Experiment title: In-siu evaluation of phase transformation and residual strain during layerwise AM by operando synchrotron XRD		Experiment number: MA-3971
Beamline: ID31	**Date of experiment**: from:30-04-2018 to:04-05-2018	**Date of report**: 26-02-2020
Shifts:	**Local contact(s)**: Veijo Honkimaki	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
Peter Lee, University of Manchester (now University College London)*
Samuel Clark, University of Manchester (now University College London)*
Chu Lun Alex Leung, University of Manchester (now University College London)*
Lorna Sinclair, University of Manchester (now University College London)*
Muhammed Azeem, University of Manchester (now University of Leicester)*
Leigh Stanger, University of Sheffield*
Jon Willmott, University of Sheffield
Ian Todd, University of Sheffield
Andre Phillion, McMaster University*

Report: In this study we redesigned our bespoke LAM Process Replicator (LAMPR), adapting it to enable reciprocal space, in addition to real space synchrotron imaging of additive manufacturing. Specifically in this study we explored the rapid phase transformations during layerwise LAM resolved via synchrotron X-ray diffraction (TR-XRD) in several materials: IN1718, SS316L, Ti185 and Ti64. During the beamtime we had no significant periods of downtime attributable to the source and the beamline staff were very helpful in helping us with the arrangemnts for equiment transport and setup. An example of the multi-modal data aquaired in this experiment is shown in Figure 1. Data analysis for the Ti-1Al-8V-5Fe powder is shown in Figure 2.

Many portions of the proposal were highly successful there were however some limitations of the study. The 250 fps of of the large area pilatus detector makes the study of the the highly dynamic laser powder bed fusion process challenging as the laser scasns across the sample at speeds of up to 1 m/s. This detector would be much better suited to studying the directed energy deposition (DED) additive manufacturing process where the laser is stationary. In this case the Eulerian frame of reference would allow the phase transformations and strain accumulations to be studied just by moving the stationary melt-pool systematically through the sample. To date, the work has yielded 4 conference presentations and will result in at 2 publications which are due to be submitted.

Conference Presentations:

2019 TMS San Antonio

C1. Title: Operando quantification of the phase transformations in additive manufacturing

Authors: S. J. Clark[a,b], C. L. A. Leung[a,b], Y. Chen[a,b], L. Sinclair[b,c], S. Marussi[c], A. Phillion[d], L. Stanger[e], J. Willmott[e], M. Azeem[a,b], R. C. Attwood[g], M. P. Olbinado[h], A. Rack[h], V. Honkimaki[h] and P. D. Lee[a,b].
[a] Mechanical Engineering, University College London, London, WC1E 7JE, UK
[b] Research Complex at Harwell, Harwell Campus, Didcot, OX11 0FA, UK
[c] School of Materials, The University of Manchester, Manchester, M13 9PL, UK
[d] Department of Materials Science and Engineering, McMaster University, Hamilton, L8S 4L7, Canada
[e] Department of Electronic and Electrical Engineering, University of Sheffield, Sheffield S1 4DE, UK
[f] Department of Materials Science and Engineering, The University of Sheffield, Sheffield S1 3JD, UK
[g] Diamond Light Source, The Harwell Science and Innovation Campus, Didcot, Oxfordshire OX110DE, UK
[h] European Synchrotron Radiation Facility, CS 40220, 38043 Grenoble Cedex 9, France
Abstract:
Laser Additive Manufacturing (LAM) can directly produce near net-shape metallic components using elemental and commercial alloy powders. However, the phases formed during LAM are far from equilibrium due to the ultra-fast laser-powder interaction (<50 ms), mixing of solutes during melting and micro-segregation upon rapid solidification. Our understanding of these phenomena and the resultant microstructural features formed cannot be fully elucidated using traditional *a posteriori* characterization, necessitating *in situ* and *operando* characterisation. We have developed a LAM Process Replicator (LAMPR) that allows real and reciprocal space synchrotron imaging of LAM. The diffraction study exploits the quasi-steady-state and layer-wise AM to enable the detection of the primary solidification of tracks under conditions distant from the near equilibrium transformations observed from conventional processing. This is compared with in-situ operando x-ray imaging of the same processes. The results can be used to help designing new alloys that exploit the full potential of LAM.

