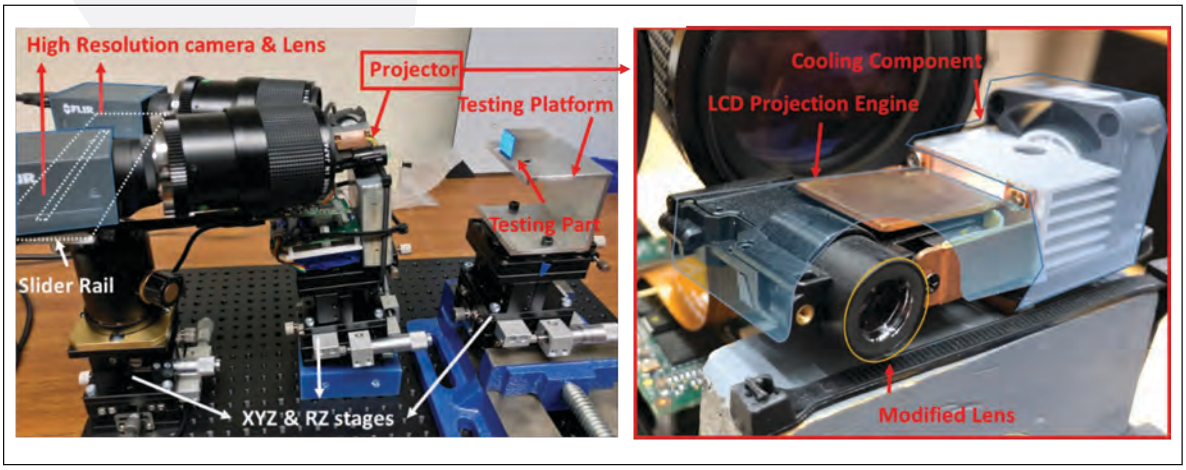


Figure. (a) The developed 3D scanner fixture design. (b) Detailed view of the modified tiny-area projector, the focal length of the original lens was increased by adding a spacer.

Q4: Can we evaluate plastic instabilities within phases under complex thermal gyrations?

Answer: In AM Ti-6Al-4V, complex thermal gyrations cause phase instabilities that have a profound impact on the 'as-built' microstructure and resulting mechanical properties.

Background and motivation: The extreme thermo-mechanical history of AM Ti-6Al-4V introduce important new microstructural phenomena that must be understood to elucidate structure-property-processing relationships. The highly non-equilibrium and cyclical nature of the thermal cycles creates the potential for novel phase transformation pathways with a fundamental influence on the resulting properties. This work provides a description of the phase transformation pathway of E-PBF Ti-6Al-4V, noting the first direct observation of the coexistence of two β phase variants within the 'as-built' condition. The implication of the phase transformation pathway on hardness is discussed in relation to chemical variation and O pickup.

Results: (i) Local β phase fractions vary significantly throughout the build. Two 'phase variants' of the high temperature β phase are retained in the 'as-built' condition, distinguishable by their relative crystallographic orientations and composition.

(ii) The final microstructure contains both β 'phase variants', α and potentially the metastable α'. The phase transformation pathway is shown in Figure 1 and begins with the formation of α' via rapid cooling from high-temperature parent β. Subsequent thermal cycling above the β-transus temperature leads to a mixed microstructure of $\alpha' + \alpha + \beta_r$, where βr denotes retained β that shares its crystallographic direction with its parent β grain. An in-situ anneal results in the decomposition of α' to $\alpha + \beta_d$, where β_d denotes β formed below the β-transus.

(iii) O was higher at the bottom of the build due to O pickup during the printing process. Oxygen pickup was found to have a predominant effect on measured hardness and its concentration should be carefully monitored.

Significance: Complex thermal gyrations associated with AM cause local variations in microstructure and the 'as-built' mechanical properties.

Reference: [1] W.J. Davids, H. Chen, K. Nomoto, H. Wang, S. Babu, S. Primig, X. Liao, A. Breen, and S.P. Ringer, *Phase transformation pathways in Ti-6Al-4V manufactured via electron beam powder bed fusion,* Acta Materialia, Vol. 215, p. 117131, (2021)

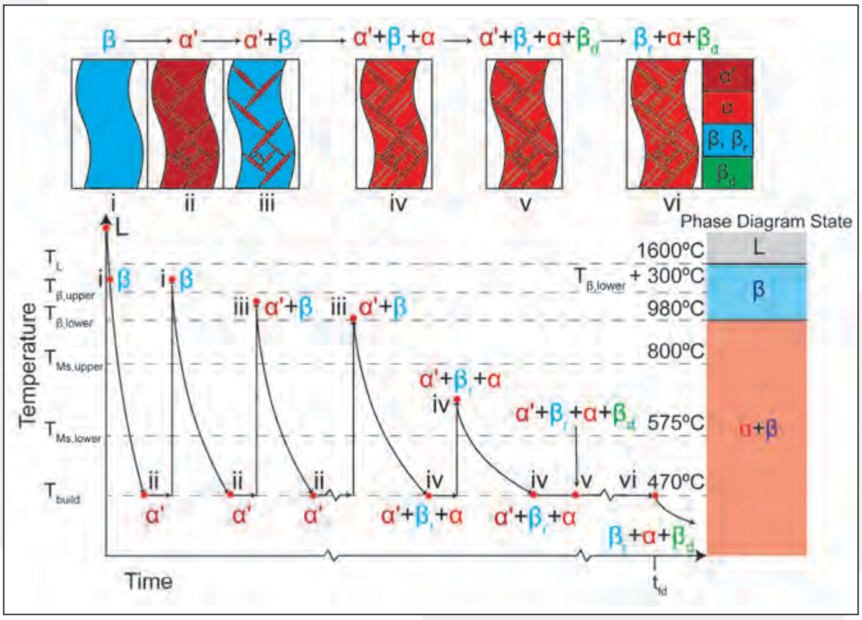
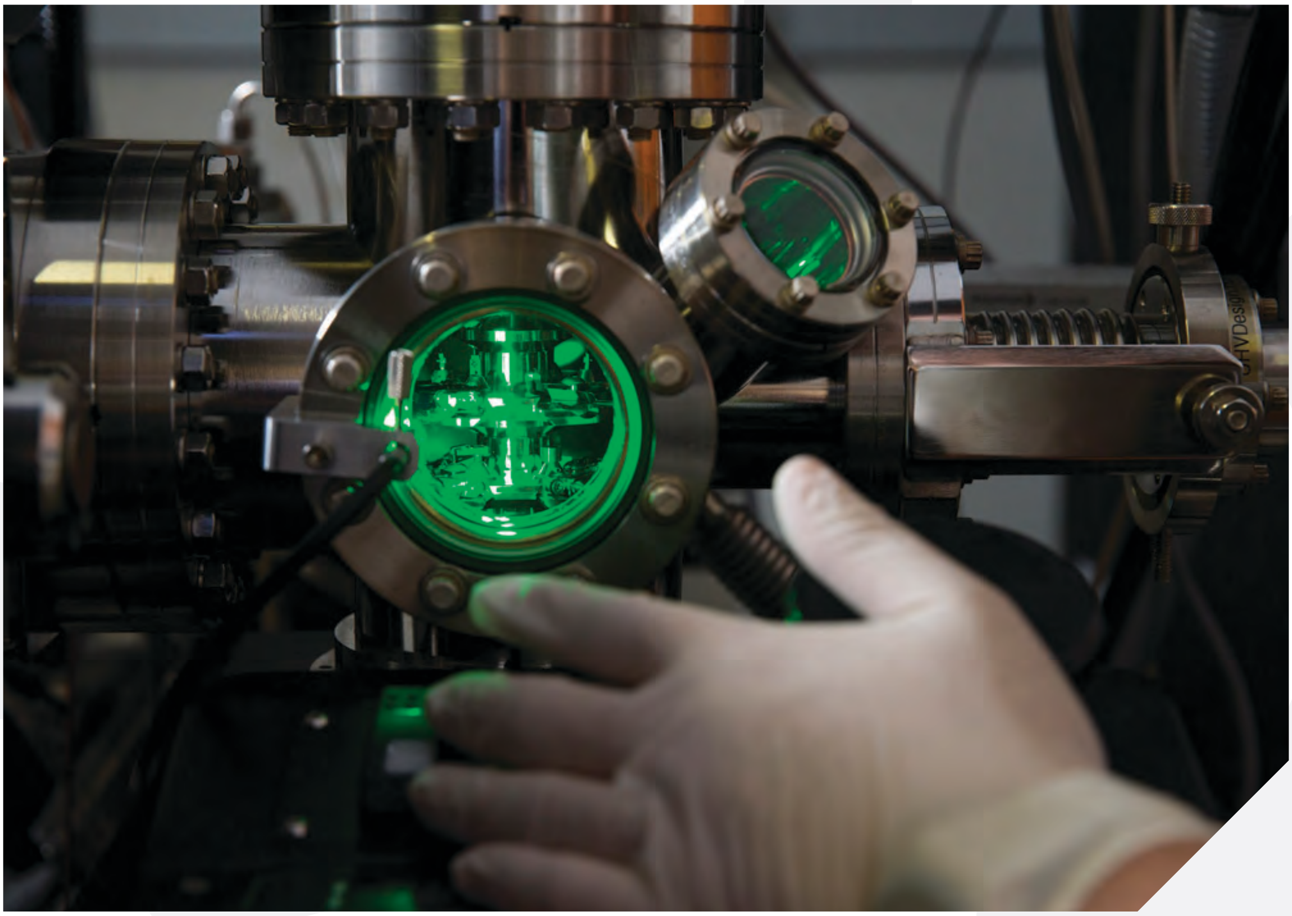


Figure. Proposed phase transformation pathway for E-PBF Ti-6Al-4V. Microstructures observed shown schematically in (i-vi) with the complete pathway written atop. (Reproduced with permission 1)


UHVDesign

6. Project Networking, Outreach and Impact

The MURI-AUSMURI collaboration has enabled the creation and dissemination of knowledge relevant to the fundamentals of AM with over 100 publications as well as transferring knowledge to industries.

Our online presence via the 3D Additive website https://www.3dadditive.com.au/ and the 'Basecamp' portal provided a collaboration point of knowledge and tools for the Industry Outreach Program. The MURI and AUSMURI teams met online every fortnight sharing presentations that were run on a rotational basis involving all eight collaborating universities.

The 3D Additive MURI-AUSMURI project has fostered collaborative research across, industries, universities and research labs in USA, Australia and other parts of the world.

Industry Networks

AUSMURI

- voestalpine ONE STEP AHEAD.
- stryker
- MITSUBISHI HEAVY INDUSTRIES
- KOBELCO
- Australian Government Department of Defence Science and Technology
- GE
- 3RDAXIS PTY LTD ADDITIVE MANUFACTURING

MURI

- ATI
- ELEMENTUM
- mag specialties
- Novelis
- BOEING
- QUAD CITY MANUFACTURING LAB
- EPRI ELECTRIC POWER RESEARCH INSTITUTE
- DePuy Synthes
- Honeywell
- NADCA NORTH AMERICAN DIE CASTING ASSOCIATION
- Q
- GE, United States Army, United States Air Force
- volunteer aerospace

Capability Networks

Sydney Manufacturing Hub based at The University of Sydney in partnerships with GE Additive and 3rd Axis Pty Ltd.

GE Additive

3RDAXIS PTY LTD ADDITIVE MANUFACTURING

Oak Ridge National Laboratory's Manufacturing Demonstration Facility for all 3D printing and Digital Factory Needs

Volunteer Aerospace (for commercial 3d printing of metals and alloys for aerospace applications)

Computational Capabilities

Thermodynamic modelling capability -ThermoCalc

Computer simulation of phase transformation and microstructure evolution in metallic systems - MATCalc

Computational Fluid dynamics (CFD) modelling capability and metal AM thermal history simulations - Flow 3D, OpenFOAM and semi-analytical heat conduction model software from Benjamin Stump at ORNL

Engineering Simulation solution for heat treatment, welding and welding assembly - SYSWELD

The Exascale Additive Manufacturing (ExaAM) simulations

Material point method (MPM) and OpenFOAM simulations of melt pool dynamics are being validated using experimental results from dynamic x-ray radiography. These simulations will feed into the other models under the Exascale computing for AM (ExaAM).

EBSD distortion correction – Python GUI

Atom probe tomography reconstruction calibration and data analysis – CAMECA AP suite 6, Matlab, Python and SIMION

Analysis of 2D EBSD data – Oxford Instruments AZtecCrystal

Crystal plasticity modelling – DAMASK

CAMECA SCIENCE & METROLOGY SOLUTIONS AMETEK

MatCalc Engineering

ECP EXASCALE COMPUTING PROJECT

DAMASK Düsseldorf Advanced Material Simulation Kit

MATLAB

esi SYSWELD

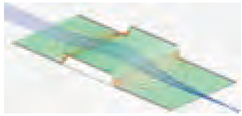
OpenFOAM

Thermo-Calc Software

FLOW-3D

OXFORD INSTRUMENTS

python



Academic Collaborations

Imperial College London

CHESS CORNELL HIGH ENERGY SYNCHROTRON SOURCE

The UNIVERSITY of OKLAHOMA

UNIVERSITY OF WOLLONGONG AUSTRALIA

Northeastern University

Carnegie Mellon University

RMIT UNIVERSITY

UNIVERSITY of NEBRASKA LINCOLN

Washington University in St. Louis

The University of Nottingham

Singapore Institute of Manufacturing Technology SIMTech

Research Laboratories and Institutes

Leveraging Additional Funding

Throughout the course of the MURI-AUSMURI program, additional funding was leveraged to maximise research impact on additive manufacturing. The following successful grant applications also demonstrate the high esteem the research team is held in and our collective capacity to conduct world-class research.

Australian Government
Australian Research Council

Australian Research Council (ARC) grants:

ARC Discovery Projects (DPs):

2023: **DP230100183 Prof. Xiaozhou Liao (USyd); Assoc. Professor Zengxi Pan (USyd)**

Metallic materials with combined chemical and structural heterogeneities.

440,520 AUD

2023: **DP230101063 Prof. Sophie Primig (UNSW); Dr. Nima Haghdadi (UNSW)**

In-situ grain boundary engineering via metal additive manufacturing.

421,760 AUD

ARC Discovery early career research award (DECRA):

2022: **DE220100527 Dr. Keita Nomoto (USyd)**

Novel high-performance copper-based materials via additive manufacturing.

420,000 AUD

ARC Linkage, Infrastructure, Equipment and Facilities (LIEF):

2024: **LE240100120 Prof. Simon Ringer (USyd) et al.**

Powder Manufacturing Facility for Additive Manufacturing.

546,254 AUD

2022: **LE220100182 Prof. Simon Ringer (USyd) et al.**

Metallurgical Facility for Solid-State Additive Manufacturing.

851,607 AUD

ARC Linkage Projects

2021: **LP210301261 Prof. Simon Ringer (USyd) et al.**

Towards use-as-manufactured titanium alloys for additive manufacturing. In collaboration with Stryker European Operations Ltd.

442,371 AUD

2019: **LP190101169 Prof. Sophie Primig (UNSW); Prof. Simon Ringer (USyd) et al.**

Structure-property relationships of next generation aero-engine Materials. In collaboration with voestalpine Bohler Edelstahl GmbH & Co KG (Austria).

570,000 AUD

Australian Nuclear Science and Technology Organisation (ANSTO) beamtime grants:

2021: ACNS Neutron Beam Instrument Grant

B. Lim, M. Reid, A. Paradowska, A.J. Breen, S.P. Ringer (USyd)

Proposal 9637: Interaction between residual stress and complex internal features in powder bed fusion Haynes-282.

49,320 AUD

2021: Australian Institute of Nuclear Science and Engineering (AINSE) Post Graduate Research Award (PGRA)

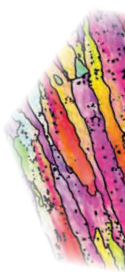
W.Davids, A.Paradowska, S.P. Ringer (USyd)

Scan pattern induced property heterogeneity in Ti-6Al-4V manufactured via electron beam powder bed fusion.

6,190 AUD

2020: **ANSTO Kowari beamtime grant' (10 days)**

M. Moyle (UNSW)



Other grants

2022: **FFG Energieforschung 8. Ausschreibung (Austria)**

T. Klein, P. Spoerk-Erdely, S. Primig (UNSW)

Novel advanced Ti superalloys for additive manufacturing

Scientific partners: LKR Ranshofen (Austria), Montanuniversitaet Leoben (Austria)

49,000 € (apportioned), 500,000 € total over 3 years

2022: **DASA Omnibus scheme**

S. Primig (UNSW); N. Haghdadi (UNSW); D. Agius C. Wallbrink

Digital Engineering Materials for Damage Tolerance

120,000 AUD over 1 year

The Austrian Research Promotion Agency (FFG) proposal 'NovelTi' on Ti alloy development for wire additive manufacturing HYPERLINK "https://auc-word-edit.officeapps.live.com/we/(https:/www.ffg.at/8-Ausschreibung-Energieforschung)"(https://www.ffg.at/8-Ausschreibung-Energieforschung) submitted with Austrian partners (LKR Ranshofen and Montanuniversitaet Leoben) (UNSW)

This project has leveraged the Center for Advanced Nonferrous Structural Alloys (CANFSA), and the National

This project has leveraged the Center for Advanced Nonferrous Structural Alloys (CANFSA), and the National Science Foundation program entitled "Industry/University Cooperative Research Center. (ISU).

Internal ISU funding from the Department of Materials Science and the College of Engineering.

Office of Naval Research, (2021-2024), "Intelligent Toolpathing for Part Repair via Hybrid Wire Arc Additive Manufacturing." Amount: $598,903 (personal share: $245,831). Position: Co-PI James Kong (VT).

Navy funded work with Lockheed Martin (ISU).

Laboratory Directed Research & Development Seed Grand from the Department of Energy (OSU).

The Austrian Research Promotion Agency (FFG) proposal 'NovelTi' on Ti alloy development for wire additive manufacturing (https://www.ffg.at/8-Ausschreibung-Energieforschung) submitted with Austrian partners (LKR Ranshofen and Montanuniversitaet Leoben) (UNSW)

7. Team Awards and Recognitions

Prof. Sudarsanam Suresh Babu (UTK)

- 2023: R&D 100 award for the development of operando neutron diffraction for AM
- 2023: MUSE-Knoxville: Lifelong Learner Core Value Award
- 2022: UT-ORII catalyst award for serving as founding educational director and helping the formation of the UT-Oak Ridge Innovation Institute
- 2022: Invited E. G. Ramachandran Distinguished Lecture, IIT Madras, Chennai, India
- 2022: Distinguished Alumni, PSG College of Technology, Coimbatore, India
- SME College Fellow 2021
- Appointed as the member of the National Science Board by the President of the United States of America, 2020

Prof. Simon Ringer (USyd)

- Pro-Vice-Chancellor (Research Infrastructure) 2022
- Elected as Fellow of the Royal Society of New South Wales (FRSN) 2021
- Awarded Materials Australia silver medal in 2020
- Elected as Fellow of Australia's Academy of Technology and Engineering 2020

Prof. Sophie Primig (UNSW)

- Promotion to full Professor, Jan 2023
- Dean of Science Research Excellence Staff Award', UNSW Sydney, 2022
- CO-lead organiser APICAM 2023 Conference at USyd 2022
- Lead guest editor for the special edition of Journal of Materials Science 2022
- Postgraduate Research Supervisor Nominee Award, UNSW ARC Postgraduate Council, 2021
- Dean of Science Collaboration & Partnership award, UNSW Sydney, 2020
- Promoted to Associate Professor 2020
- Awarded UNSW Scientia Fellowship, 2019
- Future Women Leaders Conference award, Monash University, Melbourne, 2019

Prof. Tresa Pollock (UCSB)

- 2023, Acta Materialia Gold Medal
- Honorary Membership in Société Française de Métallurgie et de Matériaux (SF2M), 2021
- TMS – Institute of Metals Robert Franklin Mehl Award, 2020
- TMS – AIME Champion H. Mathewson Award, (2019)
- TMS Morris Cohen Distinguished Achievement Award, 2019

Prof. Peter Collins (ISU)

- Recipient of the Stanley Chair in Interdisciplinary Engineering, Iowa State University 2022.

Prof. Amy J. Clarke (Mines)

- Colorado School of Mines Excellence in Research Award (Senior), 2022-2023
- John Henry Moore Distinguished Professor of Metallurgical and Materials Engineering, 2021
- TMS Brimacombe Medalist, 2020
- ASM International Fellow, 2018

Prof. Zhenyu (James) Kong (VT)

- Outstanding Mentor in 2023, Graduate School, VT
- Elected fellow of the American Society of Mechanical Engineers (ASME), 2022
- Recognized as one of the 20 Most Influential Academics in Smart Manufacturing honored by SME's Smart Manufacturing magazine, 2021
- Elected fellow of the Institute of Industrial and Systems Engineers (IISE), 2020
- Recipient of the Dean's Award for Excellence in Research, College of Engineering, Virginia Tech, 2019
- Finalist of the Best Paper Award with PhD student Bo Shen, Quality Statistics and Reliability (QSR) Section, INFORMS Annual Meeting, Nov. 8-11, 2020.

Asst. Prof. Carolin Fink (OSU)

- 2022 Prof. Koichi Masubuchi Award presented by the American Welding Society (AWS)

Nana Adomako (UNSW)

- Recipient of the UNSW Scientia PhD scholarship 2020

Andrew Breen (USYD)

- Work selected for cover image in Ultramicroscopy, vol: 243 (2023)

Chris Blackwell (OSU)

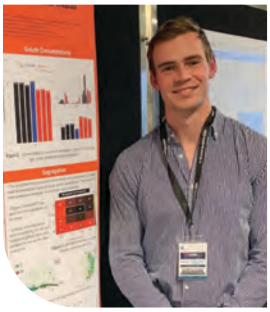
- Graduated the Class of 2020 with Research Honors Distinction. His thesis was titled- "Investigation of the Microstructure of Additively Manufactured IN738

Alexandra Bradley (UNSW Honour's student)

- 1st presentation award' Materials Australia NSW undergrad student presentation competition 2023

Kirk (Kangwei) Chen

- **Kirk (Kangwei) Chen** was awarded the Best Oral Presentation Awards for his oral presentation on 'Effect of laser powder bed fusion process and powder characteristics for Cu-10Sn alloy on microstructure and mechanical properties' at the 20th International Microscopy Congress (IMC20) held at Busan, September 10-15, 2023. Kirk is supervised by Prof. Simon Ringer and Dr. Keita Nomoto at the University of Sydney



Will Davids (USyd)

- Winner of the AINSE PGRA 2 Minute Thesis Competition 2021
- Winner of poster award at APICAM

Ryan DeMott (UNSW)

- First PhD graduate of the AUSMURI program - 2021

Garrett Fields and Raymond Wysmierski (UTK)

- Honorable Mention Award - Center for Materials Processing (CMP) – UT Knoxville – Summer 2021 Poster Session

Raghav Gnanasambandam (VT)

- Outstanding Doctor of Philosophy Award, Industrial and Systems Engineering, Virginia Tech (2023)

Michael Haines (UNSW PhD student)

- 3rd price in UNSW PGSOC postgrad poster competition 2023

Chris Jasien, (Mines)

- Recipient the Department of Energy National Nuclear Security Agency Stewardship Science Graduate Fellowship (SSGF), 2022-present

Rakesh Kamath (UTK)

- TMS Best Paper Contest - Graduate Division, 2nd Place awarded for the paper titled "In-situ, dynamic synchrotron x-ray radiography studies on melt pool evolution and solidification kinetics during laser fusion processing of Ti-6Al-4V"- 2022
- TMS Student Trivia Competition - 2nd Place- 2019
- UT Knoxville Graduate Student Senate Travel Award (TMS) - 2018, 2019 and 2020
- Selected to attend the 20th National School on X-ray and Neutron scattering (NXS) for a week each in Argonne National Lab and Oak Ridge National Lab- 2018

Bryan Lim (USyd) and Meiyue Shao (OSU)

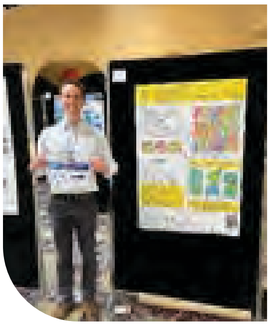
- Won best poster awards at the Micoroscopy and Microanalysis 2019 (Portland, USA)

Ming Luo (UNSW PhD student)

- People's choice award in UNSW PGSOC postgrad poster competition 2023
- People's choice award in UNSW PGSOC postgrad poster competition 2022

Amamchukwu Ilogebe (ISU)

- Recipient of MSE Department Wayne G. Basler Graduate Scholarship Award, 2022



Maxwell Moyle (UNSW)

- 3rd Place Poster Presentation Competition HI-AM online conference 2021
- Best 1 minute thesis' in the School of Materials Science and Engineering at UNSW in 2020
- Australian Government Research Training Program Scholarship UNSW - 2019
- Dean's award for Outstanding PhD Thesis, UNSW graduate research school, 2023
- 1st price in UNSW PGSOC postgrad poster competition 2022
- People's choice poster award, CAMS conference Melbourne, 2022
- Awarded the 'people's choice' poster award for his poster presentation on 'Microstructure and texture investigations in laser powder bed fusion 316L stainless steel' at the Materials Australia CAMS conference held at the University of Melbourne, June 1-3, 2022

Alivia Mourot (OSU)

- Best Graduate Student Poster Award at the 2023 Ohio State Materials & Manufacturing Conference

Dr. Keita Nomoto (Usyd)

- Awarded the prestigious three year DECRA fellowship from ARC, commencing Jan 2022

Ruben Ochoa (Mines)

- Graduate Assistance in Areas of National Need Fellowship, U.S. Department of Education, 2022
- National Science Foundation
- (NSF) Student Grant Recipient, PowderMet2022 & AMPM2022 conferences - 2022

Katie O'Donnell (ISU)

- Recipient of the Graduate Dean Scholarship

Brian Rodgers (Mines)

- Recipient the Department of Energy National Nuclear Security Agency Laboratory Residency Graduate Fellowship (LRGF), 2021-present

Alec Saville (Mines)

- National Academies/National Research Council Postdoc, National Institute of Standards and Technology, 2023
- National Science Foundation (NSF) Student Grant Recipient, PowderMet2022 & AMPM2022 conferences - 2022
- Awarded the NSF Graduate Research Fellowship (GRFP), 2019-present
- NSF Student Grant Recipient, PowderMet2019 & AMPM2019 conferences - 2019
- Proton Radiography for Materials Discovery Workshop Travel Grant- 2018

Liam Stephenson (UNSW)

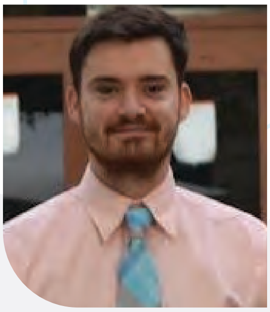
- University of New South Wales Medal 2019
- 2nd prize at the Materials Australia NSW branch undergrad thesis competition 2019

Dr. Hao Wang (Usyd)

- Publication on transformation twins in titanium alloys, in the Journal of Materials Research Letters, was selected as 'Editors choice' papers in 2021
- Dr. Hao Wang wins the Thermo Scientific Cowley-Moodie Award by the Australian Microscopy & Microanalysis Society, that is conferred to an Early to Mid-Career researcher for original research in physical sciences involving electron microscopy. Hao wins this award for his work on the micro-structural studies of additively manufactured metals.
- Hao Wang's paper Introducing C Phase in additively manufactured Ti-6Al-4V: a new oxygen-stabilized face-centred cubic solid solution with improved mechanical properties got accepted in Materials Today recently. This journal has an impact factor of 26.94

Rongxuan Wang (VT)

- IISE Manufacturing and Design Division Best Track Paper Competition Winner 2023 "Highresolution Sub-surface Thermal Measurement in Laser Powder Bed Fusion (L-PBF) Using Novel Fiber Optics and Machine Learning."



Logan White (UTK)

- Denver X-ray Conference Student XRD Poster Session Winner, Denver X-ray Conference, Virtual, August 2021.
- Los Alamos National Laboratory Student Symposium Presentation Winner for Materials Science. Los Alamos, NM, August 2021.
- First Place for Design, Exhibition of Undergraduate Research and Creative Achievement (EUReCA), Research Poster Competition at UT, April 2021.
- Office of undergraduate Research and Fellowship 2nd Place, Exhibition of Undergraduate Research and Creative Achievement (EUReCA), Research Poster Competition at UT, April 2021.
- Fulbright Research Grant Recipient, Czech Republic, April 2021
- Extraordinary Professional Promise, Tickle College of Engineering, UT, April 2021
- Extraordinary Academic Achievement, Tickle College of Engineering, UT, April 2021
- Top Collegiate Scholar, Tickle College of Engineering, UT, April 2021
- Outstanding Junior Award, Department of Materials Science and Engineering, UT, April 2020
- Barry Goldwater Scholarship Recipient, March 2020.

UNSW PGSOC poster competition

- UNSW AUSMURI contributors took home three out of the five poster prizes.

- Our 3 AUSMURI PhD graduates **Hao Wang**, **William Davids** and **Bryan Lim** with **Prof. Xiaozhou Liao** and **Prof. Simon Ringer**

In 2022, the University of Sydney Rocketry team successfully launched their flagship rocket Bluewren, becoming the first Australian team to win the overall Spaceport America Cup –the world's largest intercollegiate rocket engineering conference and competition. The Sydney Manufacturing Hub team were proud contributors to the USyd team's design, providing AM manufactured parts to the rocket.


TMS
2020

8. Personnel and Careers

Participant's List - Research Staff

Andrew Breen
Senior Research Associate (USyd)

Advanced atom probe characterisation of additively manufactured materials

Carl Cui
Research Fellow (USyd)

Ab initio computational methods, including density functional theory and molecule dynamics of different alloys

Maria Quintana
Research Scientist (MSE ISU)

Advanced Characterisation techniques, microscopy (SEM, TEM, FIB) in AM Ti64 and IN738

Supriya Pillai
Project Manager (USyd)
From Oct 2021

AUSMURI project co-ordination and management

Currently Operations Manager for the Digital Sciences Initiative at USyd

Sriram Vijayan
Research Associate Engineer (OSU)

Developing in situ Characterisation strategies to study thermally activated phenomena under AM conditions inside the TEM

Currently assistant professor at Michigan Tech

Suqin Zhu
Research Fellow (USyd)

Metallurgy and characterisation techniques, such as state-of-the-art S/TEM and APT

Postdoc and PhD Students

Priyanka Agrawal

Postdoc research associate (Ameslab ISU)

Advanced microscopy (SEM, TEM, FIB, PED) in AM Ti64

Currently Assistant Professor, University of North Texas

Nana Adomako

PhD (UNSW)

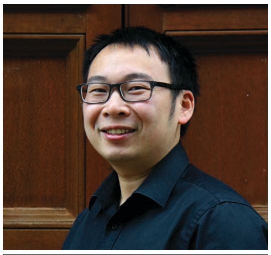
Simulation of Ni-Based superalloys via Gleeble/ in-situ TEM characterisation of AM Ni-based superalloys

Gus Becker

PhD (CSM)

Image analysis and segmentation of synchrotron x-ray imaging obtained during simulated additive manufacturing of metallic alloys

Currently a postdoc at the University of Colorado Boulder



Zibin Chen

Postdoc (USyd)
Until Oct 2021

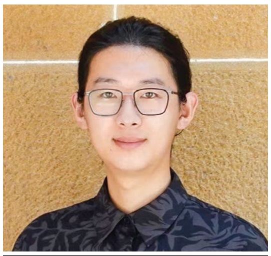
Develop and characterising new AM Ti alloys

Currently Assistant Professor at The Hong Kong Polytechnic University

Hansheng Chen

Postdoc (USyd)

In-situ experimentation Inconel 738/ Stainless steel/ CrMnFeCoNi High entropy alloys



Kangwei Chen

PhD (USyd)

The investigation of sample dimension Dependence on mechanical properties of brass

The Effect of Beam Scan Strategies on the structure property relationships in EBM Inconel 738

Christopher Blackwell

Undergraduate (Honors) (OSU)

Currently PhD Student @ University of Wisconsin-Madison

Phase transformation pathways in additively manufactured Ti-6Al-4V

Will Davids

PhD (USyd)

Currently a consultant at Infravue

Atom probe tomography reconstruction and analysis development

Alec Day

PhD (Usyd)

Until 2020

Currently a senior mechanical engineer at Outpost

3D Characterisation of AM Ti-6Al-4V via 3D-EBSD

Ryan DeMott

PhD (UNSW)
Until Oct 2021

Currently at Sandia National Lab

Multi-Layer Modelling of Solidification in the Powder Bed Fusion Process

James Dingle

PhD (USyd)

Currently an engineer at ResMed

Sensor instrumentation for additive manufacturing

Chaoran Dou

PhD. student (VT)

Katja Eder

Postdoc/

Project Manager (0.5 FTE)
Until June 2021

Manufacturing of Ceramic samples and AUSMURI project management

Currently Senior Engineer at the GE Additive Sydney Manufacturing Hub at USyd

Adriana Eres Castellanos

Postdoc (CMS)

In-situ synchrotron x-ray imaging of AM Ni-based superalloys, Al, and Ti, microstructure characterization, and solidification modelling.

Danny Galicki

PhD (UTK)
Former

Effect of elemental transfer from gas to liquid stainless steel during laser additive manufacturing and its effects.

Currently working for BWXT

David Garcia

PhD (VT)
Former

Metal material Characterisation and modelling

Currently a post-doc at Pacific Northwest National Lab

Raghav Gnannasambandam

PhD (VT)

Physics informed machine learning for additive manufacturing

Nima Haghdadi

Postdoc

Promoted to lecturer in 2021 (UNSW)

Structure-property relationships in additively manufactured duplex stainless steels and superalloys

Appointed Senior Lecturer at Imperial College, London (Feb 2024-).

Michael Haines

PhD (UNSW)

Thermal and stress gradients and their related impact on microstructure formation and phase selection within Ni-based superalloys.

Xinyi He

PhD (UNSW)

Micromechanisms of phase transformations in additively manufactured stainless steels and superalloys

Kayla Hepler

Undergraduate Researcher (OSU)

High throughput workflows for analysis of defects using optical microscopy datasets

Currently PhD student @ University of Delaware

Oliver Hesmondhalgh

Current PhD (CSN) student

In-situ/ex-situ visualization and characterization of microstructure evolution in aluminum and titanium alloys under additive manufacturing conditions to inform modeling, processing parameters, and alloy design

Amamchukwu Ilogebe

PhD (ISU)

Characterisation of defects and spatial quantification of phases in EBM IN738 and Haynes 282

Chris Jasien

PhD (CSM)

Process modelling of AM with SYSWELD and Flow-3D, solidification modelling, and comparisons with synchrotron x-ray imaging and microstructure characterisation of metastable beta-Ti alloys

Study of melt pool dynamics, solidification kinetics and resultant microstructure in spot melting of Ti-6Al-4V alloy using in situ radiography and *ex situ* diffraction

Rakesh R. Kamath

PhD candidate (UTK)

Currently post-doctoral researcher at Argonne National Laboratory

In-situ synchrotron x-ray imaging during simulated AM and electron microscopy of Ni-based alloy single crystals and related solidification modelling.

Jonah Klemm-Toole

Postdoc (CSM)
Until 2020

Currently Assistant Professor, Mines

Study on the transients in plastic instabilities during thermo-mechanical reversals in AL Ti-6Al-4V alloy.

Sabina Kumar

PhD (UTK) Former

Currently lead additive engineer at Eaton Research Lab, Michigan

James Lamb

PhD (UCSB)

TriBeam Tomography for additively manufactured Co and Ni based superalloys

Additive manufacturing simulation and surrogate modelling

Andrew Law

PhD (VT)

Currently Research Scientist at IoTeX, CA

Megan Le Corre

PhD (CMS)

Laser track melting and additive manufacturing of refractory alloys, solidification modelling, and microstructure characterization

Controlling microstructure-property heterogeneities in AM Ni-based superalloys

Brian Lim

PhD (USyd)

Currently Postdoctoral Research Associate at Oak Ridge National Laboratory

Statistical analysis for real-time quality monitoring and control

Chenang Liu

PhD (VT)
Former

Currently an assistant professor at Oklahoma State University

David Loyola

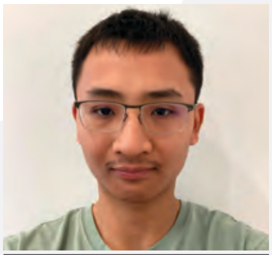
Current PhD (Mines) student

Characterization of arc and electron beam wire feed additive manufacturing of non-ferrous metals and alloys

Ming Luo

PhD (UNSW)

Grain boundary engineering via metal AM



Understanding grain boundary segregation and cohesion behaviour in alloys

Han Mai

PhD (Usyd)

Until 2022

Currently a postdoc at Max Planck Institute for Iron Research

Deep learning for additive manufacturing analysis

Maede Maftouni

PhD (VT)

Currently Senior Data Scientist at Quantum-Si

Maxwell Moyle

PhD (UNSW)

Microstructural Evolution in 17-4 PH Stainless Steel during Laser Powder Bed Fusion

Now working as a consultant at AME

Keita Nomoto

Postdoc (0.2 FTE) (USyd)

High resolution TEM analysis of AM alloys

Ruben Ochoa

PhD (CSM)

Control of microstructure development of AM Ni-based superalloys by alloying, processing, and inoculation

Katie O'Donnell

PhD (ISU)

Until 2023

Characterisation of defects and compositional variations in EBM Ti64

Now a postdoc at Carnegie Mellon University

Brian Rodgers

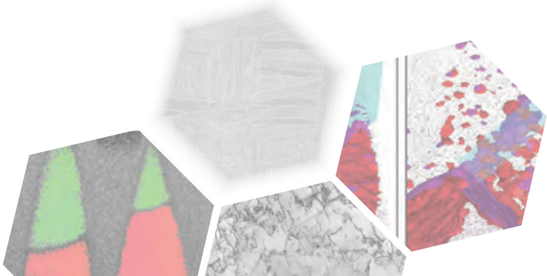
PhD (CSM)

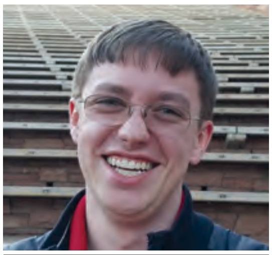
In-situ synchrotron x-ray imaging during simulated AM and electron microscopy and related solidification modelling.

Samia Razzaq

PhD (USyd)

Advanced microscopy techniques to study the microstructure and properties of 3D-printed equiatomic CoCrNi medium entropy alloy





Crystallographic texture analyses in Ti-6Al-4V as a function of scan strategy and build height with neutron diffraction and large-scale electron backscatter diffraction

Alec Saville

PhD (CSM)

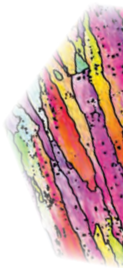
Currently a National Academies/National Research Council Postdoc at the National Institute of Standards and Technology (NIST)

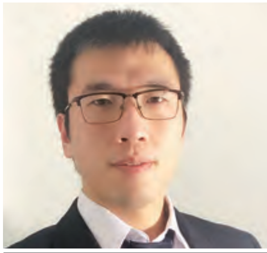
The effect of beam scan strategies on the structure property relationships in EBM Ti-6Al-4V

Meiyue Shao

PhD (OSU)
Former

Worked as a materials scientist at Headway Technologies and is currently at Intel





Advanced data analytics and machine learning for AM sensor data analysis

Bo Shen

PhD (VT)

Currently Asst. Prof. At New Jersey Institute of Technology

Brandon Solsbee

Current MS/PhD (UTK) student

Study of plastic deformations and their gradients during fusion- and solid-state additive manufacturing Currently focussing on wire arc additive manufacturing of metals

Felix Theska

Postdoc (0.2FTE)

Until June 2023

(UNSW)

High resolution characterisation of AM Al and superalloys

Microstructural evolution in additively manufactured metallic materials

Hao Wang

PhD (USyd)

Currently a postdoc at Monash University

Zijian Yu

PhD (USyd)

Microstructure and mechanical properties of AM Ni-Ti alloy

Rongxuan Wang

PhD (VT)

Optical sensors and smart AM platform development

Currently Asst. Prof. at Auburn University

Haoruo (Rosie) Zhou

PhD (USyd)
From 1st March 2022

Microstructure evolution during the processing of hardmetal tool inserts

Research Assistants / Master's Students

Microstructural evolution of Ni-based superalloys during AM

Ally Bradley

Research Assistant

Honours (Feb – Dec 2023)

(UNSW)

Currently a Process Engineering Intern at InfraBuild Australia

Development & implementation of statistically-based decision-making frameworks for material property/process optimization

Rohan Casukhela

Master's student (OSU)

In-situ radiography study on the effect of laser power on fluid flow during raster melting on Ti-6Al-4V

Garrett Fields

Undergraduate Research Assistant (UTK)

Site-specific microstructural evolution in additively manufactured 17-4 PH stainless steel

Singgih Gunarso

Master's student (UNSW)

From Aug 2023

Quantification of microstructural heterogeneities in EBM Haynes 282

Avi Gupta

Master's student (OSU)

Currently a materials scientist at Cummins Inc

Microstructural characterisation of AM alumina and Ti-64 via scanning electron microscopy

James Hansen

Research Assistant (USyd)

Currently a graduate trader at Optiver Asia Pacific

Evan Hass

Undergraduate Researcher (OSU)

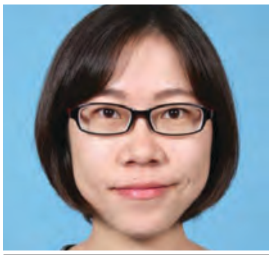
High throughput workflows for analysis of defects using optical microscopy datasets

Currently Technical Solutions Engineer, Epic

Ryan Heldt

Undergraduate Research Assistant (UTK)

In situ radiography study on the effect of laser power on melting and solidification kinetics in spot melts on Ti-6Al-4V



Qianwei Huang

Research Assistant (USyd)

Electron microscopy investigation of additively manufactured Ti alloys

Engineer at Protochips

Matt Kenney

MS Student (MSE ISU)

Advanced microscopy, X-ray CT, and 3D reconstruction in AM Ti64

Philipp Kroeker

visiting Master's student (UNSW)

Feb-Dec 2023

Sensitization of 3D printed duplex steels, in collaboration with TU Berlin (Germany)

Evangelia Lampiri

Research Assistant (UNSW) Former

Analysis of atom probe microscopy data

Carina Ledermüller

Research Assistant (UNSW)

Until Feb 2024

Preparation and characterisation of samples using SEM, EBSD and FIB

Alivia Mourot

Master's (OSU)

Effect of scan strategy on microstructure and microhardness in EBM-PBF Haynes 282

Olivia Schmid

Undergraduate Researcher (OSU)

Characterisation of EBM Ti-6Al-4V Builds

Jeremy Shin

Master's (CSM)

In-situ synchrotron x-ray imaging during simulated AM of Inconel 738

Currently an Associate Consultant, GEP Worldwide

Matthew Valderrama

Undergraduate Research Assistant (UTK)

Study of fluid flow in spot melts on PH steels using in situ radiography

Currently an undergraduate student at UTK

Luke Venter

Honours (UNSW)

Novel additively manufactured PH stainless steels and their microstructures

Sample preparation and characterisation of AM alloys

Biying Wang

Research Assistant (USyd) Former

Currently a PhD student at UNSW

In situ radiography study on the effect of composition on melting and solidification kinetics in PH steels

Logan White

Undergraduate Research Assistant (UTK)

Currently Fulbright Research Grantee at Charles University, Czech Republic

Raymond Wysmierski

Undergraduate Research Assistant (UTK)

In-situ radiography study on the effect of dwell time on melting and solidification kinetics in conduction-mode spot melts on Ti-6Al-4V

Jingdan Zhang

Research Assistant (USyd)

Microscopy of 3D printed dual-phase materials

Honours Students/Interns

Chris Cooper

Intern (USyd)

Microstructural observation of E-PBF Ti-6Al-4V alloys by electron backscatter diffraction

Currently a software engineer at Nearmap

Cameron Durrant

Honours (USyd)

Microstructural observation of E-PBF Ti-6Al-4V and AM yttria stabilised zirconia

Currently an acoustical engineer at Pyrotek

Victor Hugueville

Intern (USyd)

Atom probe tomography of AM Ni-based superalloys

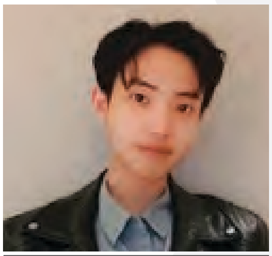
Went on to be an intern at Ariane Group

Jashuva Koppolu

Intern (USyd)

Printing and processing AM ceramics on the Lithoz Cerafab 7500 Dental

Now a research and development engineer at Davcor



Hubert Lee

Casual staff (UNSW)

Aug 2022-Apr 2023

Literature review for AM book chapter

Yingluo Li

Honours (USyd)

The measurement of alloying elements of E-PBF Ti-6Al-4V alloys by atom probe tomography

Siyu Mao

Honours (USyd)

Effects of cyclic thermal loading on the icrostructural evolution of Ti-6Al-4V using EBSD and TKD

Currently a PhD student at Singapore National University

Archie Robertson

Intern (UNSW)

Grain boundary character distribution in AM materials

Currently software engineer at Neara

Liam Stephenson

Honours (UNSW)

Effect of scanning strategy on variant selection in AM Ti-6Al-4V

Currently a consultant at Boston Consulting Group

Matthew Webb

Honours (USyd)

Mechanical testing of AM yttria stabilised zirconia

Now an engineer at Ascent Professional Services

Edward Whitelock

Intern (UNSW)

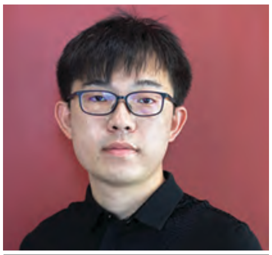
In-situ controlled e-beam melting heat treatment to induce desired strengthening in Ni-based superalloy

Currently with PGH Bricks and Pavers

Bailey Wilmot

Honours (UNSW)

Microstructural evolution during 3D printing of PH stainless steels



Effect of manufacturing route and heat treatment on the microstructure of duplex stainless steels

Bin Yu

Honours (UNSW)

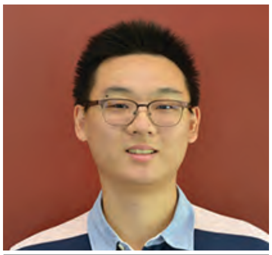
Currently at LMATS

Microstructural Evolution of AM Ti-6Al-4V by Laser Power Bed Fusion

Leon Yang

Honours (USyd)

Now a software engineer at Commsec



Microstructure characteristics of austenitic stainless steel processed by laser powder bed fusion

Mike Zou

Honours (UNSW)

Career Trajectories

The MURI-AUSMURI project has enabled knowledge and expertise that spans across, manufacturing science, metallurgy, in-situ, ex-situ, optical, X-ray, neutron, electron Characterisation, advanced microscopy and microanalysis, computational modeling and data sciences. The multi-disciplinary activities and the extensive collaborations facilitated through this project have created great opportunities for our team members in creating leadership opportunities and career paths. Faculty members and students have received many awards from societies for their research papers and service to community as mentioned in the earlier section of this report. Our project alumni have also found exciting roles in industry and academia in related fields, and some have transitioned to higher research degrees on the same project.

