

36D.0 CHARACTERIZING ADDITIVELY MANUFACTURED INCONEL 718/738

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36D.1 Project Overview and Industrial Relevance

This Multidisciplinary University Research Initiative (MURI) project funded by the Office of Naval Research (ONR) will focus, in part, on additively manufactured (AM) samples of Inconel 718 and Inconel 738. The goal is to analyze microstructural development, texture, and mechanical anisotropy that comes from the layer-by-layer build process, and to reveal potential variations with respect to build parameters. Experiments will be done at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL) to simulate the melt pool and solidification experienced during the metal additive process. From these results, solidification behavior will be extracted and related to microstructural evolution. Samples from the Manufacturing Demonstration Facility (MDF) at Oak Ridge National Laboratory (ORNL) will also be characterized to clarify the role of scan strategy on microstructural and mechanical anisotropy. This work is of interest to the aerospace sector, as Ni-based superalloys are desirable for their creep behavior and oxidation resistance, making them excellent candidates for gas turbines and other internal propulsion parts. Inconel 718 and 738 are already heavily used alloys for these applications, and understanding the differences between the microstructures produced by AM and conventional methods will lead to the manufacturing of improved parts with controlled microstructures and properties.

Ni-based superalloys are commonly used in aerospace applications for their high-temperature applications, and current alloy design focuses on AM to produce parts with complex geometries and near-net shapes through a layer-by-layer process [36D.1]. The aim of AM parts is to eliminate processing steps to save resources and costs for production. The microstructural evolution of printed parts is poorly defined, as thermal cycling and large thermal gradients from the build process differ from legacy manufacturing techniques such as casting and forging. The AM process also changes many factors; common techniques for metals include selective laser sintering (SLS), selective laser melting (SLM), and electron beam melting (EBM). Defects common to these AM processes, such as voids and lack of fusion between printed layers, negatively affect mechanical properties [36D.2]. **Figure 36D.1** shows variation of structure and properties along the build direction for an AM build of Ti-6Al-4V made with EBM [36D.3]. Therefore, it is important to understand fundamental materials science phenomena through *in-situ* and *ex-situ* experiments to better understand the end-result microstructures and material properties. The *in-situ* experiments will consist of simulating the melt pool and solidification during a typical AM build process using the APS facilities, while *ex-situ* experiments will consist of electron microscopy, electron backscatter diffraction (EBSD), and neutron diffraction to understand microstructural condition as a function of build height.

36D.2 Previous Work

As the MURI project was initiated in Fall 2018, experiments similar to the scope of this project were conducted a year ago at the APS in March 2019 with model ternary Ni-alloys and Inconel 738. The model alloys used were single-crystal specimens with differing Ni, Mo, and Al contents, evaluated at different laser powers. The R2 samples (Ni-1.9Mo-6.6Al (wt %)) had either a $\langle 110 \rangle$ or $\langle 111 \rangle$ orientation parallel to the build direction, while the R4 samples (Ni-22.2Mo-2.8Al (wt %)) had either a $\langle 100 \rangle$ or $\langle 110 \rangle$ orientation parallel to the build direction. The radiography data was processed using an ImageJ script to minimize background noise and to more clearly track the solid-liquid interface. Solidification velocities were determined at different power settings and compared with post-mortem electron microscopy to analyze the role of laser power on the columnar to equiaxed transition (CET) [36D.4].

The EBM Inconel 738 samples made at the MDF were focused on relating the scan strategy of the build to the degree of texture along the build direction. The EBM samples were made with three scan strategies: Random, Linear, and DeHoff methods. The Random method spot melts the powder material in random locations until the layer is completely melted. The Linear method raster melts the powder material from edge to edge in a straight path, while the DeHoff

method is more complicated by spot melting powder material every 11th voxel until layer completion. Only the Random samples have been manufactured, while the other samples with linear and DeHoff scan strategies are to be delivered in the future from the MDF [36D.5].