2020 TMS San Diego
C2. Title: Observing the Phase Evolution during Selective Laser Melting of a High-Fe β-Ti Alloy from Elemental Powders via In-Situ Synchrotron X-Ray Diffraction
Authors: F.F. Ahmed[1], S.J. Clark[2,3], C.L.A. Leung[2,3], Y. Chen[2,3], L. Sinclair[2,3], S. Marussi[2,3], V. Honkimaki[4], N. Haynes[5], H.S. Zurob[1], P.D. Lee[2,3], A.B. Phillion[1]
[1]*Department of Materials Science and Engineering, McMaster University, Hamilton, L8S 4L7, Canada*
[2]*Mechanical Engineering, University College London, London, England, WC1E 7J3, United Kingdom*
[3]*Research Complex at Harwell, RAL, Didcot, OX11 0FA, United Kingdom*
[4]*European Synchrotron Radiation Facility, 38000 Grenoble, France*
[5]*Collins Aerospace, Oakville, L6L 5Y7, Canada*
Abstract:
Fe is a low-cost alloying element for β-Ti alloys, potentially increasing tensile and fatigue strengths. However, the low cooling rates during casting cause Fe-rich precipitates known as β-flecks to develop during solidification. Using Selective Laser Melting (SLM) one can produce high-Fe β-Ti alloys free of β-flecks as the rapid solidification rates constrain Fe segregation. To design an optimal production route of high-Fe β-Ti alloys, the phase transformation sequence during printing must first be understood. In this study, real-time synchrotron X-Ray Diffraction was employed to characterize phase transformations during the SLM of a high-Fe β-Ti alloy. Infrared images were collected concurrently and converted to temperature. Temperature profiles were matched with the identified XRD peaks to determine the phase evolution. To further reduce costs, elemental powders rather than a pre-alloyed powder were utilized as the starting material.

C3. F. Ahmed, H. Zurob, S. Clark, P.D. Lee, A. Leung, Y. Chen, L. Sinclair, S. Marussi, V. Honkimaki, A. B. Phillion, "Phase Evolution During Selective Laser Melting of a High-Fe β-Ti Alloy From Elemental Powders," UofT-Tokyo-McMaster Materials Science and Engineering Workshop, Toronto, Canada, June, 2019, 2 pages.

C4. F. F. Ahmed, H. S. Zurob, A. B. Phillion, S. J. Clark, C. L. A. Leung, Y. Chen, L. Sinclair, S. Marussi, P. D. Lee, and V. Honkimaki, The Microstructural Development During Selective Laser Melting Of Ti-185 With In-Situ Alloying," COM Conference, Toronto, August 2020, 5 pages.

Publications

The publications in preparation are:

1. S.J. Clark, C.L.A. Leung, Y. Chen, L. Sinclair, S. Marussi, V. Honkimaki, P.D. Lee, "Operando quantification of the phase transformations in additive manufacturing", to be submitted to Additive Manufacturing.
2. F.F. Ahmed[1], S.J. Clark[2,3], C.L.A. Leung[2,3], Y. Chen[2,3], L. Sinclair[2,3], S. Marussi[2,3], V. Honkimaki[4], N. Haynes[5], H.S. Zurob[1], P.D. Lee[2,3], A.B. Phillion[1], "Phase Evolution during In situ alloying of a High-Fe beta Ti alloy via Selective Laser Melting", to be submitted to Acta Materialia.