Industry /Defence

Dr. Ryan DeMott who is the 1st PhD student from the AUSMURI team from UNSW, Sydney, is currently working at the Sandia National Labs, USA.

Dr. Sabina Kumar has moved on to pursue a career in AM as lead engineer at Eaton Research Lab, Michigan following her PhD at UTK.

Dr. Meiyue Shao, former PhD student from OSU started as a Materials Scientist at Headway Technologies and is currently at Intel.

Ms. Avantika Gupta, former Master's student from OSU is currently a Materials Scientist at Cummins Inc.

Mr. Liam Stephenson, former undergraduate Honours Student from UNSW Sydney, worked with the Defence Science and Technology Group (Adelaide) from 2020-2022 and is now a Consultant at Boston Consulting Group.

Ms. Qianwei Huang, former Research Assistant from USyd, is currently working as an Engineer at Protochips, providing in-situ TEM solutions.

Dr. Maede Maftouni, a former PhD from VT, is now a Senior Data Scientist at Quantum-Si.

Dr. William Davids, a former PhD from the AUSMURI team at USyd, worked as a Consultant at RPS from 2022 – 2023 and is currently a Consultant at Infravue.

Dr Andrew Law, a former PhD from VT, is now a Research Scientist at IoTeX.

James Dingle transitioned from being a AUSMURI Research Assistant to PhD candidate at USyd and is now working as an Engineer at ResMed.

Cameron Durrant, a former Honours student at USyd is now an Acoustical Engineer at Pyrotek.

Matthew Webb, a former Honours student at USyd is now an Engineer at Ascent Professional Services.

Victor Hugueville, a former intern at USyd went on to be an intern at ArianeGroup in France.

Leon Yang, a former Honours student at USyd is now a Software Engineer at Commsec.

Jashuva Koppolu, a former research intern at USyd is now a Research and Development Engineer at Davcor.

James Hansen, a former Research Assistant at USyd is now a Graduate Trader at Optiver Asia Pacific.

Chris Cooper, a former intern at USyd, is now a Software Engineer at Nearmap.

Dr Danny Galicki, a former Postdoctroral Researcher from UTK, is now working for BWXT.

Dr Han Mai, a former PhD from USyd, is now a Postdoctroral Researcher at Max Planck Institute for Iron Research in Germany.

Dr Alec Day, a former PhD from USyd, worked as a Mechanical Engineer/Applications Scientist at Steam Instruments and is now a Senior Mechanical Engineer at Outpost.

Dr Maxwell Moyle, a former PhD from UNSW, is now a Consultant (steel coordinator) at AME.

Mr Evan Hass, a former Undergraduate Researcher at OSU, is currently a Technical Solutions Engineer at Epic.

Mr Jeremy Shin, a former Master's student at Mines, is currently an Associate Consultant at GEP Worldwide.

Ms Ally Bradley, a former Research Assistant and undergraduate Honours student at UNSW is now a Process Engineering Intern at InfraBuild Australia.

National Laboratory

Dr. Rakesh Kamath from UTK has joined Argonne National Laboratory and serving as Postdoctroral Researcher in the beam line facilities within Argonne Photon Source.

Dr. Bryan Lim from USyd joined Oak Ridge National Laboratory as Postdoctroral Researcher in the Manufacturing Demonstration Facility.

Dr. David Garcia, a former PhD from VT is now a Material Scientist at the Pacific Northwest National Laboratory.

Dr. Andrew Polonsky, a former PhD from UCSB is currently a Staff Scientist at Sandia National Laboratory.

Dr. Nima Haghdadi was promoted from a Postdoctroral Researcher to a Lecturer at UNSW in 2021 and has recently take a position as Senior Lecturer at Imperial College, London (Feb 2024-).

Dr. Katja Eder, who was a Postdoctroral Researcher and Project Manager for the AUSMURI team at USyd is currently working as a Senior Engineer at the Sydney Manufacturing Hub at USyd.

Dr. Jonah Klemm-Toole, has moved from being a Postdoctroral Researcher to an Assistant Professor at the Colorado School of Mines.

Dr. Priyanka Agarwal, a Postdoctoral Researcher at Ameslab, ISU is currently Assistant Professor at University of North Texas.

Dr. Zibin Chen, former Postdoctroral Researcher from USyd is now at The Hong Kong Polytechnic University as Assistant Professor from Nov 2021.

Dr. Chenang Liu, a former PhD from VT is now an Asstistant Professor at Oklahoma State University.

Dr. Sriram Vijayan, previous Postdoctroral Researcher from OSU, is now an Assistant Professor at Michigan Technological University.

Dr. Katie O'Donnell, former PhD from ISU, is now a Postdoctroral Researcher at Carnegie Mellon University.

Dr. Bo Shen, a former PhD from VT is now an Assistant Professor at New Jersey Institute of Technology.

Dr. Rongxuan Wang, a former PhD from VT, is now an Assistant Professor at Auburn University.

Mr. Raghav Gnanasambandam, a current PhD at VT will join as Asst. Professor at Florida State University (August 2024).

Dr. Felix Theska, a former PhD from UNSW, is now a Senior Technical Officer (atom probe sample preparation) at USyd.

Dr. Supriya Pillai, a former AUSMURI Research Development Manager based at USyd, is now Operations Manager for the Digital Sciences Initiative at USyd.

Dr. Hao Wang, a former PhD student from USyd is now a Postdoctroral Researcher at Monash University.

Dr. Alex Saville, a former PhD from Mines, is now a National Academies/National Research Council Postdoctoral Researcher at the National Institute of Standards and Technology (NIST)

Dr Gus Becker, a former PhD from CSM, is now a Postdoctroral Researcher at the University of Colorado Boulder.

Higher Degree Research

Mr. Logan White (previously an Undergraduate Research Assistant at UTK) is currently a Fulbright Research Grantee at Charles University, Czech Republic pursuing research in materials science. Logan plans to move on to Northwestern University for a PhD in materials science.

Two AUSMURI research assistants have transitioned to a PhD after having worked on this project previously. **Ms. Haoruo Zhou (Rosie)** at USyd and **Ms. Xinyi He (Cindy)** from USyd to UNSW.

Mr. Kangwei Chen, former Master's student at USyd, is currently pursuing a PhD at the same organisation, on a related topic.

Ms. Kayla Hepler, undergraduate researcher and Mr. Christopher Blackwell an undergraduate honors student, both from OSU, are currently pursuing PhDs at The University of Delaware and the University of Wisconsin-Madison respectively.

Mr. Logan While, former undergraduate research assistant at UTK, is currently a Fulbright Research Grantee at Charles University, Czech Republic.

Ms. Biying Wang, a former Research Assistant at USyd, is currently a PhD at UNSW.

MURI-AUSMURI Teams - Member Experiences

Award of the AUSMURI project has enabled me to build an exceptional team of talented students and early career researchers who are leaders in microstructure design via additive manufacturing. This project provided a solid foundation for establishing my fundamental research program and was a strong pillar in my promotion application to Associate Professor. I am ready and keen to apply our findings to solve current and future materials design challenges by industrial partners and defence. – Prof. Sophie Primig – Lead CI from UNSW

The AUSMURI project provided a very fruitful avenue for my PhD research. It provided me with opportunities to learn and develop new experimental and analytical techniques while making lasting connections in the international research community. The success of the project directly contributed to my earning a Postdoctroral Researchertoral position and further career opportunities at a US national lab. - Dr. Ryan DeMott former PhD Student at UNSW and currently a scientist at Sandia National Labs, USA.

The AUSMURI project has been an amazing experience unlike any other. This collaborative project across different universities has given me an opportunity to interact, learn and work with some of the finest researchers in the world of AM – Dr. Sriram Vijayan, current Research Associate Engineer at OSU

Working on this project helped me gain valuable Characterisation experience while building computational tools designed to aid data analysis of large 3D EBSD datasets of additively manufactured materials – James Lamb, current PhD student at UCSB

The MURI project has given me a great opportunity to interact and work side-by-side with scientists from diverse backgrounds to contribute effectively to solving problems in AM. The experiences through this project have helped me build a strong foundation which I strongly believe will enable me to do well in future materials research roles that I plan to pursue. – Rakesh R. Kamath, current PhD candidate/Graduate Research Assistant at UTK

The MURI acted as a fantastic catalyst for understanding complicated technical concepts, including crystallographic texture, and enabled me to establish a fruitful network of fellow collaborators, many of which I actively work with to this day. – Alec Saville, current PhD student at Mines

Working on this project allowed me to broaden my skill set and expand my professional network. It was an immensely positive experience that almost certainly advanced my career goals. – Dr. Jonah Klemm-Toole, ex post-doc at Mines and currently an Assistant Professor, Mines

My experiences with the MURI project, including the experimental side at Argonne National Laboratory and the analytical side at the University of Tennessee, have helped me to become a more well-rounded and competitive researcher. - Logan White, Undergraduate Research Assistant at UTK

It was a joy working and being a part of the team. Being given this opportunity, I learned quickly how much our department supports and prepares us for the world of research. As part of this team, I sharpened my ability to inquire, find, and understand academic literature. - Matthew Valderrama, Undergraduate Research Assistant at UTK

Conducting research with Dr. Choo's group has been such a rewarding experience. Not only have I seen the work of myself and teammates improve models of additive manufacturing technology, but I am also constantly learning new technical and professional skills that will help me throughout my entire career. - Raymond Wysmierski, Undergraduate Research Assistant at UTK

I have thoroughly enjoyed working with my team on this project while enriching my knowledge of melt pool dynamics. - Garrett Fields, Undergraduate Research Assistant, UTK

Working with the MURI project has broadened my knowledge on the various Characterisation capabilities available to study the complex physics of metal Additive Manufacturing. It has truly been an honor working with a worldwide collaboration and a talented team. My research work ultimately led to the current position I hold at Eaton Research Labs as a Lead Additive Engineer! -Sabina Kumar, former PhD Student at UTK and currently a lead AM engineer at Eaton Research Lab, Michigan

Working in this project as allowed me to develop research skills. This collaborative effort has also boarded my knowledge of additive manufacturing and the work being done in the field. – Ruben Ochoa, current PhD student at Mines

Involvement in the AUSMURI project was a fantastic way to gain exposure to the international research community and contribute to the under-studied field of metal additive manufacturing. The technical skills I gained, as well as the many opportunities to communicate my findings to local and international colleagues gave me functional experience when I continued my career into defence research. - Liam Stephenson – former honours student at UNSW, currently

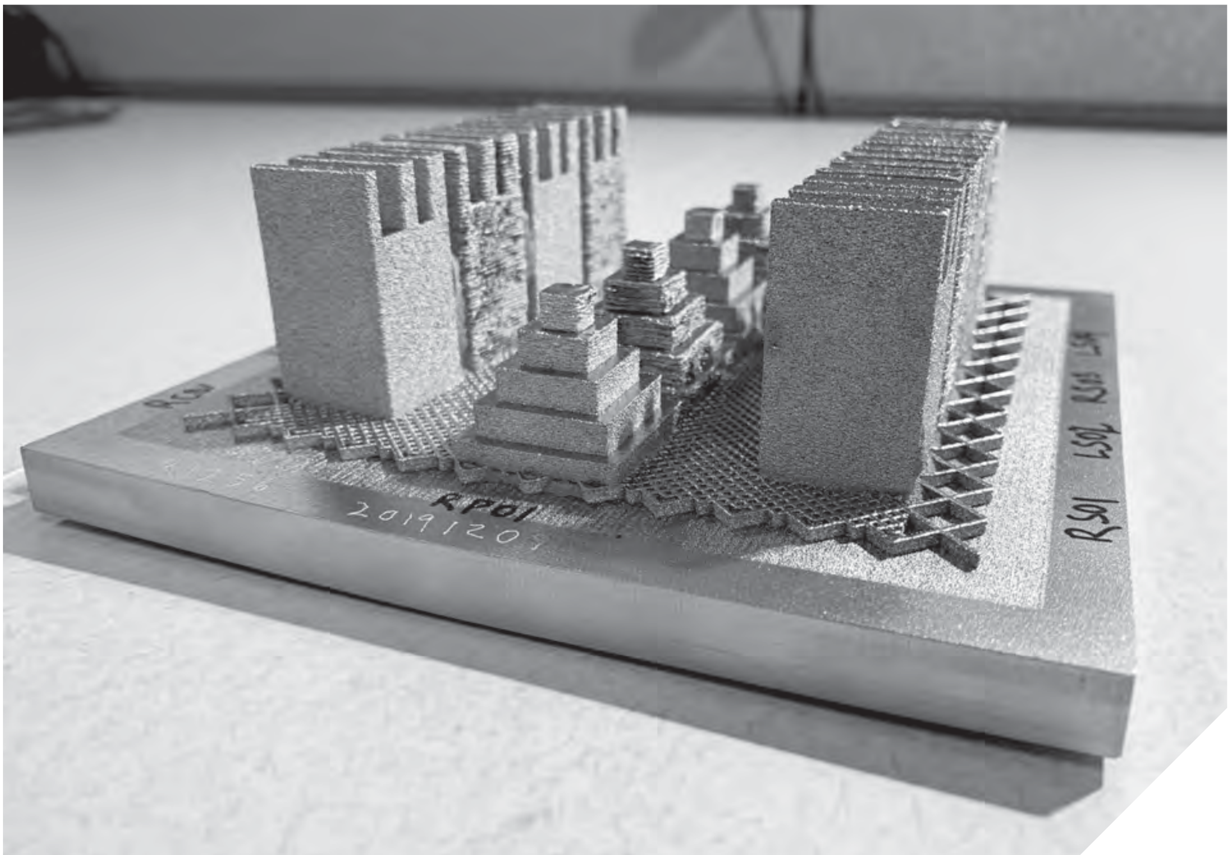
I very much enjoyed working with the AUSMURI team in my role as postdoc and project manager. Because of regular meetings and discussions, I was able to learn a lot about everyone's research and challenges, which helped me get a better understanding about AM. My work with the team helped me to get a role as technical staff at the Sydney Manufacturing Hub, where I am now looking after AM equipments and help researchers and industry clients with their projects. - Katja Eder – former post-doc and project manager at USyd, currently Senior Engineer at the GE Additive Sydney Manufacturing Hub at USyd.

Involvement in the AUSMURI team has provided me opportunities to gain hands-on research experiences and connect with leading researchers as an undergraduate student at the University of Sydney, where I concluded my first project by publishing a first-author journal paper. This multi-university collaboration was instrumental in me joining UNSW as a PhD student, where I am now continuing to expand my research network and fostering communication skills with both national and international colleagues by being part of this project. - Xinyi He (Cindy) – former honours student at USyd and current PhD student at UNSW

The AUSMURI project provided me with a unique experience in additive manufacturing, from which my horizons have significantly expanded. The inter-university and inter-country collaborations allowed me to work with the most reputational scientists globally, letting me stay connected with the state-of-the-art mindsets and techniques in the field. My current research stands on the foundation of the AUSMURI project and my experience in the AUSMURI team will definitely be a valuable resource for my future career development - Zibin Chen, former post-doc at USyd

Others

> *"Working on this project has helped to significantly increase my knowledge of metal-based AM processes and create a network of collaborators from around the world."*
>
> *"Being a part of the MURI project has allowed me to delve deeply into the problem of defects in AM Ti64, focusing on pores, voids, and compositional variations, and enabled me to contribute to the scientific understanding of these defects in the field."*
>
> *"Being part of this multi-university project has opened doors in terms of collaboration, access to results and analysis using different techniques and equipment, and has allowed me to know other professors, researchers and students involved in the project, and hopefully these will be connections that will continue for years to come."*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9. 3D Additive MURI-AUSMURI Publications

The MURI-AUSMURI project has resulted in 107 published articles during the period starting from 2019 to Feb 2024.

MURI-AUSMURI 3D Additive Project Phase 2 Publications (as at Feb 2024)

1a. V. V. Rielli, M. Luo, E. Farabi, N. Haghdadi, S. Primig, Interphase boundary segregation in IN738 manufactured via electron-beam powder bed fusion, Scripta Materialia Vol. 244, pp. 116033, (2024)

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2. J. H. Warner, S. P. Ringer, G. Proust, Strategies for metallic powder reuse in powder bed fusion: A review, Journal of Manufacturing Processes, V. 110, 31 January 2024, Pages 263-290
3. M. S. Moyle, N. Haghdadi, V. Luzin, F. Salvemini, X. Z. Liao, S. P. Ringer, S. Primig, Correlation of microstructure, mechanical properties, and residual stress of 17-4 PH stainless steel fabricated by laser powder bed fusion, Journal of Materials Science & Technology, accepted for publication (2024.01.29) - Q4
4. N. K. Adomako, N. Haghdadi, X. Z. Liao, S. P. Ringer, S. Primig, Thermal cycle induced solid-state phase evolution in IN718 during additive manufacturing: A Gleeble study, Journal of Alloys and Compounds Vol. 976, pp. 173181, (2024) - Q3
5. M. Haines, M. S. Moyle, V. V. Rielli, N. Haghdadi, S. Primig, Site-specific Cu clustering and precipitation in laser powder-bed fusion 17-4 PH stainless steel, Scripta Materialia Vol. 241, pp. 115891, (2024) – Q3
6. M. Luo, X. Z. Liao, S. P. Ringer, S. Primig, N. Haghdadi, Grain boundary network evolution in electron-beam powder bed fusion nickel-based superalloy Inconel 738, Journal of Alloys and Compounds, Vol. 972, pp. 172811, (2024.01.25) – Q3
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10. K.O'Donnell, M.J.Quintana, M.J.Kenney, P.C.Collins. "Using defects as a 'fossil record' to help interpret complex processes during additive manufacturing: as applied to raster-scanned electron beam powder bed additively manufactured Ti–6Al–4V", Journal of Materials Science, 2023.08.27, 58, 13398–13421, https://doi.org/10.1007/s10853-023-08838-0, ISSN 1573-4803 and 0022-2461
11. N. K. Adomako, N. Haghdadi, J. F. L. Dingle, E. Kozeschnik, X. Z. Liao S. P. Ringer, S. Primig, Predicting solid-state phase transformations during metal additive manufacturing: A case study on electron-beam powder bed fusion of Inconel-738, Additive Manufacturing, Vol. 76, pp. 103771, (2023) - Q4

12. M. Haines, M. S. Moyle , V. V. Rielli, V. Luzin, S. Primig, Experimental and Computational Analysis of Site-Specific Formation of Phases in Laser Powder Bed Fusion 17-4 Precipitation Hardening Stainless Steel, Additive Manufacturing Vol. 73, pp. 103686 (2023) – Q2
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Phase 2 Publications (2022 - 2024)

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Interphase boundary segregation in IN738 manufactured via electron-beam powder bed fusion

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ABSTRACT

Electron-beam powder bed fusion (EPBF) has been demonstrated to enable crack-free additive manufacturing of the traditionally non-weldable IN738 Ni-based superalloy. This is related to grain boundary (GB) segregation and precipitation phenomena during EPBF thermal cycling. We investigate such GB microstructures around typical interphase boundaries in IN738. Cyclic reheating results in γ' dissolution, reprecipitation, and formation of layers enriched in refractory elements at γ'-γ' interfaces. Interfacial excess shows that >10 atomic layers of Cr and 3.5 of Co at the GB suppress the segregation of W, B, and C. GBs around heterogeneously nucleated γ grains are decorated with less Cr and Co. This is linked to microsegregation of carbide and boride-forming elements, facilitating diffusion of minor elements during cooling. A heterogeneous interfacial excess profile at a γ'-γ' interface is reported. These findings improve the current understanding of interphase boundaries and segregation in EPBF-manufactured IN738, possibly contributing to crack-free additive manufacturing.

The Ni-based superalloy IN738 is widely used in modern gas turbines due to its excellent high-temperature strength [1,2]. It is considered a non-weldable alloy due to its high Ti and Al contents and a high-volume fraction of γ', which promote cracking [3,4]. This has made additive manufacturing (AM) of this alloy a challenge. 3D printing crack-free parts requires careful control of the solidification conditions and microstructure evolution. Electron-beam powder bed fusion (EPBF) has seen the most success in fabricating crack-free IN738 builds. The preheated substrate and precise control over the scanning strategy contribute to lower thermal gradients and minimum residual stress in the final builds. In addition, unique thermal cycles lead to in-situ γ' precipitation, solute segregation, and secondary phase formation at the grain boundaries (GBs) [5–7].

The AM of Ni-based superalloys is commonly associated with the segregation of alloying elements along both GBs and interphase boundaries [8–11]. Understanding of the interface chemistry is important to achieve desired intergranular properties [12–14]. Segregation has been shown to lead to undesirable GB phenomena such as GB decohesion and embrittlement by some authors [10,14–16], however, others report improved GB strength and creep properties [12,17,18]. Several studies have focused on the GB segregation at γ-γ and γ-γ' interfaces in AM Ni-based superalloys [6,10,11,18]. Cr, Co, Mo, and W are known to segregate at these interfaces [19]. Kontis et al. [10] reported that AM processing parameter control can achieve a fine-grained superalloy microstructure with moderate GB segregation. Cloots et al. [20] reported the segregation of Zr at GBs as the main cause of solidification cracks in laser powder bed fusion (LPBF) IN738LC. More complex secondary phases, such as carbides and borides may form in AM IN738 due to the excessive segregation of C and B, which may deteriorate the mechanical properties of the builds, such as ductility and strength [12,18]. Although some general knowledge on segregation at γ-γ GBs in AM IN738 is available [2,11,21,22], segregation phenomena in its complex GB microstructure with γ'-γ' boundaries, γ'-boride, and γ'-carbide interfaces remains largely unexplored. This remains an important knowledge gap as there is a clear relationship between these phenomena and many properties of the final build, including resistance to crack propagation and corrosion properties [13,23–26]. Through a systematic study, the current work aims to provide a better understanding of interphase boundary segregation in EPBF IN738.

Pre-alloyed gas-atomised IN738 powder with particle diameters of 60 ± 7 μm was provided by Astro Alloys Inc [5]. The chemical composition of the powder measured by inductively coupled plasma atomic emission spectroscopy was Ni-16.0Cr-8.8Co-3.3Ti-3.3Al-1.7Mo-2.6W-1.8Ta-0.8Nb-0.1C-0.01B-0.13Si-0.15Zr (wt.%) [5]. IN738 build with

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Deep Gaussian Process for Enhanced Bayesian Optimization and its Application in Additive Manufacturing

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Abstract

Engineering design problems typically require optimizing a quality measure by finding the right combination of controllable input parameters. In additive manufacturing (AM), the output characteristics of the process can often be non-stationary functions of the process parameters. Bayesian Optimization (BO) is a methodology to optimize such "black-box" functions, i.e., the input-output relationship is unknown and expensive to compute. Optimization tasks involving "black-box" functions widely use BO with Gaussian Process (GP) regression surrogate model. Using GPs with standard kernels is insufficient for modeling non-stationary functions, while GPs with non-stationary kernels are typically over-parameterized. On the other hand, a Deep Gaussian Process (DGP) can overcome GPs' shortcomings by considering a composition of multiple GPs. Inference in a DGP is challenging due to its structure resulting in a non-Gaussian posterior, and using DGP as a surrogate model for BO is not straightforward. Stochastic Imputation (SI) based inference is promising in speed and accuracy for BO. This work proposes a bootstrap aggregation-based procedure to effectively utilize the SI-based inference for BO with a DGP surrogate model. The proposed BO algorithm *DGP-SI-BO* is faster and empirically better than the state-of-the-art BO method in optimizing non-stationary functions. Several analytical test functions and a case study in metal additive manufacturing simulation demonstrate the applicability of the proposed method.

Keywords: "Black-box" functions; Surrogate Modeling; Deep Gaussian Process; Bootstrap Aggregation; Additive Manufacturing.

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Review

Strategies for metallic powder reuse in powder bed fusion: A review

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ABSTRACT

Metal additive manufacturing is a rapidly growing field because it enables extensive freedom in design coupled with the production of new materials that would be impossible to synthesise by conventional manufacturing. Successful powder bed fusion processes require careful monitoring of the powder since changes there can influence part properties and performance. This raises the issue of powder reuse, which is crucial for improving the financial viability and lowering the environmental impact of the process. As powder is widely known to degrade during reuse, powder reuse strategies must therefore be carefully devised. Here, we review three of the most common powder reuse methods: the single batch and collective ageing methods, the top up method, and the refreshing method. The effects that these reuse methods have on common powder and part properties, as well as an evaluation of powder traceability and powder handling procedures are discussed and evaluated. It is our purpose to set out the merits and challenges of the various powder reuse methods to frame a basis for their selection in academic research and industrial applications.

1. Introduction

Additive manufacturing is a term representing a number of technologies that allow for the production of objects by the deposition of material rather than subtraction, as is the case in many traditional manufacturing techniques [1]. The popularity of additive manufacturing has its origins in its ability to produce components of geometric complexity that would otherwise be difficult or impossible to create through conventional manufacturing techniques such as machining [2]. More recently, there is a recognition that additive manufacturing is a scalable route to production of new alloy materials. One of the more common additive manufacturing technologies is powder bed fusion (PBF), which utilises the continued deposition of thin powder layers selectively melted by either a laser or electron beam according to a CAD design until a final product is built. PBF is primarily responsible for the industrialisation of metal additive manufacturing, and applications in the aeronautical, biomedical, automotive, and aerospace industries now abound [3,4].

One of the considerable advantages with PBF is the ability for powder to be reused. Commonly, a significant fraction of the machine's powder stock will remain unused in the production of a given part. The amount of powder remaining unconsumed varies between builds, depending on part geometry and build chamber dimensions.

The reuse of powder presents both economic and environmental benefits. It has been estimated that Nickel Alloy 718 powder can cost about 90 USD/kg [5], while more expensive alloys such as Ti-6Al-4V can cost up to 400 USD/kg [6]. This pricing and the issues around resource utilisation demand a powder reuse strategy, as the process would be unviable if the non-consumed powder were to be wasted. Since the powder cost typically comprises 10–30 % of the build cost [7,8], substantial increases in the cost per part follow from not reusing powder. This is especially true when some powder suppliers recommend that only virgin powder be used [9]. Moreover, compliance with increasingly sophisticated emissions regulations require reductions in the energy consumption of manufacturing processes and this is a key focus for industry. Since it has been estimated that aluminium requires 224 MJ of energy to atomise 1 kg of powder [10], powder reuse is able to assist in meeting these requirements. In addition to this, powder reuse represents a significant easing of the logistical considerations surrounding powder based additive manufacturing. Lower powder quantities need to be purchased, shipped, stored, and tracked, reducing both the administrative load accompanying inventory management and the risk associated with storing large quantities of potentially dangerous material. These considerations all factor especially into industrial applications, where

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Correlation of microstructure, mechanical properties, and residual stress of 17-4 PH stainless steel fabricated by laser powder bed fusion

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Abstract

17-4 precipitation hardening (PH) stainless steel is a multi-purpose engineering alloy offering an excellent trade-off between strength, toughness, and corrosion properties. It is commonly employed in additive manufacturing via laser powder bed fusion owing to its good weldability. However, there are remaining gaps in the processing-structure-property relationships for AM 17-4 PH that need to be addressed. For instance, discrepancies in literature regarding the as-built microstructure, subsequent development of the matrix phase upon heat treatment, as well as the as-built residual stress should be addressed to enable reproducible printing of 17-4 builds with superior properties. As such, this work applies a comprehensive characterisation and testing approach to 17-4 PH builds fabricated with different processing parameters, both in the as-built state and after standard heat treatments. Tensile properties in as-built samples both along and normal to the build direction were benchmarked against standard wrought samples in the solution annealed and quenched condition (CA). When testing along the build direction, higher ductility was observed for samples produced with a higher laser power (energy density) due to the promotion of interlayer cohesion and, hence, reduction of interlayer defects. Following the CA heat treatment, the austenite volume fraction increased to ~35%, resulting in a lower yield stress and greater work hardening capacity than the as-built specimens due to the transformation induced plasticity effect. Neutron diffraction revealed a slight reduction in the magnitude of residual stress with laser power. A concentric scanning strategy led to a higher magnitude of residual stress than a bidirectional raster pattern.

Keywords: Additive Manufacturing; 17-4 PH Stainless Steel; Mechanical Properties; Residual Stress

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PUBLICATIONS PHASE 2

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Thermal cycle induced solid-state phase evolution in IN718 during additive manufacturing: A physical simulation study

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ABSTRACT

Additive manufacturing (AM) is well-known for its capability to produce and repair intricate parts e.g., Ni-based superalloy components used in aero-engine applications. However, a comprehensive understanding of the microstructure evolution and its link to properties remains elusive, with gaps in the through-process evolution of γ grains, γ', γ'', δ, and other phases in response to thermal cycling. The outstanding challenge here is to observe these transformations dynamically during the 3D printing process, as this usually requires complex in-situ measurements. Here we apply a different approach to systematically reveal the impact of thermal cycling on the solid-state microstructure evolution of IN718, via a combination of physical simulations and thermo-kinetic modelling.

We replicate the thermal cycles IN718 experiences during laser directed energy deposition. Solid-state thermal cycles are shown to have minor effects on the grain morphology due to short holding times at critical temperatures, however, they do induce plastic strain accumulation. Significantly, we show how thermal cycling influences the evolution of γ', γ", and δ phases. While high initial peak temperatures inhibit the precipitation of γ', γ", and δ phases, prolonged thermal cycling to gradually decreasing peak temperatures promotes their precipitation. The δ phase forms along grain and twin boundaries, while both γ' and γ" predominantly precipitate in Nb-enriched regions, causing heterogeneities in hardness. Our results underpin the suitability of physical simulations for replicating superalloy AM, highlighting its potential as a tool to advance the current limited understanding of the microstructure and property evolution.

1. Introduction

Additive manufacturing (AM) is an increasingly popular manufacturing route for making 3D components in a layer-by-layer manner, using powder, wire, or other types of feedstocks [1–3]. It enables the fabrication of complex geometries with short lead times, high efficiency, and minimal waste. Laser directed energy deposition (L-DED) in particular is a metal AM technique that has seen rapid growth over the past decade, with widespread applications across various industries due to its ability to fabricate larger components than powder bed fusion techniques [4–9]. Additionally, ability of L-DED to add to existing components and its suitability for repair make it an economically preferable choice [10,11]. IN718 is a Ni-based superalloy commonly used for manufacturing engineering parts such as gas turbines for aircrafts and power generation [12,13]. Consequently, DED repairs using IN718 for components such as turbine blisks are of significant industrial interest [14,15]. The popularity of IN718 can be attributed to its excellent weldability and manufacturability, combined with its exceptional properties, including high strength, corrosion resistance, and remarkable microstructural stability at high temperatures [16]. The as-printed IN718 microstructure consists of fine dendrites due to micro-segregation of solute elements, and solidification phases such as Laves and carbides [17,18]. In addition, it may comprise nano-scale, ordered intermetallic phases of γ'- ($L1_2$ Ni_3(Al, Ti)) and γ''- ($D0_{22}$ Ni_3(Nb)) as well as the µm-scale orthorhombic δ Ni_3(Nb,Ti) ($D0_a$) phase [19,20]. These phases form during solid-state phase transformations, and their presence and morphology determine the mechanical properties of the component.

Despite the increasing enthusiasm by the aerospace industry in L-

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Site-specific Cu clustering and precipitation in laser powder-bed fusion 17–4 PH stainless steel

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ARTICLE INFO

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ABSTRACT

Thermal gyrations inherent to metal additive manufacturing (AM) result in complex microstructural heterogeneities that are linked to the process parameters, geometry, and alloy composition. AM 17–4 precipitation hardened (PH) stainless steel displays heterogeneities due to its multi-phase microstructure where different as-built phases have been reported. These phases include austenite, martensite, and ferrite, as well as varying degrees of AM induced Cu clustering and/or precipitation. To enable a systematic study of the evolution of these strengthening phases, we utilize a non-standard concentric scan strategy to achieve variations in thermal conditions across a build. Using atom probe microscopy and thermal modeling, we show that the response of Cu varies across the build from no clustering at the outer regions to an increased tendency for Cu precipitation towards the center. Our results will enable the tuning of processing parameters to achieve homogenous Cu precipitation, and thus improve mechanical properties of AM 17–4 PH.

A major hallmark of powder bed fusion (PBF) additive manufacturing (AM) is the unprecedented microstructural variation possible. Due to the impacts related to processing parameters and geometry, variations in grain structure, phase fractions, and precipitate morphology can be seen within a single build geometry [1–4]. Progress has been made in advancing the understanding of the evolution of grain structure and texture control. However, due to the complexity of phase transformations during AM, there is still a paucity in understanding the site-specific control of microstructural features such as µm-scale secondary phases and nm-scale precipitation phenomena [5]. Explanations for the differences in complexity between grain control versus phase control can be given based on solidification conditions and the inherent thermal gyrations exhibited in AM. While site-specific grain structure control is primarily reliant on thermal gradients (G) and solidification velocities (R), the phase and precipitation evolution is dependent not only on the G and R, effecting liquid-solid transformations, but also the proceeding thermal gyrations effecting solid-solid transformations [6, 7].

As a multi-phase alloy, 17–4 precipitation hardened (PH) stainless steel has shown to be particularly sensitive to changes in the processing parameters, geometry, and composition during laser-PBF (L-PBF) [4, 8–11]. The matrix microstructure in 17–4 PH has been characterized within the literature as being either mostly martensitic with small amounts of austenite (FCC) or a mixture of austenite and ferrite (BCC) with diverse reported phase fractions [12–14]. Greater complexity is added when considering Cu precipitates. Only a few studies have reported the formation of Cu precipitates in as-built conditions [15,16]. Numerous other studies, in contrast, report the need for an aging step to form Cu precipitates [12,17–19]. Naturally, Cu clustering and precipitation are strongly dependent on the thermal conditions. Slower cooling rates will provide more time for Cu to diffuse, form clusters, and precipitate. As Cu precipitates are the main strengthening phase in 17–4 PH, it is important to understand the thermal histories that lead to their formation [20]. To build a better understanding, the current work utilizes a non-standard concentric scan strategy that elicits a variety of thermal signatures. This enables a systematic study of the precipitation response of Cu in L-PBF 17–4 PH. The experimental results are reasoned through thermal simulations eliciting an understanding between how the local thermal history influences the behavior of Cu. A framework is presented for using the concentric scan strategy as a method for studying precipitate evolution in other alloys due to its wide variation in thermal history.

17–4 PH samples were produced using powder provided by 3D Systems with the composition given in Table 1. Builds were printed using a 3D System ProX 300 L-PBF machine. An inward travelling concentric scan strategy was utilized as outlined by Moyle et al. [4]. The

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Grain boundary network evolution in electron-beam powder bed fusion nickel-based superalloy Inconel 738

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ABSTRACT

Additive manufacturing (AM) of alloys has attracted much attention in recent years for making geometrically complex engineering parts owing to its unique benefits, such as high flexibility and low waste. The in-service performance of AM parts is dependent on the microstructures and grain boundary networks formed during AM, which are often significantly different from their wrought counterparts. Characteristics such as grain size and morphology, texture, and the detailed grain boundary network are known to control various mechanical and corrosion properties. Advanced understanding on how AM parameters affect the formation of these microstructural characteristics is hence critical for optimising processing parameters to unlock superior properties. In this study, the difficult-to-weld nickel-based superalloy Inconel 738 was fabricated via electron-beam powder bed fusion (EPBF) following linear and random scanning strategies. Random scanning resulted in finer, less elongated, and crystallographically more random grains compared to the linear strategy. However, both scanning strategies achieve unique high grain structure stability up to 1250 °C due to the presence of carbides pinning the grain boundaries. Despite significant difference in texture and morphology, majority of grains terminated on {100} habit planes in both linear and random built samples. The results show potential for controlling grain boundary networks during EPBF by tuning scan strategies.

1. Introduction

Inconel 738 is a precipitation-hardened nickel-based superalloy that is commonly used in high-temperature conditions of up to 980°C for applications in gas turbines and aerospace engine parts owing to its excellent microstructure stability, corrosion resistance and creep properties at elevated temperatures [1–3]. The outstanding high-temperature properties of Inconel 738 are predominantly derived from precipitation, solid-solution and grain boundary hardening [4]. For instance, the precipitation hardening of Inconel 738 is connected to the formation of the coherent ordered face-centred cubic ($L1_2$) intermetallic $Ni_3(Al, Ti)$ γ' phase within the face-centred cubic γ matrix [5]. The high amount of alloying elements and high volume fraction of γ' phase in Inconel 738 contribute to improved microstructure stability and elevated temperature strength [6]. However, it is commonly known that Inconel 738 suffers from poor castability and weldability due to its high Ti and Al contents, which leads to solidification cracking, liquation cracking and ductility-dip cracking [7,8]. Therefore, it is desirable to overcome these challenges by reducing metallurgical and thermo-mechanical drivers that promote cracking during manufacturing of Inconel 738 engineering parts.

Additive manufacturing (AM) now unlocks a new avenue to fabricate crack-free builds of conventionally difficult to weld superalloys with desirable microstructures, via targeted manipulations of thermo-mechanical profiles during printing [9–12]. Inconel 738 has been successfully fabricated by many techniques such as directed energy deposition (DED) and laser powder bed fusion (LPBF) [13,14]. However, LPBF does not achieve in-situ γ' precipitation owing to high thermal gradients, and DED promotes significant liquation cracking because of the high residual stress and incipient melting. Electron beam powder bed fusion (EPBF), also known as electron beam melting, is another common AM process that has been successfully applied to print nickel-based superalloys with little to no cracking [12]. EPBF operates by using a high-energy electron beam to melt and sinter a powder bed in a layer-by-layer manner, building up a 3D part. EPBF is usually carried out under high-temperature pre-heat conditions of the powder bed

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Analysis of the high cracking resistance of a Co Ni superalloy during laser additive manufacturing

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ABSTRACT

A newly developed CoNi-based superalloy is demonstrated to be tolerant of a wide range of laser powder bed fusion print conditions, with limited defect content and high relative densities. These promising traits motivated investigation of the structural uniformity in samples printed at the upper end of the scan velocity processing window (>1000 mm/s). A new misorientation metric, starting reference orientation deviation (SROD), was calculated from mm^3-scale 3D microstructure information to examine structure evolution during epitaxial growth of columnar grains. Misorientations of $>20°$ were observed within the large columnar grains that grew through multiple build layers. The SROD observations highlight the CoNi superalloy's ability to plastically deform to high levels, as evidenced by misorientation gradients, across the range of temperatures experienced in laser additive manufacturing. Misorientation gradients within the columnar grain structure are high relative to Ni base superalloys, allowing for strengthening with high volume fractions of $L1_2$ precipitates.

The flexibility in part geometry and microstructure afforded by additive manufacturing (AM) has driven significant interest and research on this advanced fabrication technique [1–5]. High temperature structural applications, such as gas turbine engines and heat exchangers where complex geometries in small batches are commonplace, can benefit immensely from AM processes. However, the superalloys used in these applications are generally difficult to print, readily forming a variety of cracks (solidification cracks, liquation cracks, strain-age cracks, ductility-dip cracks) and other defects (keyholes, lack-of-fusion, balling) during the process [6–10]. Solutions for overcoming the limited printability of superalloys can be summarized by two approaches: (1) design of new alloys that are tailored for AM to limit their likelihood of developing cracks and other defects [11–13], and (2) development of new scanning strategies and AM processing methods that suppress defect formation for legacy Ni-based superalloys [14–18]. To maximize the utility of the former approach, scan strategies that produce fully-dense microstructures for AM-amenable alloy chemistries can be developed. An ideal AM superalloy candidate can be printed with minimal defects across a wide range of process parameters such that site-specific microstructure can be tailored as needed. There are currently only a small number of alloys with which this has been achieved [11–13,19,20]. One such alloy, a CoNi-base superalloy, has been shown to produce fully-dense parts over a wide range of processing parameters through powder bed fusion (PBF) using both a laser (-L) and an electron beam (-EB) heat source [20–23]. Intended for gas turbine applications, the CoNi-based superalloy has inherent oxidation resistance and high temperature strength facilitated by γ' volume fractions of $\sim 70\%$. Further, the alloy has a narrow freezing range and exhibits a low tendency to chemically segregate during PBF, minimizing solidification-induced stresses and the formation of deleterious phases during printing, thereby decreasing the susceptibility for hot cracking [22–25].

In order to assess the robustness of the CoNi-based superalloy in PBF-L processing, a parameter study was conducted (shown in Fig. 1). 10 x 10 x 10 mm blocks of the CoNi-based alloy (shown in Fig. 2b) with varying hatch spacing, laser power, and scan speed were printed using

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PUBLICATIONS PHASE 2

Article

Strong and ductile titanium–oxygen–iron alloys by additive manufacturing

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Titanium alloys are advanced lightweight materials, indispensable for many critical applications[1,2]. The mainstay of the titanium industry is the α–β titanium alloys, which are formulated through alloying additions that stabilize the α and β phases[3–5]. Our work focuses on harnessing two of the most powerful stabilizing elements and strengtheners for α–β titanium alloys, oxygen and iron[1–5], which are readily abundant. However, the embrittling effect of oxygen[6,7], described colloquially as 'the kryptonite to titanium'[8], and the microsegregation of iron[9] have hindered their combination for the development of strong and ductile α–β titanium–oxygen–iron alloys. Here we integrate alloy design with additive manufacturing (AM) process design to demonstrate a series of titanium–oxygen–iron compositions that exhibit outstanding tensile properties. We explain the atomic-scale origins of these properties using various characterization techniques. The abundance of oxygen and iron and the process simplicity for net-shape or near-net-shape manufacturing by AM make these α–β titanium–oxygen–iron alloys attractive for a diverse range of applications. Furthermore, they offer promise for industrial-scale use of off-grade sponge titanium or sponge titanium–oxygen–iron[10,11], an industrial waste product at present. The economic and environmental potential to reduce the carbon footprint of the energy-intensive sponge titanium production[12] is substantial.

Most industrial titanium (Ti) alloys possess microstructures based on the two basic phases of Ti, the hexagonal close-packed (HCP) α and the body-centred cubic (BCC) β. Represented by Ti–6Al–4V (wt% used throughout unless specified), α–β Ti alloys are the backbone of the Ti industry[1,2]. They can form microstructures comprising[2–5] (1) lamellar α–β with a near-Burgers orientation relationship, (2) equiaxed α and β or (3) globular α among the α–β lamellae. Each of these microstructures has merits and drawbacks, making α–β Ti alloys versatile for diverse industrial applications[1–5]. Of these, the lamellar α–β microstructure has been commonly applied.

The α–β Ti alloys are formulated by alloying Ti with α-phase and β-phase stabilizers. The α-phase stabilizers are limited to Al, N, O, C, Ga and Ge (refs. 3–5), of which N and C are tightly controlled impurities (0.05% N, 0.08% C)[2,3], whereas Ga and Ge are not commercially viable. Hence, as well as Al, O is the only other practical option. Supplementary Table 1 lists the main α–β Ti alloys using Al as the α-phase stabilizer. Notably, O outshines Al in (1) strengthening the α-phase by a factor of about 20 (calculated according to the data given in Table 4 on page 16 of ref. 1), (2) stabilizing the α-phase by a factor of about 10 (based on the aluminium equivalence formula given on page 380 of ref. 5) and (3) restricting the growth of prior-β grains during solidification by a factor of more than 40 (10.8 versus 0.26)[13]. However, these attributes of O have remained underused in the development of α–β Ti alloys.

The issue with O as a principal α-phase stabilizer in Ti is its embrittling effect owing to its strong interactions with dislocations during deformation[6,7]. Furthermore, O changes the phase equilibria, promoting the formation of the embrittling α_2-phase (Ti_3Al)[14]. These constraints have led to the following empirical design rule for industrial Ti alloys: Al + 10(O + C + 2N) + 1/3Sn + 1/6Zr < 9.0% (ref. 5). For Ti–6Al–4V, this design rule requires less than 0.12% O (ref. 15) at 0.05% N and 0.08% C, which was relaxed to 0.13% O for Grade 23 Ti–6Al–4V and 0.20% O for Grade 5 Ti–6Al–4V. Following this rule, a lower Al content allows for a higher O content. Indeed, the latest industrial α–β Ti alloy ATI 425 (Ti–4.5Al–3V–1.8Fe–0.3O)[16], allows 0.3% O maximum because of its lower Al content, for which the above empirical rule accepts a maximum of 0.31% O. If no Al is included, this rule allows a maximum of 0.72% O.

More options exist for β-phase stabilizers in Ti (refs. 3–5), with Fe being the most effective and inexpensive one[17,18]. Furthermore, Fe is the second lightest β-phase stabilizer. However, its use has been constrained by the formation of Fe-stabilized β-flecks during ingot solidification[9] (up to centimetres in size; Supplementary Note 1), which can markedly affect the mechanical properties[9]. Therefore, the use of

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materials

Article

Understanding the Effect of Electron Beam Melting Scanning Strategies on the Aluminum Content and Materials State of Single Ti-6Al-4V Feedstock

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Abstract: Research on the additive manufacturing of metals often neglects any characterization of the composition of final parts, erroneously assuming a compositional homogeneity that matches the feedstock material. Here, the composition of electron-beam-melted Ti-6Al-4V produced through three distinct scanning strategies (linear raster and two point melting strategies, random fill and Dehoff fill) is characterized both locally and globally through energy-dispersive spectroscopy and quantitative chemical analysis. As a result of the different scanning strategies used, differing levels of preferential vaporization occur across the various parts, leading to distinct final compositions, with extremes of ~5.8 wt.% Al and ~4.8 wt.% Al. In addition, energy-dispersive spectroscopy composition maps reveal specific features in both the XY and XZ planes (with Z being the build direction) as a result of local inhomogeneous preferential vaporization. The subsequent change in composition significantly modifies the materials' state of parts, wherein parts and local regions with higher aluminum contents lead to higher hardness levels (with a ~50 HV difference) and elastic property values and vice versa. While varying scan strategies and scan parameters are known to modify the microstructure and properties of a part, the effect on composition cannot, and should not, be neglected.