36D.3 Recent Progress

36D.3.1 Literature Review

Both Inconel 718 and Inconel 738 are precipitation strengthened alloys, consisting of an γ FCC austenite matrix with γ' precipitates, $\text{Ni}_3(\text{Al}, \text{Ti})$, of the L_{12} structure. Inconel 718 also contains γ'' precipitates that have a BCT structure [36D.6]. AM production of Inconel 738 is difficult, due to excessive thermal stresses upon rapid solidification, leading to hot cracking. This occurs when the growth of dendrites restricts liquid flow to the interdendritic regions during solidification. **Figure 36D.2** illustrates a crack along the build direction due to hot cracking [36D.7]. The high concentration of Al and Ti, about 6.7 wt% in Inconel 738, causes elemental segregation at the grain boundaries (GBs). This results in hot cracking during solidification and strain age cracking during post build heat treatment [36D.8]. Segregation of Zr also promotes hot cracking, due to an increase in the solidification temperature range and resulting promotion of grain boundary liquid films to lower temperatures [36D.9]. One method to control hot cracking is to change the power and travel velocity of the heat source to affect solidification conditions. One study using pulsed laser bed fusion (P-LBPF) shows that decreasing the power density yields a much finer microstructure with less hot crack susceptibility, because it provides a smaller “liquid volume critical to hot cracking” [36D.10]. It was also shown that use of an environmental chamber at elevated temperatures (900 °C) yields higher ductility, countering the residual stresses that would cause hot cracking [36D.11].

Solidification under typical AM conditions in Ni-based alloys often results in columnar dendritic growth with a $\langle 100 \rangle$ fiber texture parallel to the build direction [36D.12], which can contribute to mechanical property anisotropy. Highly textured columnar solidification microstructures can be avoided by promoting randomly oriented equiaxed grains through the CET. The thermal gradient (G) and the solidification velocity (V) can be controlled to fine-tune the microstructure between columnar and equiaxed grain structures. **Figure 36D.3** shows the experimental range for G - V with respect to grain structure [36D.13]. Studies of typical G - V values for Inconel 718 made by SLM have shown a range of 20-100 $\text{K}^*\text{s}/\text{mm}^2$ [36D.14]. In general, the microstructure will show columnar/elongated grains near the bottom of the melt pool, due to higher thermal gradients and lower solidification velocities, and equiaxed grains at the top of the melt pool, due to lower thermal gradients and higher solidification velocities. By adding inoculants, it is expected that more heterogeneous nucleation will occur ahead of the solid/liquid front, leading to a larger fraction equiaxed grains in the solidification microstructure. **Figure 36D.4** shows an example of as-built Inconel 718 with inoculants. Grains appear to have nucleated from an inoculant particle, indicating the feasibility of using inoculants to promote the CET [36D.15].

36D.3.2 Experiments at the APS

Beamtime at Sector 32-ID at the APS was allocated in February 2020 to observe real-time solidification dynamics using synchrotron X-ray imaging. Experiments were performed at low, medium, and high laser powers to sample a large portion of G - V space. Equiaxed grain Inconel 718 specimens, provided by MURI collaborators from the University of California Santa Barbara, were tested with and without a powder layer to observe any possible differences in the CET caused by the powder layer. Inoculated Inconel 718 as-built samples, provided by Elementum 3D, were tested with an inoculated powder layer to determine effects on the CET. Inconel 738 samples were tested with and without a powder layer as well. Interestingly, hot cracking was observed in the Inconel 738 samples with *in-situ* radiography in higher laser power conditions. All samples were tested with both spot and raster melt conditions. Other inoculated Ni-alloys (provided by Elementum 3D) were also tested including Inconel 625, Hastelloy 276, Rene 80, and CM 247LC.