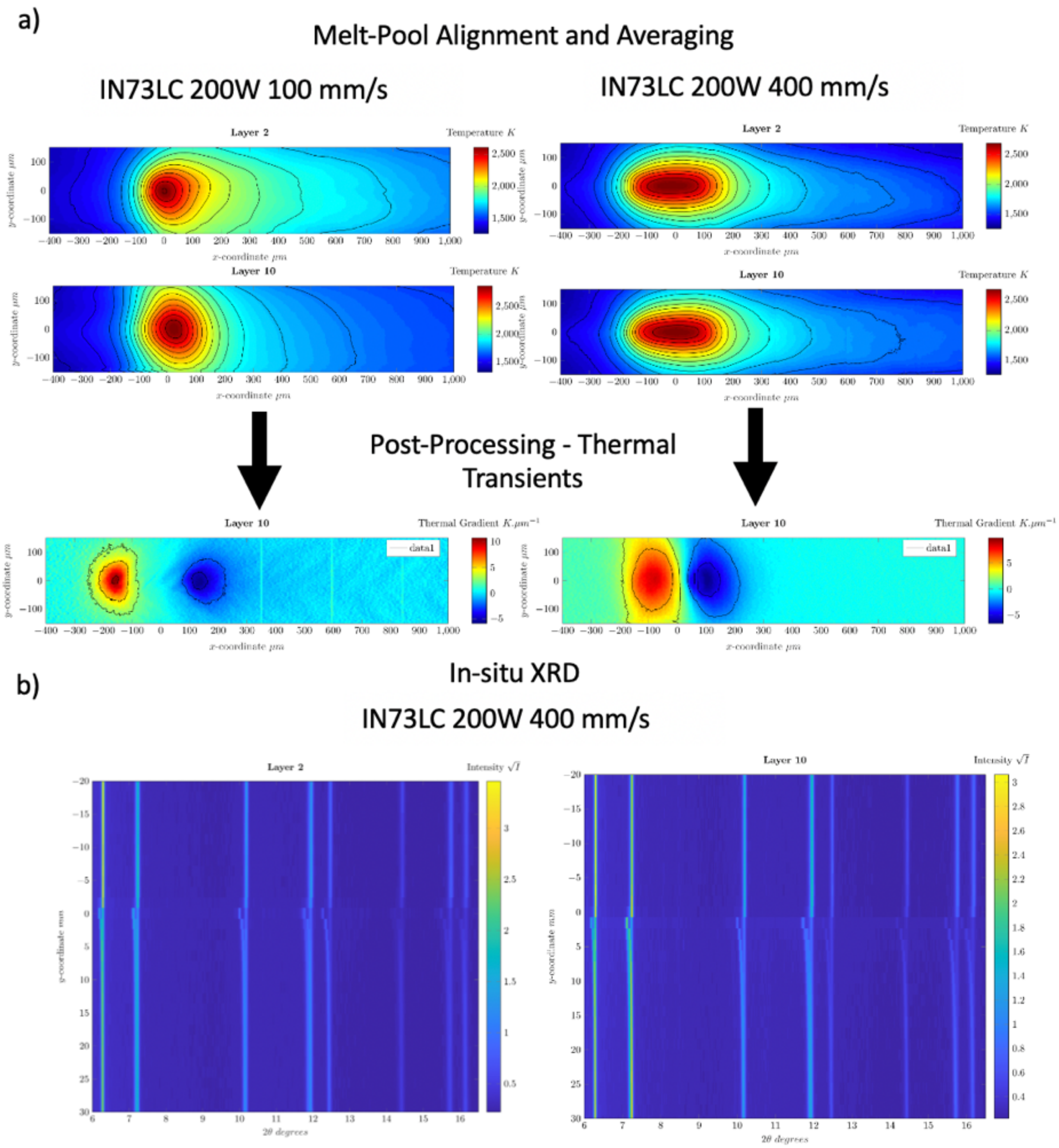
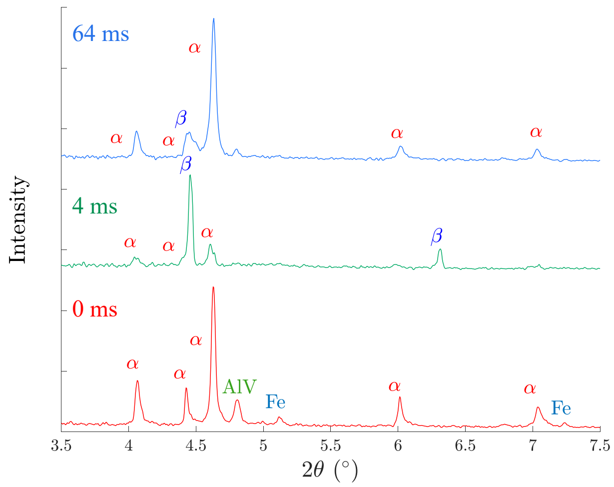