Keywords: additive manufacturing; preferential vaporization; composition; Ti-6Al-4V; feedstock materials

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1. Introduction

Additive manufacturing (AM) has been a heavily researched processing technique over the last few decades due to its many advantages over traditional processing techniques, including reduced waste and the ability to produce complex geometries [1–6]. Literature studies on AM have been primarily focused on obtaining the microstructure/texture and mechanical properties required for structural applications [7–11], neglecting other aspects of AM parts, such as composition. There is often an assumption that the final part will have the same composition as the feedstock material used in the process, with literature studies focusing on optimizing the mixing of powder and parameters to obtain the adequate melting of all powder components, including when delving into gradient materials [12–14]. However, there are thermodynamic phenomena applicable to AM that influence the final composition and that cannot, and should not, be ignored when using single feedstock powder.

Preferential vaporization is a well-studied phenomenon wherein, at high temperatures, elements with higher vapor pressures vaporize first, and at a faster rate than the other constituents of the material [15–17]. Examples of elements and alloy systems that are known to preferentially vaporize, both in conventional and additive manufacturing processes, include zinc and gallium, aluminum in titanium alloys, and chromium in stainless steels

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Metals & corrosion

Using defects as a 'fossil record' to help interpret complex processes during additive manufacturing: as applied to raster-scanned electron beam powder bed additively manufactured Ti–6Al–4V

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ABSTRACT

Defects in parts produced by additive manufacturing, instead of simply being perceived as deleterious, can act as important sources of information associated with the complex physical processes that occur during materials deposition and subsequent thermal cycles. Indeed, they act as materials-state 'fossil' records of the dynamic AM process. The approach of using defects as epoch-like records of prior history has been developed while studying additively manufactured Ti–6Al–4V and has given new insights into processes that may otherwise remain either obscured or unquantified. Analogous to 'epochs,' the evolution of these defects often is characterized by physics that span across a temporal length scale. To demonstrate this approach, a broad range of analyses including optical and electron microscopy, X-ray computed tomography, energy-dispersive spectroscopy, and electron backscatter diffraction have been used to characterize a raster-scanned electron beam Ti–6Al–4V sample. These analysis techniques provide key characteristics of defects such as their morphology, location within the part, complex compositional fields interacting with the defects, and structures on the free surfaces of defects. Observed defects have been classified as banding, spherical porosity, and lack of fusion. Banding is directly related to preferential evaporation of Al, which has an influence on mechanical properties. Lack-of-fusion defects can be used to understand columnar grain growth, fluid flow of melt pools, humping, and spattering events.

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Predicting solid-state phase transformations during metal additive manufacturing: A case study on electron-beam powder bed fusion of Inconel-738

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ABSTRACT

Metal additive manufacturing (AM) has now become the perhaps most desirable technique for producing complex shaped engineering parts. However, to truly take advantage of its capabilities, advanced control of AM microstructures and properties is required, and this is often enabled via modeling. The current work presents a computational modeling approach to studying the solid-state phase transformation kinetics and the microstructural evolution during AM. Our approach combines thermal and thermo-kinetic modelling. A semi-analytical heat transfer model is employed to simulate the thermal history throughout AM builds. Thermal profiles of individual layers are then used as input for the MatCalc thermo-kinetic software. The microstructural evolution (e.g., fractions, morphology, and composition of individual phases) for any region of interest throughout the build is predicted by MatCalc. The simulation is applied to an IN738 part produced by electron beam powder bed fusion to provide insights into how γ′ precipitates evolve during thermal cycling. Our simulations show qualitative agreement with our experimental results in predicting the size distribution of γ′ along the build height, its multimodal size character, as well as the volume fraction of MC carbides. Our findings indicate that our method is suitable for a range of AM processes and alloys, to predict and engineer their microstructures and properties.

1. Introduction

Additive manufacturing (AM) is an advanced manufacturing method that enables engineering parts with intricate shapes to be fabricated with high efficiency and minimal materials waste. AM involves building up 3D components layer-by-layer from feedstocks such as powder [1]. Various alloys, including steel, Ti, Al, and Ni-based superalloys, have been produced using different AM techniques. These techniques include directed energy deposition (DED), electron- and laser powder bed fusion (E-PBF and L-PBF), and have found applications in a variety of industries such as aerospace and power generation [2–4]. Despite the growing interest, certain challenges limit broader applications of AM fabricated components in these industries and others. One of such limitations is obtaining a suitable and reproducible microstructure that offers the desired mechanical properties consistently. In fact, the AM as-built microstructure is highly complex and considerably distinctive from its conventionally processed counterparts owing to the complicated thermal cycles arising from the deposition of several layers upon each other [5,6].

Several studies have reported that the solid-state phases and solidification microstructure of AM processed alloys such as CMSX-4, CoCr [7,8], Ti-6Al-4V [9–11], IN738 [6], 304L stainless steel [12], and IN718 [13,14] exhibit considerable variations along the build direction. For instance, references [9,10] have reported that there is a variation in the distribution of α and β phases along the build direction in Ti-alloys. Similarly, the microstructure of an L-PBF fabricated martensitic steel exhibits variations in the fraction of martensite [15]. Furthermore, some of the present authors and others [6,16–20] have recently reviewed and

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Experimental and computational analysis of site-specific formation of phases in laser powder bed fusion 17–4 precipitate hardened stainless steel

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ABSTRACT

Additive manufacturing allows for the production of intricate geometries with reduced material waste. However, due to the complex thermal gyrations that are linked to the processing parameters and geometry, inconsistencies in microstructures and mechanical properties occur from build to build. These inconsistencies are exemplified in multi-phase alloys such as 17–4 precipitation hardened (PH) stainless steel where the formation of ferrite, austenite, and martensite is sensitive to the powder composition and thermal conditions. The work presented here investigates a concentric scan strategy's impact on thermal conditions within laser powder bed fusion (PBF-LB/M) processed 17–4 PH stainless steel, and the resulting phase formation and morphologies that occur. Through the combined use of computational materials science and experimental characterization, complex phase transformation routes were identified as being dependent on the site-specific thermal gyrations within the builds. Within the outer regions of the builds, a primarily δ-ferrite solidification microstructure was identified with two notable morphologies of austenite detected, allotriomorphic and Widmanstätten austenite. Within the center of the sample, however, a primarily austenite microstructure that nucleated from the prior δ-ferrite was identified. Small fractions of α-ferrite and/or martensite were potentially identified within the austenite grains. These results are rationalized using computational thermodynamics and kinetics in conjunction with proposals for pathways towards site-specific control of phase formation and morphology in PBF-LB/M stainless steels.

1. Introduction

The enthusiasm around metal additive manufacturing (AM) is due to its ability to produce highly complex geometries with less material waste that are not straightforward or impossible to be made via traditional subtractive manufacturing methods [1]. Despite the numerous advantages of AM, its adoption has not been immediate due to process variations that make it hard to produce consistent microstructures and mechanical properties [2–4]. Multi-phase (polymorphic) alloys are more sensitive to these process variations than single phase ones. 17–4 precipitation hardened (PH) stainless steel, for example, is particularly sensitive to process variations. Under traditional manufacturing methods, 17–4 PH is a martensitic precipitation hardened stainless steel after applying a standard heat treatment that has found use in the aerospace, chemical, and nuclear industries due to its combination of good ductility, high strength, and excellent corrosion resistance [5]. Under AM, the final microstructure has been shown to vary greatly depending on processing conditions, with microstructures ranging between δ-ferrite, austenite, martensite, and combinations thereof [6].

The specific causes for the large variations in the reported microstructure in 17–4 PH are related to its composition and sensitivity to cooling rate due to process variations. For instance, Meredith et al. [7] tested several powders from different powder suppliers and observed resulting retained austenite that varied from 0 to 81 vol% in their builds. These differences were attributed to the powder composition. Sun et al. [8] showed variations in retained austenite from 13.1% to 30.5% that appeared to be dependent on the sample geometry and sample position on the build plate. In their study, the variation in retained austenite was due to the impact that sample geometry and position had on the thermal history. Moyle et al. [9] demonstrated the impact of processing

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Self-Scalable Tanh (Stan): Multi-Scale Solutions for Physics-Informed Neural Networks

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***Abstract*—Differential equations are fundamental in modeling numerous physical systems, including thermal, manufacturing, and meteorological systems. Traditionally, numerical methods often approximate the solutions of complex systems modeled by differential equations. With the advent of modern deep learning, Physics-informed Neural Networks (PINNs) are evolving as a new paradigm for solving differential equations with a pseudo-closed form solution. Unlike numerical methods, the PINNs can solve the differential equations mesh-free, integrate the experimental data, and resolve challenging inverse problems. However, one of the limitations of PINNs is the poor training caused by using the activation functions designed typically for purely data-driven problems. This work proposes a scalable tanh-based activation function for PINNs to improve learning the solutions of differential equations. The proposed Self-scalable tanh (Stan) function is smooth, non-saturating, and has a trainable parameter. It can allow an easy flow of gradients and enable systematic scaling of the input-output mapping during training. Various forward problems to solve differential equations and inverse problems to find the parameters of differential equations demonstrate that the Stan activation function can achieve better training and more accurate predictions than the existing activation functions for PINN in the literature.**

***Index Terms*—Activation function, physics-informed neural networks, differential equations, inverse problem.**

I. Introduction

DEEP Learning (DL) has revolutionized many facets of our modern society, such as self-driving vehicles, recommendation systems, and web search tools. Apart from the end-user applications, DL is also accelerating solutions to problems in engineering and science, such as fluid dynamics [1], transportation systems [2], geotechnical engineering [3], manufacturing [4], biomedical engineering [5], biology [6], and physics [7]. Despite the successes when analyzing complex physical systems, much of the DL literature is purely data-driven, ignoring a vast amount of prior knowledge. This knowledge is typically in the form of differential equations from the principled physical laws.

From the seminal work on solving differential equations with a Neural Network (NN) [8] and its major revival as Physics-informed Neural Network (PINN) [9], it is striking that a simple NN can inherently solve any general differential equation (with boundary conditions). Since then, a wide variety of applications (e.g., [10], [11], [12], [13], [14], [15], [16], [17]) used PINNs to model processes with little to no data. PINN is typically a multi-layered fully-connected neural network [18] designed for the mapping between the input spatio-temporal location and the output, which is the solution to be learned. The loss function embeds the differential equations together with the fitting loss of available data (like initial/boundary conditions or experimental data), such that learning the weights (and biases) of the NN directly implies learning the solution of the differential equation.

Traditional methods to numerically solve differential equations, like the Finite Element Methods (FEM) [19], create a mesh to interpret the solution approximately at discrete points in the spatio-temporal domain. On the other hand, PINNs can solve the differential equations mesh-free, provide a pseudo closed-form solution, and can also combine the data from actual experiments to generate a more accurate model [20]. Further, PINNs can solve the inverse problem of discovering the parameters of the differential equations provided the observed experimental data [9], [18]. The inverse problem is unsolvable with the traditional numerical methods and not well explored by the standard DL methods.

Despite the extraordinary advantages, the success of PINN is limited to a handful of representative problems, and broader adoption is still far-fetched due to a wide range of issues. The unconventional loss function with multiple competing objectives explains the poorly understood loss landscape and substandard training [21], [22]. One line of work addresses the convergence of the loss function through adaptive hyper-parameters (e.g., [21], [23], [24], [25], [26]). Wang et al. [21] proposed a learning-rate annealing procedure to adaptively weigh the different parts of the loss function to overcome the difference in the convergence between these parts, whereas McClenny et al. [23] provided a way to adaptively weigh the sampled points that constitute the loss function. Both of these works use the tanh activation function and rely on the statistics

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Anomaly detection in additive manufacturing processes using supervised classification with imbalanced sensor data based on generative adversarial network

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Abstract
Supervised classification methods have been widely utilized for the quality assurance of the advanced manufacturing process, such as additive manufacturing (AM) for anomaly (defects) detection. However, since abnormal states (with defects) occur much less frequently than normal ones (without defects) in a manufacturing process, the number of sensor data samples collected from a normal state is usually much more than that from an abnormal state. This issue causes imbalanced training data for classification analysis, thus deteriorating the performance of detecting abnormal states in the process. It is beneficial to generate effective artificial sample data for the abnormal states to make a more balanced training set. To achieve this goal, this paper proposes a novel data augmentation method based on a generative adversarial network (GAN) using additive manufacturing process image sensor data. The novelty of our approach is that a standard GAN and classifier are jointly optimized with techniques to stabilize the learning process of standard GAN. The diverse and high-quality generated samples provide balanced training data to the classifier. The iterative optimization between GAN and classifier provides the high-performance classifier. The effectiveness of the proposed method is validated by both open-source data and real-world case studies in polymer and metal AM processes.

Keywords Additive manufacturing (AM) · Generative adversarial network (GAN) · Imbalanced data · Supervised learning · Anomaly detection

Introduction

Advanced manufacturing processes, such as additive manufacturing (AM), are widely applied in various fields, including aerospace, healthcare, and the automotive industry (Liu et al., 2019). However, a major barrier preventing broader industrial adoption of the processes is the quality assurance of products. For example, surface defects exist, such as underfill from the Fused Filament Fabrication (FFF) process shown in Fig. 1. It is due to highly complex interactions in consecutive layers during printing. The defect causes a deficiency in the mechanical properties of the final product, such as density, tensile strength, and compressive strength (Hajalfadul and Baumers, 2021). Therefore, timely detection of the anomaly in the process is necessary so that corrective actions can be taken to mitigate the defects and improve the quality of products (Makes and Collaborative, 2017).

Recently, the development of sensor technologies and computational power offered online process data, providing excellent opportunities to achieve effective quality assurance through a data-driven approach (Liu et al., 2021). Specifically, the sensor data are usually in high volume in terms of both dimensionality and sampling frequency, having plenty of information about the manufacturing processes (Zhou et al., 2020). Therefore, it is very beneficial to investigate the underlying relationships between the sensor data and process quality state based on a data-driven perspective (Li et al., 2021). Utilizing the sensor data, supervised classification methods have been widely applied to online anomaly detection in the manufacturing process (Banadaki et al., 2020; Jin et al., 2019; Guo et al., 2019). This is because these

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Despite the extraordinary advantages, the success of PINN is limited to a handful of representative problems, and broader adoption is still far-fetched due to a wide range of issues. The unconventional loss function with multiple competing objectives explains the poorly understood loss landscape and substandard training [21], [22]. One line of work addresses the convergence of the loss function through adaptive hyper-parameters (e.g., [21], [23], [24], [25], [26]). Wang et al. [21] proposed a learning-rate annealing procedure to adaptively weigh the different parts of the loss function to overcome the difference in the convergence between these parts, whereas McClenny et al. [23] provided a way to adaptively weigh the sampled points that constitute the loss function. Both of these works use the tanh activation function and rely on the statistics

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Morphological stability of solid-liquid interfaces under additive manufacturing conditions

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A R T I C L E I N F O

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A B S T R A C T

Understanding rapid solidification behavior at velocities relevant to additive manufacturing (AM) is critical to controlling microstructure selection. Although *in-situ* visualization of solidification dynamics is now possible, systematic studies under AM conditions with microstructural outcomes compared to solidification theory remain lacking. Here we measure solid-liquid interface velocities of Ni-Mo-Al alloy single crystals under AM conditions with synchrotron X-ray imaging, characterize the microstructures, and show discrepancies with classical theories regarding the onset velocity for absolute stability of a planar solid-liquid interface. Experimental observations reveal cellular/dendritic microstructures can persist at velocities larger than the expected absolute stability limit, where banded structure formation should theoretically appear. We show that theory and experimental observations can be reconciled by properly accounting for the effect of solute trapping and kinetic undercooling on the velocity-dependent solidus and liquidus temperatures of the alloy. Further theoretical developments and accurate assessments of key thermophysical parameters – like liquid diffusivities, solid-liquid interface excess free energies, and kinetic coefficients – remain needed to quantitatively investigate such discrepancies and pave the way for the prediction and control of microstructure selection under rapid solidification conditions.

1. Introduction

The ability to accurately understand, predict, and control solidification behavior in metals and alloys is crucial to achieve advanced manufacturing. Historically, theoretical and experimental analyses of solidification have focused on relatively slow to moderate solid-liquid interface velocities relevant to directional solidification, experienced for instance during casting processes. The fundamental understanding of microstructure selection at high velocities was primarily built on welding or quenching experiments and subsequent microstructural analyses of solidified samples. Seminal studies have identified crucial phenomena in high-velocity pattern formation, such as banding instabilities [1–4]. More recently, the emergence of additive manufacturing (AM) – relying on technologies that often involve relatively high solidification rates – has renewed the general interest in rapid solidification. In the meantime, the use of techniques for *in-situ* imaging of metals processing has also grown substantially [5–9]. These advances have paved the way for a deeper look and updated analyses on the classical assumptions and models used for the rapid solidification of metals and alloys, based on sound, well-controlled, and monitored experiments. Here we analyze measurements from *in-situ* synchrotron X-ray imaging of simulated laser powder bed fusion (PBF-LB), involving laser melting and rapid solidification of model ternary Ni-Mo-Al alloy single crystals and discuss them in light of existing solidification theories.

For a given alloy composition, the expected solidification pattern

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Full length article

Phosphorus and transition metal co-segregation in ferritic iron grain boundaries and its effects on cohesion

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ABSTRACT

The phenomenological interplay in the segregation of phosphorus (P) and transition metal (TM) elements at grain boundaries (GBs) in steels has long been suspected to be the main contributor to temper embrittlement. However, many of the details remain unclear. Here, we investigate the segregation, co-segregation and cohesion effects of TMs (Co, Cr, Cu, Mn, Mo, Ni, Nb, Ti, V, W) along with P in various ferritic iron (α-Fe) GBs utilising density functional theory and simulations of kinetics. Our findings demonstrate that P is unlikely to cause intergranular fracture via weakened interfacial bonding when segregated by itself. Nevertheless, the stronger segregation binding of P compared to TMs can explain the ubiquitous presence of P segregated at GBs. We find that most P-TM interactions at ferritic GBs are repulsive and differ significantly from the corresponding interactions in the bulk. Due to the repulsive interactions and strong segregation binding of P, the enrichment of P over time at GBs leads to the depletion of prior-segregated cohesion-enhancing solutes at general GBs. Additionally, certain P-TM co-segregation combinations that are cohesion-lowering are energetically favoured at such GBs. We posit these mechanisms act in tandem as critical causalities of P-induced temper embrittlement in alloyed steels. Finally, we reveal a contradiction in the predicted cohesion effects of segregated P calculated in the Rice–Thomson–Wang theory of interfacial embrittlement compared to that as assessed by chemical bonding strength, calculated in the DDEC6 bond-order framework. These findings have important implications for GB engineering for interfacial cohesion.

1. Introduction

Temper embrittlement is one of the oldest unresolved problems in the metallurgy of steels. The phenomenon is characterised by a brittle failure mode that occurs via intergranular fracture after an alloy steel undergoes prolonged exposure to temperatures in the range of 400–600 °C [1]. Temper embrittlement can cause the sudden, unexpected catastrophic failure of steel alloys [2]. It is well-accepted that grain boundary (GB) segregation of impurities and alloying additions play a central role in temper embrittlement [1,3–5]. This segregation can alter the cohesion of the GBs through chemical effects, allowing for them to be strengthened or weakened, depending on the specific elements segregated. As a result, extensive research efforts, experimental and theoretical, have been devoted to studying the effects of segregation of varying alloying additions and impurities on GB cohesion in steels. Despite this extensive body of research, there remains significant uncertainty regarding the specific mechanisms by which temper embrittlement occurs [6].

One of the most well-studied impurities with regard to temper embrittlement in steels is P, since it has long been suspected to be the main culprit behind the phenomenon. Pioneering experimental efforts observed the consistent presence of P on intergranularly-fractured surfaces of temper-embrittled steels [7–14], leading to the conclusion that temper embrittlement could be mostly attributed to P-segregation to GBs. However, its exact role in temper embrittlement remains unclear [6].

Importantly, temper embrittlement manifests prominently in structural alloy steels, and does not occur in pure unalloyed steels [1]. Transition metals (TMs) are one of the most important groups of alloying elements for steels, employed for purposes such as grain size stabilisation [e.g. Mn [15]], imparting corrosion resistance [e.g. Cr [16]] or their potential to drive clustering-induced strengthening [e.g. Nb [17], Ti [18]]. As a result, significant research has been devoted to investigating the role that alloying TMs play in temper embrittlement.

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Heat treatment for metal additive manufacturing

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A B S T R A C T

Metal additive manufacturing (AM) refers to any process of making 3D metal parts layer-upon-layer via the interaction between a heating source and feeding material from a digital design model. The rapid heating and cooling attributes inherent to such an AM process result in heterogeneous microstructures and the accumulation of internal stresses. Post-processing heat treatment is often needed to modify the microstructure and/or alleviate residual stresses to achieve properties comparable or superior to those of the conventionally manufactured (CM) counterparts. However, the optimal heat treatment conditions remain to be defined for the majority of AM alloys and are becoming another topical issue of AM research due to its industrial importance. Existing heat treatment standards for CM metals and alloys are not specifically designed for AM parts and may differ in many cases depending on the initial microstructures and desired properties for specific applications. The purpose of this paper is to critically review current knowledge and discuss the influence of post-AM heat treatment on microstructure, mechanical properties, and corrosion behavior of the major categories of AM metals including steel, Ni-based superalloys, Al alloys, Ti alloys, and high entropy alloys. This review clarifies significant differences between heat treating AM metals and their CM counterparts. The major sources of differences include microstructural heterogeneity, internal defects, and residual stresses. Understanding the influence of such differences will benefit industry by achieving AM metals with consistent and superior balanced performance compared to as-built AM and CM metals.

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Focused Ion Beam induced hydride formation does not affect Fe, Ni, Cr-clusters in irradiated Zircaloy-2

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ABSTRACT

Room temperature focused ion beam (FIB) milling is known to potentially promote the formation of hydrides in zirconium and its alloys. We used atom probe tomography (APT) to determine the composition of irradiated and as-produced Zircaloy-2 fuel cladding. We consistently found $\sim$ 50 at% hydrogen in all room temperature FIB-milled specimens run in voltage pulsing APT measurements. Crystallographic analysis of APT data however showed slightly better agreement with δ-hydride (ZrH_2, FCC, $\sim$ 60–66.7 at% H) than γ-hydride (ZrH, FCT, $\sim$ 50 at% H). Electron energy loos spectroscopy (EELS) measurements prior to APT analyses confirmed the presence of δ-hydride. Hence, APT gives a systematic underestimation of hydrogen for Zr-hydride. Milling at cryogenic temperatures was found to not cause such hydride formation. However, we did not find significant differences in the clusters formed by segregation of the alloying elements Fe, Cr and Ni to irradiation induced a-loops whether the material was identified as α-Zr or hydride. Therefore, analyzing irradiation-induced redistribution of alloying elements in Zr fuel cladding using APT does not rely on FIB preparation at cryogenic temperatures. However, in conjunction with voltage pulsing APT cryo-FIB can be worthwhile if one aims at investigating hydrogen distribution or hydrides.

1. Introduction

Fuel cladding in boiling water nuclear reactors is manufactured from alloyed α-Zr (HCP crystal structure) since Zr has a low thermal neutron capture cross section and Zr-alloys show good corrosion properties. The most relevant degradation mechanisms in the reactor environment are corrosion, hydrogen pick-up (HPU) and irradiation damage. Irradiation by fast neutrons creates vacancies and interstitials, which rearrange and agglomerate, leading to the formation of dislocation loops, which in alloyed Zr can have diameters smaller than 5 nm [1]. These loops lie on certain crystallographic planes [2,3]. Their number, size and type evolve with increasing neutron dose [1,4]. After a short period of irradiation (days to weeks) [1,4] a-loops with Burgers vector $1/3 < 11\bar{2}0 >$ form on close-to prismatic planes and align in layers parallel to the basal plane [2] and reach a saturation density after roughly one year [1]. Later – at the time when significant dimensional changes take place – c-loops with Burgers vector $1/6 > 20\bar{2}3 >$ form [3]. These are purely of vacancy type, lie in the basal plane, and are larger than a-loops in the same material [3].

Zircaloy-2 type alloys are the most widely used as fuel cladding in boiling water reactors (BWRs). Alloying elements are Sn, Fe, Cr, Ni and O. While Sn and O stay in solid solution, nearly the entirety of the transition metals Fe, Cr and Ni – due to their solubility of less than approximately 10 ppm by weight [5] (in agreement with APT data from Zircaloy-4 [6]) – form precipitates that are referred to as secondary phase particles (SPPs). During irradiation, these SPPs are amorphized and dissolve. Fe, Cr and Ni then segregate around dislocation loops [1,7] and form clusters throughout the α-Zr matrix that vary in composition, shape and size [8]. FeCr-clusters are often spheroidal [8,9] while FeNi-clusters are often disk- [8] or ring-shaped [10].

Hydride formation is detrimental to the structural integrity of fuel cladding since ductility is reduced and crack initiation is enhanced [11]. It occurs during operation (as hydrogen is released during Zr oxidation in water) and during cooling (when hydrogen solubility decreases) [11]. The known Zr-hydrides are: ζ-$ZrH_{0.5}$ (FCC, metastable), γ-ZrH (FCT, likely metastable), δ-$ZrH_{1.5-1.7}$ (FCC), and ε-ZrH_2 (FCT) [12]. Of these, δ- and γ-hydride are most often related to mechanical failures [12]. The maximum solubility of hydrogen in Zr is only a few atomic ppm at room temperature

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Revealing latent pole and zone line information in atom probe detector maps using crystallographically correlated metrics

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ABSTRACT

Poles and zone lines observed within atom probe field evaporation images are useful for a range of atom probe crystallography studies, including calibration of the reconstruction and crystallographic characterisation of microstructural features such as grain boundaries. However, this information is not always readily apparent. Techniques for plotting crystallographically correlated metrics contained within atom probe data to enhance pole and zone line contrast across the detector space are developed. This includes consideration of the electric field, molecular ions, lattice structure retained within the reconstruction, specific elemental species, the number of pulses between detection events, and the lateral distance between sequential detection events. These approaches are then applied to experimental atom probe tomography datasets on technically pure Al, nanocrystalline Al, highly doped Si, and additively manufactured Inconel 738, Haynes 282, and Ti–6Al–4V. The results facilitate the extension of atom probe crystallography studies to a broader range of crystalline datasets where crystallographic information is not readily apparent from existing methods, as well as a deeper understanding of field evaporation behaviour during an atom probe experiment.

1. Introduction

Atom probe tomography (APT) enables the 3D position and chemical identity of millions of individual atoms to be reconstructed with sub-nm spatial accuracy from a material specimen [1]. When using appropriate experimental conditions and analysis methods, information about the underlying crystal structure and orientation of crystal grains in the analysed specimen can sometimes be obtained and used for a variety of useful atom probe crystallography [2] studies. These include the calibration of the tomographic reconstruction [3–6] as well as direct crystallographic measurements of microstructural features such as grain boundaries [7–9] and elemental site preferencing behaviour in atomically ordered crystal structures [10,11] that are often of particular interest due to their influence on engineering material properties [12,13]. Observation of the so-called 'poles and zone lines' [14,15] in the field evaporation image (FEI) of an APT experiment on a single- or poly-crystalline specimen is typically the critical first step in performing an atom probe crystallography study, but these features are not always readily apparent in modern atom probe datasets using conventional analysis tools. Poles and zone lines are caused by the change in the local electric field environment of lattice terraces on the surface of the specimen resulting in a density variation of ion hits on the detector. Without these features, it becomes very challenging to determine the orientation of the crystal projection on the detector, rendering any crystallographic analysis problematic.

Crystallographic information, including in the FEI but also directly in the lattice structure contained within the reconstruction [16], is highly dependent on the chosen experimental parameters and material properties of the specimen being analysed [17]. In general, voltage pulsed experiments performed on metals with fewer solutes held at lower temperatures will work towards crystallographic information being more readily apparent within the dataset [17–19]. However, such conditions are not often practical for many modern atom probe studies. Lower temperature voltage pulsed experiments typically result in lower experimental yield [1], so the experimentalist often turns to laser pulsing and higher specimen temperatures for the sake of larger datasets, albeit compromising on the spatial resolution and the opportunity to more easily perform crystallographic analysis. Increasingly more complicated crystalline materials with many solute additions are also being analysed [20], which complicates the sequence of evaporation and

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Real-time process monitoring and closed-loop control on laser power via a customized laser powder bed fusion platform

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A R T I C L E I N F O

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A B S T R A C T

Additive manufacturing (AM) is one of the most effective ways to fabricate parts with complex geometries using various materials. However, AM also suffers from printing quality issues resulting from the defects such as over-melt, lack of fusion, swelling, etc. One of the root causes of those issues is that the process parameters remain constant during the entire printing process, regardless of the dynamic heat accumulation and various printing feature sizes. For instance, raster is the most common scanning strategy in the laser powder bed fusion (L-PBF) process. The length of the raster line varies depending on the printing feature size. When scanning small features, the raster line is short, resulting in heat accumulations and over-melt. These variabilities may cause severe quality issues and thus suggest adaptive process parameters be applied. Aiming to address this challenge, this study develops a closed-loop control system to regulate the laser power based on melt pool thermal emission to avoid over-melt, balling, and high surface roughness. The control target is determined by correlating the printing quality (dimensional printing error in this study) with the thermal emission through thin-line printing trials using variable power. A high-speed thermal sensor and controller are designed, tuned, and implemented on a newly developed L-PBF testbed. The system successfully maintains a low dimensional error by regulating the laser power at 2 *kHz*. A significant improvement in printing quality was achieved, as validated by both microscopic imaging and 3D scanning.

1. Introduction

1.1. Background

The popularity of additive manufacturing (AM) has increased dramatically in the past decade, given the advantage that it can effectively fabricate parts with complex geometry. Among the AM processes, laser powder bed fusion (L-PBF) has been intensively studied because it is ideal for metal 3D printing. L-PBF forms the part geometry using a high energy density laser beam to selectively melt metal powder onto the substrate [1–4]. This technology has been widely used in many industries, such as aviation, medical, and military [5,6]. However, hindered by a large number of process parameters and non-steady-state printing, high-quality printing is still hard to achieve. Specifically, the L-PBF process parameters include hatch spacing, hatch pattern, hatch angle, layer height, powder size, laser spot size, laser power, laser speed, delay time, protection gas flow rate, etc. In addition, the non-steady state of the printing can be caused by the variation of powder packing, re-melting, heat accumulation, spatters, un-uniform powder spreading, and others.

L-PBF fabricates parts lay by layer, and each layer is filled by melt tracks. Raster, a tool path generation strategy filling area by parallel lines, is the most common scanning pattern in L-PBF. However, using a fixed laser power regardless of raster length and printing feature size (thin wall or pointy regions less than 400 μm are commonly referred to as small features in L-PBF) will cause several quality issues [7]. Miniature features or sharp corners have short raster lines. In this scenario, heat will accumulate and cause over melt (see Fig. 1a). The root cause of

Abbreviations: L-PBF, Laser powder bed fusion; *PID*, Proportional-integral-derivative controller; *FOV*, Field of view; *LDR*, Light-dependent resistor (photoresistor); *ROI*, Resolution of interest; *TEI,*, Thermal emission index (the reading of the proposed sensor).

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On the interplay of internal voids, mechanical properties, and residual stresses in additively manufactured Haynes 282☆

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ABSTRACT

Complex geometries and topology optimisations for weight and materials savings are leading drivers for the additive manufacturing of Ni-based superalloys through electron beam powder bed fusion (PBF-EB). However, there is a marked departure in these geometrically complex components with respect to the thermal signatures understood in commonly studied prismatic PBF-EB test coupons. This often results in unaccounted site-specific microstructure-property variations in complex PBF-EB builds. Here, the effects of topological changes, such as intentionally engineered internal voids, on the mechanical performance of an as-fabricated Haynes 282 monolith is revealed. The internal voids serve as representative physical models for changing thermal boundary conditions with build height. Complementary local nanoindentations, multi-scale microscopy, and residual stress measurements were used to understand the mechanisms behind geometry-structure-property relationships. The results highlight the effectiveness and influence of changing thermal conditions on the local mechanical property response of PBF-EB Haynes 282.

1. Introduction

Ni-based superalloys are commonly used in high temperature power generation and aerospace applications due to their outstanding high temperature mechanical performance and chemical inertness [1]. Superalloys often contain over 10 solute additions which promote their high temperature performance [2]. However, the raw material include costly refractory elements and platinum group metals which currently have supply risk issues [2]. Additive manufacturing (AM) can reduce raw material requirements and minimise the need for subtractive machining in the fabrication of Ni-based superalloys. This is especially important for aerospace applications where buy-to-fly ratios (the amount of raw materials purchased to the amount used in-service) of sub-components can be reduced to nearly 1:1 through powder bed fusion (PBF) AM methods [3]. This is in part, due to the ability to fabricate near-net complex shaped parts inaccessible with traditional subtractive machining methods via topological optimisations.

Haynes 282 (H282), is a current generation Ni-based superalloy that has comparable tensile and creep properties to other common superalloys such as Rene-41 or Waspaloy whilst having significantly lower γ' phase fractions (~ 19 %) [4]. This low γ' fraction and chemistry that leads to smaller solidification temperature range ($\Delta T = T_{Liquidus} - T_{Solidus}$) reduces H282's susceptibility to liquation and solidification cracking during fabrication [5,6]. It is marked as one of the forerunners for the next generation of advanced ultra-supercritical turbine use in power plants [7,8]. As the presence of γ' provides the high temperature strength of Ni-based superalloys [9], this low γ' phase fraction and comparable mechanical property set of H282 makes it an ideal 'easy-to-fabricate'

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Robust Tensor Decomposition Based Background/Foreground Separation in Noisy Videos and Its Applications in Additive Manufacturing

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***Abstract*— Background/foreground separation is one of the most fundamental tasks in computer vision, especially for video data. Robust PCA (RPCA) and its tensor extension, namely, Robust Tensor PCA (RTPCA), provide an effective framework for background/foreground separation by decomposing the data into low-rank and sparse components, which contain the background and the foreground (moving objects), respectively. However, in real-world applications, the video data is contaminated with noise. For example, in metal additive manufacturing (AM), the processed X-ray video to study melt pool dynamics is very noisy. RPCA and RTPCA are not able to separate the background, foreground, and noise simultaneously. As a result, the noise will contaminate the background or the foreground or both. There is a need to remove the noise from the background and foreground. To achieve the three components decomposition, a smooth sparse Robust Tensor Decomposition (SS-RTD) model is proposed to decompose the data into static background, smooth foreground, and noise, respectively. Specifically, the static background is modeled by the low-rank tucker decomposition, the smooth foreground (moving objects) is modeled by the spatio-temporal continuity, which is enforced by the total variation regularization, and the noise is modeled by the sparsity, which is enforced by the ℓ_1 norm. An efficient algorithm based on alternating direction method of multipliers (ADMM) is implemented to solve the proposed model. Extensive experiments on both simulated and real data demonstrate that the proposed method significantly outperforms the state-of-the-art approaches for background/foreground separation in noisy cases.**

***Note to Practitioners*—This work is motivated by melt pool detection in metal additive manufacturing where the processed X-ray video from the monitoring system is very noisy. The objective is to recover the background with porosity defects and the foreground with melt pool in the presence of noise. Existing methods fail to separate the noise from the background and foreground since RPCA and RTPCA have only two components, which cannot explain the three components in the data. This paper puts forward a smooth sparse Robust Tensor Decomposition by decomposing the tensor data into low-rank, smooth, and sparse components, respectively. It is a highly effective method for background/foreground separation in noisy case. In the case studies on simulated video and X-ray data, the proposed method can handle non-additive noise, and even the case of high noise-ratio. In the proposed algorithm, there is only one tuning parameter λ. Based on the case studies, our method achieves satisfying performance by taking any $\lambda \in [0.2, 1]$ with anisotropic total variation regularization. With this observation, practitioners can apply the proposed method without extensive parameter tuning work. Furthermore, the proposed method is also applicable to other popular industrial applications. Practitioners can use the proposed SS-RTD for degradation processes monitoring, where the degradation image contains the static background, anomaly, and random disturbance, respectively.**

***Index Terms*— Robust tensor decomposition (RTD), smooth sparse decomposition, spatio-temporal continuity, total variation regularization, low-rankness.**

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Nomenclature

H, W, T	The height, width, and number of an image frame
(r_1, r_2, r_3)	The multi-linear rank in Tucker Decomposition
λ	The balance coefficient in the proposed objective function
$\mathcal{X}$	The order three tensor in $\mathbb{R}^{H\times W\times T}$ represented by $\{\mathbf{X}_1, \cdots, \mathbf{X}_T\}$
$\mathbf{X}_t$	t-th image frame in $\mathbb{R}^{H\times W}$
$\mathcal{L}$	The low-rank tensor (static video background)
$\mathcal{S}$	The smooth tensor (smooth moving objects)
$\mathcal{E}$	The noise tensor (all kinds of noise)
$\mathcal{X} \times_n \mathbf{U}$	The mode-n multiplication of a tensor $\mathcal{X}$ with a matrix $\mathbf{U}$
$\mathcal{G}$	The core tensor in tucker decompsition
$\mathbf{U}_1, \mathbf{U}_2, \mathbf{U}_3$	The factor matrices in tucker decompsition
$\boldsymbol{f}$	The auxiliary variable
$\mathbf{D}_h, \mathbf{D}_v, \mathbf{D}_t$	Three vectorizations of the difference operation along the hori-zontal, vertical, and temporal directions
$\mathbf{D}$	The the concatenated difference operation, i.e, $[\mathbf{D}_h^\top, \mathbf{D}_v^\top, \mathbf{D}_t^\top]^\top$
$\|\cdot\|_F$	The Frobenius norm

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Introducing C phase in additively manufactured Ti-6Al-4V: A new oxygen-stabilized face-centred cubic solid solution with improved mechanical properties

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An oxygen-rich face-centred cubic (FCC) Ti phase was engineered in the microstructure of a Ti-6Al-4V alloy via additive manufacturing using laser powder bed fusion. Designated 'C', this oxygen-rich FCC phase has a lattice parameter of 0.406 nm and exhibits an orientation relationship with the parent α' phase as follows: $(0001)_{\alpha'}//\{111\}_C$, and $\langle 1\bar{2}10\rangle_{\alpha'}//\langle 1\bar{1}0\rangle_C$. We propose that the formation of the C phase is facilitated by the combined effect of thermal gradients, deformation induced by the martensitic transformation, and local O enrichment. This enables an in-situ phase transformation from the hexagonal close-packed α' phase to the C phase at elevated temperatures. Our density functional theory calculations indicate that oxygen occupancy in the octahedral interstices of the FCC structure is energetically preferred to corresponding sites in the α' phase. The in-situ mechanical testing results indicate that the presence of the FCC phase significantly increases the local yield strength from 1.2 GPa for samples with only the α' phase to 1.9 GPa for samples comprising approximately equal volume fractions of the α' and FCC phases. No loss of ductility was reported, demonstrating great potential for strengthening and work hardening. We discuss the formation mechanism of the FCC phase and a pathway for future microstructural design of titanium alloys by additive manufacturing.

Keywords: FCC Ti; Additive manufacturing; Interstitial strengthening; Mechanical properties

Introduction

Titanium and its alloys – especially Ti-6Al-4V – exhibit a remarkable combination of high specific strength, and excellent corrosion resistance such that they are widely used in critical applications ranging from aerospace to biomedical implants. Their capacity for plastic deformation is limited, in large part, by the hexagonal close-packed (HCP) nature of the low-temperature α phase. Speculation that a face-centred cubic (FCC) form of Ti can be stabilized at room temperature goes back several decades [1–13]. Multiple controversies have emerged from these propositions [1–3,5,12], such that today it is widely agreed that the FCC forms of Ti are either intermetallic

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Electron and laser-based additive manufacturing of Ni-based superalloys: A review of heterogeneities in microstructure and mechanical properties

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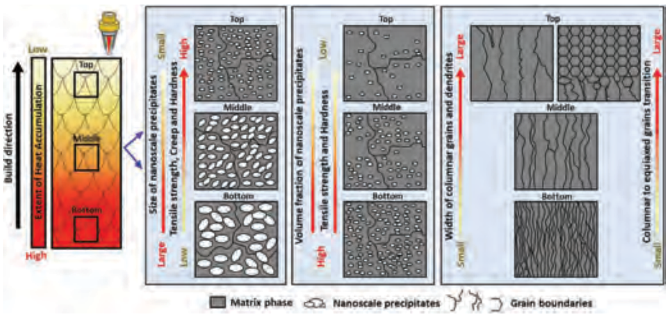
HIGHLIGHTS

- The heterogeneities in the microstructure and mechanical properties of AM Ni-based superalloys are reviewed.
- The origins of heterogeneities are linked to the variations in thermal conditions throughout the build.
- A short case study is presented.
- Strategies to minimize microstructure heterogeneity are discussed.

GRAPHICAL ABSTRACT



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ABSTRACT

The adaptation of additive manufacturing (AM) for Ni-based superalloys has gained significance in aerospace and power-generation industries due to the ability to fabricate complex, near-*net*-shape components on-demand and with minimal material waste. Besides its advantages, challenges remain in metal AM, especially for printing complex alloys such as superalloys. These challenges are often linked to heterogeneity in the as-fabricated parts and continue to limit the practical applications of AM products. A thorough understanding of the relationship between the complex AM process and the resulting microstructure heterogeneity needs to be established before mitigation strategies can be developed. The ability to fabricate more homogeneous Ni-based superalloy parts is expected to unlock not only better mechanical properties but also additional fields of applications.

This review aims to summarize the current understanding of heterogeneities in the microstructure and mechanical properties of AM Ni-based superalloys. Microstructure heterogeneities discussed include heterogeneity in the chemical composition, phase constitution, porosity, grain and dendrite morphology, and solid-state precipitates. Related heterogeneities in hardness, tensile, creep, fatigue, and residual stress are discussed to represent mechanical properties, and mitigation strategies are summarized. The origins of heterogeneity in the as-fabricated parts are linked to the variations in AM thermal conditions caused by the complex thermal histories.

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Additively manufactured Haynes-282 monoliths containing thin wall struts of varying thicknesses

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ABSTRACT

Magnitude and distribution of residual stresses in additively manufactured Ni-based superalloys may impact the mechanical performance of as-fabricated parts. Though electron beam powder bed fusion (E-PBF) can produce components with minimal defects and residual stresses compared to laser powder bed fusion and directed energy deposition, variations of them may occur within the complex geometry of a component, due to inherent variations of thermal signatures and the evolution of section modulus along the build direction. This work reveals the residual stress distribution, characterised from neutron diffraction, of an as-fabricated Haynes 282 monolith containing internal cube voids and thin wall struts of varying thicknesses. Complementary local hardness measurements and multi-scale microscopy were used to investigate the geometry-structure-property relationships. Observed variations in hardness were attributed to a combination of type I macro-scale residual stresses and variations in bimodal γ' precipitation behaviour. The results highlight the influence of residual stresses and microstructure on the mechanical properties of E-PBF Haynes 282.

1. Introduction

Haynes 282 (H282) is a current generation Ni-based superalloy, developed and adopted for hot section parts in gas turbines for aircraft and power generation [1,2]. It is considered one of the forerunning candidates for turbine components in new, advanced ultrasupercritical power plant applications [3,4]. These powerplants aim for net plant efficiencies of ~47%, up from current-generation powerplant designs of 37% efficiency [5], attained through steam operating temperatures and pressures up to 760 °C and 31 MPa, respectively. This increase in thermal efficiency was estimated to reduce CO_2 and fuel-related emissions from 0.85 to 0.67 tonnes/MWh, a significant ~21% reduction [5].

H282's high-temperature strength is achieved primarily through intermetallic $L1_2$ γ' precipitation. However, compared to other common Ni-based superalloys such as Waspaloy and Rene-41, fully aged H282 has a comparably lower γ' phase fraction of 19%, whilst retaining equivalent tensile and creep properties [2,6]. This severely reduces the strain-age cracking tendency – greatly increasing its fabricability and weldability compared to other superalloys [6]. Furthermore, long term thermal exposure at 760 °C shows increased room temperature yield strength, without the formation of topologically close packed (TCP) phases. It is noteworthy that the TCP phases are brittle and deleterious and often found in other Ni-based superalloys such as Rene-41 [1]. With the design and development of new generation of steam and gas turbines

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Review

Powder bed fusion additive manufacturing of Ni-based superalloys: a review of the main microstructural constituents and characterization techniques

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ABSTRACT

Metal additive manufacturing (AM) has unlocked unique opportunities for making complex Ni-based superalloy parts with reduced material waste, development costs, and production lead times. Considering the available AM methods, powder bed fusion (PBF) processes, using either laser or electron beams as high energy sources, have the potential to print complex geometries with a high level of microstructural control. PBF is highly suited for the development of next generation components for the defense, aerospace, and automotive industries. A better understanding of the as-built microstructure evolution during PBF of Ni-based superalloys is important to both industry and academia because of its impacts on mechanical, corrosion, and other technological properties, and, because it determines post-processing heat treatment requirements. The primary focus of this review is to outline the individual phase formations and morphologies in Ni-based superalloys, and their correlation to PBF printing parameters. Given the hierarchal nature of the microstructures formed during PBF, detailed descriptions of the evolution of each microstructural constituent are required to enable microstructure control. Ni-based superalloys microstructures commonly include γ, γ', γ'', δ, TCP, carbides, nitrides, oxides, and borides, dependent on their composition. A thorough characterization of these phases remains challenging due to the multi-scale microstructural hierarchy alongside with experimental challenges related to imaging secondary phases that are often nanoscale and (semi)-coherent. Hence, a detailed discussion of advanced characterization techniques is the second focus of this review, to enable a more complete understanding of the microstructural evolution in Ni-based superalloys printed using PBF. This is with an expressed goal of directing the research community toward the tools necessary for a thorough investigation of the processing-microstructure-

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Intergranular precipitation and chemical fluctuations in an additively manufactured 2205 duplex stainless steel

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ABSTRACT

Fluctuations in energy distribution during additive manufacturing (AM) can result in spatial and temporal thermal transients. These transients can lead to complexities, most significantly when alloys with multi phases are subjected to AM. Here we unveil such complexities in a duplex stainless steel, where we report an unanticipated formation of a Ni-Mn-Si rich phase at grain boundaries and a local fluctuation in Cr and Fe concentrations in regions close to grain boundaries, providing Cr-rich precursors for Cr_2N formation after laser powder bed fusion (LPBF). The formation of these phases is believed to be due to severe thermal gyrations and thermal stresses associated with LPBF resulting in a high-volume fraction of ferrite supersaturated with N and Ni, and a high density of dislocations accelerating diffusion and phase transformations.