36D.4 Plans for Next Reporting Period

All samples from the previous APS run will undergo post-mortem microstructural analysis, including:

- EBSD on spot and raster scans of Inconel 718 and Inconel 738 to determine the effects of varying laser power;
- Neutron diffraction measurements on Inconel 738 samples at Los Alamos National Laboratory to analyze texture and differences along the build height;
- Define a hot-cracking regime for Inconel 738 under LPBF methods using solidification velocity profiles from *in-situ* radiography data and post-mortem electron microscopy;

36D.5 References

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36D.6 Figures

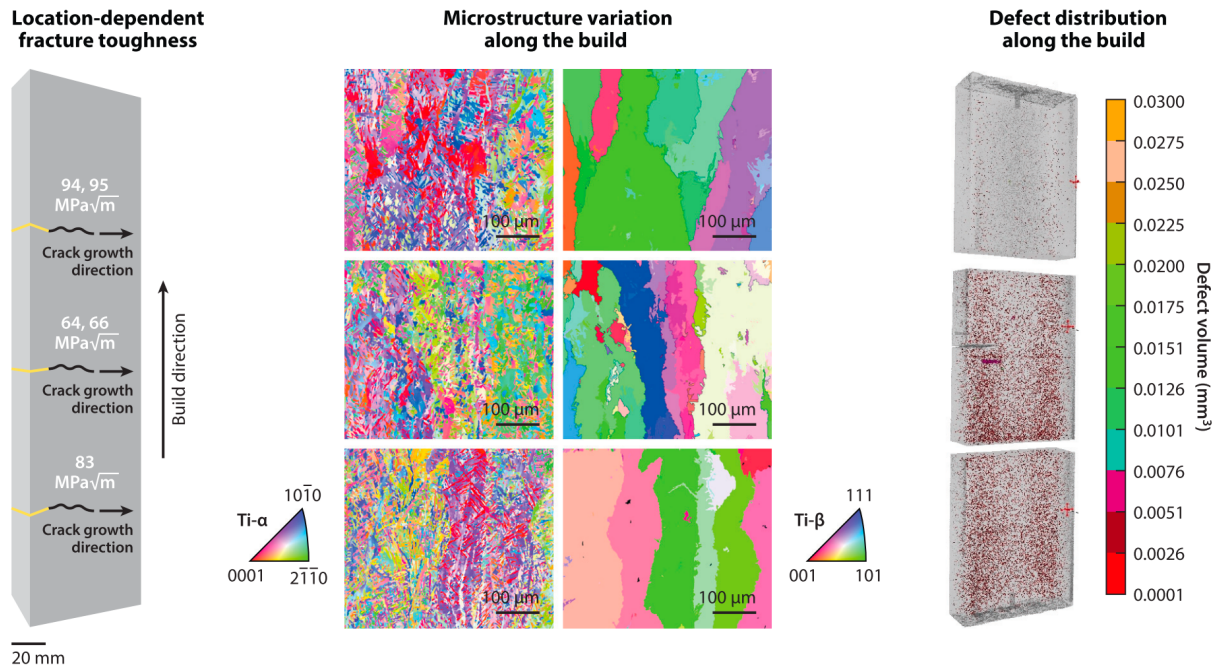


Figure 36D.1: Differences in underlying microstructure for an AM build of Ti-6Al-4V. Values for fracture toughness and defect distribution are also shown with respect to the build direction [36D.3].