Figure 1 Exemplar multi-modal data collected during this beamtime: a) thermeography of the meltpool b) XRD of the phase transformations

XRD Analysis on Ti-1Al-8V-5Fe

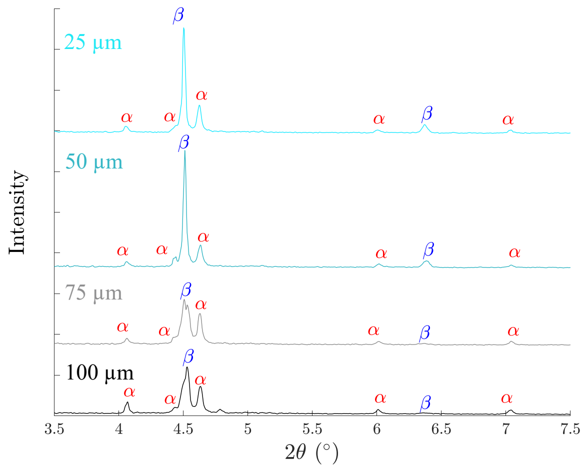
Melt Pool XRD



Powders (α-Ti, Fe, AlV) $\xrightarrow{\text{Laser}}$ Liquid (~2900 K)

→ β+ low α → α+ low β

Single Layer Tracking of Phase Evolution



α+β $\xrightarrow{\text{Laser}}$ β + low α → α + β

EBSD Analysis

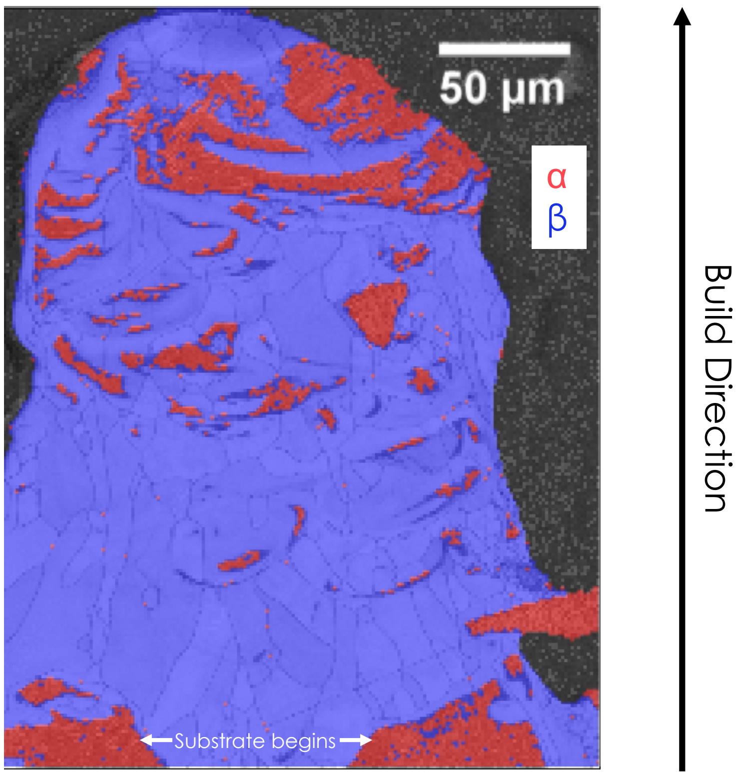


Figure 2 Analysis of Acquired in situ XRD on the Ti-1Al-8V-5Fe alloy. Upper Left shows the phase evolution in structure within the melt pool during Selective Laser Melting. Upper Right shows the phase evolution tracking a layer through successive builds as it gets further away from the powder layer. Lower shows microscopy analysis (EBSD characterization) to identify the alpha and beta phase locations.