Recent advancements in additive manufacturing (AM) of metals have made it possible to 3D-print duplex stainless steels (DSSs) into complex shaped parts, with mechanical and corrosion properties comparable to those of wrought counterparts [1–7]. While offering several benefits such as short lead time, design flexibility and raw material savings, metal AM results in non-equilibrium microstructures and micro/macro instabilities due to its inherent thermal gyrations and high cooling rates [8,9]. Thus, significant deviations from equilibrium microstructures have been reported in DSSs, after laser powder bed fusion (LPBF) AM [2,3,5,7]. For instance, contrary to the usual 50/50 austenite-ferrite microstructure, 2205 DSS after LPBF is mainly ferritic with a small volume fraction of austenite (<5%) precipitating at ferrite-ferrite boundaries. Another major difference of LPBF DSS compared to its wrought counterparts is the supersaturation of N and other austenite forming elements in ferrite that, combined with small fractions of austenite, can lead to the formation of unexpected precipitates [3,7]. While *in-situ* Cr nitride formation is now a well-known phenomenon in AM of DSS, formation of other precipitates, e.g., those enriched in Ni and Si, in the as-deposited condition has not yet been reported.

Precipitation of Cr nitrides plays an important role in controlling the corrosion and mechanical properties of DSSs [3,10]. For traditionally manufactured DSS, it is widely accepted that Cr nitrides form at the austenite-ferrite boundaries due to an abundance of N and Cr and accelerated diffusion therein, where the interphase boundaries act as heterogenous nucleation sites lowering the energy barrier for nucleation [11]. Due to the low volume fraction of austenite in LPBF DSS [3], there are a very limited number of austenite-ferrite boundaries available that may act as nucleation sites. Furthermore, most of the austenite−ferrite boundaries in the as-LPBF condition exhibit the low energy Kurdjomov-Sachs orientation relationship [3] that has been reported to make these boundaries resistant to third phase precipitation [12]. Up to now, the formation of Cr nitrides in LPBF has been reported to occur at ferrite-ferrite boundaries or intragranularly, driven by cyclic heating/cooling and supersaturation of N within ferrite, respectively [3,13,14]. The role of spatial and temporal transients inherent to AM leading to chemical fluctuations and how they may potentially contribute to the formation of Cr_2N has not been studied yet.

Here we show, using a combination of scanning transmission electron microscopy (S-TEM) and atom probe tomography (APT), the LPBF-

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High Throughput Characterization to Quantify Microstructural Heterogeneities in Additively Manufactured Haynes 282

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Haynes 282 is a precipitation strengthened nickel (Ni) based superalloy that finds extensive application in industrial gas turbine engines used in Advanced Ultra Supercritical (A-USC) powerplants, primarily due to its excellent high temperature creep resistance and thermal stability. These properties in combination with good weldability make this alloy an ideal candidate for fabrication by Additive Manufacturing (AM). AM offers the advantage of near-net shape fabrication of complex geometries while optimizing the amount of material used.

Electron Beam Melting (EBM) is a powder bed fusion-based AM process which uses a high energy electron beam to melt metal powder particles spread over a bed, in a layer-by-layer fashion to generate a near-net shaped component. During EBM processing, the material experiences steep thermal gradients and rapid thermal cycling, which results in reduced partitioning of solute elements and formation of columnar grains with non-equilibrium phases. Recent reports have shown that spatio-temporal thermal transients during AM can be controlled by varying the beam scanning strategy (energy input) to produce a columnar to equiaxed transition [1-4]. Previous research on Ni-based superalloys has shown that γ' precipitation is strongly influenced by solidification rates and thermal treatments. And precipitation distribution influences mechanical properties such as hardness and tensile strength [5-6]. A wider adoption of AM processes in industry is often challenged by a lack of significant datasets to generate coherent process-structure-property linkages. In order to enable industrial qualification of EBM deposited Haynes 282 components, the effects of varying thermal gradients and solidification rates on γ' precipitation and mechanical properties needs to be systematically analyzed. Studies investigating evolution of γ' as a function of varying EBM parameters are still very limited [7].

In this work, we use high throughput electron microscopy characterization to quantify γ' size distribution, and micro hardness testing to evaluate the impact on mechanical properties. AM build and sample geometry, as well as sites for detailed microstructural characterization are shown in the schematics in Figure 1. Three columns with the same build height (10 mm) but with different thickness of 2, 3 and 4 mm, respectively, were investigated to understand the impact of build geometry. A 2-mm-high section at the base of the build was included in the study to investigate the impact of a change in scan velocity. In order to perform systematic site-specific characterization, the sample was classified into regions according to build height and column thickness (as shown in Figure 1b).

A low acceleration voltage (1 kV) and low working distance (5 mm) as well as in-column detectors were used in the scanning electron microscope (SEM) allowing for optimum resolution and contrast to resolve γ' precipitates [8]. Both backscattered electron (Z contrast), and secondary electron (topological contrast) imaging was used to characterize γ' precipitates at each location. MIPAR software was used for thresholding to obtain quantitative values for γ' size. Automated image collection using FEI MAPS software was performed to generate a statistically significant dataset. As seen in the graph in Figure 2a,

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In situ TEM Observations of Thermally Activated Phenomena in Materials Under Far-From-Equilibrium Conditions

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Structural materials, that have been fabricated using additive manufacturing (AM) or have been welded using metal joining processes, experience varying spatial and temporal thermal transients due to a very local but high energy heat source. Similarly, components experience varying thermal transients when 'in service', e.g. in next generation nuclear reactor cores, gas turbine engines, re-entry space vehicles and solder joints in micro-electronic packages. These varying thermal transients (extreme thermal gradients (10^4 - 10^6 K/m) and/or rapid thermal cycling (10^2- 10^3 K/s)) cause microstructural changes due to solid-solid phase transformations under far-from-equilibrium conditions forming metastable phases with unknown properties.

In order to improve materials performance, dynamic microstructural evolution under such thermal processing or 'in service' conditions need to be better understood. Currently, this information can only be obtained through *post-mortem* characterization of parts. To study these dynamic processes at high spatial resolution and correlate them to material properties and performance, the development of new *in-situ* heating stages is needed. Previously, in-situ transmission electron microscopy (TEM) heating experiments have been used to study thermally activated phenomena such as defect-solute interactions, diffusion-controlled and interface-controlled phase transformations in structural materials [1-3]. Micro-electro-mechanical-system (MEMS)-based heating stages have the potential to enable such studies. Benefits of MEMS microheaters include fast heater response times, low thermal mass, and high temperature homogeneity [4]. These MEMS heating stages have enabled in-situ investigations of steady-state phenomena at high spatial resolution under isothermal conditions.

Recently, we have modified a commercially available MEMS microheater to enable the generation of a thermal gradient across a TEM sample [5]. Ex-situ infra-red thermography and the in-situ 'Ag nanocube sublimation' technique [4] were used to confirm the temperature distribution and reveal a large thermal gradient (10^6 K/m) across the area of interest [6]. Additionally, the rapid heating and cooling rates of up to 10^3 K/s of MEMS devices allows us to mimic processing and/or 'in service' like conditions for a wide range of applications, inside the TEM.

Here, we show that MEMS based heating stages can be used to replicate transient thermal conditions by adopting three simple strategies, as shown in Figure 1(a-c). *In situ* TEM case studies that mimic the processing and/or 'in service' thermal conditions for three critical applications are presented.

Ductility dip cracking (DDC) is a solid-state phenomenon that occurs in re-heated weld metal or heat affected zones of base metals [7]. DDC is a major problem in Ni-Cr alloy filler metals due to

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Gamma Prime Characterization in Additively Manufactured Haynes 282 after One-Step and Two-Step Post-Process Heat Treatments

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Haynes 282 is a precipitation strengthened Ni-based superalloy with applications in industrial gas turbine engines due to high temperature creep resistance and thermal stability. Its improved performance at high temperatures has been attributed to the existence/appearance of the gamma prime (γ’) phase [1]. Additionally, the high weldability of Haynes 282 lends itself to the fabrication by metal-based additive manufacturing (AM) techniques, such as electron beam powder bed fusion (EBM-PBF). AM is advantageous for producing near net shapes of complex geometries with reduced material waste.

Despite these advantages, there are some fundamental challenges related to the physical metallurgy of AM fabrication processes that still need to be understood. The non-equilibrium EBM process conditions (large thermal gradients and rapid temperature cycling) result in metastable microstructures with a columnar morphology that differ significantly from microstructures obtained by conventional processing routes. Major research efforts are directed towards identifying AM processing and post-processing routes to fabricate builds with equiaxed microstructures, even in complex build geometries, to achieve AM builds exhibiting anisotropic properties.

Previous reports have shown that high temperature properties of Haynes 282 processed in the conventional (non-AM) manner can be significantly improved by a post-process heat treatment (i.e., ageing) which includes a two-step ageing process at 1010°C for 2 hours with subsequent air cooling plus a second step at 788 °C for 8 hours and subsequent air cooling [2]. More recently, Unocic et al. reported that a simpler one-step ageing (2, 4, 6, or 8 hours at 800 °C) can yield comparable mechanical properties in Haynes 282 as in samples after a two-step ageing when fabricated conventionally [3] or by EBM [4]. In one of our earlier studies [5], we examined the variations in microstructure and hardness across an EBM Haynes 282 build composed of thin walls of different thickness and similar height over a base. Our characterization study revealed that γ’ and Vickers hardness of these builds were inversely correlated. Subsequent heat treatments under two different ageing conditions (one-step and two-step) resulted in no significant variation in Vickers hardness, compared to the as-fabricated EBM sample. However, the microstructure showed an anomalous decrease in γ’ size in certain locations of the build after both ageing treatments.

Here, we perform a systematic evaluation of the microstructural evolution in γ’ size and morphology at different time steps, in both the one-step ageing and two-step ageing case. The microstructure of samples obtained from different sections of the build, shown in Figure 2(b), are investigated using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). For each sample, images were taken 1 mm away from the top and bottom edges to give a representative area to characterize the γ’ precipitates, shown in Figure 2(b). The mean γ’ size in the as-deposited build was found to be 0.078 μm in Section 2, taken at 12 locations with 180 images. After completing of the one-step and two-step ageing heat treatment, mean γ’ size decreased to 0.040 and 0.048 μm, respectively. Analysis of γ’ precipitates was achieved by high-resolution SEM imaging using an immersion lens at low kV(2 kV) in an Apreo LoVac SEM (Thermo Fisher Scientific Inc). The sample was placed at a low working distance of ∼ 5 mm and the ‘in-column’ T3 as well as the ‘in-lens’ T1 detectors were used. Thereby, both backscattered electron (Z contrast) and secondary electron (topographical contrast) imaging contrast were used to characterize γ’. The volume fraction, γ’ size, and other quantitative microstructural information were determined through the image processing software MIPAR [6, 7].

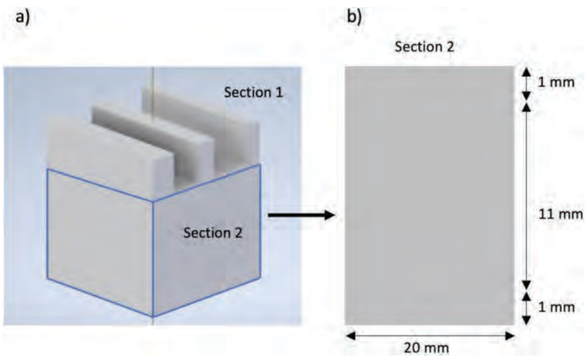

Fig. 1. (a) Schematic of EBM Haynes 282 build showing sections 1 and 2. Section 2 is the focus area for the γ′ characterization after post processing.

PUBLICATIONS PHASE 2

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Microscopy AND Microanalysis

Proceedings

Microstructural and Mechanical Property Differences Resulting from Melt Pool Interactions with the Electron Beam Chamber Environment

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Additive manufacturing (AM) of the popular titanium alloy Ti-6Al-4V continues to displace traditional manufacturing methods in multiple industries, due to benefits that include a reduction of waste and an increase in the complexity of allowable geometries. Recent publications have focused on the characterization of materials states related to the thermomechanical cycles experienced during the AM process (i.e., the liquid-solid, solid-solid transformations resulting from the layer-by-layer nature of the process) through build heights [1-5]. While the cyclic nature of AM processes is important in setting the microstructure, the interactions of the melt pool with the surrounding chamber environment are equally important and have significant impacts on the resulting materials state.

Literature often carries the assumption that compositions are homogeneous in single-feedstock parts, as the powder remains the same throughout the build height [1, 6]. However, during the initial and subsequent melting stages, melt pool interactions with the chamber environment include the evaporation and recondensation of elements, leading to local variations in composition [7-11] that are often neglected in the literature. The work presented here contains examples of samples that show evidence of preferential Al vaporization in electron beam melted Ti-6Al-4V, and the effect that the resulting local variations in composition have on microstructures and mechanical properties.

Ti-6Al-4V square prisms (15mm (X) x 15mm (Y) x 25mm (Z, build direction)) were produced at Oak Ridge National Laboratory Manufacturing Demonstration Facility (ORNL MDF) using an ARCAM EBM Q10plus system. Details of the raster and random scan strategies used are described in [1, 12-14].

The as-built samples were sectioned using electrical discharge machining (EDM). The EDM'd surfaces were ground and polished using traditional metallographic sample preparation techniques, completely removing the EDM recast. Samples were imaged using an FEI Teneo LoVac field-emission scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS). MATLAB was used to plot EDS maps for easier comparison. Optical microscopy (OM) mosaics were obtained using a Zeiss inverted optical metallographic microscope after the sample was etched with Kroll's reagent.

Preferential vaporization is a well-studied phenomenon, whereas, at high temperatures, elements with higher vapor pressure, vaporize first and at a faster rate [7, 10, 11]. These local fluctuations in composition are significant, as they can lead to local variations in the materials state of a part. Examples of local compositional changes in both scan strategies (raster and random) are shown in Figs. 1(a) and (b). Both samples contain regions of high Al (~6wt.%) and low Al (~4-5wt.%), showing that the assumption of homogeneous composition from the literature is incomplete (as V remains fairly consistent, EDS maps depicting this element are not shown). In the raster scan strategy, these compositional changes have a primarily banded structure (Fig. 1(a)) due to the morphologies of the resulting melt pools, as has been observed previously in other work [7-10]. In the random strategy, the compositional changes are not as 'structured' and primarily follow the shapes of the melt pools (Fig. 1(b)). Furthermore, the compositional variations can also be seen in etched optical micrographs of the raster scan strategy sample (Fig. 1(c)), in the form of alternating dark and light microstructural bands, but cannot be detected using the same etching procedure in the random scan strategy sample (Fig. 1(d)). Kroll's reagent, the etchant used, preferentially attacks the β phase in Ti alloys, indicating a higher fraction of the β-phase in the darker regions. The darker bands have also been shown to correspond with lower Al content [15], leading to the higher fraction of β in these regions as compared to the lighter banded regions.

ORIGINAL RESEARCH ARTICLE

Heterogeneity and Solidification Pathways in Additively Manufactured 316L Stainless Steels

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A unique microstructural feature often referred to as ''fish-scale'' has been reported in 316L austenitic stainless steel parts made by laser powder bed fusion (L-PBF) technique. Because the final microstructure is predominantly austenitic, with a face-centered cubic (γ-fcc) crystal structure, the ''fish-scale'' structures were originally assumed to be based on etching response due to crystallographic orientations of the solidified γ grains. This research evaluated this assumption through multi-length scale and site-specific characterization using optical microscopy, hardness mapping, X-ray diffraction, electron back-scattered diffraction imaging, and scanning transmission electron microscopy. The nanoscale compositional measurements suggest that the ''fish-scale'' structures are related to a phase selection phenomenon that occurs during solidification due to spatial and temporal variation of thermal gradients and liquid–solid interface velocity. This phenomenon triggers the transition from γ-fcc to body-centered cubic (bcc) δ-ferrite solidification and then subsequent solid-state phase transformations of this bcc to fcc at low temperature. The significance of these phase transformation pathways with reference to deployment of additively manufactured 316 stainless steel components for harsh environments relevant to power generation is discussed.

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On the high-temperature stability of the Al_8Cu_3Ce intermetallic in an additively manufactured Al-Cu-Ce-Zr alloy

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ABSTRACT

High-temperature resistant eutectic Al alloys are crucial materials for lightweight and energy efficient design in the automotive and aviation industries. Additive manufacturing offers a pathway to refine eutectic microstructures and develop novel alloys with superior high-temperature strength. High-volume fraction intermetallic Al-Cu-Ce alloys have been developed to deliver high-temperature strength in combination with reduced hot-tearing susceptibility. Zr is added to provide additional strengthening via nanoscale Al_3Zr precipitation, and to stabilize and avoid coarsening of the Al_8Cu_3Ce phase. However, the detailed interaction between Zr and Al_8Cu_3Ce remains unexplored. In this work, we show with synchrotron X-ray diffraction that laser powder bed fusion fabricated Al-Cu-Ce and Al-Cu-Ce-Zr alloys contain predominantly the Al_8Cu_3Ce intermetallic in the as-fabricated condition. Heat treatment of the Al-Cu-Ce alloy results in the $Al_8Cu_3Ce \rightarrow Al_8Cu_4Ce$ phase transformation. In the Al-Cu-Ce-Zr alloy, minor fractions of $(Al,Cu,Si)_4Ce$ and Al_2Cu-θ are found in the as-fabricated condition, while Al_8Cu_3Ce remains stable during heat treatment. Atom probe microscopy quantifies intermetallic stoichiometries and reveals how Zr is enriched at the Al-matrix/Al_8Cu_3Ce interface acting as a diffusion barrier against solute exchange. Calibrated thermodynamic modeling underpins this as a kinetic effect. A qualitative microstructural model summarizes, how Zr stabilizes Al_8Cu_3Ce against phase transformations and coarsening.

1. Introduction

The high specific strength of Al alloys makes them attractive materials for light weight mechanical design in the aviation and automotive industries [1–3]. However, a major challenge is to unlock high-temperature mechanical properties for service above 200 °C, or ~0.5 of the homologous melting temperature [4,5]. Layer-by-layer fabrication of parts via additive manufacturing (AM) is one pathway promising superior properties in comparison to conventionally processed Al alloys [5]. Unique cooling and solidification conditions during AM enable flexibility in the design of components with refined microstructures and improved mechanical properties [6,7]. The high cooling rates during AM unlock novel alloy compositions with potential to increase supersaturation in solid solution and thus, promote increased volume fractions of strengthening phases [8,9]. In a comprehensive review, Michi et al. [5] identified high-temperature precipitation strengthened alloys, high-volume fraction intermetallic alloys and ceramic dispersion alloys as the most promising candidates. Al-Cu-Ce-(Zr) alloys belong to the group of high-volume fraction intermetallic alloys. Dense networks of Ce-rich intermetallic phases provide strengthening at temperatures reaching up to 400 °C [10,11], or coarsening resistance up to 500 °C [12].

The alloying elements Cu, Ce, Zr and Si have gained interest in recent years. Cu and Si are additions with high solubility in the Al-matrix [4]. Si promotes the formation of a eutectic which is beneficial for processability through avoiding solidification cracking [13], and Si in solid solution provides additional strengthening, particularly when high cooling rates in AM result in extended solubility [14,15]. Cu promotes the formation of various intermetallic phases, and Al_3Cu-θ'', Al_2Cu-θ', and Al_2Cu-θ are most frequently reported [16,17]. Zr exhibits limited solubility in the Al-matrix, but experimental observations have shown the potential for supersaturation during AM processing, as mentioned above [9]. Thus, strengthening $L1_2$-ordered Al_3Zr precipitates may be formed during heat treatments [18–21]. Ce exhibits near-zero solubility in the Al-matrix and forms various intermetallic phases [22]. Al_4Ce is usually formed as a high-temperature phase, whereas $Al_{11}Ce_3$ or Al_3Ce

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Evidence of *in-situ* Cu clustering as a function of laser power during laser powder bed fusion of 17–4 PH stainless steel

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A R T I C L E   I N F O

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Additive Manufacturing
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Precipitation

A B S T R A C T

The use of intrinsic heat treatments to control the microstructural evolution during additive manufacturing could eliminate the need for costly post-build processing. Using atom probe microscopy and cluster search algorithms, this study investigates the degree of Cu clustering and precipitation in 17–4 precipitate hardening (PH) stainless steel fabricated by laser powder bed fusion (LPBF). It was found that LPBF samples exhibit a greater than random degree of Cu clustering, irrespective of the laser power during fabrication. It is further shown that using a higher laser power (161.5 W rather than 127.5 W) led to a higher number density of Cu clusters, Cu precipitates, and higher hardness due to the greater heat input. The observations of Cu-rich clusters and precipitates within as-printed LPBF samples and its laser power dependence are novel and show potential for inducing desired strengthening phases directly during LPBF, mitigating the need for post-fabrication heat treatments.

17–4 precipitation hardening (PH) stainless steel possesses high strength and corrosion resistance, allowing it to be applied widely, for example in the aerospace, automotive, and marine industries [1–3]. Due to its good weldability [4], many of the studies around laser powder bed fusion (LPBF) of stainless steels have centred around this alloy [5,6]. During conventional processing, 17–4 PH is solution annealed in the austenite phase field, quenched to form a martensitic microstructure, and then aged to form nanoscale Cu rich precipitates, which enable the attainment of its high strength [2]. Continued ageing of conventionally fabricated 17–4 PH results in several changes to the crystal structure of the Cu precipitates. Initially, they nucleate and grow with a body-centred cubic (BCC) structure inherited from the matrix, before becoming incoherent and face-centred cubic (FCC) via a series of intermediate structures such as the 3R and 9R crystal structures [7,8]. This precipitation sequence also holds true for other PH stainless steels that form Cu rich phases after ageing, such as 15–5 PH [9]. Strength and hardness in these Cu precipitation hardening steels is reported to peak when the precipitates attain diameters of 1.5 – 3 nm [10–12], which is typically when they exhibit the 9R crystal structure [9,10]. This ageing response may occur faster in highly dislocated structures due to the enhanced effect of dislocation pipe diffusion [13].

Alloys such as 17–4 PH may experience an intrinsic heat treatment (IHT) during fabrication via various additive manufacturing routes [14]. In LPBF, as the laser melts subsequent layers of the build, previously melted material outside new melt tracks experiences cyclic reheating with each pass of the laser. Although not sufficient to melt these regions, this reheating can still result in thermally activated microstructural changes in many alloys [14]. Examples include precipitation [14–19], phase transformations [15,19,20], recrystallisation [21], and grain reorientation [22].

The potential to leverage the IHT during LPBF to induce solute clustering and even precipitation hardening in PH stainless steels is largely unexplored. Atom probe microscopy (APM) and transmission electron microscopy have been used to quantify the precipitation sequence in conventionally produced 17–4 PH during thermal ageing [10–12]. Our goal was to design a LPBF process that leveraged IHT to induce Cu clustering and precipitation. We have used APM to quantify the level of clustering and precipitation which we have sought to correlate to the LPBF laser power. This can pave the way for targeted tuning of printing parameters to develop precipitation hardened 17–4 PH parts through IHT and reduce the requirement for post processing heat treatments. The wider context of our goal is the more general desire to directly manufacture (print) precipitation hardened 17–4 PH parts with little or no requirement for post-processing heat treatments.

17–4 PH stainless steel samples were fabricated using a 3D Systems ProX 300 LPBF machine. The process was carried out in an N_2

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PUBLICATIONS PHASE 2

Formation of a transition V-rich structure during the α' to $\alpha + \beta$ phase transformation process in additively manufactured Ti-6Al-4 V

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ABSTRACT

Ti-6Al-4V parts fabricated by laser powder-bed fusion (L-PBF) additive manufacturing often suffer from poor ductility and low toughness due to the predominance of the acicular α' martensitic phase in their microstructures. This challenge can be overcome by manipulation of the L-PBF thermal history to introduce an $\alpha' \rightarrow \alpha+\beta$ decomposition, resulting in a fine lamellar $\alpha+\beta$ structure with a combination of excellent strength and ductility. Understanding the details of the $\alpha' \rightarrow \alpha+\beta$ phase transformation process is critical for fabricating titanium alloys with excellent mechanical properties. Through a systematic electron microscopy characterisation of the microstructural evolution of a Ti-6Al-4V alloy fabricated by L-PBF, here we reveal that the $\alpha' \rightarrow \alpha+\beta$ phase transformation occurs in two steps: $\alpha' \rightarrow \alpha + \alpha_{HME}$ (high in Mo_{eq}) and $\alpha_{HME} \rightarrow \beta$, in which α_{HME} is a newly discovered non-equilibrium structure with a hexagonal close-packed structure and with a composition close to that of the β phase.

1. Introduction

Metal additive manufacturing (AM) brings about highly non-equilibrium processing such that alloys are subjected to complex thermal and stress gyrations that are far from those encountered in conventional metallurgical processing. The large thermal gradients and rapid rates of temperature change create new process dynamics [1–4], affecting material strength, ductility, fatigue life, and many other critical properties [5–8]. Laser powder-bed fusion process (L-PBF) is a premier AM technique [1,4,9–11], which offers distinct advantages over conventional manufacturing, including design freedom, near-net or net shape production, efficient use of materials, short lead time, and substantial cost saving in many cases [1,4,12]. A wide variety of metallic materials, including stainless steels [2,6,13], high-entropy alloys [14–16], aluminium alloys [17], and titanium alloys [12], have been processed to date using L-PBF.

Titanium and its alloys, especially Ti-6Al-4V, exhibit a remarkable combination of high specific strength and excellent corrosion resistance such that they are widely used in critical applications ranging from aerospace components to biomedical implants. The benchmark dual-phase Ti-6Al-4V demonstrates excellent manufacturability by various AM processes [4,7,18–21]. Depending on the processing thermal history, Ti-6Al-4V can have different microstructures and phases: (i) the hexagonal close-packed (HCP) α' martensitic phase (P63/mmc, a = 0.293 nm, c = 0.468 nm) from rapid cooling [12,22,23], (ii) the dual-phase HCP α phase (P63/mmc, a = 0.293 nm, c = 0.467 nm) [22–25] + body-centred cubic (BCC) β phase (Im$\bar{3}$m, a = 0.319 nm) structure from moderate cooling or equilibrium conditions [22,25], and (iii) the orthorhombic soft martensite α" (Cmcm, a = 0.301 nm, b = 0.497 nm, c = 0.466 nm) from quenching from relatively low temperatures (between 750 and 900 °C) [22,26,27]. Samples with a fully α' phase can achieve a yield strength of > 1300 MPa, but the total tensile elongation (ductility) is below the minimum threshold of ~10% recommended for critical structural applications [22,28]. The poor ductility is caused by the HCP nature of the low temperature supersaturated α' phase with a slightly distorted lattice. Post heat treatment is often applied for ductility improvement [29]. Ti-6Al-4V parts that are processed by solution treatment at 940 °C followed by ageing at 480 °C for 8 h and then cooling in air contain fine lamellar $\alpha+\beta$ and exhibit an excellent combination of yield strength (> 1100 MPa) and tensile elongation (13%) [28]. Recent studies showed that the corrosion resistance of AM Ti-6Al-

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TECHNICAL ARTICLE

Check for updates

A Comparison of Statistically Equivalent and Realistic Microstructural Representative Volume Elements for Crystal Plasticity Models

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Abstract
Two methods used to construct a microstructural representative volume element (RVE) were evaluated for their accuracy when used in a crystal plasticity-based finite element (CP-FE) model. The RVE-based CP-FE model has been shown to accurately predict the complete tensile stress–strain response of a Ti–6Al–4V alloy manufactured by laser powder bed fusion. Each method utilized a different image-based technique to create a three-dimensional (3D) RVE from electron backscatter diffraction (EBSD) images. The first method, referred to as the realistic RVE (R-RVE), reconstructed a physical 3D microstructure of the alloy from a series of parallel EBSD images obtained using serial-sectioning (or slicing). The second method captures key information from three orthogonal EBSD images to create a statistically equivalent microstructural RVE (SERVE). Based on the R-RVEs and SERVEs, the CP-FE model was then used to predict the complete tensile stress–strain response of the alloy, including the post-necking damage progression. The accuracy of the predicted stress–strain responses using the R-RVEs and SERVEs was assessed, including the effects of each microstructure descriptor. The results show that the R-RVE and the SERVE offer comparable accuracy for the CP-FE purposes of this study.

Keywords Crystal plasticity · Finite element modelling · Statistically equivalent representative volume element · 3D-EBSD · Titanium · Laser powder bed fusion

Introduction

Integrated computational materials engineering (ICME) is increasingly being used to fast-track materials innovation, qualification and integration. This requires modelling across scales, from the atomic to the continuum, to predict a material's mechanical properties using the composition, processing conditions, and microstructural features as input variables, which can then be used for design and testing purposes. Such capability requires the coupling of micro-mechanics models with representative volume elements (RVEs) that represent the material's microstructure. Azhari et al. [1] recently developed a crystal plasticity-based finite element (CP-FE) model to predict the complete tensile stress−strain response of the laser powder bed fusion (L-PBF) Ti−6Al−4V alloy using an RVE to capture the essential attributes of its microstructure. This material is anisotropic and inhomogeneous, which has a major effect on its plastic deformation and failure. The CP-FE model developed by Azhari et al. [1] is similar to many other CP-FE models that have been developed to predict the mechanical response of titanium alloys from

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Exploration of atom probe tomography at sub-10K

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ABSTRACT

The operating temperature is a critical parameter in atom probe tomography experiments. It affects the spatial precision, mass resolving power and other key aspects of the field-evaporation process. Current commercially available atom probes operate at a minimum temperature of ~25 K when measured at the specimen. In this paper, we explore and implement changes to the mechanical design of both the LEAP® and EIKOS™ atom probe microscope systems manufactured by CAMECA® to enable a specimen temperature in the sub-10 K regime. We use these modified instruments to analyze four materials systems: pure Al (in both pulsed-voltage and pulsed-laser mode), pure W (pulsed-voltage mode only), doped Si, and GaN (pulsed-laser mode only). The effects of conducting atom probe experiments in the sub-10 K regime were assessed with reference to a range of quantitative analysis metrics related to spatial precision, mass resolving power, stoichiometry and charge-state ratio. We demonstrate that the spatial precision is significantly improved with decreasing temperature, whilst the effect on mass resolving power is relatively minor. The enhanced spatial precision is significant insofar as it enables lattice planes from the doped Si samples to be resolved. Furthermore, mass spectral analysis, lower noise floors and changes in the field evaporation process enabled more accurate GaN compositional measurements. We discuss the significance of these findings for the semiconductor and metallurgical industries and the potential opportunities for further investigations of this parameter space.

1. Introduction

In 1955, Kanwar Bahadur and Erwin Müller observed atoms for the first time in real space using the field ion microscope (FIM) [1,2]. This momentous achievement for science and technology in general, and microscopy in particular, was a critical stepping stone in the journey towards the development of the technique now widely known as atom probe tomography (APT) [3,4]. A key enabler for this important achievement was the cryogenic instrumentation that provided these experimentalists with access to liquid nitrogen (~72 K) operating temperature at their FIM samples [1,2]. Although the exact mechanisms affecting the resolution in FIM were not known at the time, it was determined that the operating temperature was indeed a key factor. Later in 1969, Weizer and Forestieri [5] determined that the resolution in FIM continued to improve with decreasing temperature, reporting a maximum at the lowest operating temperature of their experimental setup, at ~5 K. These findings were independently confirmed in 1972 by Adachi et al., who demonstrated a direct correlation between the lower bound operating temperature to both the imaging resolution and the image brightness (4x) in FIM experiments at 4.2 K compared to 25 K [6]. However, as FIM evolved into atom probe tomography (APT), technical difficulties in developing instrumentation capable of operating in these temperature regimes have restricted any significant progress in exploring the effects and potential benefits of a sample temperature in the same low-temperature space (<10 K) in APT. Since ~1970, almost all atom probe instrumentation built, whether it be commercial, or an in-house laboratory set-up, have used closed-cycle He refrigerators with complex cryo-paths between the refrigerator cold-head and the specimen tip, with the result that base sample temperatures no lower than ~20 K are achieved and more usually the base temperature is in the range 25–30 K [3,4,7]. The first-generation LAWATAP is one notable outlier with a base sample temperature of ~10 K, however a systematic investigation on the effect of temperature in this regime on APT has not been reported [8].

APT operates through the ionization of atoms from the apex surface of a needle-shaped specimen of radius ~20–200 nm. This ionization is

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Toward online layer-wise surface morphology measurement in additive manufacturing using a deep learning-based approach

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Abstract

Layer-wise surface morphology information plays a critical role in the quality monitoring and control of additive manufacturing (AM) processes. 3D scan technologies can provide effective means to obtain accurate surface morphological data. However, most of the existing 3D scan technologies are time consuming due to either contact mode or algorithm complexity, which are not capable of obtaining the surface morphology data in an online manner during the printing process. To implement online layer-wise surface morphological data acquisition in AM processes, one practical solution is to model the correlation between 2D images and 3D point cloud data. In practice, since this correlation is usually highly complex due to the high dimensionality and non-linearity, it is usually impractical to find an explicit mathematical transfer function to quantify this correlation effectively. To address this issue, a deep learning-based model is developed in this study, in which a powerful deep learning algorithm, namely, convolutional neural network (CNN), is incorporated. With the trained CNN model, the 3D surface data can be predicted directly without the relatively time consuming triangulation computation by the 3D scanner. Thus, the speed of surface data acquisition and morphology measurement can be improved. To validate the effectiveness and efficiency of the proposed methodology, both simulation and real-world AM case studies were performed. The results show that the prediction accuracy using the proposed method is promising. In terms of averaged relative prediction error, it can be mostly lower than 10% in the experiments. Therefore, the proposed method has a great potential for online layer-wise surface morphology measurement in AM.

Keywords Additive manufacturing · Surface morphology · Online layer-wise measurement · Deep learning

Introduction

In a large variety of advanced manufacturing applications, the surface morphology information plays a very significant role as one of the most critical determinant of functional integrity (Jiang, 1973), particularly, in additive manufacturing (AM, also known as 3D printing) (ACFOAMTSFO Terminology, 2012). Due to the layer-wise fabrication framework of AM, the occurring surface quality issues in each layer, such as the deformation and defects, may significantly deteriorate the products quality (Everton et al., 2016; Agarwala et al., 1996). Thus, accurate monitoring and evaluation of layer-wise surfaces morphology, particularly, the surface roughness (e.g., Ra), which is one key component of surface morphology, are critical to effectively track and control the process quality of AM.

Currently, with the fast-growing of metrology devices, various measurement technologies are capable of capturing the 3D geometric information of an object, such as the coordinate-measuring machine (CMM) (Hocken & Pereira, 2016), 3D laser scan (Benner, 2016), and structured light scan (Rocchini et al., 2001). However, most of these existing measurement technologies are either using contact mode or are time consuming, which are not suitable for online layer-wise surface morphology measurement in AM. Although they are accurate, the time to scan one layer cannot be neglected compared with the layer printing time. For example, in this study, the structured light 3D scan may take about 30 s to scan one

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A PERSPECTIVE OF THE NEEDS AND OPPORTUNITIES FOR COUPLING MATERIALS SCIENCE AND NONDESTRUCTIVE EVALUATION FOR METALS-BASED ADDITIVE MANUFACTURING

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ABSTRACT

This paper presents a perspective of the needs and opportunities associated with the multidisciplinary problem of nondestructive evaluation (NDE) of additive manufacturing (AM). Recognizing the multidisciplinary nature of the problem, as well as the need to bridge knowledge between the different communities, the paper is structured to provide brief backgrounds and details relevant to both communities, as well as present an assessment of the state of the art. This paper, in some respects, is meant to be a primer of the different landscapes, as well as a catalyst for making future connections. At the end, it will be clear that there is much more work to be done, but that the work that is ongoing is exciting, and the potential to exploit NDE techniques for metals-based AM is very high.

KEYWORDS: additive manufacturing, nondestructive evaluation, materials state, measurement techniques, materials physics

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Introduction

Increasingly, there is an awareness that the paradigm-changing nature of additive manufacturing (AM) requires a reassessment of both materials science and nondestructive evaluation (NDE). Traditionally, these technical specialties/disciplines are separated, as their role in the development, manufacture, and use of parts and components in advanced technical systems, such as vehicles, aircraft, defense, and energy systems, is notably different. However, it is also becoming clear that there is a significant opportunity if these traditionally separate subject matter experts can collaborate in the area of AM.

The causes associated with why these technical experts are separated is worth a brief discussion. First, there is the typical role that these experts play in any organization. A materials scientist plays important roles in the development and optimization of new materials, often long before those materials are qualified and become part of the design and manufacturing ecosystem. A materials engineer may then be highly involved in certain aspects of the manufacturing ecosystem, providing subject matter expertise related to process controls and destructive testing to assure specific metrics of quality (for example, mechanical testing or microscopy). The NDE experts often receive a handoff of parts and components, and then apply their skill sets to ensure that the quality of parts is known to an acceptable degree of uncertainty, monitoring parts over their lifetime in service. In certain organizations, the NDE experts can play a role in the design of the parts if philosophies such as design for inspectability are part of the organization's culture. Second, there are the types of data these different subject matter experts typically manage. For the materials scientist or engineer, the spatial domain dominates the characterization techniques, enabling the direct observation of grains, texture, precipitates, and defects. For the NDE expert, the tools invariably rely upon measurements involving time, and are thus in the frequency domain, which can be converted into the spatial domain using various techniques. Lastly, the NDE experts are trained to use statistics (that is, probability of detection) to pursue rare events and are, by their occupation, risk averse. Conversely, the research of many materials scientists is primarily focused on the initial stages of new materials development, where it is not uncommon to imagine in an almost unbridled sense the possibilities of the new materials under study.

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Metal Additive Manufacturing

Preface to the special issue: microstructure design in metal additive manufacturing—physical metallurgy revisited

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Metal additive manufacturing (AM) now offers unmatched freedom to manufacture engineering parts using a wide range of metals and alloys, to design highly complex shapes, and to do this with little to no waste and short lead times. However, many questions remain, e.g., how (local) properties of metal AM parts are achieved and, relatedly, if and how they can be designed? This kind of knowledge is necessary to make AM parts perform better, a prerequisite to unlock them for new types of demanding applications. On the other hand, most physical metallurgy textbook knowledge currently available assumes equilibrium conditions when discussing the microstructural evolution, and this assumption often fails during AM, requiring us to revisit this knowledge in an AM context.

With recent advances in AM technologies as well as in our ability to handle large 3D datasets, one focus of methodology development has been on advancing microstructural analyses to reveal local processing–structure–property relationships. Opportunely, the three of us have strong backgrounds in microstructure characterization, and we see AM as a fantastic opportunity to design microstructures and to achieve better material performances.

The topic of this special issue of Journal of Materials Science was obvious: 'Microstructure design in

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RESEARCH ARTICLE

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Quantification of extreme thermal gradients during in situ transmission electron microscope heating experiments

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Abstract

Studies on materials affected by large thermal gradients and rapid thermal cycling are an area of increasing interest, driving the need for real time observations of microstructural evoultion under transient thermal conditions. However, current in situ transmission electron microscope (TEM) heating stages introduce uniform temperature distributions across the material during heating experiments. Here, a methodology is described to generate thermal gradients across a TEM specimen by modifying a commercially available MEMS-based heating stage. It was found that a specimen placed next to the metallic heater, over a window, cut by FIB milling, does not disrupt the overall thermal stability of the device. Infrared thermal imaging (IRTI) experiments were performed on unmodified and modified heating devices, to measure thermal gradients across the device. The mean temperature measured within the central viewing area of the unmodified device was 3–5% lower than the setpoint temperature. Using IRTI data, at setpoint temperatures ranging from 900 to 1,300°C, thermal gradients at the edge of the modified window were calculated to be in the range of 0.6×10^6 to 7.0×10^6°C/m. Additionally, the Ag nanocube sublimation approach was used, to measure the local temperature across a FIB-cut Si lamella at high spatial resolution inside the TEM, and demonstrate "proof of concept" of the modified MEMS device. The thermal gradient across the Si lamella, measured using the latter approach was found to be 6.3×10^6°C/m, at a setpoint temperature of 1,000°C. Finally, the applicability of this approach and choice of experimental parameters are critically discussed.

KEYWORDS
in situ heating, in situ TEM, nonequilibrium processes, thermal gradient, thermometry

1 | INTRODUCTION

In situ heating holders are widely used to observe thermally activated processes inside a transmission electron microscope (TEM) at high spatial resolution (Butler, 1979; Jungjohann & Carter, 2016). Early in situ TEM heating experiments were performed using furnace type heating stages, which have a large thermal mass (Butler, 1979). Observations of dynamic processes at high spatial resolutions were challenging, as the differences in thermal conductivity and thermal expansion coefficient between the "sample cup" around the furnace heater and the sample created a thermal lag, which increased thermal equilibration times, and thereby, increased sample drift. This was a major limitation while observing the initial stages of a thermally activated process during an in situ TEM heating experiment, as critical information was lost before the sample thermally equilibrated. The new generation of micro-electro-mechanical systems (MEMS)-based heating holders were developed to mitigate these limitations and are, therefore, nowadays widely used for in situ TEM studies (Allard

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TECHNICAL ARTICLE

A Framework for the Optimal Selection of High-Throughput Data Collection Workflows by Autonomous Experimentation Systems

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Abstract
Autonomous experimentation systems have been used to greatly advance the Integrated Computational Materials Engineering paradigm. This paper outlines a framework that enables the design and selection of data collection workflows for autonomous experimentation systems. The framework first searches for data collection workflows that generate high-quality information and then selects the workflow that generates the highest-value information as per a user-defined objective. We employ this framework to select the optimal high-throughput workflow for the characterization of an additively manufactured Ti–6Al–4V sample using a deep-learning based image denoiser. The selected workflow reduced the collection time of backscattered electron scanning electron microscopy images by a factor of 5 times as compared to the case study's benchmark workflow, and by a factor of 85 times as compared to the workflow used in a previously published study.

Keywords ICME · Materials informatics · Autonomous experimentation systems · Decision science · Workflow design/engineering · High-throughput experimentation

Introduction

Autonomous experimentation (AE) (including autonomous simulation) is being explored as a strategy to accelerate materials design and reduce product development cycles [1–5]. Autonomous experimentation is defined by Stach et al. as "...an iterative research loop of planning, experiment, and analysis [that is] carried out autonomously." [1]. Materials AE research is a rapidly advancing field. Powerful applications of AE in materials science include implementing Bayesian optimization principles in AE systems to quickly optimize material properties of interest [6–11], as well as utilizing AE systems to perform high-throughput experimentation (HTE) for rapid materials discovery and optimization for polymers, metals, ceramics, and more [12–19].

We consider AE in a broader context and pose a futuristic scenario where scientific discovery proceeds from a human investigator giving a simple command to an autonomous system, such as identifying the likely root cause of failure for an example component. While this thought experiment borders on science fiction, it is instructive to consider the steps the autonomous system must complete to arrive at a final conclusion. For this autonomous exploration to be carried out, the system must:

1. Parse the verbal instructions into a quantifiable objective that meets the requirements of the user
2. Identify the necessary information required to achieve the objective
3. Design a workflow to collect relevant information. In the materials realm this might include, for example, a sequence of testing, characterization, and simulation steps

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Single crystal elastic constants of additively manufactured components determined by resonant ultrasound spectroscopy

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ABSTRACT

Bayesian inference with Sequential Monte Carlo was used to determine the single crystal elastic constants of additively manufactured (AM) cobalt-nickel-based superalloy specimens from only the resonant frequencies and texture data. This novel framework enables the quantification of the single crystal elastic constants for AM and polycrystalline specimens using only electron backscatter diffraction (EBSD) and Resonant Ultrasound Spectroscopy (RUS), avoiding the expense of bulk single crystal fabrication or synchrotron experiments. A parallelizable and open-source Python package (SMCPy) was used to perform Bayesian inference of the single crystal elastic constants from resonant frequencies of AM specimens. The single crystal elastic constants determined from AM cobalt-nickel-base superalloy specimens were validated with measurements of the single crystal elastic constants on a bulk single crystal specimen. EBSD texture data was used to determine the single crystal elastic constants from the resonant frequencies of AM specimens, and validated with neutron diffraction data by considering the experimental uncertainty in both the EBSD and neutron diffraction data. The robustness of this framework for varied texture orientations relative to the build direction (BD) was demonstrated for AM specimens printed at 0° and 20° BD-inclinations.

1. Introduction

Single crystal elastic constants govern the fundamental mechanical response of single crystalline and polycrystalline materials, and are necessary to inform crystal-scale property calculations. Considering the microstructural variability within additively manufactured (AM) components due to variations in process parameters, component geometry, and build conditions [1,2]; accurate knowledge of the single crystal elastic constants is critical for building inspection protocols and constitutive models.

Traditional measurements of single crystal elastic properties involve the fabrication of a bulk single crystal and subsequent mechanical [3,4] or ultrasonic [5–7] testing. However, single crystal growth is extremely difficult. Given the high thermal gradients and large solidification interface velocities [8,9] involved in AM, designing an alloy to control the columnar to equiaxed transition further increases the difficulty to grow a single crystal. Even for alloys amenable to single crystal fabrication, the production of single crystals requires specialized equipment and is expensive/ time-consuming. Consequently, methods such as time-of-flight neutron diffraction [10–12] and mechanical loading with in-situ high energy x-ray measurements [13–15] have been proposed to evaluate single crystal elastic constants from polycrystalline specimens. However, these approaches involve high cost synchrotron-based methods and access to these facilities.

It is critical to develop methods capable of quantifying the single crystalline elastic properties from polycrystalline components using rapid and cost-effective measurement techniques available in the laboratory. Of particular interest is the determination of single crystal properties from polycrystalline elastic properties [16,17]. The determination of single crystal properties from the polycrystal elastic properties involves an inverse problem, where homogenizations (e.g. Voigt/ Reuss averaging [16]) are iteratively calculated for a known microstructure. The single crystal elastic constants are varied until the calculated polycrystalline elastic constants fit the measured polycrystalline elastic constants. The precision of the calculated single crystal constants can be increased through utilizing self-consistent estimates [15,17], supplementing the elasticity with indentation data [18], and using Bayesian inference/ machine learning techniques [15,19].