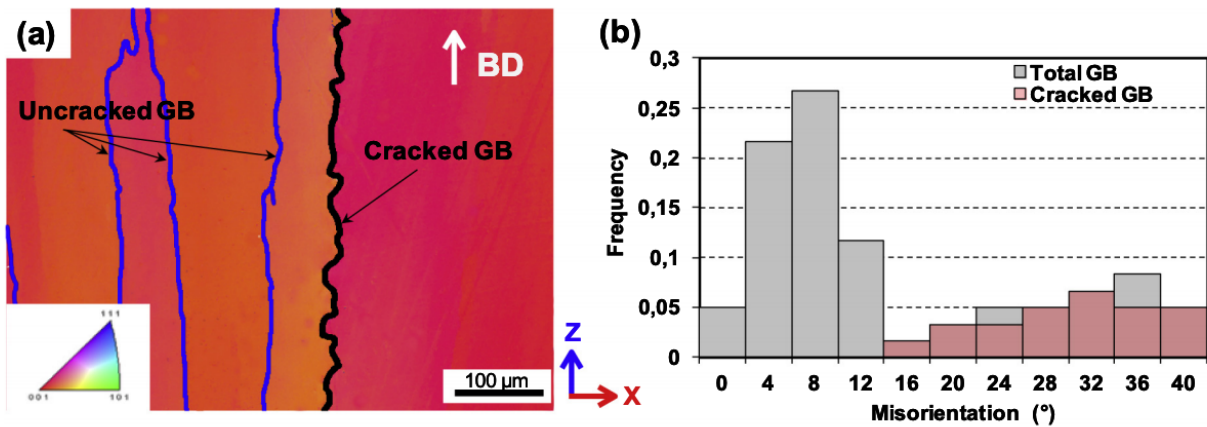


Figure 36D.2: Hot cracking along the build direction in AM Inconel 738LC. The cracks occur along high-angle grain boundaries (GBs) [36D.7].

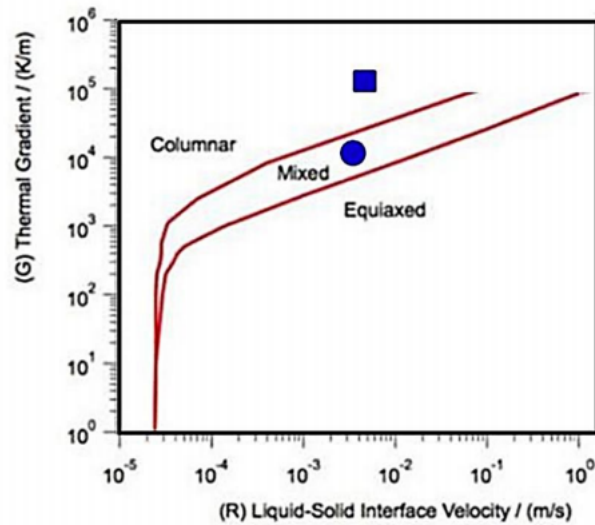
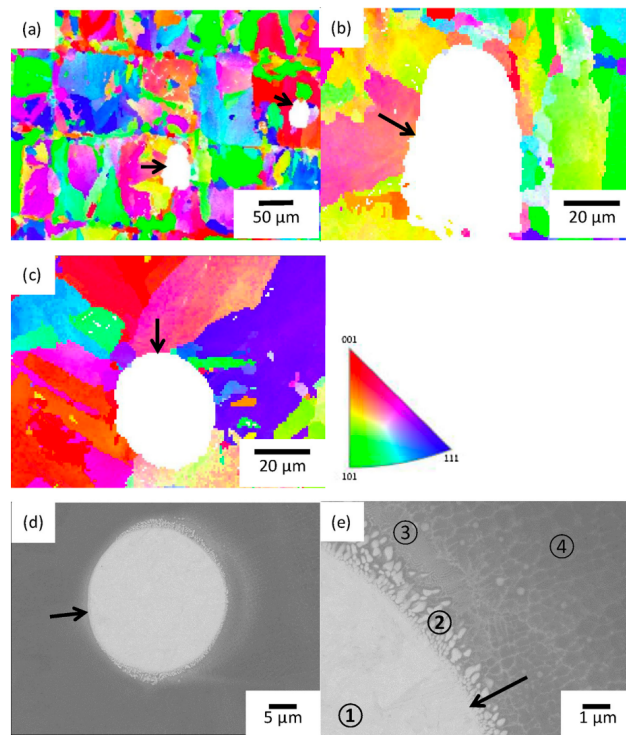


Figure 36D.3: Columnar to equiaxed transition plot for Inconel 718. The square represents a region of columnar dendritic solidification, and the circle represents a mixed region of columnar and equiaxed dendritic solidification [36D.13].



The arrows indicate the inoculants.

Figure 36D.4: EBSD inverse pole figure map of as-built Inconel 718 with inoculants along (a) the transverse direction, (b) magnified image of (a), and (c) along the longitudinal direction. (d) Backscattered electron scanning electron microscopy images of as-built IN718 with inoculants along the transverse direction. (e) Nucleation of new grains associated with the location of an inoculant particle [36D.17].