Looking toward non-destructive evaluation techniques, recent

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Schmid Factor Crack Propagation and Tracking Crystallographic Texture Markers of Microstructural Condition in Direct Energy Deposition Additive Manufacturing of Ti-6Al-4V

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Abstract

Metallic additive manufacturing (AM) provides a customizable and tailorable manufacturing process for new engineering designs and technologies. The greatest challenge currently facing metallic AM is maintaining control of microstructural evolution during solidification and any solid state phase transformations during the build process. Ti-6Al-4V has been extensively surveyed in this regard, with the potential solid state and solidification microstructures explored at length. This work evaluates the applicability of previously determined crystallographic markers of microstructural condition observed in electron beam melting powder bed fusion (PBF-EB) builds of Ti-6Al-4V in a directed energy deposition (DED) build process. The aim of this effort is to elucidate whether or not these specific crystallographic textures are useful tools for indicating microstructural conditions in AM variants beyond PBF-EB. Parent β-Ti grain size was determined to be directly related to α-Ti textures in the DED build process, and the solid state microstructural condition could be inferred from the intensity of specific α-Ti orientations. Qualitative trends on the as-solidified β-Ti grain size was also determined to be related to the presence of a fiber texture, and proposed as a marker for as-solidified grain size in any cubic metal melted by AM. Analysis of the DED Ti-6Al-4V build also demonstrated a near complete fracture of the build volume, suspected to originate from accumulated thermal stresses in the solid state. Crack propagation was found to *only* appreciably occur in regions of slow cooling with large $\alpha + \beta$ colonies. Schmid factors for the basal and prismatic α-Ti systems explained the observed crack pathway, including slower bifurcation in colonies with lower Schmid factors of both slip systems. Colony morphologies and localized equiaxed β-Ti solidification were also found to originate from build pauses during production and uneven heating of the build edges during deposition. Tailoring of DED Ti-6Al-4V microstructures with the insight gained here is proposed, along with cautionary insight on preventing unplanned build pauses to maintain an informed and controlled thermal environment for microstructural control.

1. Introduction

Metallic additive manufacturing (AM) enables the production of custom geometry parts and rapid design iteration during the overall design process [1–3]. This enables manufacturing without the

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Lifelike metallic structures using origami and compliant mechanisms ☆

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A B S T R A C T

In nature, materials and designs are smart i.e., they achieve the desired shape or size to optimize different processes without needing external devices. However, in the design of mechanical metallic structures, gaining such behavior without using special materials or external sources is still a visionary thought. Here the concept of lifelike metallic structures is introduced. The vision is to produce designs where the metallic walls can behave like human muscles. To create such structures, origami-inspired designs in combination with compliant mechanisms are proposed. For fabricating such designs with desired mechanical properties, it is also proposed to leverage additive manufacturing techniques.

1. Introduction

The evolution of industrial energy systems has been driven by the desire for enhanced energy efficiencies and has resulted in design and manufacturing innovations. This is particularly relevant at the present time where heightened sensitivities to the generation of carbon emissions from energy production exist. Some of the most promising innovations and inspiration for the design of energy systems (e.g., heat exchangers) are observed in natural biological systems. Biological systems such as the lungs and heart can respond to external stimuli without actuators or mechanical stimuli. However, structural materials systems present today do not exhibit the necessary behaviors to enable the innovative advance to biologically inspired systems.

Animate materials are defined as those that are sensitive to their environment and can adapt to fulfill their intended functions (Fig. 1a)[1]. A subset of these materials are smart materials, materials that sense and actuate in response to external stimuli with properties that are spatially and temporally correlated[2]. Network analyses of the scientific literature indicate that smart materials rely on (Fig. 1b) two approaches: (1) External sensors and actuators to bring about a shape change or (2) Special materials (e.g., shape memory alloys that rely on reversible crystallographic phase transitions). Interestingly, this analysis also reveals limited research on material systems that can react to a given stimulus without relying on special materials, sensors, actuators, automation, multiple systems with artificial intelligence at temperatures relevant to harsh environments[3].

The foundational questions are as follows: (i) Can smart metallic structures be designed to animate with reference to external stimuli i.e., changing boundary conditions such as temperature, pressure, or other stimuli without inputs from external sensors or actuators? (ii) Can a metallic structure be designed to perform like a biological muscle? The proposed solution is based on biomechanisms available in nature that can be realized via a combination of *origami inspired* designs and *compliant mechanisms*. These structures are referred to as lifelike metallic structures. Origami-inspired designs had been successfully implemented in material families such as polymers and nanomaterials[4–6]. The adoption of this technique to structural metals (e.g., stainless steel) poses challenges due to the mechanical limitations of structural metals. However, with the matriculation of additive manufacturing (AM) technologies, design methodologies, and structural materials this can become a reality as discussed below:

2. Lifelike metallic structures: Inspiration & vision

The design of the human lung makes for one of the most efficient heat exchangers with a significant surface area to volume

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In situ melt pool measurements for laser powder bed fusion using multi sensing and correlation analysis

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Laser powder bed fusion is a promising technology for local deposition and microstructure control, but it suffers from defects such as delamination and porosity due to the lack of understanding of melt pool dynamics. To study the fundamental behavior of the melt pool, both geometric and thermal sensing with high spatial and temporal resolutions are necessary. This work applies and integrates three advanced sensing technologies: synchrotron X-ray imaging, high-speed IR camera, and high-spatial-resolution IR camera to characterize the evolution of the melt pool shape, keyhole, vapor plume, and thermal evolution in Ti–6Al–4V and 410 stainless steel spot melt cases. Aside from presenting the sensing capability, this paper develops an effective algorithm for high-speed X-ray imaging data to identify melt pool geometries accurately. Preprocessing methods are also implemented for the IR data to estimate the emissivity value and extrapolate the saturated pixels. Quantifications on boundary velocities, melt pool dimensions, thermal gradients, and cooling rates are performed, enabling future comprehensive melt pool dynamics and microstructure analysis. The study discovers a strong correlation between the thermal and X-ray data, demonstrating the feasibility of using relatively cheap IR cameras to predict features that currently can only be captured using costly synchrotron X-ray imaging. Such correlation can be used for future thermal-based melt pool control and model validation.

Laser powder bed fusion (L-PBF) is a popular metal additive manufacturing (AM) method (also known as metal 3D printing)[1–4]. It uses a laser beam to form the part by repeatedly melting thin powders onto the substrate surface. Due to its layer-wise and location-specific deposition manner, L-PBF can create complex geometries that traditional manufacturing methods cannot. Furthermore, in L-PBF, site-specific material property control can be achieved by assigning site-specific printing parameters[5]. Given the above advantages, L-PBF has already been used in many industries such as medical, aerospace, and defense[6,7].

However, L-PBF still suffers from large residual stress, deformation, delamination, and porosity[8–10]. The lack of understanding of the melting and solidification process under non-equilibrium conditions is the main barrier to achieving a high-quality deposition[11,12]. The melt pool and keyhole evolution must be well studied to enable local microstructure control and minimize defect formation[13]. More specifically, measurements of the thermal gradient and the solid–liquid boundary velocity are necessary.

The melt pool is the liquid-phase material bounded by the mobile solid–liquid boundary. The material properties of a 3D printed part are determined by the microstructure resulting from the thermal gradient and liquid to solid boundary velocity during the solidification process[14]. The keyhole is the vapor-depression zone in the center of the melt pool caused by overheating, vaporization, and the resulting vapor recoil pressure. The keyhole severity is critically related to the trapped gas porosity level during the melting and solidification process[15,16]. Note that lack of fusion and hot cracking can introduce additional porosity, but these issues are at a much larger scale that is typically considered unacceptable; therefore, they are not covered in this paper.

This work focuses primarily on single spot melting and solidification. While this method has limitations in applicability to traditional line scanning, it does offer a similar melt pool condition to random spot melting.

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In Situ X-ray Radiography and Computational Modeling to Predict Grain Morphology in β-Titanium during Simulated Additive Manufacturing

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Abstract: The continued development of metal additive manufacturing (AM) has expanded the engineering metallic alloys for which these processes may be applied, including beta-titanium alloys with desirable strength-to-density ratios. To understand the response of beta-titanium alloys to AM processing, solidification and microstructure evolution needs to be investigated. In particular, thermal gradients (Gs) and solidification velocities (Vs) experienced during AM are needed to link processing to microstructure development, including the columnar-to-equiaxed transition (CET). In this work, in situ synchrotron X-ray radiography of the beta-titanium alloy Ti-10V-2Fe-3Al (wt.%) (Ti-1023) during simulated laser-powder bed fusion (L-PBF) was performed at the Advanced Photon Source at Argonne National Laboratory, allowing for direct determination of Vs. Two different computational modeling tools, SYSWELD and FLOW-3D, were utilized to investigate the solidification conditions of spot and raster melt scenarios. The predicted Vs obtained from both pieces of computational software exhibited good agreement with those obtained from in situ synchrotron X-ray radiography measurements. The model that accounted for fluid flow also showed the ability to predict trends unobservable in the in situ synchrotron X-ray radiography, but are known to occur during rapid solidification. A CET model for Ti-1023 was also developed using the Kurz–Giovanola–Trivedi model, which allowed modeled Gs and Vs to be compared in the context of predicted grain morphologies. Both pieces of software were in agreement for morphology predictions of spot-melts, but drastically differed for raster predictions. The discrepancy is attributable to the difference in accounting for fluid flow, resulting in magnitude-different values of Gs for similar Vs.

Keywords: in situ radiography; additive manufacturing; solidification modeling; beta-titanium; CET modeling

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1. Introduction

The continued development of metal additive manufacturing (AM) over the past couple of decades has expanded the applications and material classes in which these processes can be used. Titanium (Ti) alloys have been at the center of this development, due to their superior properties, particularly for aerospace and defense applications. Although Ti-6Al-4V (Ti-64) has typically dominated in terms of use and research pertaining to metal AM processes, metastable β-Ti alloys have begun to find increased use over Ti-64 (an $\alpha + \beta$ alloy), due to their increased strength-to-density ratios, among other properties [1]. These

Smooth Robust Tensor Completion for Background/Foreground Separation with Missing Pixels: Novel Algorithm with Convergence Guarantee

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Abstract

Robust PCA (RPCA) and its tensor extension, namely, Robust Tensor PCA (RTPCA), provide an effective framework for background/foreground separation by decomposing the data into low-rank and sparse components, which contain the background and the foreground (moving objects), respectively. However, in real-world applications, the presence of missing pixels is a very common and challenging issue due to errors in the acquisition process or manufacturer defects. RPCA and RTPCA are not able to recover the background and foreground simultaneously with missing pixels. This study aims to address the problem of background/foreground separation with missing pixels by combining video recovery and background/foreground separation into a single framework. To achieve this goal, a smooth robust tensor completion (SRTC) model is proposed to recover the data and decompose it into the static background and smooth foreground, respectively. An efficient algorithm based on tensor proximal alternating minimization (tenPAM) is implemented to solve the proposed model with a global convergence guarantee under very mild conditions. Extensive experiments on actual data demonstrate that the proposed method significantly outperforms the state-of-the-art approaches for background/foreground separation with missing pixels.

Keywords: Robust Tensor Completion (RTC), Spatio-temporal Continuity, Low-rank Property, Tensor Proximal Alternating Minimization (tenPAM), Global Convergence

Nomenclature

H, W, T	The height, width, and number of image frames
(r_1, r_2, r_3)	The multi-linear rank in Tucker Decomposition
λ	The balance coefficient in the proposed objective function
Ω	The index set of the observed elements
$\boldsymbol{\mathcal{X}}$	The order-3 tensor in $\mathbb{R}^{H\times W\times T}$ represented by $\{\mathbf{X}_1, \cdots, \mathbf{X}_T\}$
$\mathbf{X}_t$	t-th image frame in $\mathbb{R}^{H\times W}$
$\boldsymbol{\mathcal{L}}$	The low-rank tensor (static video background)
$\boldsymbol{\mathcal{S}}$	The smooth tensor (smooth moving objects)
$\boldsymbol{\mathcal{X}} \times_n \mathbf{V}$	The mode-n multiplication of a tensor $\boldsymbol{\mathcal{X}}$ with a matrix $\mathbf{V}$

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Super Resolution for Multi-Sources Image Stream Data Using Smooth and Sparse Tensor Completion and Its Applications in Data Acquisition of Additive Manufacturing

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ABSTRACT
Recent developments of advanced imaging systems spur their applications in many areas, ranging from satellite remote sensing for geographic information to thermal imaging analysis for manufacturing process monitoring and control. Due to different specifications of imaging systems, the resulting image stream data (videos) have different spatial and temporal resolutions. This proposed work is based on the image stream data captured by multiple imaging systems for the same object with different but complementary spatial and temporal resolutions. For example, one system has high spatial but low temporal resolutions while the other one has opposite resolutions. The goal of this article is to develop a new super resolution method that integrates these different types of image stream data to improve both spatial and temporal resolutions, which is critical to obtaining more insightful information for more effective quality control of targeted processes or systems. To fulfill this goal, a new tensor completion model is developed by considering both smooth and sparse features simultaneously and is thus termed smooth and sparse tensor completion (SSTC). The results of the extensive case studies illustrate the superiority of our method over the elaborately selected benchmark methods.

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KEYWORDS
CANDECOMP/PARAFAC (CP) decomposition; Rank estimation; Smooth and sparse decomposition

1. Introduction

Advanced imaging systems have been widely applied to many areas, such as images for manufacturing processes monitoring and control (Liu et al. 2019; Shen, Xie, and Kong 2020), spectral images in remote sensing applications (Shaw and Burke 2003). The spatial resolution and acquisition rate (temporal resolution) are the keys to the performance of imaging systems for capturing the evolution insight of the targeted object. Different imaging systems generate image stream data with different spatial and temporal resolutions, and thus have complementary performances. For example, in thermal imaging analysis of a metal additive manufacturing (AM) process (Haines et al. 2018), a small thermal camera can be mounted inside of the 3D printer chamber to obtain high spatial resolution images (the distance between camera and the region of interest could be very short, resulting a small field of view), but the resulting images will have low temporal resolution (the small size limits its capability of high acquisition rate of images). On the contrary, an advanced thermal camera with high temporal resolution is typically very bulky and has to be mounted outside of the 3D printer chamber, thus the resulting spatial resolution is low (the distance between the camera and the region of interest is long and thus results in a large field of view). It is vital to obtain image stream data with both high spatial and high temporal resolutions so that subtler information, which is perhaps hidden from us if using just one of the above imaging systems, can be captured and analyzed.

This challenge motivates the research presented in this article, namely to develop a method that fuse images streams with complementary spatial and temporal resolutions to obtain high spatial and high temporal image stream. By applying our proposed method, we can achieve a cheap sensing system to acquire expensive sensor data. In general, either high spatial and low temporal, or low spatial and high temporal sensors are easy to obtain, while high spatial and high temporal sensors are quite expensive or even do not exist for some stringent specifications. For example, a thermal gradient is a critical physical quantity that describes the most rapid changing rate of the temperature around a particular location in heating/cooling processes (Raplee et al. 2017), which has spatial temporal structure. According to the literature (Al-Bermani et al. 2010), the microstructure can be predicted by the thermal gradient. Thermal gradient information can be obtained by thermography. However, there is no such equipment to obtain thermal gradient because of the unique property of the melt pool (Cheng et al. 2018) in the selective laser melting (SLM) process. The melt pool is small (100–200 μm) and the dwell time is short (0.5–1.5 ms). The temporal resolution (frame rate) of the camera should at least be 20k Hz because the solidification can be even shorter than the dwell time. The spatial resolution needs to be around 3 μm per pixel and this can give a detailed thermal gradient of the melt pool, for example, a keyhole in melt pool in SLM is normally around 10 μm in diameter (Cunningham et al. 2019), and 3 μm spatial resolution can give at least 3 point measurement along one direction for the

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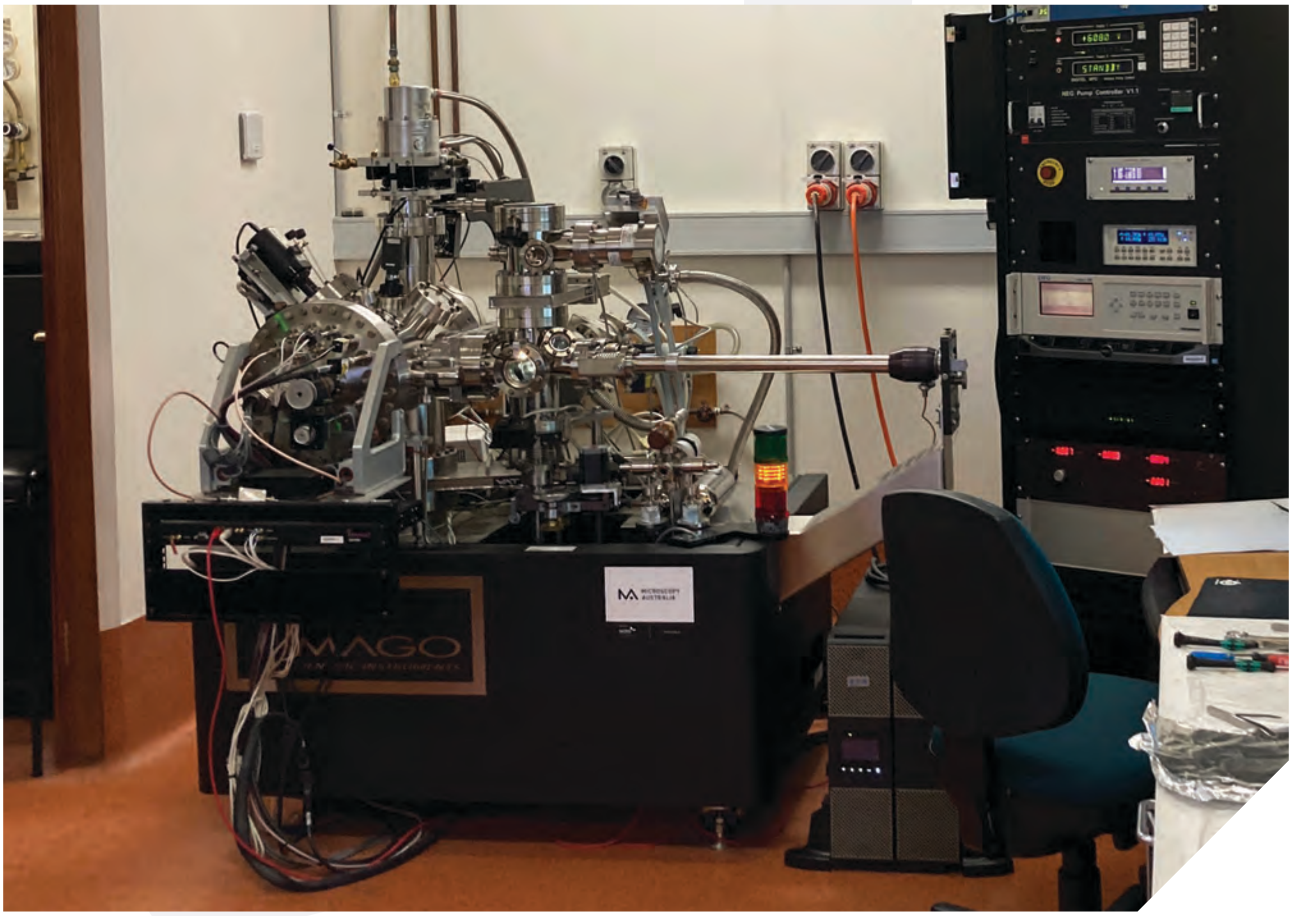

9. 3D Additive MURI-AUSMURI Publications

Phase 1 of the MURI-AUSMURI project has resulted in 56 published articles during the period starting from 2019 to April 2022.

MURI-AUSMURI 3D Additive Project Publications (as at April 2022)

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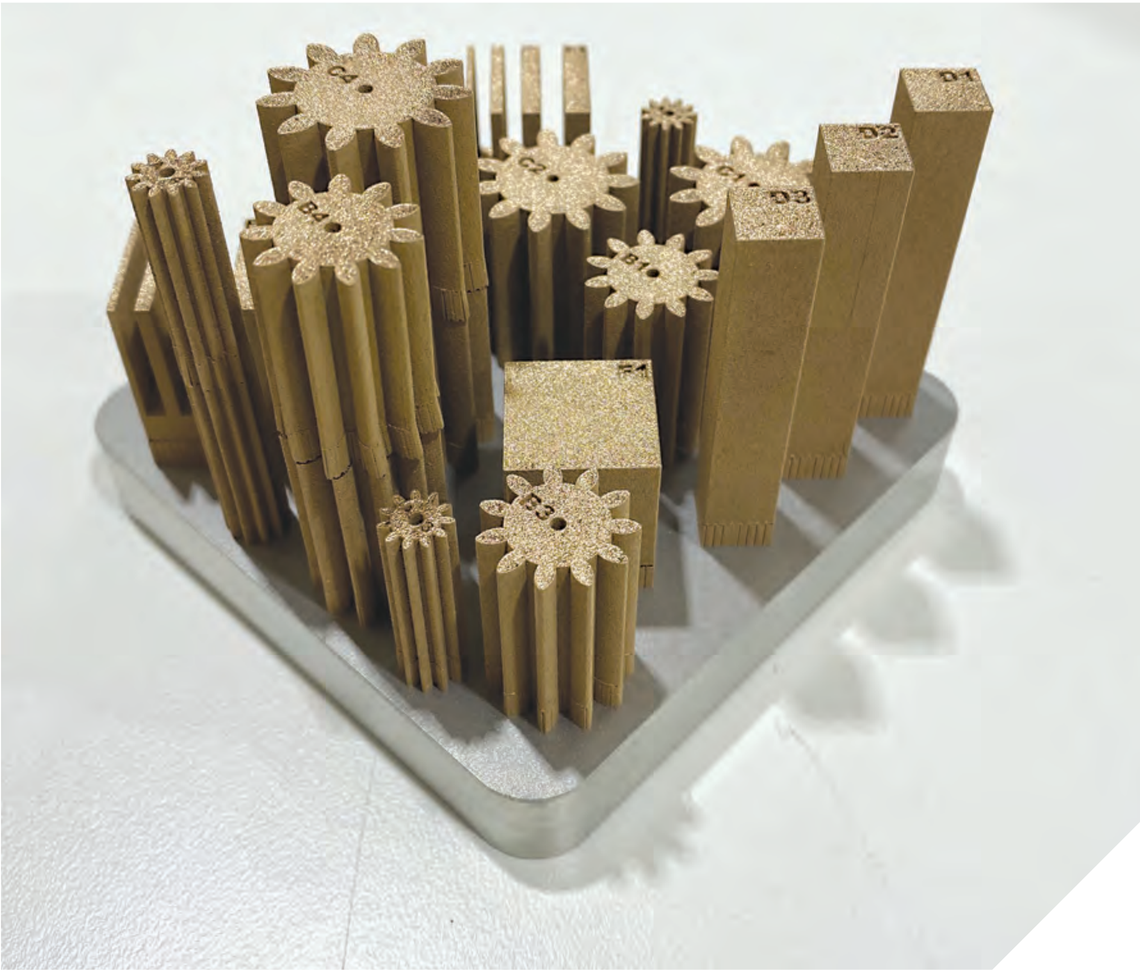
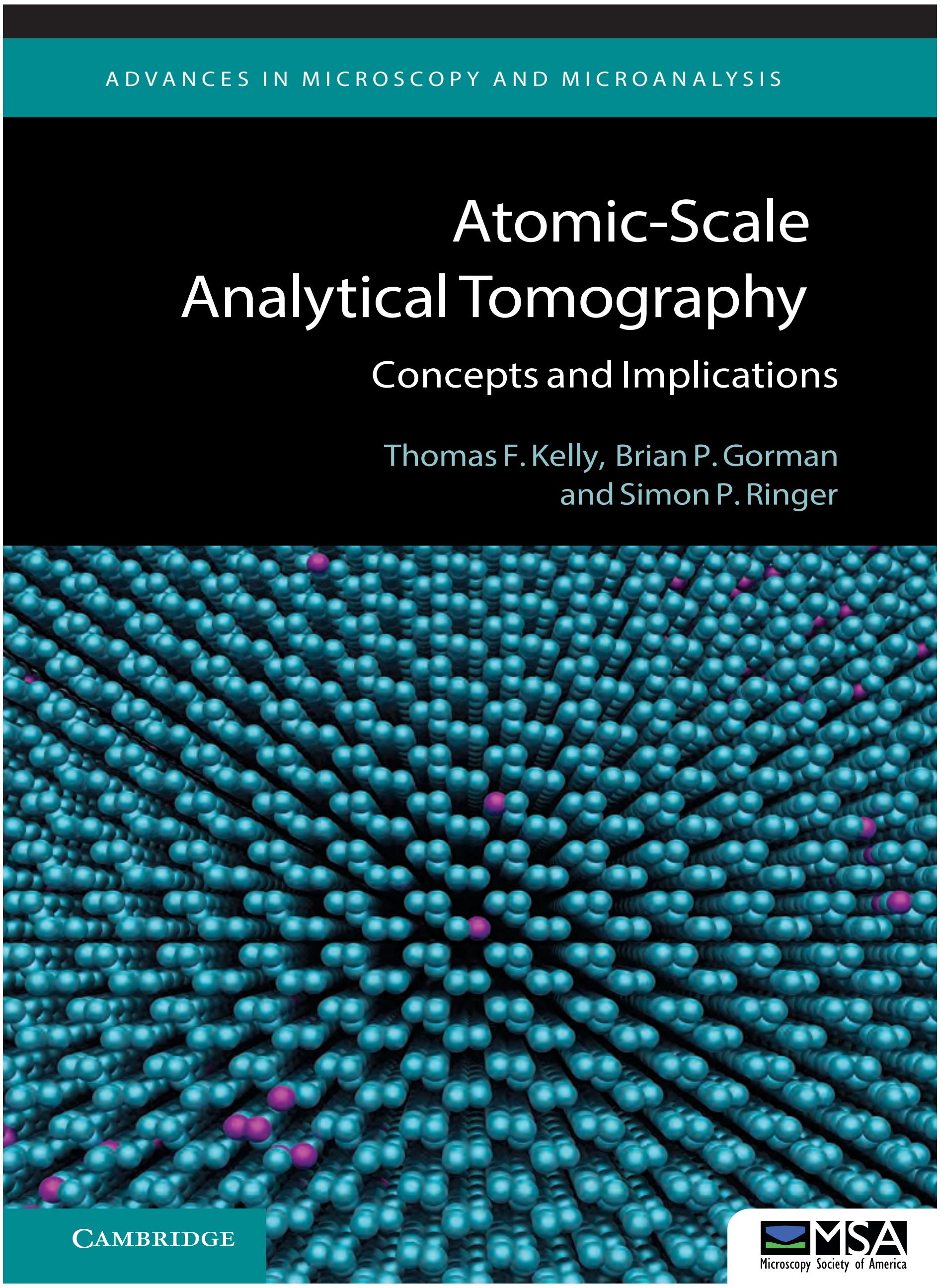
Phase 1 Publications (2018 - 2022)





Thomas F. Kelly, Steam Instruments, Inc., **Brian P. Gorman,** Colorado School of Mines, **Simon P. Ringer,** University of Sydney

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Book Description

A comprehensive guide on Atomic-Scale Analytical Tomography (ASAT) that discusses basic concepts and implications of the technique in areas such as material sciences, microscopy, engineering sciences and several interdisciplinary avenues. The title interrogates how to successfully achieve ASAT at the intersection of transmission electron microscopy and atom probe microscopy. This novel concept is capable of identifying individual atoms in large volumes as well as in 3D, with high spatial resolution. Written by leading experts from academia and industry, this book serves as a guide with real-world applications on cutting-edge research problems. An essential reading for researchers, engineers and practitioners interested in nanoscale characterisation, this book introduces the reader to a new direction for atomic-scale microscopy.

Review

'The quest for making better materials is ultimately linked to understanding the role of each individual atom in its place and its chemical environment. Three pioneers in the field guide us through a comprehensive scenario of how this can be achieved by combining the two most advanced atomic-scale characterization techniques: atom probe tomography and transmission electron microscopy.'

Joachrim Mayer - RWTH Aachen University

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Research Article

Correlation between precipitates evolution and mechanical properties of Al-Sc-Zr alloy with Er additions

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ABSTRACT

Correlation between precipitates evolution and mechanical properties of Al-Sc-Zr alloy with Er additions during isothermal ageing were investigated by microhardness measurements, transmission electron microscopy, atom probe tomography and density functional theory-based simulations. The results demonstrate that the Er additions significantly improve the hardness during elevated temperature ageing, especially at 400 °C. This is mainly because Er additions increase the nucleation rate of the Al_3(Er,Sc,Zr) precipitates, resulting in a higher density of fine and uniform dispersion of $L1_2$ structured nanoparticles. First-principles calculations demonstrate that the second nearest neighboring solute-solute interactions for the species Sc, Zr and Er are energetically favored – a key feature to rationalize the observed precipitate structure and the underlying formation mechanism. The sequential formation of the core/shell precipitates in the Er-free alloy and core/double-shell precipitates in the Er-containing alloy arises due to the different solute-solute and solute-vacancy interaction energies, and the relative diffusivities of the Er, Sc and Zr species in Al. These results shed light on the beneficial effects of Er additions on the age-hardening behavior of Al-Sc-Zr alloy and provide guidance for designing the ageing treatments for the Al-Sc-Zr(-Er) alloys.

1. Introduction

There is a strong demand for the manufacture of low-density, heat-resistant machine components in automotive applications such as brake discs and engines parts, and this is driving the design of new, lightweight Al alloys that exhibit high thermal stability and strength. This presents a great scientific and technological challenge because precipitation strengthening is the most common strengthening mechanism in traditional Al alloys and yet most of the second-phase precipitates that occur in these Al alloys are metastable, undergoing coarsening or even dissolution at temperatures $\geq$ 200 °C [1–4]. The precipitate coarsening results in a dramatic drop in the strength and structural integrity, limiting the thermal stability and creep resistance of traditional heat treatable Al alloys to $\leq$ 180 °C. Therefore, there is great interest in the development of new Al alloys with both high thermal stability and high strength.

Micro-alloying with rare earth and transition metal elements is an effective approach to improve the performances of Al alloys [3,5,6]. In particular, the additions of Sc and Zr in the Al alloy have received much recent attention due to the significant strengthening effects and high thermal stability of the Al_3Sc and Al_3Zr precipitate phases [3,7–11]. For example, it has been reported that the addition of 0.23 wt.% Sc and 0.11 wt.% Zr to Al alloys can increase the strength by ~150 MPa [8]. Combined additions of Sc and Zr in Al alloys can form core/shell Al_3(Sc,Zr) precipitates in which Sc segregates to the precipitate core and Zr segregates to the outer shell [11–13]. These nanostructured core/shell Al_3(Sc,Zr) precipitates exhibit high coarsening resistance due to the low diffusion rate of Zr in α-Al, thus contributing positively to the thermal stability of Al alloys [14–16]. It was reported that the strength of an Al-0.06Sc-0.06Zr (at.%) alloy did not decrease even when the sample was exposed to temperatures as high as 400 °C due to the very low coarsening rate of these core/shell Al_3(Sc,Zr) precipitates [17]. Although such Al-Sc-Zr alloys demonstrate potential as high

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Research Article

On the microstructure and texture evolution in 17-4 PH stainless steel during laser powder bed fusion: Towards textural design

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ABSTRACT

Additive Manufacturing (AM) of metals allows the production of parts with complex designs, offering advanced properties if the evolution of the texture can be controlled. 17-4 precipitation hardening (PH) stainless steel is a high strength, high corrosion resistance alloy used in a range of industries suitable for AM, such as aerospace and marine. Despite 17-4 PH being one of the most common steels for AM, there are still gaps in the understanding of its AM processing–structure relationships. These include the nature of the matrix phase, as well as the development of texture through AM builds under different processing conditions. We have investigated how changing the laser power and scanning strategy affects the microstructure of 17-4 PH during laser powder bed fusion. It is revealed that the matrix phase is δ-ferrite with a limited austenite presence, mainly in regions of the microstructure immediately below melt pools. Austenite fraction is independent of the printing pattern and laser power. However, reducing the time between adjacent laser passes during printing results in an increase in the austenite volume fraction. Another effect of the higher laser power, as well as additional remelting within the printing strategy, is an increase in the average grain size by epitaxial ferrite grain growth across multiple build layers and the development of a mosaic type microstructure. Changes to the scanning strategy have significant impacts on the textures observed along the build direction, while ⟨100⟩ texture along the scanning direction is observed consistently. Mechanisms for texture formation and the mosaic structure are proposed that presents a pathway to the design of texture via AM process control.

1. Introduction

The field of metal additive manufacturing (AM) has gained much interest in recent years. Its advantages over traditional processing routes, such as casting and forming, include a low lead time for prototype generation as well as a greater degree of design freedom, with many previously unachievable part geometries now possible through AM [1]. The microstructures produced within AM builds govern their properties and thus a detailed understanding of the processing microstructure relationship is of great interest. Due to the high magnitude of the thermal gradients (G) and solidification rates (R) during AM as well as the incremental nature of solidification, AM microstructures have distinct differences from those produced by conventional methods. In each layer, solidification occurs within one melt track at a time, constraining the microstructure to form around melt pool boundaries (MPBs). Solidification may be either columnar or equiaxed, depending on the process parameters and the resulting ratio between G and R [2]. The overall high magnitude of G and R can result in the formation of finer microstructures than those typically produced by conventional methods [3,4]. The thermal history experienced by the material during the AM process can also result in the formation of unexpected phases, for example through microsegregation [5,6] or rapid heating and cooling rates bypassing the formation of certain phases [7]. The layer-by-layer melting or deposition process in AM technologies can lead to epitaxial grain growth [8]. This is the process by which a new solid grows into the liquid nucleating from existing solid, i.e. previously solidified layers and tracks in the case of AM [8,9].

It has been found that the solidification mode can be changed between columnar and equiaxed, with suitable control of the pro-

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Measuring oxygen solubility in Ni grains and boundaries after oxidation using atom probe tomography [☆]

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ABSTRACT

Poor oxidation resistance is a key contributor to material failure within extreme environments. Understanding oxygen solubility is important for computation aided design of new high strength, high-temperature oxidation resistant alloys. Oxygen solubility within pure metals, such as Ni, has been studied using a multitude of techniques, but Atom Probe Tomography (APT) has not been used for such a measurement to date. APT is the only technique offering both a high chemical sensitivity (<10 ppm) and resolution (<1 nm) allowing for a composition measurement within nms of the oxide/metal interface. APT was employed to measure the oxygen content at different depths from the oxide/metal interface as well as grain boundaries for a high and low purity Ni sample oxidized at 1000 °C for 48 h. The results reveal <10 s of ppm oxygen solubility within Ni metal at all depths and 100 s of ppm oxygen within GBs.

The demand for efficiency enhancements and emission reductions in the power generation and transportation industries is driving operating temperatures higher than before [1,2]. Enabling multiple energy sources requires new high temperature materials that can withstand increasingly harsher environments [3]. Consequently, high temperature corrosion is amongst one of the life-limiting degradation mechanisms that must be addressed during alloy development.

Recent progress in computation hardware and software has enabled computation aided alloy design to be an efficient and effective method for designing new alloys [4–9]. However, modeling high-temperature corrosion is extremely challenging due to the local nature of the processes that can be influenced by a combination of factors. Although there has been significant progress in predicting oxidation induced degradation [6,10,11], there is no comprehensive model to predict multicomponent alloy high temperature oxidation behavior. Most high temperature alloys rely on surface chromia or alumina scales for oxidation protection [12]. Formation of the desired scale depends on the oxide thermodynamic stability, oxygen solubility, diffusivity of oxygen and element(s) forming the oxide scale, temperature, oxygen partial pressure, and oxide particle nucleation density [13,14]. Quantitative information on each of these parameters is essential to oxidation resistant alloy development. Yet, the experimental determination of dissolved oxygen has received limited attention because of challenges with detecting light elements in low concentration with high spatial resolution [15–18].

There are several studies that measured oxygen solubility in pure Ni metal, including those by Seybolt and Fullman [15], Alcock and Brown [19], and Park and Altstetter [16]. These three pivotal studies showed 100 s of ppm oxygen solubilities for temperatures ranging from 700–1200 °C, agreeing well with expected theoretical solubilities in Ni (Sievert's equation) [20]. The Ni was polycrystalline in these studies making the delineation of GB effects impossible.

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Multi-task Gaussian process upper confidence bound for hyperparameter tuning and its application for simulation studies of additive manufacturing

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ABSTRACT

In many scientific and engineering applications, Bayesian Optimization (BO) is a powerful tool for hyperparameter tuning of a machine learning model, materials design and discovery, etc. Multi-task BO is a general method to efficiently optimize multiple different, but correlated, "black-box" functions. The objective of this work is to develop an algorithm for multi-task BO with automatic task selection so that only one task evaluation is needed per query round. Specifically, a new algorithm, namely, Multi-Task Gaussian Process Upper Confidence Bound (MT-GPUCB), is proposed to achieve this objective. The MT-GPUCB is a two-step algorithm, where the first step chooses which query point to evaluate, and the second step automatically selects the most informative task to evaluate. Under the bandit setting, a theoretical analysis is provided to show that our proposed MT-GPUCB is *no-regret* under some mild conditions. Our proposed algorithm is verified experimentally on a range of synthetic functions. In addition, our algorithm is applied to Additive Manufacturing simulation software, namely, Flow-3D Weld, to determine material property values, ensuring the quality of simulation output. The results clearly show the advantages of our query strategy for both design point and task.

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Multi-task Gaussian process upper confidence bound; automatic task selection; *no-regret*; hyperparameter tuning

1. Introduction

In machine learning, an appropriate setting of the hyperparameters in the algorithms (for example, regularization weights, learning rates, etc.) is crucial to achieve a satisfactory performance. A poor setting of the hyperparameters may result in a useless model, even when the model structure is correct. In materials design and discovery, how to choose the chemical structure, composition, or processing conditions of a material to meet design criteria is a key problem. There are many other examples of design problems in advertising, healthcare informatics, manufacturing, and so on. Any significant advances in automated design can result in immediate product improvements and innovation in a wide area of domains.

Bayesian Optimization (BO) (see, Jones *et al.* (1998); Shahriari *et al.* (2015)) has emerged as a powerful tool for these various design problems. Fundamentally, it is a general method to efficiently optimize "black-box" functions. There is only weak prior knowledge available, typically characterized by expensive and noisy function evaluations, a lack of gradient information, and high levels of non-convexity. BO is impacting a wide range of areas, including combinatorial optimization (see Williams *et al.* (2000)); Hutter *et al.* (2011), automatic machine learning (see Bergstra *et al.* (2011); Snoek *et al.* (2012), material design (Frazier and Wang, 2016), and reinforcement learning (Brochu *et al.*, 2010).

BO is a sequential model-based approach to solving the "black-box" optimization problem. For a given task, the method iterates over the following steps until the available computational budget is exhausted:

1. A a set of evaluated points is used to learn a probabilistic regression model $p(f)$ of the objective function f (typically in the form of a Gaussian Process (GP) (Williams and Rasmussen, 2006)).
2. $p(f)$ is used to induce a proper acquisition function that leverages the uncertainty in the posterior to trade-off exploration and exploitation.
3. The acquisition function is optimized to determine the next query point to be evaluated.
4. The regression data set in task 1 is augmented with the newly evaluated point.

Different from single-task BO introduced above, Multi-Task Bayesian Optimization (MTBO) (Swersky *et al.*, 2013) is a general method to efficiently optimize multiple different but correlated "black-box" functions. The settings for multi-task Bayesian optimization widely exist in many real-world applications. For example, the K-fold cross-validation (Bengio and Grandvalet, 2004) is a widely used technique to estimate the generalization error of a machine learning model for a given set of hyperparameters. However, it needs to retrain a model K times using all K training-validation

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PUBLICATIONS PHASE 1

Robust Tensor Decomposition Based Background/Foreground Separation in Noisy Videos and Its Applications in Additive Manufacturing

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***Abstract*—Background/foreground separation is one of the most fundamental tasks in computer vision, especially for video data. Robust PCA (RPCA) and its tensor extension, namely, Robust Tensor PCA (RTPCA), provide an effective framework for background/foreground separation by decomposing the data into low-rank and sparse components, which contain the background and the foreground (moving objects), respectively. However, in real-world applications, the video data is contaminated with noise. For example, in metal additive manufacturing (AM), the processed X-ray video to study melt pool dynamics is very noisy. RPCA and RTPCA are not able to separate the background, foreground, and noise simultaneously. As a result, the noise will contaminate the background or the foreground or both. There is a need to remove the noise from the background and foreground. To achieve the three components decomposition, a smooth sparse Robust Tensor Decomposition (SS-RTD) model is proposed to decompose the data into static background, smooth foreground, and noise, respectively. Specifically, the static background is modeled by the low-rank tucker decomposition, the smooth foreground (moving objects) is modeled by the spatio-temporal continuity, which is enforced by the total variation regularization, and the noise is modeled by the sparsity, which is enforced by the ℓ_1 norm. An efficient algorithm based on alternating direction method of multipliers (ADMM) is implemented to solve the proposed model. Extensive experiments on both simulated and real data demonstrate that the proposed method significantly outperforms the state-of-the-art approaches for background/foreground separation in noisy cases.**

***Note to Practitioners*—This work is motivated by melt pool detection in metal additive manufacturing where the processed X-ray video from the monitoring system is very noisy. The objective is to recover the background with porosity defects and the foreground with melt pool in the presence of noise. Existing methods fail to separate the noise from the background and foreground since RPCA and RTPCA have only two components, which cannot explain the three components in the data. This paper puts forward a smooth sparse Robust Tensor Decomposition by decomposing the tensor data into low-rank, smooth, and sparse components, respectively. It is a highly effective method for background/foreground separation in noisy case. In the case studies on simulated video and X-ray data, the proposed method can handle non-additive noise, and even the case of high noise-ratio. In the proposed algorithm, there is only one tuning parameter λ. Based on the case studies, our method achieves satisfying performance by taking any $\lambda \in [0.2, 1]$ with anisotropic total variation regularization. With this observation, practitioners can apply the proposed method without extensive parameter tuning work. Furthermore, the proposed method is also applicable to other popular industrial applications. Practitioners can use the proposed SS-RTD for degradation processes monitoring, where the degradation image contains the static background, anomaly, and random disturbance, respectively.**

***Index Terms*—Robust tensor decomposition (RTD), smooth sparse decomposition, spatio-temporal continuity, total variation regularization, low-rankness.**

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Nomenclature

H, W, T	The height, width, and number of an image frame
(r_1, r_2, r_3)	The multi-linear rank in Tucker Decomposition
λ	The balance coefficient in the proposed objective function
$\mathcal{X}$	The order three tensor in $\mathbb{R}^{H\times W\times T}$ represented by $\{\mathbf{X}_1, \cdots, \mathbf{X}_T\}$
$\mathbf{X}_t$	t-th image frame in $\mathbb{R}^{H\times W}$
$\mathcal{L}$	The low-rank tensor (static video background)
$\mathcal{S}$	The smooth tensor (smooth moving objects)
$\mathcal{E}$	The noise tensor (all kinds of noise)
$\mathcal{X} \times_n \mathbf{U}$	The mode-n multiplication of a tensor $\mathcal{X}$ with a matrix $\mathbf{U}$
$\mathcal{G}$	The core tensor in tucker decompsition
$\mathbf{U}_1, \mathbf{U}_2, \mathbf{U}_3$	The factor matrices in tucker decompsition
$\boldsymbol{f}$	The auxiliary variable
$\mathbf{D}_h, \mathbf{D}_v, \mathbf{D}_t$	Three vectorizations of the difference operation along the hori-zontal, vertical, and temporal directions
$\mathbf{D}$	The the concatenated difference operation, i.e, $[\mathbf{D}_h^\top, \mathbf{D}_v^\top, \mathbf{D}_t^\top]^\top$
$\|\cdot\|_F$	The Frobenius norm

Electron Microscopy Methods

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Introduction

In this article, we aim to give an overview of electron microscopy methods that are currently used or regarded as suitable for characterizing the complex microstructures of alloys during additive manufacturing (AM). This disruptive technology is also known as 3D printing, because of its ability to manufacture complex shaped engineering parts in a layer by layer manner. This unlocks unprecedented design freedom and little waste, by using feedstock materials such as powders, wires or sheets, and high energy beams such as lasers or electron beams. The advantages of AM become most apparent when considering applications requiring low numbers of complex-shaped, custom-built parts, for example biomedical implants, or replacement or repair of high-value parts in aerospace applications, such as nozzles in jet-engines (Bajaj *et al.*, 2020; Raplee *et al.*, 2017). However, despite its many advantages, the applications of AM techniques for manufacturing metal parts are still far from being widespread in industry. This is because many initial attempts were carried out following trial and error type approaches, neglecting detailed studies of the multiscale microstructural evolution beyond observations by light optical microscopy, and their links to the properties of the final AM builds.

Thus, in order to achieve more widespread applications of AM in additional industrial sectors, 3D printing of parts needs to be enabled with similar or even better properties than achieved via conventional processing routes such as casting, forging and machining. To achieve this goal, the underlying physical metallurgy phenomena need to be understood in more detail. Although there are certain similarities to conventional casting, powder metallurgy, welding, and thermo-mechanical processing, it is now established that steady-state conditions, as are assumed during conventional processing of alloys, for example during iso-thermal heat treatments, will fail in AM. This is due to the presence of steep thermal gradients and large thermal gyrations brought about by the sudden and frequent changes in energy delivery modes during 3D printing. These parameters depend on the type of beam scanning strategy which can be modified through varying the energy source and scanning pattern, and which is not static for any given geometry (Raplee *et al.*, 2017).

Electron microscopy (EM) is the perhaps most versatile tool for shedding more light on the complex multiscale microstructural evolution during AM. We will review EM characterization techniques from the large (mm – μm) down to the atomic scale for imaging and chemical analyses of typical AM microstructures based on scanning and transmission electron microscopy (SEM and TEM). We will then explore selected state-of-the art 3D imaging techniques suitable for characterization of typical AM microstructures, e.g., 3D electron back-scatter diffraction (3D EBSD) in a focused ion beam (FIB) microscope. A short section on correlative microscopy will outline approaches that enable full crystallographic and chemical analyses of features such as interfaces, applying atom probe microscopy (APM). In-situ heating and micro-mechanical testing approaches for SEM and TEM will be introduced. These techniques are now capable of simulating thermal cycles typical to AM in a TEM. A short outlook on where this field may be headed will be provided at the end.

2D Characterization Methods

The following section will review common 2D characterization methods suitable for multi-scale imaging of AM microstructures going from the larger (mm – μm) scale down to the nm and atomic scale. Fig. 1, which is provided at the end of this section, is a schematic aimed at giving an overview over individual techniques that may be suitable at different length scales, alongside with typical features found in AM microstructures that can be characterized using these techniques. For example, on the large scale, melt pool boundaries and secondary phase particles may be imaged using SEM. Individual crystallographic orientations of grains, subgrains, and secondary phases can be determined via EBSD. Further crystallographic information on the nm scale is revealed via

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Super Resolution for Multi-Sources Image Stream Data Using Smooth and Sparse Tensor Completion and Its Applications in Data Acquisition of Additive Manufacturing

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ABSTRACT

Recent developments of advanced imaging systems spur their applications in many areas, ranging from satellite remote sensing for geographic information to thermal imaging analysis for manufacturing process monitoring and control. Due to different specifications of imaging systems, the resulting image stream data (videos) have different spatial and temporal resolutions. This proposed work is based on the image stream data captured by multiple imaging systems for the same object with different but complementary spatial and temporal resolutions. For example, one system has high spatial but low temporal resolutions while the other one has opposite resolutions. The goal of this article is to develop a new super resolution method that integrates these different types of image stream data to improve both spatial and temporal resolutions, which is critical to obtaining more insightful information for more effective quality control of targeted processes or systems. To fulfill this goal, a new tensor completion model is developed by considering both smooth and sparse features simultaneously and is thus termed smooth and sparse tensor completion (SSTC). The results of the extensive case studies illustrate the superiority of our method over the elaborately selected benchmark methods.

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CANDECOMP/PARAFAC (CP) decomposition; Rank estimation; Smooth and sparse decomposition

1. Introduction

Advanced imaging systems have been widely applied to many areas, such as images for manufacturing processes monitoring and control (Liu et al. 2019; Shen, Xie, and Kong 2020), spectral images in remote sensing applications (Shaw and Burke 2003). The spatial resolution and acquisition rate (temporal resolution) are the keys to the performance of imaging systems for capturing the evolution insight of the targeted object. Different imaging systems generate image stream data with different spatial and temporal resolutions, and thus have complementary performances. For example, in thermal imaging analysis of a metal additive manufacturing (AM) process (Haines et al. 2018), a small thermal camera can be mounted inside of the 3D printer chamber to obtain high spatial resolution images (the distance between camera and the region of interest could be very short, resulting a small field of view), but the resulting images will have low temporal resolution (the small size limits its capability of high acquisition rate of images). On the contrary, an advanced thermal camera with high temporal resolution is typically very bulky and has to be mounted outside of the 3D printer chamber, thus the resulting spatial resolution is low (the distance between the camera and the region of interest is long and thus results in a large field of view). It is vital to obtain image stream data with both high spatial and high temporal resolutions so that subtler information, which is perhaps hidden from us if using just one of the above imaging systems, can be captured and analyzed.

This challenge motivates the research presented in this article, namely to develop a method that fuse images streams with complementary spatial and temporal resolutions to obtain high spatial and high temporal image stream. By applying our proposed method, we can achieve a cheap sensing system to acquire expensive sensor data. In general, either high spatial and low temporal, or low spatial and high temporal sensors are easy to obtain, while high spatial and high temporal sensors are quite expensive or even do not exist for some stringent specifications. For example, a thermal gradient is a critical physical quantity that describes the most rapid changing rate of the temperature around a particular location in heating/cooling processes (Raplee et al. 2017), which has spatial temporal structure. According to the literature (Al-Bermani et al. 2010), the microstructure can be predicted by the thermal gradient. Thermal gradient information can be obtained by thermography. However, there is no such equipment to obtain thermal gradient because of the unique property of the melt pool (Cheng et al. 2018) in the selective laser melting (SLM) process. The melt pool is small (100–200 μm) and the dwell time is short (0.5–1.5 ms). The temporal resolution (frame rate) of the camera should at least be 20k Hz because the solidification can be even shorter than the dwell time. The spatial resolution needs to be around 3 μm per pixel and this can give a detailed thermal gradient of the melt pool, for example, a keyhole in melt pool in SLM is normally around 10 μm in diameter (Cunningham et al. 2019), and 3 μm spatial resolution can give at least 3 point measurement along one direction for the

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Role of scan strategies and heat treatment on grain structure evolution in Fe-Si soft magnetic alloys made by laser-powder bed fusion

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ABSTRACT

A major goal in printing soft magnetic Fe-Si steels using additive manufacturing is to take advantage of the potential for complex geometric designs and site-specific grain control. One major step in the processing of these alloys is understanding how processing parameters might impact how the as-built microstructure responds to annealing (i.e. the annealing response). The impact of scan strategy on the annealing response for thin wall geometries is specifically explored. Two scan strategies were explored for a thin wall geometry that produced a strongly columnar grain structure and equiaxed grain structure. Samples from both scan strategies annealed at 1200 °C showed a marked difference in annealing response with the more equiaxed sample seeing full recrystallization and grain growth, while the more columnar grain structure saw little change in microstructure. After analysis through characterization techniques and thermal-mechanical simulations Differences in internal energy within the grains were ruled out because calculated GND density values were similar for both samples. The formation of secondary particles was ruled out as a contributing factor due to the type of oxide formations and their size. It was concluded that the contributing factor to the difference in the annealing response were a difference in the resulting grain size and the density of high angle grain boundaries. These two differences were largely attributed to differences in the thermal gradient conditions due to grains preferentially growing in the direction of the steepest thermal gradient.

1. Introduction

Metal additive manufacturing (AM) processes have transitioned from prototype to structural applications because of their ability to construct complex and light weight geometries with tailored microstructures designed for complex loading conditions. A next step is to extend the same ability to impart site-specific functional properties based on electrical and magnetic phenomena. If proven, the ability to impact functional and mechanical properties in complex geometries opens up new avenues for the adoption of AM. Recent publications have considered various ferromagnetic alloys including Fe-Co [1–4] and Fe-Ni [2,5,6] due to their importance in electrical transformer cores and electrical motor applications. Soft-magnetic Fe-Si alloys, the focus of current research, should exhibit high magnetic permeability and electrical resistivity which is desirable for reducing power losses [7]. Generally, on increasing of the silicon content up to 6.5 wt% we can increase magnetic permeability and electrical resistivity [8]. The fundamental questions explored in this paper: can we produce site-specific microstructures in Fe-Si alloys through modification of laser scanning strategy during the laser powder bed fusion (L-PBF) process and subsequent heat treatment. Second, what is the role of solidification microstructure, solid-state phase transformations and evolution of plastic strain gradients due to spatially and temporally varying thermo-mechanical signatures during AM?

For soft magnetic steels, reduction of power losses usually involves the minimization of hysteresis and eddy current losses [8]. Hysteresis losses are dependent on crystallographic texture, grain size and material properties. To decrease the hysteresis losses, materials will be processed to have a crystallographic texture aligned in the < 001 > direction known as the easy magnetization direction [7]. Hysteresis losses are also

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Multi-scale characterisation of microstructure and texture of 316L stainless steel manufactured by laser powder bed fusion

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ABSTRACT

316L stainless steel produced by additive manufacturing (AM) offers superior mechanical and corrosion properties compared to parts of the same steel processed by conventional methods. However, thorough understanding of the detailed microstructural evolution during various additive manufacturing techniques is lacking when compared to conventionally produced counterparts. As such, the present work contributes to filling this gap in knowledge by using advanced microscopy techniques to analyse the microstructure of 316L stainless steel builds produced by laser powder bed fusion. The results show that changes in processing parameters lead to variations in grain structures, texture, and hardness. For instance, the dominant texture and grain structure is heavily influenced by changes to the laser scanning rotation between individual layers, while increasing laser power reduces hardness. The current study also provides additional insights into how grains in AM 316L steel accommodate strain. Due to the complex strain fields in austenite grains, they are usually split into complex-shaped regions separated by dislocation boundaries with characters similar to deformation- and micro-band boundaries in conventionally deformed austenite. Through electron probe micro-analysis over a large area, the current study reveals the tendency of Mo and Si concentrations to fluctuate around the cell and melt pool boundaries.

1. Introduction

Additive manufacturing (AM) offers promising new avenues to produce high performance metal parts with intricate features [1,2]. In AM, 3D parts are usually made layer by layer using different feedstock materials such as wires or powders, and high energy power sources such as lasers or electron beams [3]. AM is currently used to manufacture parts made from a variety of engineering alloys such as steels, Ti, Ni, Al, and Mg alloys [4]. Among these, steels have received the perhaps highest level of attention in research, currently comprising about one third of the metal AM publications in literature [5]. As steels account for ~80 wt % of all metallic parts used for engineering purposes, there is still much more study required to comprehensively understand the microstructure-property relationships of AM steels. Stainless steels are of particular interest due to their highly advantageous combination of mechanical and corrosion properties. This is underpinned by the fact that ~55% of all steel powders used in AM manufacturing in 2018 were stainless steels [6]. Among different stainless steels grades, 316L is the most popular in AM with a broad range of applications such as in construction, oil and gas, automotive, aerospace, and dentistry [7].

Laser powder bed fusion (LPBF) is the most common AM technique used to process 316L stainless steel [6]. LPBF 316L is usually characterised by columnar grains comprised of sub-micron scale cells decorated with segregation of alloying elements [8–11]. There are different theories regarding the sequence of formation and elemental segregation resulting in these cellular structures. Some authors reported that these structures are formed due to cellular solidification and its associated elemental segregation [10]. Others have criticised this mechanism, and instead have attributed cell wall segregation to dislocation rearrangements into low-angle boundaries followed by absorption of alloying elements into these dislocation walls [11]. Irrespective of their formation mechanisms, such low angle grain boundaries and the related high dislocation density have a substantial influence on mechanical and corrosion properties of 316L stainless steels [8–11].

Although segregation might account for the formation of cells, it cannot explain the complex misorientation gradients within a grain in AM microstructures. In a recent study by Bertsch et al. [12], it has been shown that dendrite formation during solidification and related micro-

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Evolution of microstructure and mechanical properties in 2205 duplex stainless steels during additive manufacturing and heat treatment

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ABSTRACT

Metal additive manufacturing (AM) offers exceptional design freedom, but its high thermal gradients often generate non-equilibrium microstructures with chemical and interfacial instabilities. Steels that solidify as δ-ferrite often experience a further solid-state phase transformation to austenite during AM. The detailed nature of this phase transformation during AM is yet to be fully understood. Duplex stainless steel, which is known for its unique combination of high corrosion resistance and mechanical properties, is a suitable alloy to further study this phase transformation.

The current study aims to gain novel insights into solid-state phase transformations and mechanical properties of duplex stainless steels during laser powder-bed fusion (LPBF). As-printed microstructures exhibit significant deviations when compared to conventionally manufactured counterparts in terms of phase balance and morphology, elemental partitioning, and interface character distribution. During LPBF, only a small fraction of austenite forms, mostly at the ferrite-ferrite grain boundaries, via a phase transformation accompanied by diffusion of interstitials. Austenite/ferrite boundaries are shown to terminate on $\{100\}_F//\{111\}_A$ planes. This is due to the character of parent ferrite-ferrite boundaries which is dictated by the sharp <100> texture and geometry of austenite grains induced by directional solidification and epitaxial growth of ferrite. Benchmarking mechanical properties against a wrought counterpart demonstrates that AM offers high strength but relatively low ductility and impact toughness. A short heat treatment reverts the microstructure back to its equilibrium state resulting in balanced tensile and toughness properties, comparable to or even better than those of wrought counterparts.

1. Introduction

Duplex stainless steels (DSSs) are designed to synergistically combine the superior properties of ferrite and austenite to achieve advanced mechanical and corrosion properties [1]. These steels solidify initially as δ-ferrite, which partially transforms to austenite upon cooling to temperatures lower than ~1370 °C. Control of the chemical composition and the ferrite-to-austenite phase transformation path are the main means to adjust the volume fraction, morphology, and chemistry of the two phases [2–4]. Traditionally, roughly equal fractions of austenite and ferrite are considered as the optimum phase balance, and steels with such microstructures are widely used in harsh environments such as in offshore infrastructures e.g., for oil and gas industries, paper and pulp industries, and desalination plants [1]. Although the majority of duplex steel parts currently in use are manufactured via casting and forming, there are currently ongoing efforts to employ alternative state-of-the-art manufacturing processes such as 'additive manufacturing' to fabricate DSS components. DSSs have a low thermal expansion coefficient and relatively high thermal conductivity compared to austenitic stainless steels, making them fit for additive

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Metal Additive Manufacturing

Texture evolution in a CrMnFeCoNi high-entropy alloy manufactured by laser powder bed fusion

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ABSTRACT

Additive manufacturing (AM) techniques including laser powder bed fusion have been widely used to produce metallic components with microstructures and mechanical properties distinctly different from the conventionally manufactured counterparts. Understanding how AM parameters affect the evolution of microstructure, including texture, of these AM metallic components is critical for appropriate manipulation of their processing and therefore their mechanical properties. Here we conducted a systematic investigation of texture evolution of a face-centred cubic CrMnFeCoNi high-entropy alloy cuboid fabricated using laser powder bed fusion. Our results showed that the texture evolutions along the build direction were different between the corner and central parts of the sample. Detailed analysis suggested that the texture evolution is closely related to local thermal gradient, which is a property that can be manipulated through changing AM parameters. The different textures lead to the significant variations of mechanical properties within the sample.

Introduction

Additive manufacturing (AM) is an advanced technology for rapid production of components with complex geometries. Opposed to the conventional subtractive manufacturing approaches where unwanted materials are removed from an over-dimensioned bulk piece, AM is an incremental process based on layer-by-layer deposition of materials from a feedstock of wires or powders that is often selectively melted by a high power focused laser or electron beam [1–4]. As a near-net or net shape fabrication technology, AM significantly reduces the materials cost by only consuming the required amount of feedstock materials and therefore is ideal

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Enhancing the repassivation ability and localised corrosion resistance of an additively manufactured duplex stainless steel by post-processing heat treatment

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ABSTRACT

Duplex stainless steel (DSS) 2205 produced by laser powder bed fusion (LPBF) additive manufacturing was found to possess poor repassivation ability and weak resistance to localised corrosion. This was due to the nonequilibrium phase composition and elemental distribution in the as-built LPBF DSS affected by the rapid solidification inherent to LPBF processing. A short-time post-processing heat treatment at 1000 °C for 10 min followed by water quenching to room temperature was shown to be able to enhance the repassivation ability and localised corrosion resistance by retrieving the equilibrium dual-phase microstructure. A discussion is given on the mechanism behind such behaviour.

1. Introduction

Additive manufacturing (AM) has attracted considerable attention during the past decade as a new means of producing engineering metallic materials, in particular components with intricate geometries [1–3]. However, a major challenge associated with AM metallic materials is their nonequilibrium and sometimes anisotropic microstructures compared to their conventionally manufactured counterparts which may affect the material properties and deteriorate their performance [4,5]. This is even more significant in AM of materials where their microstructure evolution is highly dependent on the cooling rate during solidification, e.g., dual- and multi-phase materials. Duplex stainless steels (DSSs) are among such materials whose phase composition in the as-built AM state has been reported to deviate from the equilibrium ferritic-austenitic microstructure. This has been attributed to the rapid solidification inherent to AM [6]. Achieving a balanced microstructure is believed to be important for DSSs because a combination of high mechanical strength and excellent corrosion resistance is generally considered to be acquired with a roughly 50:50 vol fraction of δ-ferrite and austenite phases and a high proportion of alloying elements [7–10]. Indeed, the microstructural changes imposed during manufacturing processes (e.g., welding) have been found to undermine the mechanical and corrosion properties of DSSs [11,12].

Suitable post-processing heat treatments are required to restore the dual-phase microstructure of DSSs in order to obtain the optimised properties of interest. Table 1 summarises the phase balance of different grades of DSSs produced by laser powder bed fusion (LPBF) AM technology and changes induced by common post-LPBF heat treatments. As can be seen, the as-built material exhibits a high proportion of the ferrite phase in all cases. Such ferrite-dominated microstructure in the as-built state is changed to a ferritic-austenitic microstructure after heat treatment, with the extent of phase transformation depending on the detailed heat treatment conditions.

Concerning the corrosion properties of DSSs the formation of ferrite and precipitation of secondary phases (e.g., Cr-rich nitrides) are considered to be of great importance because of their susceptibility to attack in corrosive environments [13–17], however, the significance of such microstructural features on the repassivation properties of DSSs has not been sufficiently understood. Most of the previous studies on the corrosion of AM DSSs are mainly concentrated on the pitting corrosion characterisation in a stagnant solution using conventional electrochemical techniques such as potentiodynamic polarisation measurements [18–22]. For instance, Jiang et al. [22] reported that pitting characteristics for the as-built LPBF 2205 DSS and LPBF 2507 super DSS

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Advanced quantification of the site-occupancy in ordered multi-component intermetallics using atom probe tomography

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ABSTRACT

A new experimental method is proposed to quantify the site-occupancy of substitutional solute elements in multi-component intermetallics from atom probe tomography data and is applied to the $L1_2$ ordered γ' phase in the top, middle and bottom regions of an electron powder bed fusion produced IN-738LC build. Ti, W and Ta are found to substitute almost exclusively for the β sites. Cr and Mo show mixed behaviour with a higher proportion substituting for the β sites. Co also shows mixed behaviour but with a higher proportion substituting for the α sites. While γ' maintains an almost constant chemistry and site-preference behaviour throughout the build, the relative site-occupancy ratio is observed to change, particularly for Co and Mo. The results suggest that local changes in thermal history inherent to metal additive manufacturing processes may induce changes to the resultant site-occupancy of γ'. The method described here improves the experimental quantification of the local atomic site-occupancy, enabling an assessment of the substitutional solute element fractions occurring at the α and β sites in $L1_2$ ordered structures. This is important in multi-component intermetallics because of the burgeoning interest in relating the elastic and plastic properties of these structures to their site-occupancy.

1. Introduction

Intermetallics play a vital role in the design of many engineering materials. Their chemistry and structure has significant influence over the resulting mechanical [1–3] and functional [4–6] properties. This notion of structure necessarily includes long-range ordering, and the nature of the site-preferencing behaviour of any solute species that may have been added, as well as the morphology of intermetallic precipitates. An archetype example is γ' which is the predominant high-temperature strengthening phase in many Ni-based superalloys used in the hottest components of gas turbine engines [3]. The high bonding strength (cohesive energy) within γ' brought about the $L1_2$ ordered nature of the phase [7,8] give γ' its strength imparting properties [3,9,10].

γ' has a stoichiometric base composition of Ni_3Al with limited non-stoichiometric compositional variation [8]. Ni atoms occupy the centre of the unit cell faces (or α positions), while Al atoms occupy the unit cell corners (or β positions) in an ordered fashion, as shown in Fig. 1a. Alternating (002) lattice planes contain either Ni atoms (α sites) or an equal planar density of Ni and Al atoms (α and β sites). When substitutional solute elements are added, site-preferencing to either the α or β sites is typically observed [11,12]. The magnetic interaction [13–15] and the strain energy [16] induced by incorporating a solute atom into the $L1_2$ crystal are the predominant factors influencing this behaviour. This behaviour is of particular interest because many studies have reported an enhancement of mechanical properties when solute additions participate in the ordered γ' precipitates of Ni-based superalloys [17–20].

The site-preferencing of a solute species can be predicted through a combination of techniques including consideration of the ternary phase field extents of γ' and the relative size of the added solute species relative to Al and Ni [3]. Many first-principles studies have also been performed to study sub-lattice site-occupancy characteristics of ternary alloying additions but most of these calculations have been performed at 0 K which exclude the influence of temperature. Wu and Li [18] and Jiang and Gleeson [21] have addressed this problem by combining

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Overview article

The segregation of transition metals to iron grain boundaries and their effects on cohesion

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ABSTRACT

The segregation of transition metal elements to grain boundaries in steels plays a critical role in determining their cohesion. Here, we investigate the segregation, co-segregation, and cohesion effects of various transition metals (Co, Cr, Cu, Mn, Mo, Ni, Nb, Ti, V and W) to different grain boundary characters in ferritic-iron (α-Fe) through a systematic, brute-force style configurational analysis utilising density functional theory calculations. We demonstrate that differing grain boundary characters change not only transition metal segregation and co-segregation behaviours, but also their effects on cohesion. The effects of co-segregated solutes on cohesion can be substantially different from their summed individual parts. We show that solute-solute interactions at grain boundaries vary significantly as a function of grain boundary character. These interactions are shown to be substantially different from those that occur in the bulk. We introduce a novel quantitative method for assessing effects of segregated elements on interfacial cohesion through calculating the strength of bonds at a grain boundary in the DDEC6 bond-order framework. It is shown that work of separation quantities calculated through rigid separation of surfaces better captures the strength of bonding in most cases, and thus more accurately depicts intergranular fracture. Collectively, these results offer valuable insight towards rational grain boundary engineering in steels.

1. Introduction

Grain boundaries (GBs) are an important class of defects that play a significant role in the properties of engineering alloys. The control and engineering of GBs via thermomechanical processing, known as GB engineering [1], has attracted considerable attention as a method to control alloy properties. GB segregation refers to the presence of elevated concentrations of foreign elements accumulated near GBs. The segregation of either impurities and/or alloying elements to GBs has been shown to dramatically alter the cohesion of GBs [2,3]. It is also well known that GB character is a major factor in the determination of the segregation behaviour of different elements [4,5]. Importantly, various transition metals are often used in alloying to impart specific properties to the final alloy. The segregation of transition metals to GBs plays a key role in the stabilisation of nanocrystalline iron-based alloys [6,7]. Undesirable phenomena, such as temper embrittlement [8] and hydrogen embrittlement [9], mediated by GB segregation, can cause sudden catastrophic failure in the form of intergranular fracture in steels. Thus, understanding the segregation behaviour of transition metals to different types of GBs and their effects on cohesion is a prerequisite for knowledge-based GB engineering of steels with a resistance to such phenomena. However, behaviours that arise due to segregation effects can be difficult to isolate experimentally due to the complex nature of real alloys, despite recent advances [10,11]. To this end, many computational studies have focused on the elemental segregation of transition metal solutes to Fe GBs, and how they may affect GB cohesion [12–20]. However, such studies can contain seemingly conflicting information for even well-studied elements, as evidenced by a comprehensive review by Lejček et al. [21].

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A Crystallography-Mediated Reconstruction (CMR) Approach for Atom Probe Tomography: Solution for a Singleton Pole

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ABSTRACT

Spatially accurate atom probe tomography reconstructions are vitally important when quantitative spatial measurements such as distances, volumes and morphologies etc. of nanostructural features are required information for the researcher. It is well known that the crystallographic information contained within the atom probe data of crystalline materials can be used to calibrate the tomographic reconstruction. Specifically, the crystallographic information projected into the field evaporation images is used. This offers a powerful and accurate enhancement of the atom probe technique. However, this is often difficult to do in practice. In previously reported approaches, it was necessary to index at least two poles to compute the image compression factor 'ξ' and observe crystallographic planes in at least one of the pole regions to obtain a measure of the field factor 'k_f' while also manually accounting for a change in reconstruction parameters throughout the dataset. Not only is this error-prone and time consuming, it does not work for materials that exhibit limited crystallographic information in their field evaporation image. Here, we extend the applicability of the crystallographic calibration of atom probe data by proposing a reconstruction methodology where only one pole with observable lattice planes is required in the projected detector image. Our proposal also accounts for dynamic variations in the reconstruction parameters throughout the 3D dataset. The method is simpler and significantly faster to implement and is applicable to more atom probe situations than previously approaches. Our single-pole crystallography mediated reconstruction (SP-CMR) utilizes the Hawkes-Kasper projection model (equivalent to the equidistant-azimuthal projection model) and the direct fourier (DF) fit algorithm to determine the precise reconstruction parameters required to produce flat atomic planes. It is applied to experimental Al and highly Sb-doped Si data. The discrepancies between the spatial dimensions of the SP-CMR reconstructions compared to uncalibrated reconstructions are visually apparent. Consistent plane spacings and angles between crystallographic directions matching the theoretically known values for each crystal structure are demonstrated.

1. Introduction

One of the most powerful aspects of atom probe tomography (APT) as a microanalysis technique is its ability to reveal three-dimensional (3D) spatial information on the atomic-scale architecture of materials. It is from this 3D data that APT has achieved a significant impact and utility in efforts to understand dopant distributions [1], solute segregations to grain boundaries and dislocations [2], crystallographic structure and relationships [3], solute clustering and the early stages of precipitation [4], and other phenomena in materials science. However, the raw data captured during an APT experiment is not in 3D. It is collected as a set of 2D ion impact coordinates across the face of a detector, in mm scale, with each impact assigned an associated time-of-flight. The process of converting this raw data into a tomographic reconstruction is a critical step in the analysis workflow in the APT technique.

1.1. Reconstruction fundamentals

The reconstruction process in APT converts the ion coordinates in the 2D detector frame of reference (X_D, Y_D), as well as the detector hit-sequence number 'N_D' and specimen radius 'R' to the 3D coordinates

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Towards the Development of a Multi-Modal Community-Based AM Database

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Metal additive manufacturing (AM) is increasingly being sought after in critical military, aerospace, and biomedical manufacturing applications for its capability to create near-net shaped parts while minimizing time and material cost. In order to fabricate critical parts with orientation dependent properties required by industries, a complete understanding of microstructural heterogeneities (MH) and the ability to control these MH using AM process parameters is needed. These knowledge gaps are a serious impediment to AM part qualification and industry adoption [1]. Additionally, the non-equilibrium process conditions during AM of parts have led to the development of new alloys suitable for AM processes [2]. Therefore, strategies to accelerate AM part qualification is a major challenge that faces the AM community. The reasons for these barriers to qualification of AM parts are 1) the lack of spatial understanding in the hierarchy of defects and MH found in AM parts, 2) the lack of data-driven approaches for discovering new materials/alloys suitable for AM and 3) the lack of standardized high-volume process-structure-property (PSP) datasets for AM builds.

Overcoming these barriers necessitates the aggregation of multi-format, multi-dimensional datasets (often spanning multiple terabytes) required to develop such standards. These datasets must contain experimental, computational, and empirically derived data about processing pedigree, microstructural features, and performance parameters of parts spanning multiple builds. Moreover, the data must be able to be accessed in such a way that the community can readily derive relationships in the PSP space. In recent years, these challenges have been addressed in several ways. The creation of the propnet Python module has been created to empirically derive secondary properties from datasets and is a way to increase the dimensionality and informational footprint of the data collected [3]. Another is the advent of quality standards for the management and stewardship of scientific data known as the FAIR guiding principles [4]. FAIR data stands for Findable, Accessible, Interoperable, and Reusable/Reproducible data. These principles dictate how metadata is to be represented within a federated system. HyperThought™ is designed with features to allow users to upload data to a secure location on the cloud and ensure that their data meets FAIR standards [5]. HyperThought™ is capable of accepting data from users via REST API, attaching relevant metadata tags to files uploaded, run community-created algorithms on files for processing, and most importantly, represent the overall structure of an industrial process or scientific study through its process-modeling sub-application, Workflow. HyperThought™ enables users to develop a persistent machine-representation of knowledge gained from their data collection efforts.

This study will present a systematic workflow to collect multi-modal datasets, with secondary properties of the material system derived using propnet. The entire dataset with explained pedigree will then be uploaded onto HyperThought™. The data will be available for public access. This dataset will consist of spatially collected microstructural data obtained from an AM block using a high-throughput characterization methodology established in our previous work [6]. The extent of spatial variations in microstructure due to process conditions across the sample will then be related to the changes in local performance parameters within the sample. We envision that this could further stimulate a community-wide effort to create a collection of datasets that can be used as a starting point for AM

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Full Length Article

Formation and 3D morphology of interconnected α microstructures in additively manufactured Ti-6Al-4V

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ABSTRACT

Three-dimensional characterization methods, such as 3D electron backscatter diffraction (3D-EBSD), have been used to reveal phase transformation and microstructural evolution mechanisms in multi-phase materials such as steel or titanium alloys. While 3D techniques have enabled many findings in steels, fine dual phase microstructures in titanium alloys such as the basketweave structure have been challenging to resolve. Now, advances in 3D-EBSD methods using sectioning with a plasma focused ion beam have allowed in-depth analyses of fine α microstructures. We apply 3D-EBSD to investigate the microstructures formed in Ti-6Al-4V by electron powder bed fusion (E-PBF) using different scanning strategies. Basketweave, acicular, and colony microstructures are produced from linear, Dehoff, and random scanning strategies, respectively. Different types of 3D interconnectivity were revealed in each microstructure including within clusters of platelets in the basketweave microstructure, within a grain boundary allotriomorph in the acicular microstructure, and between platelets in colonies. These observations are discussed in terms of the formation mechanisms of interconnectivity, including sympathetic nucleation, impingement, and morphological instability. Morphological instability was found to potentially play a role in both the basketweave and colony structures while the interconnectivity in the acicular structure likely forms via sympathetic nucleation or impingement. This information allows for a more complete description of the phase evolution of Ti-6Al-4V during thermal cycling in E-PBF than previously available and represents new insights into the complex branching reported in different titanium microstructures.

1. Introduction

Three-dimensional microscopy techniques, while far from new, have lately gained increased interest due to improvements in resolution and computational reconstruction and visualization techniques [1]. 3D imaging and analysis have enormous potential to provide crucial insights into the true morphology and evolution of complex microstructures. These techniques can now be applied to reveal the microstructural evolution of titanium alloys, which can form a variety of phases in highly complex 3D morphologies under certain processing conditions. This potential has been demonstrated previously in answering several open questions within the field of steels, which undergo a similar array of phase transformations and form similar complex structures, though generally on a coarser scale.

In case of steels, these include the morphology and origin of proeutectoid Widmanstätten ferrite and cementite, the origins of bainite/acicular ferrite, and the formation mechanism of pearlite. Deep etching [2] and serial sectioning [3] revealed how proeutectoid cementite plates in fact originated at an austenite grain boundary (GB) even when they appeared to be intragranular, as well as dendrite-like 3D morphologies within cementite films on austenite GBs. A major controversy in this area was whether Widmanstätten side plates formed on GB allotriomorphs via sympathetic nucleation [4] or branching due to morphological instability [5]. Sympathetic nucleation is the nucleation of a precipitate at the interface of another precipitate of the same phase in order to continue a phase transformation after the phase interface can no longer move, or to replace the high energy phase interface with a lower energy GB [4,6–8]. Morphological instability can occur when perturbations in a migrating phase boundary cause the interface to become unstable and form branches [9–11].

Though in situ experiments and many observations of low angle grain boundaries (LAGBs) between side plates have contributed to the conclusion that sympathetic nucleation generally dominates in this case

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3D characterization of microstructural evolution and variant selection in additively manufactured Ti-6Al-4 V

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ABSTRACT

Ti-6Al-4 V is a popular alloy in additive manufacturing (AM) due to its applications in the biomedical implants and aerospace industries where the complex part geometries allowed by AM provide cost and performance benefits. Ti-6Al-4 V goes through a $\beta \rightarrow \alpha'$ transformation after solidification which is known to experience variant selection, e.g., through the formation of clusters of variants which, when situated together, partially accommodate the strain of the phase transformation. During electron beam powder bed fusion AM, an in situ decomposition of α' martensite occurs during the cyclic reheating caused by melting successive layers, resulting in $\alpha + \beta$ microstructures. How variant selection influences the evolution beyond the initial rapid cooling remains an open question. Using 3D electron backscatter diffraction, we provide a clearer understanding without ambiguity from sectioning effects of how α' decomposes into microstructures with distinct morphologies and variant/intervariant distributions. We extract quantitative 3D information on the various intervariant boundaries networks formed in samples printed using three different electron beam scanning strategies. This shows that differing mechanisms during the decomposition result in a shift from self-accommodating clusters in an acicular microstructure, to either the preferred growth of six variants in a basketweave microstructure, or to a colony microstructure where variant selection is determined by prior-β grain boundaries. We propose a new representation of the misorientations arising from the Burgers orientation relationship, which we refer to as intervariant network diagram, to reveal how variant selection during the martensitic transformation and subsequent decomposition leads to the intervariant boundary networks observed. This holistic understanding of the microstructural evolution has the potential to allow tailoring of microstructures and properties for specific applications.

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SHORT PULSED LASERS FOR MATERIALS MODIFICATION, CHARACTERIZATION, AND SYNTHESIS

Recent Developments in Femtosecond Laser-Enabled TriBeam Systems

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Streams of multimodal three-dimensional (3D) and four-dimensional (4D) data are revolutionizing our ability to design and predict the behavior of a broad array of advanced materials systems. Over the last 10 years, a new 3D imaging platform consisting of a femtosecond (fs) pulsed laser coupled with a focused ion beam scanning electron microscope (FIB SEM) has been developed by UC Santa Barbara in collaboration with Thermo Fisher Scientific (formerly FEI). The femtosecond-laser-enabled FIB SEM, called the TriBeam, has become one of the only 3D serial sectioning methods available that can gather millimeter-scaled multimodal datasets at sub-μm voxel resolutions; these length scales are critical for many materials problems. Multimodal chemical, crystallographic, and morphological information can be gathered rapidly on a layer-by-layer basis and reconstructed in 3D. Large (gigabyte to terabyte scale) 3D datasets have been generated for a broad array of materials systems, including metallic alloys, ceramics, biomaterials, polymer- and ceramic-matrix composites, and semiconductors. The research tasks performed have resulted in a completely new design, operating with a dual-wavelength femtosecond-pulsed laser on a plasma focused ion beam (PFIB) platform.

INTRODUCTION, BACKGROUND, AND HISTORY

The convergence of rapidly advancing computational capabilities, new theoretical approaches for property prediction, three-dimensional (3D) and four-dimensional (4D) characterization techniques, and the ability to rapidly print and evaluate materials has suddenly opened many new horizons for the discovery and development of previously inaccessible advanced materials. On the characterization front, a range of microscopes and x-ray methods have been developed to gather 3D microstructural information from the nanoscale to centimeter scale for a range of materials. While the scope of this work is not intended to comprehensively review the available 3D approaches, many significant advances have been made that are extending the spatial capabilities and detection modalities for 3D and 4D data acquisition. The wide suite of synchrotron x-ray imaging approaches have been broadly summarized previously,[1–3] including in a recent special issue on 3D materials science.[4] Many materials tomography challenges are becoming more routine at synchrotron sources, such as grain mapping by diffraction contrast tomography (DCT)[5] or high-energy diffraction microscopy (HEDM),[6,7] subgrain stress measurements[3] and imaging of the localization of plasticity via topotomography,[1,8,9] and dark-field x-ray microscopy for defect imaging.[10,11] Advancements in grain mapping with DCT using laboratory x-ray sources has improved dramatically from early work, which focused on the capture of

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On the pitting corrosion of 2205 duplex stainless steel produced by laser powder bed fusion additive manufacturing in the as-built and post-processed conditions

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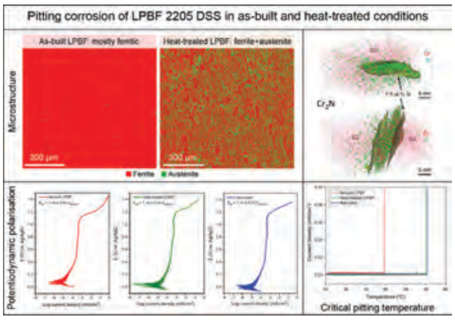
HIGHLIGHTS

- 2205 DSS shows similar pitting potentials and passive current densities at RT irrespective of processing route.
- Cr is depleted adjacent to intergranular Cr_2N particles but remains above the critical value for passivation.
- As-built LPBF 2205 DSS shows a lower CPT compared to its hot-rolled counterpart.
- Post-AM heat treatment enhances the critical pitting temperature to the level of the hot-rolled DSS.

GRAPHICAL ABSTRACT



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ABSTRACT

The effects of additive manufacturing (AM) and post-AM heat treatment on microstructural characteristics and pitting corrosion of 2205 duplex stainless steel were studied and benchmarked against its conventionally hot-rolled counterpart. The rapid solidification and possible loss of N associated with AM resulted in a non-equilibrium microstructure dominated by δ-ferrite with a minor fraction of austenite and abundant Cr_2N precipitation. Atom probe tomography revealed that no depletion of Cr occurs around intragranular Cr_2N. A deduction in Cr was observed adjacent to intergranular Cr_2N particles, however, Cr content in these regions remained above the critical value of 13 wt%. Post-AM heat treatment was effective in restoring the duplex microstructure while dissolving the Cr_2N precipitates. Although the pitting resistance in the as-built AM specimen was lower than that of its hot-rolled counterpart, it was fully recovered after post-AM heat treatment.

1. Introduction

Following the great progress in additive manufacturing (AM) during the past decades, many industry sectors now attempt to use this technology to enable the production of complex 3D

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Qualification pathways for additively manufactured components for nuclear applications

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ABSTRACT

This research paper evaluated three pathways for qualification of 316 L stainless steel components made by laser powder bed fusion additive manufacturing (AM). Comprehensive and consistent process flows with computational modeling, in-situ measurements, ex-situ characterization and mechanical testing with simple- and complex- geometries were explored. The role of post-process hot isostatic pressing (HIP), and solution anneal treatment were evaluated. By using HIP, the scatter in 316 L steel AM properties within single and complex components was minimized to meet the requirement of existing industry standards. For applications where HIP may not be feasible and with some extent of defect tolerance, alternative qualification methodologies of deploying L-PBF AM parts were also explored with samples made with and without engineered porosities. The data generated in this research will be relevant to deployment of AM components for emerging nuclear energy applications.

1. Introduction

Nuclear power plant equipment manufacturers are moving towards deployment additive manufacturing (AM) methods to produce reactor internal components. This move is driven by the unique capability of AM to generate complex geometries rapidly with the potential for improved performance, while reducing the cost and time to market, with wide range of materials including metals, polymers, ceramics and hybrid components [1–3]. In the last two decades extensive progress has been made in the science and technology of AM and being disseminated through research publications, reviews and books [4–9]. At the same time, standards, codes and regulatory bodies are contemplating on the reliability of these AM components for real-life service due to potential scatter in metallurgical and mechanical properties emanating from machine specific and process variations [10]. For example, Hull points out the following interests from US Nuclear Regulatory Commission (NRC) including reliability of AM processing, quality of parts, properties of parts, structural performance of AM parts and their inspectability, service performance and ageing degradation of AM parts, and emerging manufacturing processes [11]. Although current efforts to develop qualification methods and standards (e.g., ASTM F42 and AWS) are based on fabrication/testing of coupons [12–22], *there is no clear, concise methodology for component and process-based certification*. For example, even when two identical parts are made with the same processing equipment and powder composition, variations in properties are observed due to stochastic variations in laser energy interaction, associated effects leading to inefficient melting and defect formation, as well as spatial and temporal variations of thermal signatures [23–26]. Therefore, many researchers have been working on a rapid component level qualification method that combines AM with high fidelity process optimization and control through integrated computational materials engineering (ICME) [27]. This research paper evaluates the feasibility of this approach by considering all aspects from start to finish in making an AM component and deploying the same. The proposed qualification methodologies integrate legacy knowledge

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Spherical pores as 'microstructural informants': Understanding compositional, thermal, and mechanical gyrations in additively manufactured Ti-6Al-4V

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ABSTRACT

Detailed analysis of defects such as spherical porosity can act as informants, providing some information regarding the complex and often hidden physics associated with additive manufacturing. Variation in the presence and nature of these defects can shed new insights into the AM process. In this paper, the compositional, crystallographic, microstructural, and morphological characteristics surrounding gas pores in Electron Beam Melted Ti-6Al-4V have been assessed and correlated with different scanning strategies (raster and two point melting ones, Dehoff and random). The large spherical pores (>25μm), exclusively present in raster scan, exhibit perturbations normal to the vertical sidewalls of the pores that are likely the result of elastic instabilities resulting from chemical and crystallographic variations and initiated by vertical compression caused by thermal stresses related to the cyclic process – effectively a form of microbuckling. Electron backscatter diffraction maps support the theory that these perturbations occur at elevated temperatures and prior to the final solid-solid phase transformation.

Additive manufacturing (AM) has many advantages relative to traditional manufacturing processes [1–6], including the ability to produce net- or near-net-shape parts, the potential to eliminate processing steps, faster times between part conception to realization, lower buy-to-fly ratios, and the ability to produce complex structures with the desired topology or composition gradient. Despite these benefits, the presence of different types of porosity and defects in AM builds has been a limiting factor when it comes to the widespread commercial implementation of these products [1,7,8].

Porosity is generally viewed as a deleterious feature, reducing the ability of a part to withstand the intended loads during service, leading to premature failure or for the part to be rejected during quality control assessments prior to use [9–12]. Typical solutions when managing/eliminating porosity [13–16] include optimizing and/or redefining the manufacturing process (and/or the process variables), redesigning the part or sections of it, post-processing to remove zones with defects (if they are consistently in the same location), changing the selected material, or incorporating a 'knock down' factor on some critical property. There exists a relatively robust body of research on the effects of various sizes and fractions of porosity on the attending mechanical properties in AM builds [8,11,17].

Despite the drawbacks of porosity in the engineering behavior of a part, these defects can serve as microstructural "informants", revealing considerable information on the processing-microstructure relationships that exist during AM processes, the mechanisms that create these defects, and the correlations between process parameters, location, and other dynamics of the additive manufacturing process, including compositional, thermal, microstructural, and mechanical gyrations. What makes pores, especially spherical pores originating from the presence of a gas of some type, behave as high-fidelity informants is that they: are defined by the presence of a free surface/volume; are distinguishable from the surrounding microstructure; and, behave predictably given that they are partially removed from the constraint of adjacent material. This paper investigates the types of details that these microstructural informant spherical pores can reveal regarding the additive manufacturing process, specifically the ARCAM electron beam melting (EBM) powder bed fusion (PBF) method.

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Role of thermo-mechanical gyrations on the α/β interface stability in a Ti6Al4V AM alloy[☆]

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ABSTRACT

Fluctuating energy distributions experienced during Additive Manufacturing yield an evolution of spatial and temporal transients within a part. In general, the in-situ monitoring of these transients is near to impossible during manufacturing. In order to then gain perspective into the impact on these localized thermo-mechanical transients on the interface stability, rapid thermo-mechanical reversals with known boundary conditions are imposed on an AM Ti6Al4V alloy which resulted in a phase transformation leading to an increased β phase stability. Our goal with this study is to comprehend the kinetics of this phase transformation with concepts of stored energy due to plastic strain accumulation and diffusion kinetics. Atom Probe Tomography is employed to study the partitioning of the solute elements across the interface. As expected, the thermo-mechanically cycled samples showed a reduced Vanadium concentration across the β phase. This concentration profile across the interface, alongside a full-width-half-max analysis, provided insight on the potential phase transformation kinetics involved in the $\alpha \rightarrow \beta$ transformation subject to thermo- mechanical gyrations.

Additive Manufacturing (AM) is ideal for the fabrication of complex geometries with less material waste and unique properties [1]. As a result of the incremental layer-by-layer building of a part, each and every voxel within the part experiences multiple thermal gyrations between the liquidus, solidus, and the pre-heat temperatures. These signatures are also complicated by spatially and temporally varying heating and cooling rates. In principle, these gyrations provide opportunities for site-specific control of microstructures within these voxels. A classic example of site-specific control of solidification texture was achieved by alternating the AM melt scan path between linear- raster to spot-fill patterns [2]. With an emerging geometry, there occurs a remelt of layers and multiple thermal gyrations, leading to spatial and temporal signatures evolving in the part [3–6]. These rapid and fluctuating evolving transients are prone to dynamic mechanical constraints and the non-linear interactions may result in complex thermo-mechanical conditions, i.e., transient variations of thermal stress between compressive and tensile and associated plastic strains. This research explores the role of these thermo-mechanical-metallurgical interactions on phase stability in the Ti6Al4V alloy. We postulate that these thermo-mechanical gyrations can provide additional control of the overall microstructure and properties by manipulating the solid-state phase transformation, as well as plastic strain distributions within these phases. Although one can attempt to validate this hypothesis by a detailed 3D characterization of AM parts, accurate correlation of the thermo-mechanical signatures to microstructural features still eludes us due to the enormity of the characterization task and the need for high-fidelity modeling/sensing of thermo-mechanical signatures at the same locations [7–12]. Therefore, we have developed an alternate pathway

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Microstructure-property gradients in Ni-based superalloy (Inconel 738) additively manufactured via electron beam powder bed fusion

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ABSTRACT

Electron beam powder bed fusion (E-PBF) can produce nickel-based superalloy components with minimal cracking and post-processing. This is due to the reduced thermal gradients and high print temperatures accessible through innovative beam scanning strategies compared to other AM processes. However, variations in thermal signature along the build direction inherent in alloys printed using E-PBF can drive significant changes in the microstructure and associated mechanical properties. In this work, through complementary local property measurements we observed a 127 – 145% increase in mean elastic modulus and 7–9% increase in mean hardness, as a function of build height, of an as-fabricated non-weldable Ni-based superalloy, Inconel 738. These properties were attributed primarily to variations in the γ' character with build height, revealed through a multi-scale microstructural characterisation. The results highlight the influence of the thermal signatures on the microstructural-property relationships of E-PBF Inconel 738.

1. Introduction

Inconel-738 (IN738) is a Ni-based superalloy used predominantly for turbine blades in the high-temperature compressive stage of stationary gas turbines and turbojet engines [1]. IN738 is a precipitation-hardened alloy that routinely operates at temperatures up to ~980 °C due to its excellent high-temperature performance (e.g.) high creep rupture, yield strength, and improved hot corrosion, oxidation and sulfidation resistance [2]. Its high-temperature strength is predominantly derived from the precipitation of the coherent ordered $L1_2$ intermetallic $Ni_3(Al,Ti)$ γ' phase within a face-centred cubic (FCC) γ matrix [3–5]. Substantial strengthening is imparted by γ', restricting $< 110 >$ dislocations from entering γ' without the formation and subsequent removal of an anti-phase boundary (APB) [3]. Dislocations must travel through the γ' phase in pairs, with the second dislocation removing the APB introduced by the first. The creation of APBs incurs an significant energy increase and is a great barrier for glissile dislocations [6]. In addition, solid solution strengthening of the γ phase is obtained from Cr, Co and refractory metals including Nb, Ta, Mo and W, whilst grain boundary strengthening is also obtained from Zr, borides, and carbides [3,4,7].

As the presence of γ' provides the requisite elevated temperature strength, much effort has been devoted to the production of superalloys with high γ' volume fractions (55–70%) that can withstand increasingly higher temperatures and stresses [8]. It is well known that 'non-weldable' superalloys containing high amounts of γ', such as IN738, conventionally suffer from poor castability and weldability due to their high Al and Ti content, which makes them prone to substantial solidification cracking, liquation and strain-age cracking [9–13]. Therefore, it is desirable to overcome these issues by minimizing both mechanical and metallurgical drivers for these cracks [14].

Additive manufacturing (AM) provides an avenue to produce crack and defect-free parts from non-weldable Ni-based superalloys by controlling the thermo-mechanical conditions [15]. Superalloys such as IN738 are amenable to gas or water atomisation for feedstock to the printing process [16]. There have been many studies optimising the printing parameters to limit solidification and liquation cracking of AM non-weldable superalloys [16–18]. Though IN738 has been successfully printed by laser powder bed fusion (L-PBF) and directed energy

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An integrated manifold learning approach for high-dimensional data feature extractions and its applications to online process monitoring of additive manufacturing

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ABSTRACT

As an effective dimension reduction and feature extraction technique, manifold learning has been successfully applied to high-dimensional data analysis. With the rapid development of sensor technology, a large amount of high-dimensional data such as image streams can be easily available. Thus, a promising application of manifold learning is in the field of sensor signal analysis, particular for the applications of online process monitoring and control using high-dimensional data. The objective of this study is to develop a manifold learning-based feature extraction method for process monitoring of Additive Manufacturing (AM) using online sensor data. Due to the non-parametric nature of most existing manifold learning methods, their performance in terms of computational efficiency, as well as noise resistance has yet to be improved. To address this issue, this study proposes an integrated manifold learning approach termed multi-kernel metric learning embedded isometric feature mapping (MKML-ISOMAP) for dimension reduction and feature extraction of online high-dimensional sensor data such as images. Based on the extracted features with the utilization of supervised classification and regression methods, an online process monitoring methodology for AM is implemented to identify the actual process quality status. In the numerical simulation and real-world case studies, the proposed method demonstrates excellent performance in both prediction accuracy and computational efficiency.

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KEYWORDS
Additive manufacturing; integrated manifold learning; isometric feature mapping (ISOMAP); multi-kernel metric learning (MKML); online process monitoring

1. Introduction

With the ever-increasing demands on product complexity and accuracy, sensor-based quality control techniques play a critical role in manufacturing industries. Currently, fast-improving sensor technologies are capable of generating a massive amount of real-time process data, providing excellent opportunities to achieve effective quality assurance through data analytics. However, due to process and design complexities, as well as an inherent noisy environment, providing high accuracy analysis with a satisfactory computational efficiency for the online sensor data still remains a major challenge, particularly for high-dimensional data such as images (Tootooni *et al.*, 2016; Liu, Law, *et al.*, 2019). Consequently, the objective of this study is to develop an effective feature extraction methodology for high-dimensional sensor data and then apply it to online process monitoring of an Additive Manufacturing (AM) process.

Based on the rapid development of machine learning techniques in recent decades, it has become highly effective to identify the process quality status based on sensor data using appropriate supervised classification or regression methods (Rao *et al.*, 2015; Bastani *et al.*, 2016). However, since sensor data has a high dimensionality and also is contaminated with certain levels of noise, classification analysis using the raw data or ineffective feature extraction of the data may cause inaccurate results in real-world applications such as AM and other advanced manufacturing processes, e.g., results in a large number of false alarms or an extremely high miss detection rate. Therefore, a noise-resistant dimension reduction and feature extraction method is urgently required.

To achieve this goal, this article proposes a new approach for feature extraction and dimension reduction, termed Multi-Kernel Metric Learning-embedded ISOmetric feature MAPping (MKML-ISOMAP) based on the manifold learning method (Cayton, 2005), which is known for its effectiveness in nonlinear data dimension reduction. This approach aims to overcome the above-mentioned shortcomings by achieving the following two major goals:

1. From the methodological point of view, it addresses the limitations existing in the current manifold learning literature and improves the feature extraction accuracy and computational efficiency for high-dimensional data analysis.

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TECHNICAL ARTICLE

MAUD Rietveld Refinement Software for Neutron Diffraction Texture Studies of Single- and Dual-Phase Materials

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Abstract

This work presents a detailed instructional demonstration using the Rietveld refinement software MAUD for evaluating the crystallographic texture of single- and dual-phase materials, as applied to High-Pressure-Preferred-Orientation (HIPPO) neutron diffraction data obtained at Los Alamos National Laboratory (LANL) and electron backscatter diffraction (EBSD) pole figures on Ti–6Al–4V produced by additive manufacturing. This work addresses a number of hidden challenges intrinsic to Rietveld refinement and operation of the software to improve users' experiences when using MAUD. A systematic evaluation of each step in the MAUD refinement process is described, focusing on devising a consistent refinement process for any version of MAUD and any material system, while also calling out required updates to previously developed processes. A number of possible issues users may encounter are documented and explained, along with a multilayered assessment for validating when a MAUD refinement procedure is finished for any dataset. A brief discussion on appropriate sample symmetries is also included to highlight possible oversimplifications of the texture data extracted from MAUD. Included in the appendix of this work are two systematic walkthroughs applying the process described. Files for these walkthroughs can be found at the data repository located at: https://doi.org/10.18434/mds2-2400.

Keywords Rietveld refinement · MAUD · Crystallographic texture · Ti–6Al–4V · Tutorial · Additive manufacturing

Introduction

The Rietveld refinement software known as Material Analysis Using Diffraction (or MAUD for short) [1] is a powerful tool for evaluating crystallographic texture and crystallographic structure across a wide range of material systems. Employing an iterative least-squares minimization fitting technique to refine calculated diffraction spectra to experimental data, MAUD can refine both neutron diffraction and X-ray diffraction (XRD) results. One of the features developed in MAUD is a "wizard" to analyze data acquired from the High-Pressure-Preferred-Orientation (HIPPO) neutron diffraction beamline at the Lujan Center at the Los Alamos Neutron Science Center (LANSCE) at Los Alamos National Laboratory (LANL).

Previously written documentation contains example spectra and gives an effective overview of using MAUD [2, 3]. Wenk et al. and Lutterotti both provide insights into the "behind the scenes" operations within MAUD, and explain the internal mechanisms of the software, while showcasing an example refinement for users to follow. However, challenges encountered by the authors using these tutorials, especially when analyzing two-phase materials, identified a gap in the current literature. Since the writing of prior tutorials [1, 2], HIPPO has been upgraded with increased detection capabilities, leaving some steps in previous tutorials

Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

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Clustered Discriminant Regression for High-Dimensional Data Feature Extraction and Its Applications in Healthcare and Additive Manufacturing

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***Abstract*— The recent increase in applications of high-dimensional data poses a severe challenge to data analytics, such as supervised classification, particularly for online applications. To tackle this challenge, efficient and effective methods for feature extraction are critical to the performance of classification analysis. The objective of this work is to develop a new supervised feature extraction method for high-dimensional data. It is achieved by developing a clustered discriminant regression (CDR) to extract informative and discriminant features for high-dimensional data. In CDR, the variables are clustered into different groups or subspaces, within which feature extraction is performed separately. The CDR algorithm, which is a greedy approach, is implemented to obtain the solution toward optimal feature extraction. One numerical study is performed to demonstrate the performance of the proposed method for variable selection. Three case studies using healthcare and additive manufacturing data sets are accomplished to demonstrate the classification performance of the proposed methods for real-world applications. The results clearly show that the proposed method is superior over the existing method for high-dimensional data feature extraction.**

***Note to Practitioners*—This article forwards a new supervised feature extraction method termed clustered discriminant regression. This method is highly effective for classification analysis of high-dimensional data, such as images or videos, where the number of variables is much larger than the number of samples. In our case studies on healthcare and additive manufacturing, the performance of classification analysis based on our method is superior over the existing feature extraction methods, which is confirmed by using various popular classification algorithms. For image classification, our method with elaborately selected classification algorithms can outperform a convolutional neural network. In addition, the computation efficiency of the proposed method is also promising, which enables its online applications, such as advanced manufacturing process monitoring and control.**

***Index Terms*— Additive manufacturing (AM), classification analysis, clustering, discriminant regression (DR), greedy algorithm, healthcare, variable selection.**

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NOMENCLATURE

N	Number of training samples.
P	Number of variables.
K	Number of clusters.
L	Number of class labels.
i, j, n	Index of variables, clusters, and samples.
$\mathbf{X}$	$\mathbb{R}^{N\times P}$ data matrix.
$\mathbf{X}_i$	ith column of $\mathbf{X}$.
$\mathbf{x}_n$	nth row of $\mathbf{X}$.
t_n	Label of $\mathbf{x}_n$, $t_n \in \{1, \dots, L\}$.
$\mathbf{Y}$	Label indicator matrix $\mathbf{Y} \in \mathbb{R}^{N\times L}$.
$\mathbf{Y}_t$	tth column of $\mathbf{Y}$.
G_j	Set of index of variables in jth cluster.
G_j^*	Optimal set of index of variables in jth cluster.
$\mathcal{G}$	Partition (or clustering) result $\{G_1, \dots, G_K\}$.
λ_j	Regularization coefficient for jth cluster.
$\mathbf{X}_{G_j}$	Data matrix only consists of variables index G_j.
$\mathbf{\Phi}_{G_j}$	$\mathbf{I}_N - \mathbf{X}_{G_j}(\mathbf{X}_{G_j}^\top \mathbf{X}_{G_j} + N\lambda_j \mathbf{I}_{\lvert G_j\rvert})^{-1}\mathbf{X}_{G_j}^\top$.
g_j	Cardinality constrain for jth cluster.
$\mathcal{W}$	Set of projection matrices from each cluster.
$\mathbf{W}_{G_j}$	Projection matrix for jth cluster.
$\lVert\cdot\rVert_F$	The Frobenius norm of a matrix.
$\mathrm{Tr}(\cdot)$	Trace of a matrix.
$\mathbf{I}_P$	$P \times P$ identicial matrix.
$\mathbf{F}_{G_j}$	Feature extracted from jth cluster.
$\mathbf{F}$	Final feature set $\mathbf{F} = (\mathbf{F}_{G_1}, \mathbf{F}_{G_2}, \dots, \mathbf{F}_{G_K})$.
$\sigma_{\max}(\cdot)$	Largest eigenvalue of a matrix.
$\sigma_{\min}(\cdot)$	Smallest eigenvalue of a matrix.
θ_g	$= \max_{\lvert G\rvert = g} \sigma_{\max}(\mathbf{X}_G \mathbf{X}_G^\top)$.
g	$g = \max_{j\in[K]} g_j$.
$\underline{\lambda}$	$= \min_{j\in[K]} \lambda_j$.
$\overline{\lambda}$	$= \max_{j\in[K]} \lambda_j$.
β	$= \max_{j\in[K]}(\theta_{g_j}/N\lambda_j)$.
$\underline{\theta}$	$= \min_{G\in[P],\lvert G\rvert\ge P-\sum_{j=1}^K g_j} \sigma_{\min}(\mathbf{X}_G \mathbf{X}_G^\top)$.
γ	$= (\underline{\theta}/(K(N\overline{\lambda} + \theta_1)(1+\beta)^2)) \log((P+1)/(P+1-\sum_{j=1}^K g_j))$.
δ	Assignment matrix $\delta \in \{0,1\}^{P\times K}$.
v^G	Objective value from greedy algorithm.
v^*	Optimal objective value.

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RESEARCH ARTICLE

MICROSCOPY RESEARCH & TECHNIQUE WILEY

Quantification of extreme thermal gradients during in situ transmission electron microscope heating experiments

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Abstract

Studies on materials affected by large thermal gradients and rapid thermal cycling are an area of increasing interest, driving the need for real time observations of microstructural evoultion under transient thermal conditions. However, current in situ transmission electron microscope (TEM) heating stages introduce uniform temperature distributions across the material during heating experiments. Here, a methodology is described to generate thermal gradients across a TEM specimen by modifying a commercially available MEMS-based heating stage. It was found that a specimen placed next to the metallic heater, over a window, cut by FIB milling, does not disrupt the overall thermal stability of the device. Infrared thermal imaging (IRTI) experiments were performed on unmodified and modified heating devices, to measure thermal gradients across the device. The mean temperature measured within the central viewing area of the unmodified device was 3–5% lower than the setpoint temperature. Using IRTI data, at setpoint temperatures ranging from 900 to 1,300°C, thermal gradients at the edge of the modified window were calculated to be in the range of 0.6×10^6 to 7.0×10^{6}°C/m. Additionally, the Ag nanocube sublimation approach was used, to measure the local temperature across a FIB-cut Si lamella at high spatial resolution inside the TEM, and demonstrate "proof of concept" of the modified MEMS device. The thermal gradient across the Si lamella, measured using the latter approach was found to be 6.3×10^{6}°C/m, at a setpoint temperature of 1,000°C. Finally, the applicability of this approach and choice of experimental parameters are critically discussed.

KEYWORDS
in situ heating, in situ TEM, nonequilibrium processes, thermal gradient, thermometry

1 | INTRODUCTION

In situ heating holders are widely used to observe thermally activated processes inside a transmission electron microscope (TEM) at high spatial resolution (Butler, 1979; Jungjohann & Carter, 2016). Early in situ TEM heating experiments were performed using furnace type heating stages, which have a large thermal mass (Butler, 1979). Observations of dynamic processes at high spatial resolutions were challenging, as the differences in thermal conductivity and thermal expansion coefficient between the "sample cup" around the furnace heater and the sample created a thermal lag, which increased thermal equilibration times, and thereby, increased sample drift. This was a major limitation while observing the initial stages of a thermally activated process during an in situ TEM heating experiment, as critical information was lost before the sample thermally equilibrated. The new generation of micro-electro-mechanical systems (MEMS)-based heating holders were developed to mitigate these limitations and are, therefore, nowadays widely used for in situ TEM studies (Allard

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ORIGINAL REPORT

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Introducing transformation twins in titanium alloys: an evolution of α-variants during additive manufacturing

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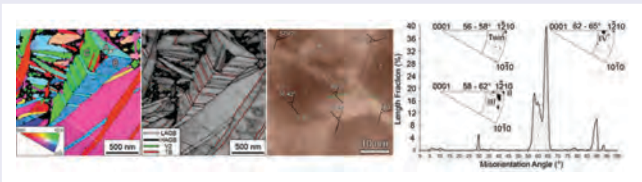
ABSTRACT

Titanium alloys can experience a cooling-induced phase transformation from a body-centred cubic phase into a hexagonal close-packed phase which occurs in 12 crystallographically equivalent variants. Among them, variant selection II, 60°/$\langle 1\bar{2}10\rangle$, is very close to the orientation of $\{10\bar{1}1\}\langle 1\bar{2}10\rangle$ twins (57.42°/$\langle 1\bar{2}10\rangle$). We propose that the cyclic thermal loading during additive manufacturing introduces large thermal stresses at high temperature, enabling grain reorientation that transforms the 60°/$\langle 1\bar{2}10\rangle$ variant boundaries into the more energetically stable 57.42°/$\langle 1\bar{2}10\rangle$ twin boundaries. This transformation twinning phenomenon follows a strain accommodation mechanism and the resulting boundary structure benefits the mechanical properties and thermal stability of titanium alloys.



IMPACT STATEMENT

A new twinning mechanism, transformation twinning, was discovered in a Ti–6Al–4V alloy fabricated by selective laser melting. The resulting high density of transformation twins impact the global mechanical properties significantly.

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KEYWORDS
Titanium alloy; additive manufacturing; twinning mechanism; variant selections

1. Introduction

Most structural alloys are polycrystalline materials with different types of grain boundaries (GBs). The structures of GBs have an undisputable effect on the mechanical properties of materials. A special type of GBs is twin boundaries (TBs) that separate twins and their matrix. While conventional GBs with random orientation relationships between neighbouring grains hamper the motion of dislocations and therefore strengthen materials, they usually deteriorate the ductility and thermal stability of materials. In contrast, TBs can simultaneously enhance the strength and ductility and do not deteriorate the thermal stability of materials [1–9]. As such, TBs are preferred over random GBs. Depending on their formation mechanism, twins can be classified as growth twins, annealing twins, or deformation twins.

Titanium (Ti) alloys have received significant attention due to their broad applications [10]. Most Ti alloys undergo a phase transformation from the high-temperature body-centred cubic (BCC) β phase to the low temperature hexagonal close-packed (HCP) α (or α') phase during cooling. The orientations of the resulting HCP grains relative to the BCC parent grains are governed by the Burgers orientation relationship [11],

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ORIGINAL ARTICLE

Development of structured light 3D-scanner with high spatial resolution and its applications for additive manufacturing quality assurance

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Abstract
Digital three-dimensional (3D) scanning is a cutting-edge metrology method that can digitally reconstruct surface topography with high precision and accuracy. Such metrology can help traditional manufacturing processes evolve into a smart manufacturing paradigm, which can ensure product quality by automated sensing and control. However, due to limitations with the spatial resolution, scanning speed, and size of the focusing area, commercially available systems cannot be directly used for in-process monitoring in smart manufacturing. For example, a metal 3D printer requires a scanner with second-level sensing, micron-level spatial resolution, and a centimeter-scale scanning region. Among the 3D scanning technologies, structured light 3D scanning can meet the scanning speed criteria but not the spatial resolution and scanning region criteria. This work addresses these challenges by reducing the field of view of a structured light scanner system while increasing the image sensor pixel resolution. Improvements to spatial resolution and accuracy are achieved by establishing hardware selection criteria, integrating the proper hardware, designing a scale-appropriate calibration target, and developing noise reduction procedures during calibration. An additively manufactured Ti-6Al-4V part was used to validate the effectiveness of the proposed 3D scanner. The scanning result shows that both melt pool geometry and overall shape can be clearly captured. In the end, the scanning accuracies of the proposed scanner and a professional-grade commercial scanner are validated with a nanometer-level accuracy white light interferometer using high-density point cloud data. Compared to the commercial scanner, the proposed scanner improves the spatial resolution from 48 to 5 μm and the accuracy from 108.5 to 0.5 μm. Compared to the white light interferometer, the proposed scanner improves the scanning and processing speed from 2 to 20 s.

Keywords 3D scanning · Structured light · Sensing · Surface metrology · Resolution improvement · Noise reduction · Calibration · Point cloud analysis · Additive manufacturing · and Smart manufacturing

1 Introduction

1.1 Background

In advanced manufacturing, automatic quality control is a key enabler for defect detection and mitigation based on sensor technologies. Additive manufacturing (AM) provides a novel way to fabricate parts by layer-wise addition of material [1]. However, its sustainability is constrained by the inherent limitations of layer-by-layer fabrication, leading to numerous defects such as balling, porosity, and distortion [2]. Therefore, online layer-wise monitoring is an important research area since defects that occur during printing may severely deteriorate the product quality [2]. The 3D surface topological information for each layer usually includes critical quality information, such as melt pool size, surface roughness, pores, other defects, or unexpected process alterations. For example, Parry et al. [3] indicated that the melt pool size directly correlates with penetration depth, residual stress, and overall geometry precision. The 3D surface topological information can be obtained by 3D scanning, which is a group

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Review

Additive manufacturing of steels: a review of achievements and challenges

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ABSTRACT

Metal additive manufacturing (AM), also known as 3D printing, is a disruptive manufacturing technology in which complex engineering parts are produced in a layer-by-layer manner, using a high-energy heating source and powder, wire or sheet as feeding material. The current paper aims to review the achievements in AM of steels in its ability to obtain superior properties that cannot be achieved through conventional manufacturing routes, thanks to the unique microstructural evolution in AM. The challenges that AM encounters are also reviewed, and suggestions for overcoming these challenges are provided if applicable. We focus on laser powder bed fusion and directed energy deposition as these two methods are currently the most common AM methods to process steels. The main foci are on austenitic stainless steels and maraging/precipitation-hardened (PH) steels, the two so far most widely used classes of steels in AM, before summarising the state-of-the-art of AM of other classes of steels. Our comprehensive review highlights that a wide range of steels can be processed by AM. The unique microstructural features including hierarchical (sub)grains and fine precipitates induced by AM result in enhancements of strength, wear resistance and corrosion resistance of AM steels when compared to their conventional counterparts. Achieving an acceptable ductility and fatigue performance remains a challenge in AM steels. AM also acts as an intrinsic heat treatment, triggering 'in situ' phase transformations including tempering and other precipitation phenomena in different grades of steels such as PH steels and tool steels. A thorough discussion of the performance of AM steels as a function of these unique microstructural features is presented in this review.

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Nima Haghdadi, Majid Laleh and Maxwell Moyle contributed equally and are therefore listed in alphabetical order.

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Grain boundary character distribution in an additively manufactured austenitic stainless steel

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ABSTRACT

The grain boundary character distribution (GBCD) in an austenitic stainless steel produced by additive manufacturing (AM) in both as-built and annealed conditions was studied. Relatively fine grains and a non-fibre texture was achieved by AM, and as-built structure showed a high population of Σ3 boundaries. A five-parameter GBCD analysis revealed that the microstructure is mostly dominated by highly incoherent Σ3 boundaries. The grain boundary network also consisted of random high angle, coherent Σ3s terminating on (111) planes with a pure twist character, and tilt Σ9 boundaries. The findings show prospects for the possibility of engineering the grain boundary network of materials *in-situ*, via the stress and heat induced by the thermal cycles during AM.

Laser powder bed fusion (LPBF) is an additive manufacturing (AM) technology that uses a high-energy laser beam to melt powder particles to consolidate a metallic part [1,2]. Upon laser irradiation of a powder bed, a melt pool forms and then solidifies at a very fast rate (up to 10^7 K/s [3]). Individual tracks join together during succeeding laser passes to form a 3D part. During LPBF, the material experiences complex thermal gradients and gyrations which subsequently result in a unique microstructure. These unique microstructural features can lead to superior mechanical properties [4,5] and corrosion resistance [6–8] e.g. in LPBF austenitic stainless steels, exceeding those of their traditionally manufactured counterparts. A current focus in the metal AM research community is directed towards understanding such complex microstructures in order to develop metals and alloys with optimised properties of interest by altering processing variables [9].

Grain boundary engineering (GBE) has been the subject of intensive research during the last 40 years with the aim of introducing coincident site lattice (CSL) and low-angle grain boundaries (GBs) to mitigate undesirable intergranular phenomena such as corrosion, embrittlement and fracture [10–14]. While GBE has been studied for traditionally manufactured stainless steels, the potential of the thermomechanical hysteresis that a material experiences during AM for engineering microstructures is yet to be explored. Typical anisotropic microstructures reported in AM materials consisted of grains elongated towards the build direction with <100> and/or <110> texture [15,16]. Such morphology can be broken down via a change in laser scanning strategy, where a hierarchical microstructure can be formed that offers outstanding mechanical properties achieved either through microsegregation resulting from cellular solidification and/or via a network of dislocation-rich sub-boundaries [4,17]. It can be hypothesized that engineering the grain boundary network in austenitic stainless steel via AM may unlock additional unique properties in terms of corrosion, embrittlement and fracture resistance. This is because diffusivity, mobility, and segregation of a grain boundary are affected by its crystallography. However, an analysis of the formation mechanism and a detailed knowledge of the crystallographic character of CSL grain boundaries in AM microstructures is currently lacking. The current work reports on the possibility of *in-situ* GBE during AM of an austenitic stainless steel, and provides a full five-parameter macroscopic description of these boundaries. The results can be extended to several other face centred cubic (FCC) metals with low-to-medium stacking fault energy (SFE).

Argon-atomised 316L austenitic stainless steel (hereinafter 316L SS) powder with particle sizes between 5 and 45 μm was used. Cubes (10 × 10 × 10 mm^3) were produced using a LPBF machine

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Full length article

Phase transformation pathways in Ti-6Al-4V manufactured via electron beam powder bed fusion

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ABSTRACT

The design of additively manufactured metallic alloys with tailored mechanical properties requires a detailed understanding of the microstructural evolution throughout the printing process. In Ti-6Al-4V, this involves a complex combination of phase transformations, leading to microstructural and property variations within a single as-fabricated build. The origin of such property variations and the sequence of phase changes occurring during the cyclic heating and cooling process remain uncertain. We have studied the phase transformation pathway by following how, in particular, the β phase growth varies within the build. Samples manufactured by electron beam powder bed fusion were analysed using electron microscopy and atom probe tomography techniques. We demonstrate that a significant β phase fraction variation occurs within a given build plane. We reveal that the high-temperature β phase can be separated into two categories, depending on whether it was retained from cooling from above the β transus temperature, or nucleated below it. This is the first direct evidence of the coexistence of both types of β transformation products in Ti-6Al-4V. The abrupt cyclic nature of the additive manufacturing process is what has facilitated this unusual transformation sequence. The work provides a complete and general description of the phase transformation pathway, informed by these observations. The implication of the phase transformation pathway on hardness is discussed in relation to chemical variation and oxygen pickup.

1. Introduction

Ti-6Al-4V is the most widely used of the titanium alloys in the aerospace and biomedical industries, due to its high strength-to-weight ratio [1,2] and excellent corrosion resistance [3]. The alloy is traditionally made through well-established methods such as forging and casting, with careful control over thermo-mechanical processing to tune the microstructure and achieve high strength and ductility. These methods have high production costs and material wastage [4,5], and often require extensive and complex machining to final shapes and dimensions [6]. Additive manufacturing (AM) is an emerging technology that enables near net shape production of complex parts at a low cost with minimal material waste in a burgeoning range of industrial applications. amongst the most common of the metal AM processes is powder bed fusion (PBF), whereby powder feedstock resting on a bed is selectively melted by either a laser (L-PBF) or an electron (E-PBF) beam. Ti-6Al-4V is amenable to both L-PBF and E-PBF processes as it is highly weldable, having a capacity to generate fine-scale microstructures upon rapid cooling [7] due to its narrow freezing range (5 K) [8]. Many details about the stability of interfaces and the evolution of phase transformations in these highly non-equilibrium processes remain unstudied.

The well-known extreme nature of the thermo-mechanical conditions associated with AM can lead to undesirable microstructures in the as-fabricated builds. Steep thermal gradients and rapid cooling rates associated with laser-based additive manufacturing can lead, for example, to the formation of a fully martensitic (α') microstructure in Ti-6Al-4V [9–11]. The resultant ductility is well below that required in many critical structural applications

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Research Paper

Solidification texture, variant selection, and phase fraction in a spot-melt electron-beam powder bed fusion processed Ti-6Al-4V

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ABSTRACT

The effect of a spot melt strategy on the solidification texture, variant selection, phase fraction, and their variations along the build height was investigated in comparison to a conventional linear melt strategy. Ti-6Al-4V alloy samples were manufactured by an electron-beam powder bed fusion process using two different melt strategies and characterized using high-energy synchrotron x-ray diffraction. The linear and spot melt cases resulted in β<100> cube and fiber textures, respectively, with the cube/fiber axis nearly parallel to the build direction. The α phase exhibited Burgers orientation relationship (BOR) with the parent β phase for both melt strategies. Although all of the key texture components and the BOR between the α and β phases were consistently observed in both cases along the build height, there were several measurable differences between the two. At the top of the build height, the β<100> build texture intensity was stronger in the linear case. However, the texture intensity in the linear case decreased gradually from the top to near the substrate, whereas the spot melt case did not show this trend. Moreover, the BOR was examined along the build height and the results showed that there is a distinct variant selection in the spot melt case, unlike the linear case. Specifically, the planar variants sharing $(\bar{1}01)_\beta \parallel (0002)_\alpha$ with $[\overline{222}]_\beta$ and $[2\bar{2}2]_\beta$ were the most prominent in the spot melt, but no preference was identified in the directional variants. The β phase fraction was slightly lower in the spot melt case compared to the linear case. In both the cases, the β phase fraction decreased moving towards the substrate but remained significantly higher than that of the powder feedstock. Overall, the novel spot melt strategy produced a more homogeneous microstructure in terms of both the phase fraction and texture across the build height.

1. Introduction

Metal additive manufacturing (AM) is a novel processing technique wherein the metallic feedstock is deposited layer-by-layer to produce a desired component. The key advantages that AM offers are significant reduction in material waste and enabling of complex part design intractable by conventional metal casting and forming techniques. The process optimization for the control of microstructure, porosity, and part distortion has been studied for various alloy systems, including the Fe, Ti, Ni, and Al alloys, using fusion-based AM techniques, such as L-PBF (laser - powder bed fusion), E-PBF (electron-beam powder bed fusion) and DED (direct energy deposition) techniques [1]. One of the challenges involved in the advancement of fusion-based AM techniques, in terms of the basic understanding of the relationship between the microstructure and key processing parameters, is the complexity in the local thermal history.

Ti-6Al-4V alloy (Ti-64) is a α + β dual-phase titanium alloy used in the aerospace, chemical, and biomedical industries due to its high strength-to-weight ratio and corrosion resistance. Recently, the processing-microstructure relationships have been studied extensively for the AM Ti-64 manufactured using PBF and DED systems [2,3]. The processing parameters, such as energy density, input power, scan speed, and scan strategy, influence microstructural features [4] and defect distribution [5]. The microstructure is also influenced by the build size and geometry, thereby resulting in local variations in microstructure and subsequent mechanical response within a complex part [6–9].

Crystallographic texture plays an important role in determining the tensile [10,11], fatigue [11–13], fracture [14], and corrosion [15] behavior of Ti-6Al-4V components. For example, the preferred alignment of basal (0002) planes perpendicular to the loading axis results in

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Texture evolution as a function of scan strategy and build height in electron beam melted Ti-6Al-4V☆

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A B S T R A C T

Metal additive manufacturing (AM) enables customizable, on-demand parts, allowing for new designs and improved engineering performance. Yet, the ability to control AM metal alloy microstructures (i.e., grain morphology, crystallographic texture, and phase content) is lacking. This work performs corroborative neutron diffraction and large-scale electron backscatter diffraction (EBSD) measurements to assess crystallographic texture in electron beam melted (EBM) Ti-6Al-4V as a function of scan strategy and build height. Texture components for one raster and two spot melt scan strategies were evaluated using a triclinic specimen symmetry to capture all possible texture components, which were found to be considerably different than previously reported values from studies employing orthotropic specimen symmetry. This finding highlights the importance of a standard method and best practice for assessing textures produced by AM. Texture was found to vary between scan strategies, but changed minimally as a function of build height. Parent phase β-Ti reconstructions obtained from as-built crystallographic orientations revealed spot melt scan strategies produced finer equiaxed/columnar grains with clear 001_β build direction fiber textures, whereas the raster scan strategy produced large columnar grains and a weaker 001_β build direction fiber texture. The observed grain morphologies agree with those predicted by solidification theory for the thermal gradients and solidification velocities experienced during the build process. The presence of a strong 001_β fiber orientation (typical of cubic solidification) produced by spot melting was found to correlate with a previously unreported $01\bar{1}2_\alpha$ fiber texture in the as-built condition and colony microstructures. The $01\bar{1}2_\alpha$ fiber texture was weakly observed for the raster scan strategy, and 001_β oriented grains preferentially transformed into α' martensite with orientations between $1\bar{1}00_\alpha$ and $11\bar{2}0_\alpha$. This shift in product α-Ti orientations has not yet been reported, and further work is recommended to understand these crystallographic signatures in the context of solid-state phase transformations. The presence of the $01\bar{1}2_\alpha$ fiber texture is proposed as a useful diagnostic for evaluating the solidification or transformed microstructure condition (e.g., grain morphology and texture) of Ti-6Al-4V AM builds via accessible techniques like laboratory X-ray diffraction.

1. Introduction

Additive manufacturing (AM) of metallic alloy components has advanced considerably over the past three decades, with breakthroughs in technologies, control systems, feedstock development, and build quality across processes like laser beam (LBPBF) powder bed fusion,

☆ Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

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3D electron backscatter diffraction characterization of fine α titanium microstructures: collection, reconstruction, and analysis methods

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ABSTRACT

3D electron backscatter diffraction (3D-EBSD) is a method of obtaining 3-dimensional crystallographic data through serial sectioning. The recent advancement of using a Xe^+ plasma focused ion beam for sectioning along with a complementary metal-oxide semiconductor based EBSD detector allows for an improvement in the trade-off between volume analyzed and spatial resolution over most other 3D characterization techniques. Recent publications from our team have focused on applying 3D-EBSD to understand microstructural phenomena in Ti-6Al-4V microstructures as a function of electron beam scanning strategies in electron beam powder bed fusion additive manufacturing. The microstructures resulting from this process have fine features, with α laths as small as 1 µm interwoven in a highly complex fashion, presenting a significant challenge to characterize. Over the course of these fundamental works, we have developed best-practice 3D-EBSD collection protocols and advanced methods for 3D data reconstruction and analysis of such microstructures which remain unpublished. These methods may be of interest to the 3D materials characterization community, especially considering the lack of standard commercial software tools. Thus, the current paper elaborates on the methods and analysis used to characterize fine titanium microstructures using 3D-EBSD and presents a detailed description of the new algorithms developed for probing the unique features therein. The new analyses include algorithms for identifying intervariant boundary types, classifying three-variant clusters, assigning grains to variants, and quantifying interconnectivity of branched α platelets.

1. Introduction

2D microscopy methods, such as light optical microscopy (LOM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM), are powerful tools for studying structure-property-performance relationships in materials across several length scales. However, there are certain microstructural features which cannot be observed from a single 2D section or projection. 3D characterization techniques have demonstrated great potential to provide important insights into the microstructural evolution of a variety of engineering materials that would be unavailable from 2D data [1]. A variety of 3D techniques exist, though they tend to be used infrequently compared to 2D microscopy techniques because of challenges in the collection and interpretation of 3D data. Due to these challenges, there is always a trade-off between collecting data from a large enough volume to be representative of the behavior of the bulk material while maintaining sufficient spatial resolution to observe all relevant features. As techniques have developed and improved, the goal is now often to probe larger volumes at higher resolutions.

Serial sectioning is a technique which involves iteratively removing

Abbreviations: 3D-EBSD, three-dimensional electron backscatter diffraction; FIB, focused ion beam; EBSD, electron backscatter diffraction; PFIB, plasma focused ion beam; E-PBF, electron beam powder bed fusion; AM, additive manufacturing; BC, band contrast; BS, band slope; MAD, mean angular deviation; LAGB, low-angle grain boundary; RHAGB, random high-angle grain boundary; RANSAC, random sample consensus; MSAC, M-estimator sample consensus; GBCD, grain boundary character distribution.

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An understanding of hydrogen embrittlement in nickel grain boundaries from first principles

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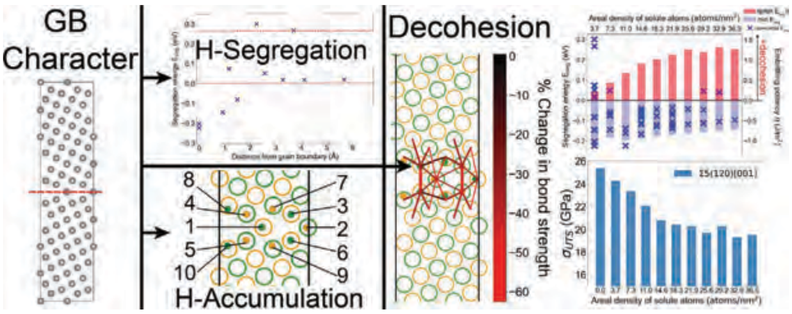
HIGHLIGHTS

- GB character controls vulnerability to hydrogen segregation and embrittlement.
- Low energy, highly coherent GBs minimise H-accumulation, embrittlement.
- H-H interactions controls amount of H segregation, accumulation at a GB.
- Bonding analysis reveals fundamental atomistic nature of H-embrittlement at GBs.

GRAPHICAL ABSTRACT



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ABSTRACT

Here, the segregation and accumulation of hydrogen in Ni grain boundaries, and its effects on cohesion and tensile mechanical strength were studied by means of density functional theory simulations. Three model grain boundaries were considered: the $\Sigma3(1\bar{1}1)[110]$, $\Sigma5(120)[001]$ and $\Sigma11(1\bar{1}0)[113]$, as representatives for the highly coherent twin, high energy random high angle, and "special" low energy highly coherent grain boundaries, respectively. Hydrogen segregation was found to be favourable in the $\Sigma5$ and $\Sigma11$ grain boundaries, but not in the $\Sigma3$. Hydrogen accumulation studied via a comprehensive site-permutation analysis revealed the mechanisms for how H accumulation capacity varies as a function of grain boundary character. We show that the interfacial cohesion of boundaries can diminish by between 6.7–37.5% at varying levels of H-accumulation. The cohesion of the grain boundaries was analysed using a novel chemical bond-order based approach, enabling a quantitative atomistic determination of the fracture paths arising from hydrogen embrittlement. These simulations explain the details of why grain boundary character is the principal determinant of the likelihood of hydrogen segregation and accumulation, and hence their vulnerability to hydrogen-enhanced decohesion. This knowledge can be used in the design of thermomechanical processes to achieve grain boundary engineering for resistance to hydrogen embrittlement.

1. Introduction

In engineering alloys, grain boundaries (GBs) are an important class of defects which can dominate the macroscopic mechanical behaviour of the material. In particular, the segregation of impurity

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PUBLICATIONS PHASE 1

DIFFERENCES IN DEFECT DISTRIBUTION ACROSS SCAN STRATEGIES IN ELECTRON BEAM AM Ti-6Al-4V

The fraction and size of pores present in EBM Ti-6Al-4V specimens varies depending on the melting strategy used, whether linear raster melting or point melting.

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In recent years, additive manufacturing (AM) has begun to displace traditional manufacturing techniques for specific applications. Notable benefits of AM include reduced times from design to product, an improved buy-to-fly ratio, lower waste, and the ability to produce complex geometries[1,2]. An additional benefit of additive manufacturing is the variety of manufacturing processes that span across heat source (e.g., laser, electron beam, plasma), input material type (e.g., powder, wire), atmosphere, and the number of axes of control among others[2-4]. This variability in processing route means that a process can be identified and optimized for a class of products or parts. Despite these various advantages, one of the primary drawbacks of AM processes is porosity within builds, which ultimately reduces the ability of a part to withstand tensile stresses and can lead to premature failure[4-6].

Electron beam melting (EBM) is a powder bed fusion technique that uses an electron beam as a heat source to melt powder particles that have been spread over a build plate[7]. Unlike laser-based processes, EBM requires the build chamber to be at vacuum, reducing the probability of porosity stemming from gases present within the build chamber[1]. Gas pores in EBM are thus typically caused by either gases present in the feedstock material (i.e., retained gas porosity) or vaporization of select elements (i.e., keyholing)[4,8]. Gas pores formed through either mechanism result in nearly spherical morphologies whose locations within the layer of a build and presence within a solidified part are influenced by the fluid dynamics of the melt pool[4,9,10].

The most common scan strategies of EBM are point-melting and variations on linear raster scan strategies, i.e., moving the electron beam in a linear fashion across the powder bed following a pattern[11]. Point-melting scan strategies, less commonly studied and used, involve point-by-point melting of small volumes of material of the powder bed. Research has shown that point-by-point melting strategies can be used for site-specific control of the resulting microstructure and texture by varying process parameters and the location and order of points melted, thereby leading to local and specific variations in mechanical properties[12,13].

OBSERVATIONS IN AS-BUILT SAMPLES

Ti-6Al-4V specimens were produced at Oak Ridge National Laboratory Manufacturing Demonstration Facility using an ARCAM EBM Q10plus system and TEKNA Ti-6Al-4V plasma atomized powder. Each specimen had a geometry of 15 x 15 x 25 mm.

Three different scan strategies were used to produce the samples: a linear raster scan (L), random point-melting (R), and what is known as the Dehoff point-melting strategy (D). The raster scan L consisted of a serpentine pattern that rotated 67.5° after the end of every layer. Each 15 x 15 mm area of R and D was segmented into coordinates. A computer-generated random

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Enhanced strength-plasticity combination in an Al–Cu–Mg alloy——atomic scale microstructure regulation and strengthening mechanisms

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ABSTRACT

A new approach to the stimulation of atomic clustering reactions shows potential to address the 'strength-ductility trade-off' in Al–Cu–Mg alloys. The evolution of the alloy microstructure during this thermomechanical processing was followed carefully using electron microscopy, X-ray diffraction, and atom probe microscopy. The distributions of the Mg and Cu solute atoms, and the characteristic distributions of Mg–Cu co-clusters, were investigated in detail. The factors affecting the strength of the alloy were assessed semi-quantitatively and the results suggest that Mg–Cu atom co-clusters contribute significant strengthening in this Al–Cu–Mg alloy, when subjected to this thermomechanical processing. The novel introduction of asymmetric rolling into the thermomechanical process increases the overall strength and we propose that this is due to the synergistic effects of cluster strengthening, dislocation strengthening, and texture engineering. The significant improvement in plasticity is attributed to a combination of the shear texture, the relatively high ratio of low-angle grain boundaries, and the avoidance of microstructural stress and strain concentrations such as those often associated with precipitation and precipitate-free-zones.

1. Introduction

Aluminium alloys used in major structural engineering applications, such as modern transportation, aerospace and defence platforms face acute design challenges. Not only are high strength, ductility and toughness required, but also are outstanding fatigue and corrosion resistance. Most metallurgical mechanisms for increasing the alloys' strength, such as precipitation strengthening, result in a loss in ductility—an effect widely referred to as the 'strength-ductility trade-off' [1]. Therefore, it is extremely challenging to design aluminium alloys that possess a comprehensive portfolio of properties.

The optimisation of alloy composition via microalloying [2–7] is an effective means by which the alloy properties may be enhanced. Microalloying research usually involves either the enhanced nucleation of existing precipitation from the base system, or the catalytic stimulation of alternative precipitation reactions. Research into microalloying of Al alloys dates back to Hardy's work [2], where trace Cd, In or Sn additions to binary Al–Cu alloys were shown to suppress the formation of G.P. zones while stimulating the nucleation of θ′ plates, which are ordinarily difficult to nucleate intragranularly in the base Al–Cu system, and tend to form on dislocations. The finer, denser distribution of the θ′ plates results in a dramatically improved mechanical strength [5]. An example of microalloying stimulating an alternative precipitation strengthening reaction is the effect of trace additions of Ag to Al–Cu–Mg alloys in stimulating the precipitation of the Ω phase, which improves the tensile properties and creep resistance at temperatures as high as 200 °C [6]. This occurs via the precipitation of the Ω phase, which is catalysed from pre-precipitate solute clustering reaction between Mg and Ag [7]. Ultimately, the approach of using multiple alloying elements in small amounts (micro-alloying) can pose challenges in terms of environmental issues and sustainability.

Grain refinement has been of tremendous research interest in the

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3D electron backscatter diffraction study of α lath morphology in additively manufactured Ti-6Al-4V

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Additive manufacturing

ABSTRACT

Titanium alloys exhibit complex, multi-phase microstructures which form during liquid-solid and solid-solid phase transformations. These phase transformations govern the microstructural evolution and are potentially more complex during additive manufacturing due to large thermal gradients and inhomogeneities. The prototypical fundamental unit of titanium microstructures are the α laths, and investigations into their three-dimensional morphology may provide new insights. A prior β-grain boundary, 3-variant clusters and interconnected laths were studied in 3D in electron-beam printed Ti-6Al-4V using a plasma FIB. These key features are of interest for studying variant selection in additive manufacturing.

1. Introduction

Titanium alloys, particularly Ti-6Al-4V, are well suited to additive manufacturing (AM) due to their applications in biomedical implants and aerospace components which benefit from the design advantages of AM. These alloys combine high corrosion resistance and biocompatibility with desirable mechanical properties, such as a high strength-to-weight ratio. The mechanical properties are highly dependent on the microstructure [1], and the details of microstructural evolution can vary widely depending on the alloy composition and processing condition [2]. Ti-6Al-4V is an α+β alloy consisting of the hexagonally close packed (HCP) α phase and a small volume fraction of body centered cubic (BCC) β phase at room temperature. Typical α morphologies include laths arranged in colonies or basket-weave structures, or equiaxed grains which are the result of thermomechanical processing [1].

During AM, large thermal gradients exist and numerous re-heating cycles occur. The magnitude of the gradients and the details of the cycles will depend upon the AM processing conditions. Such variation in processes may result in concurrent variations in the microstructural evolution which are not yet fully understood. A more complete understanding of the microstructures is required in order to potentially elucidate the details of the phase transformations and microstructural evolution that accompany the complex thermal history. Ti-6Al-4V presents an opportunity to understand repetitive solid-solid phase transformations during processing. In electron beam melting (EBM), the high temperature β phase is thought to undergo a martensitic transformation to α' and to subsequently decompose into an α+β lamellar structure as the build is held at an elevated temperature [3–5].

The Burgers orientation relationship, typically obeyed between the α and β phases in titanium alloys [6] allows for the formation of 12 possible α orientations from a parent β grain. However, usually only a few α variants are observed within one prior β grain. This preference for some α variants over others is known as variant selection. Variant selection is believed to play an important role in determining the final microstructure [7] but the role of variant selection during the microstructural evolution and its influence on properties of AM Ti-based alloys is largely unexplored.

The two well-accepted mechanisms identified for variant selection are: (i) nucleation of a certain variant at a prior β-β grain boundary to minimize the interfacial energy between the α and the two β grains based either on the disorientation of the two β grains [7–13] or the grain boundary plane [14–16]; and (ii) the simultaneous nucleation of three variants which share a $\langle 11\bar{2}0\rangle$direction in order to minimize their transformation strain by growing as a self-accommodating cluster [17–21] which has been referred to as a 'delta' distribution [18].

Abbreviations: AM, Additive manufacturing; EBM, Electron-beam melting; EBSD, Electron backscatter diffraction; FIB, Focused ion beam; LMIS, Liquid metal ion source

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PUBLICATIONS PHASE 1

Full Length Article

On the formation of spherical metastable BCC single crystal spatter particles during laser powder bed fusion

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ABSTRACT

Spatter particles, created during laser powder-bed-fusion (L-PBF) additive manufacturing process of 316 L stainless steel, solidified as single-crystal, non-equilibrium, body-centered cubic (BCC) ferrite, which has not been reported before. This phenomenon is unusual considering that the composition of stainless steel 316 L typically ensures primarily austenitic (face-centered-cubic, FCC) solidification. These particles were analyzed with multi-length scale microscopy and diffraction methods. Mechanisms for the competition between BCC and FCC phases were evaluated with computational thermodynamic and interface response function theories, as a function of thermal boundary conditions. These results indicate that the particles solidify at rapid rates and/or that conditions exist during solidification that allow for the nucleation and growth of the BCC phase that outcompete the FCC phase. The novelties of the work pertain to three aspects (i) discovery of fully single crystal BCC particles residing within spatter, (ii) rationalization of the mechanisms for this phenomenon with suites of characterization and modeling tools, as well as, (iii) the suggestion that L-PBF and associated spattering processes can be used as a synthesis route to produce metastable, single-crystal structures.

1. Introduction and motivation

Recent publications [1–3] have confirmed that many of the physical-chemical processes relevant to additive manufacturing (AM) including energy absorption by materials [4], heat and mass transfer [5–7], defect formation [8,9], distortion and residual stress evolution [10], as well as, phase transitions involving solid-solid [11], solid-liquid [12], liquid-gas [13], and gas-plasma [14–16], have commonalities to fusion welding. However, the flexibility of geometry and scanning strategies, spatially and temporally varying thermal, chemical, and mechanical conditions can lead to complex phenomena that may move the solidification process in and out of equilibrium to non-equilibrium conditions. We hypothesized that these transients can be more severe in AM than welding. In this work, we evaluated this hypothesis by processing stainless 316 L alloy powder with laser powder bed fusion (L-PBF) AM processes. By using an AM machine capable of producing laser pulses, i.e., a commercial Renishaw® machine, we imposed large transients in energy absorption and heat and mass transfer conditions that are quite different from typical fusion welding of stainless steel. Typical complexities of the boundary conditions and motivation behind this research are illustrated in Fig. 1. In the Renishaw® system, the laser energy deposition is achieved in a sequence of melted spots [17]. As a result, the transient conditions can be varied by changing the laser power, exposure time, distance between spots (d_s), and hatch spacing (h) (Fig. 1(a)). Typical in-situ IR imaging of laser and material interactions show extensive spatter formation (Fig. 1(b)).

Interestingly, these spatters emanate from the molten region and the frequency and trajectories are controlled by both the processing conditions and shielding gas flow direction. Since it is difficult to track the spatial and temporal motion of the spatter particles (Fig. 1(c)), we postulated that their trajectories will lead to complex liquid to solid phase transitions and may access phase transitions that are not accessible to traditional processing techniques. Our goal was to understand factors that control the phase selection (BCC or FCC) during solidification under these transients. The characteristics of phases, crystallography, and composition of the spatter are probed with multi-length (mm to Å) scale characterization to validate our hypothesis.

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Five-parameter characterization of intervariant boundaries in additively manufactured Ti-6Al-4V

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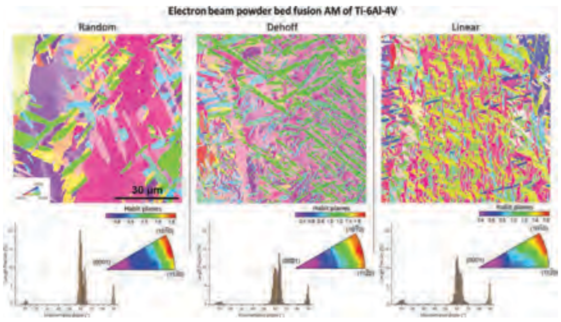
HIGHLIGHTS

- 5-parameter crystallographic characteristics of boundaries in AM Ti-6Al-4 V were studied.
- Irrespective of the morphology, there is a strict Burgers OR between α and β.
- AM Ti-6Al-4V microstructures show a maximum population of prismatic planes.
- The crystallographic constraints imposed by the BOR determine the plane characteristics.

GRAPHICAL ABSTRACT



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ABSTRACT

Additive manufacturing has emerged as a promising route to fabricate complex-shaped Ti-6Al-4V parts. The microstructural evolution and variant selection across builds in response to different printing strategies processed by electron beam powder bed fusion has been previously clarified. However, a detailed knowledge of the grain boundary plane characteristics of the α-α intervariant interfaces is still missing. The aim of this study was to reveal the full 'five-parameter' crystallographic characteristics of the intervariant boundaries. The most common α-α intervariant for colony and basketweave microstructures was 60°/[1 1 $\bar{2}$ 0], while in the acicular microstructure, the maximum was at 63.26°/[$\overline{10}$ 5 5 $\bar{3}$]. This is discussed in terms of self-accommodation during the β to α phase transformation, and the degree of coherence of the α laths in the as-deposited condition and during further growth. The grain boundary plane distributions reveal a high tendency for intervariant boundaries to terminate on prismatic and pyramidal planes rather than on low-energy basal planes. This suggests that, during additive manufacturing of Ti-6Al-4V and irrespective of the α morphology, the crystallographic constraints imposed by the Burgers orientation relationship determine the boundary plane distribution characteristics.

1. Introduction

Ti-6Al-4V is currently the most commonly used titanium alloy with a wide range of applications in industries such as aerospace, defence, biomedical, and maritime [1]. There are intense and ongoing efforts to enhance the properties and performance of this alloy via cost-effective manufacturing routes [2]. The most recent revolution in Ti-6Al-4V manufacturing has been the emergence of additive manufacturing

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Metals & corrosion

Multimodal γ' precipitation in Inconel-738 Ni-based superalloy during electron-beam powder bed fusion additive manufacturing

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ABSTRACT

Additive manufacturing is a promising alternative method for fabricating components of Ni-based superalloys which are difficult to cast, form and join. However, typical thermal cycles associated with laser powder bed-fusion techniques suppress the formation of desirable microstructures containing γ' particles, necessitating long-time post-process heat treatments. Here we report in-situ precipitation of γ' ($L1_2$-ordered) particles and carbides during electron-beam powder bed-fusion of Inconel-738. The γ' particles are homogenously distributed across the build and exhibit a *multimodal* size distribution. Based on atom-probe microscopy, we propose a eutectic reaction and multiple nucleation, growth, coarsening and dissolution bursts during thermal cycling as formation mechanism.

Introduction

Inconel 738 (INC738) is a precipitation hardened Ni-based superalloy with exceptional (~1090 MPa) tensile strength and oxidation resistance at elevated temperatures up to 980 °C for applications in modern power generation turbines and aerospace engines [1–3]. INC738 achieves these excellent high-temperature properties, specifically yield strength, mainly due to the precipitation of $L1_2$-ordered intermetallic Ni_3(Al, Ti, X) γ' particles, where X stands for alloying elements that substitute either Ni or Al sites in the lattice. INC738 and its derivatives are, however, extremely difficult to cast, weld and forge due to their high susceptibility to hot cracking [4]. For instance, in the case of welding, it has been reported that tensile stresses due to the interaction of the precipitation of γ' and cooling stresses during solidification can lead to

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Dynamic phase transformations in additively manufactured Ti-6Al-4V during thermo-mechanical gyrations ☆

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ABSTRACT

A complex interaction of process parameters, geometry and scan strategies in Additive Manufacturing (AM), can bring about spatial and temporal transients, i.e., $\Sigma T(x, y, z, time)$, within a part. Published literature focusses on fluctuating thermal cycles on the microstructure evolution. However, the microstructural variations have not been correlated to dynamic flow behavior due to the macro- and micro-scale phenomena, i.e., accumulated plastic strains brought about by large thermal gradients, transformational strains and crystallographic misfit strains. Therefore, we studied the mechanical response of Ti6Al4V alloys produced by AM under externally imposed controlled thermo-mechanical reversals in a Gleeble® thermo-mechanical simulator. The stress-strain behaviors were correlated to phase fractions, lattice strains, and also limited information on crystallographic texture using neutron diffraction techniques at the VULCAN Beamline at SNS, ORNL and also metallographic studies. The results are discussed and rationalized based on theories of static and dynamic phase transformations.

1. Introduction

Ti-6Al-4V is a well-known $(\alpha + \beta)$ titanium alloy which due to its specific strength, has found variety of applications ranging from implant devices in biomedical applications to structural parts in the aerospace sector. The high cost of titanium alloy parts made by traditional manufacturing comprising of forging and machining has made Additive Manufacturing (AM) a prime choice of fabricating complex parts and components [1]. It is of particular interest in the aerospace field, where AM could prove to be an effective technology to reduce the buy-to-fly ratio and increase the flexibility of fabrication of complex geometries compared to conventional methods. Therefore, extensive fundamental and applied research had been done to accelerate the adoption of AM of Ti-6Al-4V. A quick literature review by Web of Science shows there are more than 200+ articles related to this topic, starting with early work by Kobryn and Semiatin [2] and Kelly and Kempe [3,4]. Although thermal cycles during AM have been considered by the researchers, the effect of spatially and temporally varying ***thermo-mechanical signatures*** on phase transformations and flow properties have not been considered in any of the computational models [5]. The current paper focusses on this aspect from a fundamental perspective.

To provide context to the above problem statement a typical Electron Beam Powder Bed Fusion (E-PBF) process is briefed. The E-PBF process starts with the pre-heating of a powder layer, followed by the melting of the powders as per a path file of a geometry specified by a CAD model. A layer of powder is then raked over it and this continues till the part is completed. With the additional build-up of layers, adjacent melt areas and previous layers experience multiple thermal cycles from the liquidus temperature (~1660 °C) and the pre-heat temperature (~470 °C) [6]. Therefore, not only does a point experience multiple thermal gyrations between the Liquid-Solid transformation temperature, but also experiences multiple thermal cycles through the Solid-Solid transformation temperature, which, for Ti-6Al-4V, is at the β transus temperature $(\beta \rightarrow \alpha + \beta)$ at about ~990 °C [7]. When a two-phase alloy is subjected to multiple thermal cycles, one of the phases will tend to expand or contract relative to the other, because of the difference in their thermal expansion co-efficient and Young's moduli at different temperatures [8]. In AM, it has been shown that the melting of one layer can cause the

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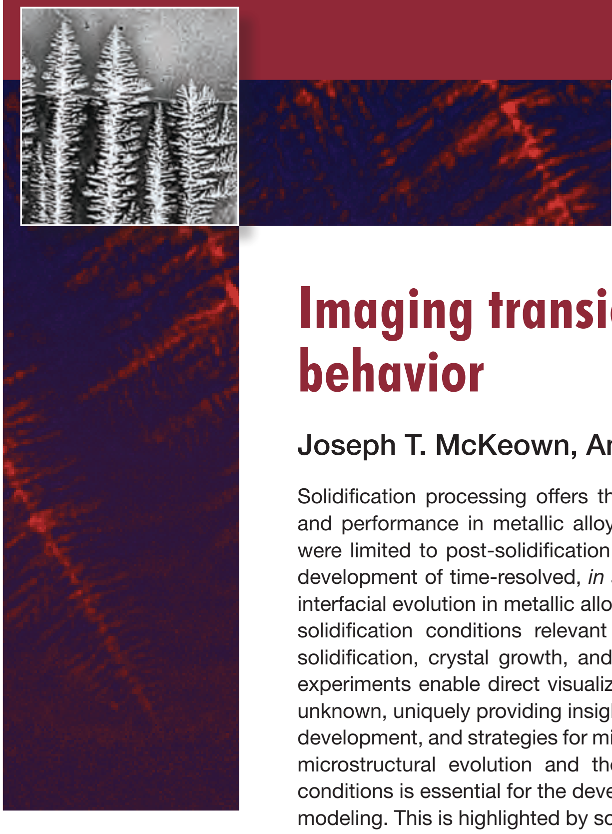

Imaging transient solidification behavior

Joseph T. McKeown, Amy J. Clarke, and Jörg M.K. Wiezorek

Solidification processing offers the first opportunity to control microstructure, properties, and performance in metallic alloy components. Until recently, microstructural evaluations were limited to post-solidification characterization by destructive methods. We review the development of time-resolved, *in situ* imaging techniques capable of capturing solid–liquid interfacial evolution in metallic alloys with high spatial and temporal resolution under diverse solidification conditions relevant for applications ranging from conventional directional solidification, crystal growth, and casting, to welding and additive manufacturing. These experiments enable direct visualization of transient behaviors that would otherwise remain unknown, uniquely providing insights into the physics that impact microstructure and defect development, and strategies for microstructural control and defect mitigation. Understanding microstructural evolution and the characteristics that form under various solidification conditions is essential for the development of multiscale, experimentally informed predictive modeling. This is highlighted by solidification simulations that utilize *in situ* measurements of solidification dynamics from state-of-the-art experimental techniques.

Introduction

Solidification processing can be traced back more than 5000 years, and the melting and casting of metals and alloys have represented major technological advances for society. However, inception of modern solidification science began only in the late 1940s and early 1950s with Turnbull's pioneering work on nucleation[1–10] and a seminal paper by Tiller et al. on solute redistribution,[11] showing that the complex process of alloy solidification could be described quantitatively. Since then, scientific advances in solidification have continued to provide innovative solutions for manufacturing and breakthrough technologies for industry and society. As an example, the evolution of nickel-based superalloys, in terms of the complexity of both alloy composition and solidification processing, has led to development of aircraft turbine blades with outstanding high-temperature strength and corrosion resistance, enabling modern jet engines with improved performance.[12–14]

During solidification, alloy microstructure is determined by processing conditions, including temperature gradient, concentration gradient, cooling rate, and growth velocity. These process conditions govern the solid–liquid interface morphology, with interface patterns ranging from planar to cellular to complex dendritic structures,[15,16] and they play a critical role in the growth competition of grains and formation of grain boundaries in polycrystalline and multiphase materials.[17,18] Thus, control of the solidification conditions provides an initial opportunity to influence microstructure development, and hence properties and performance, in metallic components.

Typically, metals and alloys are examined post-solidification by sectioning components to infer the dependence of microstructure and its evolution at elevated temperature on processing conditions. These destructive inquiries assume that the final state suitably represents an instantaneous condition at an earlier time during solidification.[17] Recently, development of real-time monitoring techniques has permitted direct observations of metallic alloys during solidification with appropriate spatial and temporal resolutions to capture microstructure evolution and responses to process condition changes that control transient solidification behaviors. In this article, examples are given that demonstrate the impact of these *in situ* techniques on our understanding of microstructure and defect development during alloy solidification, providing a path toward process strategies that enable control of structure evolution and creation of optimal properties. In addition, we demonstrate utilization of measurements from these state-of-the-art *in situ* experiments to develop multiscale predictive models for structure evolution under various processing conditions.

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In-Situ Monitoring for Defect Identification in Nickel Alloy Complex Geometries Fabricated by L-PBF Additive Manufacturing

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Published literature shows defect formation during laser powder bed fusion additive manufacturing (AM) of nickel base superalloys are sensitive to alloy chemistry, processing conditions, and geometry. In this work, ability to detect spatial distributions of defects is explored using *in-situ* monitoring of thermal signatures and surfaces. Simple and complex geometrical components were fabricated with CM247-LC® powder in an AM machine outfitted with optical and thermal sensors. The spatial and temporal variations of thermal signatures (peak intensity, decay, and number of gyrations), as well as, layer-by-layer optical images were analyzed. The observed thermal signatures were also verified with an analytical model for layer-wise heat transfer simulation that is sensitive to laser raster scan strategies. The cross-comparison data with reference to defects, obtained by X-ray tomography, were correlated with *in-situ* observations.

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I. INTRODUCTION

QUALIFICATIONS of metallic components for mission critical application[1] rely on successful completion of many steps including the ability of materials to meet the standard sets of mechanical properties depending on the service conditions. These standards were developed over many decades based on process-structure-property (P-S-P) relationships for metals made by traditional manufacturing including, casting, thermo-mechanical processing, powder metallurgy and welding. The development of P-S-P involves extraction of standard-geometry samples from a large-scale feedstock (*e.g.*, billets or forgings). This is indeed an acceptable approach, because thermal or thermomechanical cycles experienced by metals are expected to be more or less similar or within the bounds to allow for *site-independent* and *predictable* properties with *limited uncertainty*.[2] Similar approach is being extended for additive manufacturing (AM) by developing the P-S-P relationships[3,4] and associated uncertainty[5] with simple prismatic shapes[6,7] oriented in different angles[8] with reference to a build chamber. However, the validity of this extension relies on two foundational assumptions: (a) thermo-mechanical signatures are similar in all locations within these samples during AM and (b) even if there are changes in thermal signature, they do not lead to large changes in defects, microstructure and properties. Interestingly, many publications and recent work by Yoder *et al.*[9,10] and Frederick *et al.*[11] question this assumption: *are P-S-P correlations derived from simple geometry relevant to complex geometries*? This question arises because the process scanning strategies in each powder bed fusion (PBF) equipment is fixed for each part. Therefore, a small change in geometry or nesting of parts in a build may lead to changes in spatial and temporal variations of thermo-mechanical signatures,[12] which may lead to heterogeneous distribution of defects and microstructures and thereby lead to scatter in properties. Foster *et al.*[13] and Baucher *et al.*[14] suggested that the above challenge can be potentially addressed by deployment of *in-situ* monitoring of temperature of the build surface.

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Design and Tailoring of Alloys for Additive Manufacturing

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Additive manufacturing (AM) promises a major transformation for manufacturing of metallic components for aerospace, medical, nuclear, and energy applications. This perspective paper addresses some of the opportunities for alloy and feedstock design to achieve site-specific and enhanced properties not attainable by conventional manufacturing processes. This paper provides a brief overview of the role of powders, as well as solidification and solid-state phase transformation phenomena typically encountered during fusion-based AM. Three case studies are discussed that leverage the above to arrive at microstructure control. The first case study focuses on approaches to modify the solidification characteristics by *in-situ* alloying. The second case study focuses on the need for concurrent design of alloys and processing conditions to arrive at the columnar to equiaxed transition during solidification. The third case study focuses on the design of a cobalt alloy for AM, with emphasis on tailoring liquid and solid state phase transformations. The need for comprehensive knowledge of processing conditions during AM, *in-situ* and ex-situ probing of microstructure development under AM conditions, and post-print processing, characterization, and qualification are articulated for the design of future alloys and component geometries built by AM.

https://doi.org/10.1007/s11661-020-06009-3

I. INTRODUCTION

ADDITIVE manufacturing (AM)[1] offers a broad suite of new opportunities for the design and production of metallic components, particularly for the aerospace, energy, and biomedical sectors, where component geometry is complex and production volumes are low to moderate.[2] Furthermore, emerging print technologies offer the possibilities of on-demand manufacturing, customization, design complexities, part count reduction, reduction in lead time tooling and associated speedup in development, more efficient use of material, and energy and environmental benefits in terms of CO_2 reduction.[3–8] In recent years, there have been significant advances in the design and production of metallic 3D printing systems[2–6] that use either wire or powder as input stock with conventional alloys. With these advances in machines, it is indeed possible to replace existing manufacturing processes, such as casting, with AM for low-volume, high-value added and geometrically complex components. While there are many excellent studies that focus on the fundamentals of printing, heat treatment and mechanical properties of well-known printing alloys,[3–27] the focus of this perspective is on emerging opportunities for tailoring the design of alloys for AM processes. This article is not intended to be a comprehensive review, but aims to highlight pathways for expanding the suite of available alloys for AM. The above literature clearly articulates that alloy design (i.e., definition of alloy chemistry and the associated specification ranges) for AM should be guided by application requirements (*e.g.*, temperature and stress), size and complexity of printed component(s), service environment (*e.g.*, wear, corrosive, static or dynamic loading), need for dual function (*e.g.*, mechanical, neutron shielding[28] and heat transfer properties[29]), complexity of the geometry, the type of AM processing machines and infrastructure, parameter scope, ability to accommodate post-processing, such as hot isostatic pressing (HIP) and/or heat treatment,[30] and finally the business case.[31] As with conventional materials processing approaches, it is essential to confirm that a given alloy can be fabricated with acceptable levels of defects, which inevitably arise along any material processing path and ultimately limit properties.

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temperatures as low as 11 K (*6*). A more recent detailed crossed-beams study on this reaction in the collision energy range of 9.8 to 282 cm^{-1} showed that the reaction resonance peak at the energy around −40 cm^{-1} on the product side is responsible for the enhanced reactivity (see the figure) near 0 K (*7*). Because of this resonance-enhanced quantum tunneling through the reaction barrier, the rate was substantially enhanced at temperatures approaching 0 K. The quasi-bound resonance detected in the crossed-beams study was also in good agreement with the negative-ion photodetachment spectroscopic results (*8*).

The study by de Jongh *et al.* is among advances made recently in the study of quantum resonances in atomic and molecular collisions at temperatures near absolute zero. Experimental breakthroughs have mainly been enabled by emerging molecular-beam methods and better detection techniques. Strong interplay between experiment and theory has also enhanced our understanding of transient resonances in collisions to a level of spectroscopic accuracy. Dynamics studies of atomic and molecular collisions are particularly important to the understanding of energy transfer and chemical reaction processes in gas-phase systems. Such studies affect the understanding of physical and chemical processes in a wide range of systems, including terrestrial and planetary atmospheres, interstellar clouds, gas-phase lasers, semiconductor processing, plasmas, and combustion processes. ■

"...the resonances could only be accurately described with a new NO-He PES at the CCSDT(Q) level...demonstrating the exceptionally high level of the resonance model..."

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3D PRINTING

Closing the science gap in 3D metal printing

X-ray imaging and modeling reveal how metal powders absorb energy and can create defects

By **Andrew T. Polonsky** *and* **Tresa M. Pollock**

Additive manufacturing [three-dimensional (3D) printing] methodologies for high–melting point metallic materials are being used in the advanced aerospace and biomedical sectors to fabricate high-value and geometrically complex parts in moderate production volumes. One barrier to more widespread applications is the gaps in the understanding of the processes that occur during the layer-by-layer buildup by beam heating and melting of powder or wire layers. For example, the absorption of energy in powder layers that are only a few particles thick is poorly understood. On page 660 of this issue, Khairallah *et al.* (*1*) used in situ x-ray synchrotron observations of powder dynamics coupled to thermal and hydrodynamic flow modeling to study energy absorption at the scale of powder particles. The presence of the powder, relative to a flat plate without powder, improves absorptivity at low laser power, but as power approaches 200 W, the details of the powder become far less important.

In powder-bed printing, overlapping linear scans or repeated spot melts with electron or laser beams in preselected patterns form a layer of the part. The process is repeated for hundreds or thousands of layers to build up an object (see the figure, left). Ideally, the print process parameters are adjusted continuously to achieve the desired material structure in zones of the printed part. In a recent demonstration, collections of small equiaxed crystals ("grains") were printed in a background structure of large columnar crystals (*2*).

At a macroscopic level, the power supplied by the laser, the beam shape, the scan velocity and pulse duration, and the scan pattern must be tuned to achieve favorable local melting conditions. For example, printing a simple cube 2.5 cm on each edge would typically require roughly 3 to 6 km of linear track melting, or 5 million to 30 million individual spot melts. Along this print path, the physics of laser energy absorption, powder particle motion, melting, vaporization, fluid flow, heat transfer, mass transfer of alloy constituent elements, nucleation of the solid, buildup of residual stress, and evolution of the solid-state structure of the material must be predicted and controlled to ensure reproducible printing of high-quality objects (*3*–*6*).

The final properties of a material are controlled by the structure of the material derived from processing (printing) parameters as well as the size, distribution, and character of processing-induced defects. Connecting processing physics to structure and defect formation is challenging, given the complex dynamics of the powder particles. In the vicinity of the melting event, the intense heating of the powder and underlying print substrate by the laser beam creates vapor plumes that can cause particles to "recoil" away from the heated region (*7*).

For example, a short melt track through a 50-μm-thick powder layer of a cobalt-nickel alloy (*8*) that was placed on top of a sheet of the same alloy can be formed (see the figure, left). The sheet is composed primarily of one large grain that exhibits continued growth through the melted layer, except in a region at the top of the melt track where new crystallites formed because of the arrival of a powder particle on the top of the melt pool. A 3D tomographic dataset (*9*) (see the figure, right) shows that the arrival of the powder particle results in nucleation of multiple crystals of different orientations (indicated by differences in color). The physics that produce this unusual powder particle trajectory and the implications for this disturbance in structure for the next printed layer are not well understood.

Khairallah *et al.* studied such powder dynamics in relation to the formation of defects in stainless steel. A specific concern during printing is the ejection of "spatter," that is, liquid droplets or entrained powder particles that undergo expulsion from the melt pool and appear as the sparks or smoke in videos of metal 3D printing. The authors observed spatter events and associated powder dynamics at high resolution

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Texture Analysis of Additively Manufactured Ti-6Al-4V Deposited Using Different Scanning Strategies

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A limited number of features that comprise the more wholistic materials state of electron beam, powder bed additively manufactured Ti-6Al-4V have been investigated. Coupling scanning electron microscopy and orientation microscopy, the microstructure and texture of samples produced using different AM scan strategies have been studied at various positions along the build height of the samples. Both the qualitative and quantitative results, including parent beta grain orientation, alpha lath texture, and predominant type of microstructure (colony *vs* basketweave), and their length scales are included. Both the scan strategy used for the build and the time between proximal molten pool passes have been shown to significantly influence the resulting microstructure and texture.

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I. INTRODUCTION

A wide variety of additive manufacturing (AM) techniques are being adopted because of their many advantages over traditional manufacturing processes.[1] For example, AM can be used to achieve net or near-net shapes with a higher degree of complexity than through conventional techniques, thereby making topologically optimized designs practical.[2] In addition, depending upon the process and powder recycling practices, a very low buy-to-fly ratio can be achieved.[3] Beyond these design and manufacturing considerations, there are metallurgical benefits, including the possibility of producing compositionally graded structures[4,5] and potentially tailoring the composition and microstructure to achieve a certain site-specific property. Despite these advantages, there remains gaps in our understanding of the composition–microstructure–property relationships for additively manufactured materials.

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These gaps exist due to at least three principal reasons. Firstly, the complex and cyclic time-temperature nature of these processes and the multi-scale physics that govern the evolution of the materials state* are interrelated temporally and spatially in ways that our current physical relationships are ill-equipped to describe, let alone predict.[6–8] Secondly, the diverse nature of the processes themselves add to the difficulty in understanding the processing–composition–microstructure–property relationships. Fundamentally, the microstructure will be set by the solidification and subsequent phase transformations, which are governed by thermal gradients and time-temperature histories. As an example, the thermal gradients themselves vary considerably between large-scale and small-scale melt pools,[9–11] and the ratio of dominant heat transfer mechanisms varies between electron beam, laser, and plasma AM variants. Thirdly, some processes, including the electron beam powder bed ARCAM process, can be considered digital, in that the beam can be spatially and temporally controlled in discrete digital steps that are decoupled and which lead to spot melting as opposed to molten pools trailing behind the energy source. These variations lead to a rich and diverse literature base, but

*We are intentionally using the word "materials state" here to imply something broader than simply the microstructure. Materials state includes not only microstructure, but also preferential crystallographic orientations and the presence of defect structures, ranging from dislocation structures through macroscopic porosity and cracks, across a wide range of length scales (both spatial and temporal).

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RESEARCH ARTICLE

Towards process consistency and *in-situ* evaluation of porosity during laser powder bed additive manufacturing

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ABSTRACT

There is a synergy between welding and additive manufacturing with reference to spatial and temporal variations of heat transfer. In this research, *in-situ* measurements of heat transfer conditions are considered as a viable qualification methodology for additive manufacturing (AM). Infrared imaging (IR) was performed within a laser powder bed fusion (L-PBF) AM machine equipped with an IR camera. Infrared thermal signatures as a function of space and time, while processing Ti6Al4V and 316L stainless steel powders, were extracted and analysed. The analyses correlated the defect evolution at low- and high-heat input conditions to thermal decay and integrated intensities. The IR based results were validated by processing a 316L cylinder with engineered porosities and detecting the same with ground truth data from computed tomography.

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Introduction

Recent publications have confirmed that metal additive manufacturing is indeed similar to that of welding and joining [1]. While the qualification methodologies for welded components are matured by the development of welding procedure specifications (WPS), procedure qualification reports (PQR), welder qualification tests and the welder performance qualification (WPQ) methodologies, it is not clear whether these methodologies are extendible to AM. Madrid et al. [2] articulate the welding capability assessment method based on multidisciplinary design of welded structures. In this method, the process flow starts from definitions of (i) raw material (ii) weld geometries, (iii) tack welding and (iv) robotic welding to arrive at a quantitative metric of weld quality. In each of the above steps, the welding process, machine and final geometry of the components are tied together unequivocally and cannot be separated. This interdependency arises, because the quality of the welded components is defined by the spatial and temporal variations of thermo-mechanical-metallurgical conditions during single/multi-pass welding [3] that modify the solidification, solid-state transformation and also plastic strain gradients within the welds [4,5]. The challenge on extending the above weld qualification approaches is not simple due to the perceived freedom of AM that enables geometrical complexity. Currently, many trade organisations are developing qualification methodologies based on standard test samples made with nested configurations within a powder bed fusion (PBF) system and tensile testing the same [6,7]. The inherent assumption in this approach is that for given AM processing conditions, the spatial and temporal variation of thermal signature is assumed to be the same throughout the build.

This assumption has been questioned by Yoder et al. [8,9], who have shown that different geometries in a nested format within PBF system will lead to non-uniform defect formation and hypothesised that these are due to widely varying thermal signatures. This question is indeed raised by other researchers and Mazumder proposed a concept of 'certify as you build' [10] to address this question. We have listed a few of the important AM parameters (Figure 1) set by the user and also the uncertainties in parameters relevant to this concept. All these parameters affect the spatial and temporal variations of thermo-mechanical–chemical signatures and may lead to unpredictable defect and microstructure

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The effect of beam scan strategies on microstructural variations in Ti-6Al-4V fabricated by electron beam powder bed fusion

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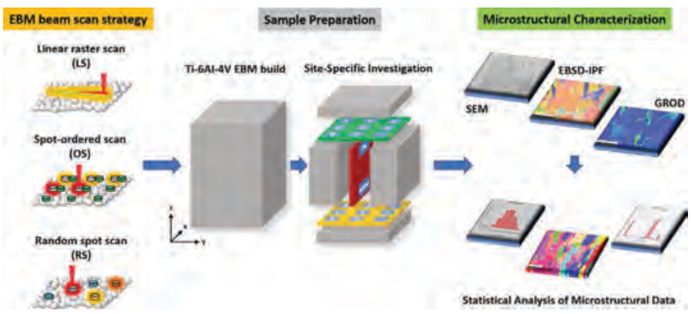
HIGHLIGHTS

- wAM Ti-6Al-4V exhibits a (more) homogenous microstructure when manufactured using an EBM PBF spot scan strategy.
- EBM spot scan strategies result in coarser α laths and finer prior β grains, compared to raster scan strategy.
- Spot scan strategies were found to result in a fiber texture, whereas raster scan results in a cube texture.
- Observed distribution of type 2 & type 4 α/α lath boundaries suggests mild to weak variant selection in all Ti64 samples.

GRAPHICAL ABSTRACT



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ABSTRACT

In electron beam melting (EBM) powder bed fusion (PBF), variations in electron beam scan strategies can be used to control thermal transients in the additive manufacturing process, both in the melt pool and in previously deposited layers. In this study, three different EBM beam scan strategies, i.e. the standard raster scan, ordered spot scan, and random spot scan patterns, were used to fabricate three identical Ti-6Al-4V blocks. Using scanning electron microscopy and electron backscatter diffraction, variations in microstructure and crystallographic texture, such as α lath thickness, prior beta β grain size and orientation, α/α lath boundary (LB) distributions, are investigated with respect to the applied scan strategy. Both spot scan strategies result in coarser α laths and smaller prior β grains with width and height < 1/3 of the value of the typical large columnar grains, observed in raster scan samples. The combined fraction of type 2 and type 4 α/α LBs measured in the three samples was found to be between 0.50 and 0.85, which is greater than the expected combined fraction of ~0.36 for a random distribution of α variants. This suggests the presence of a mild to weak variant selection in EBM Ti64.

1. Introduction

Additive manufacturing (AM) is an attractive manufacturing approach over traditional subtractive manufacturing methods due to its potential advantages [1–3,5–7], such as geometric design flexibility [3,8,9], low 'buy-to-fly' ratio, and reduced need for tooling. These advantages result in faster time-to-market for highly complex parts and significant weight reduction [3,10]. Over the past two decades, AM has moved to the forefront of industrial and academic research due to advancements in AM instrumentation and technology [1,2,6,11,12], also opening up AM for a wider selection of materials. Despite this progress, several challenges remain in metal-based AM.

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Effect of scanning strategy on variant selection in additively manufactured Ti-6Al-4V

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ABSTRACT

Additive manufacturing of the Ti-6Al-4 V alloy is increasingly popular for making complex shaped parts. This alloy undergoes a transformation from the body-centred cubic β phase to the hexagonally close-packed α phase following solidification. There are currently gaps in the understanding of relationships between the processing conditions and the final material microstructure. In particular, the role of α variant selection mechanisms within additively manufactured parts is not well enough understood to assure product quality when varying processing parameters such as the scanning strategy. In this study, Ti-6Al-4 V samples were fabricated via the electron beam powder fusion (E-PBF) process under three different scanning strategies (linear scan, random and Dehoff point fills). Electron back-scatter diffraction revealed that the scanning strategy employed directly affects which variant selection mechanism dominates during the β→α transformation. Faster cooling rates in the linear scan produce microstructures which are influenced heavily by self-accommodation, while the microstructure of the slower cooling random fill strategy is dominated more by prior β grain boundary effects. This, in turn, dictates the microstructural evolution of the material, leading to the prevalence of different microstructural features such as macrozones or intragranular 3-variant clustering. These insights will enable optimisation of processing strategies in additive manufacturing to produce tailored product microstructures.

1. Introduction

Additive manufacturing (AM) is a layer-by-layer fabrication method that enables the production of near-net shape components in a quick and cost-efficient manner [1]. This makes it especially attractive for industries that require high-cost, low volume and geometrically complex parts such as the biomedical and aerospace sectors. Electron beam powder bed fusion (E-PBF) is an AM technique that utilises a high energy electron beam to melt and bind together metallic powder feedstocks [1]. Fabrication in a high vacuum chamber makes it suitable for metals prone to oxidation, including the titanium family of alloys [2]. E-PBF, however, creates complex thermal gradients within the structure during build-up, making it difficult to predict the properties of the build. The nature of these thermal gradients can be controlled by variation of the process parameters such as the scanning strategy [3]. The widespread commercial adoption of AM parts is driving increasing interest in a more in-depth understanding of the links between E-PBF processing and the resultant microstructures so as to consistently fabricate products with desired properties.

Ti-6Al-4 V undergoes a complex microstructural evolution during the E-PBF process. This alloy has low thermal conductivity, resulting in significant temperature inhomogeneities, and undergoes a transformation from the body-centred cubic (BCC) β phase to the hexagonally close-packed (HCP) α phase (or martensitic α') after solidification [4]. The complex microstructural evolution during AM of Ti-6Al-4 V has been previously studied [5–7]. Liquid first solidifies as the β phase which tends to grow epitaxially with the <001> orientation along the build direction with a columnar morphology. The β phase then undergoes a solid-state phase transformation upon cooling. As the cooling rate in E-PBF is estimated to be above 410 Ks^{-1} [5,8], a martensitic phase transformation is expected to occur. The α' martensite subsequently goes through an in-situ decomposition into α + β due to a pseudo-annealing effect from the elevated build temperatures and cyclic reheating from the melting of subsequent layers. This results in a final

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Full length article

Effect of cyclic rapid thermal loadings on the microstructural evolution of a CrMnFeCoNi high-entropy alloy manufactured by selective laser melting

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ABSTRACT

Metallic materials produced by additive manufacturing experience complex stress and thermal gyrations along the build direction. This has the potential to produce complicated heterogeneous microstructures that may exhibit a wide variety of mechanical properties. There remains a paucity of studies on the nature and the formation mechanisms of the microstructural heterogeneity and this limits our capability for microstructural design in additively manufactured metallic materials. Here, we present an electron microscopy-based investigation of a CrMnFeCoNi high-entropy alloy produced by selective laser melting. We have focussed on a systematic investigation of the microstructural evolution along the build direction. Our results reveal a remarkable hierarchy of microstructures, including the formation of nanocrystalline grains, elemental segregation and precipitation, cellular dislocation structures, deformation twinning, and deformation-induced phase transformation. Our research clarifies the relationships amongst different features, and provides guidance for future structural manipulation of materials produced by additive manufacturing.

1. Introduction

Selective laser melting (SLM) is a powder-bed-based additive manufacturing (AM) technique [1–5]. As with other three-dimensional (3D) printing processes [1,6,7], SLM offers distinct advantages over conventional manufacturing, including design freedom, near-net or net shape production, efficient use of materials, short lead times, and substantial cost savings in many cases [2,5,8]. The SLM processes of metallic materials consist of cyclic rapid thermal loadings that affect significantly the microstructure and consequently the mechanical properties of materials. During the AM processes, different layers experienced different thermal histories, leading to microstructure variation along the build direction. These significant structural features are not observed in materials produced via traditional synthesis and processing methods.

Metallic materials, e.g., stainless steels [9–11], high-entropy alloys (HEAs) [12,13], and aluminium alloys [14], produced by 3D printing are usually of hierarchical structures that include columnar grains, melt pools, cellular dislocation structures, and precipitates [9,12]. The formation of hierarchical structures and their influence on mechanical properties are a hot topic in metallic materials manufactured by AM [5,8–10,12]. Transmission electron microscopy (TEM) studies revealed heterogeneous structures, i.e. cellular dislocation structures, chemical cells and precipitates, along the build direction, which are closely related to the cyclic thermal loadings and could be manipulated by properly adjusting the 3D printing processes [8,9,12,15]. It is still not clear how these heterogeneous structures formed and if the structures could be manipulated through adjusting the processing parameters for superior mechanical properties. We anticipate that more characterisation-based research will be needed to understand the complex phe-

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In-situ synthesis of oxides by reactive process atmospheres during L-PBF of stainless steel

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A B S T R A C T

Traditionally, reactive gases such as oxygen (O_2) and carbon dioxide (CO_2) have been avoided during laser powder bed fusion (L-PBF) of metals and alloys based on the notion that it may lead to defect formation and poor properties. Here we show that instead, these gases can be used to form sub-μm-sized oxide particles in-situ during the L-PBF process in an Fe-Cr-Al-Ti stainless steel and lead to improved room temperature and high-temperature mechanical properties. We manufactured cube samples using pure Ar and various reactive gas atmospheres, namely an O_2/Argon (Ar) mixture containing 0.2 % O_2 and CO_2/Ar mixtures containing up to 100 % CO_2. Co-axial measurements of infrared radiation emitted from the melt pool showed correlation to the presence of O_2 or CO_2 in the gas mixture. Builds produced under CO_2-containing atmosphere contained complex oxides with an average diameter of ~40 nm, an Al-rich core and a Ti-rich shell. Due to the high cooling rates typical to L-PBF, agglomeration of oxides and slag formation on the surface of the samples could almost be entirely avoided. Compression tests at temperatures up to 800 °C showed that the samples produced in 100 % CO_2 have about 20 % higher yield stress compared to samples produced in Ar. The paper concludes with a discussion of the formation mechanism of the observed oxides. Our results show that in-situ reactions during additive manufacturing processes are a promising pathway to the synthesis of particle-reinforced alloys.

1. Introduction

Laser Powder Bed Fusion (L-PBF) is an Additive Manufacturing (AM) technology that is capable of creating near-net shape parts with complex geometries through successive melting and solidification of thin layers of metal powder material, ranging from 20 to 100 μm in thickness, using a laser energy source [1]. Compared to traditional manufacturing methods, which usually involve subtractive processes such as milling, AM allows for greater part complexity while having less material waste. Full details of the AM process have been given previously [2,3]. Although this process is often presented with different acronyms, e.g., selective laser melting (SLM) or direct metal laser melting (DMLM), in this paper, we will adopt the terminology suggested by ISO standards, i.e., L-PBF.

Often, argon (Ar) is used in L-PBF as the shielding gas to protect the parts being built from oxidation. Other gases such as nitrogen have seen more recent adoption for certain types of steels due to cost considerations, but its use is limited. This is because nitrogen is not fully inert in the L-PBF process and N uptake may lead to changes in the microstructure, e.g. an increased retained austenite fraction in 17-4 PH stainless steels [4,5]. The impact of reactive gas atmospheres such as O_2 and CO_2 during the L-PBF process in particular, and in AM in general, is not well explored until now. Some works have noted the formation of oxides in steel parts during AM processing which have been claimed to be the result of residual oxygen in the atmosphere [6–8]. These cases of oxide formation were not always intentional. On the other hand, Springer et al [9]. were able to show the intentional in-situ formation of oxides and nitrides in DED (Directed Energy Deposition, another AM process) of 316 L stainless steel through exposure to air, while Wysocki et al. [10] demonstrated in L-PBF an increased oxygen content in samples when printing pure titanium with the addition of 0.2-0.4 vol. % of oxygen in the atmosphere. It should be noted that several studies note the formation of small (tens to hundreds of nm in diameter) oxide particles in L-PBF-produced steels even processed under a nominally pure Ar atmosphere. The question hence arises whether these oxides, which are sometimes claimed to improve mechanical properties, originate from oxygen introduced via the oxide layers on powder particle surfaces or from residual oxygen in the atmosphere, and further, if they

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Segregation of the major alloying elements to $Al_3(Sc,Zr)$ precipitates in an Al–Zn–Mg–Cu–Sc–Zr alloy

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ABSTRACT

Solute segregation of Zn, Mg and Cu in and around $Al_3(Sc,Zr)$ precipitates in an Al-Zn-Mg-Cu-Sc-Zr alloy was systematically studied using atom probe microscopy, aberration-corrected scanning transmission electron microscopy and first-principles simulations. Results show that the $Al_3(Sc,Zr)$ precipitates occur as a Sc-rich 'core' and Zr-rich 'shell' structure. Zn segregates to the Zr-rich shell of $Al_3(Sc,Zr)$ precipitates and substitute mainly for the Al atoms, and the Zn segregation intensifies with increasing ageing time. Mg and Cu atoms segregate to the Zr-rich shell during the early stages of ageing, while the Mg atoms prefer the Al-matrix with longer ageing time. Density functional theory calculations demonstrate that such segregation is energetically favored and highlight the diverse role of $Al_3(Sc,Zr)$ in influencing the distribution of the major alloying elements.

1. Introduction

Solute segregation within and around second-phase precipitates in Al alloys has attracted considerable interest in recent years due to the fact that it can significantly affect the properties of alloys [1–4]. For example, Sc segregation at the θ'/α-Al matrix interface increases the strengthening effect of the θ' (Al_2Cu) phase in Al-Cu-Sc alloys [5,6]. The Mg segregation was reported to occur at the coherent Al_3Sc/α-Al matrix interface, enhancing the stability of Al-Mg-Sc alloys at elevated temperatures [7,8]. The experimental and other details of a number of other instances of solute segregation to Al alloys are described in Ref. [9], including the well-studied case of Mg and Ag segregation to the Ω phase in Al-Cu-Mg-Ag alloys [10]. Thus, a detailed understanding of the solute segregation at precipitates is essential for materials optimization.

Al-Zn-Mg-Cu alloys are widely used in aerospace applications due to their high strength to weight ratio and good weldability [11–13]. It is generally accepted that the primary strengthening precipitate in Al-Zn-Mg-Cu alloys is the η' phase ($MgZn_2$) [14,15]. Recently, trace amounts of Sc and Zr have been shown to improve both the corrosion resistance [16,17] and mechanical performance [18] of Al-Zn-Mg-Cu alloys via the formation of core-shell $Al_3(Sc,Zr)$ precipitates. The core-shell structured $Al_3(Sc,Zr)$ precipitates can increase the thermal stability of Al alloys due to the highly stability of the core-shell structure [19,20]. The $Al_3(Sc,Zr)$ precipitates possess a $L1_2$ structure with a lattice parameter of 0.4095 nm which is smaller than that of Al_3Sc (0.4125 nm) [21,22], thus the lattice parameter mismatch between the precipitates and the Al matrix was decreased due to the formation of core-shell $Al_3(Sc,Zr)$. It has been suggested that alloying with Sc and Zr lowers the solubility of major alloying elements (i.e. Zn, Mg and Cu) in the α-Al matrix, resulting in an enhanced age hardening response [23,24]. In particular, the $Al_3(Sc,Zr)$ precipitates in Al-Zn-Mg-Cu-Sc-Zr alloys were shown to provide additional nucleation sites for the precipitation of the metastable η' phase and the equilibrium η phase [25–27]. Notwithstanding these important findings, the details of how Sc and Zr additions alter the nature of precipitation in the Al-Zn-Mg-Cu-Sc-Zr alloys remain unclear as it is experimentally challenging.

Alloying elements such as Zn, Mg and Cu were reported to be partially dissolved in $L1_2$ structured precipitates [28–30]. However, there is no indication as to whether these alloying elements segregate to the precipitate/α-Al matrix interface or, alternatively, become incorporated within the precipitates. More recently, Zn segregation was reported to occur within the Al_3Zr precipitates with $L1_2$ structure

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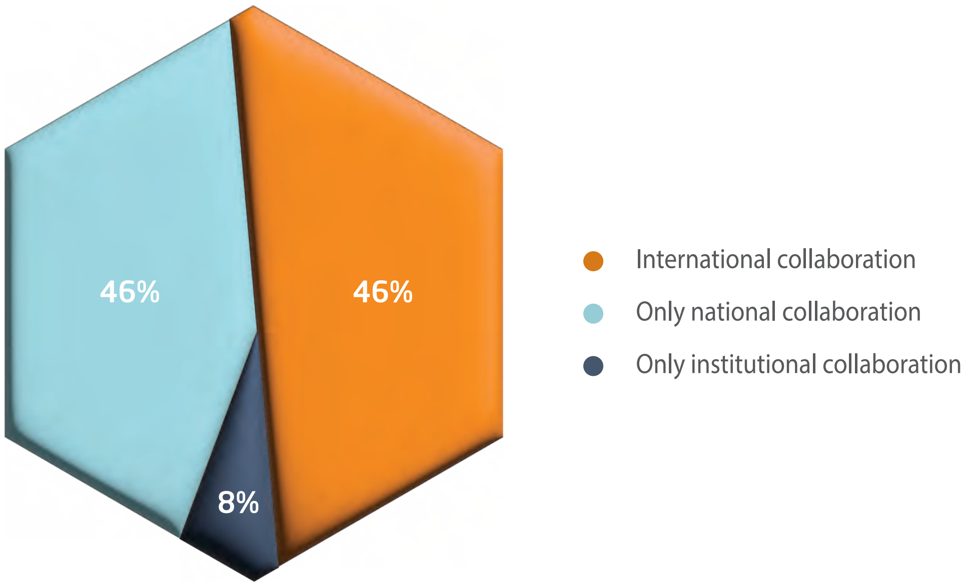
Annexure

MURI- AUSMURI Publication Analysis (as of Feb 2024)

Publication metrics of the MUR-AUSMURI project papers were analysed using SciVal. Out outputs in the Annexure, the following summarises the results of the analysis.

Overall research performance			
Citations	**Citations per Publication**	**Authors**	**FWCI***
1468	15.6	228	1.75

Research performance based on collaboration				
Collaboration type	**Scholarly Output**	**Citations**	**Citations per Publication**	**FWCI**
International	43	691	16.1	2.11
National	43	705	16.4	1.51
Institutional	8	72	9.0	1.16



Early Citation Data: Outputs in Top Citation Percentiles - 22%

(Outputs in Top Citation Percentiles indicates the extent to which an entity's publications are present in the most-cited percentiles of a data universe)

Publications in Top Journal Percentiles - 54%

(Publications in Top Journal Percentiles in SciVal indicates the extent to which an entity's publications are present in the most-cited journals in the data universe)

*Field-Weighted Citation Impact (FWCI) in SciVal indicates how the number of citations received by an entity's publications compares with the average number of citations received by all other similar publications in the data universe. A FWCI of more than 1.00 indicates above the global average for similar publications

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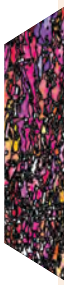

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