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Understanding the Role of Hot Isostatic Pressing Parameters on the Microstructural Evolution of Ti-6Al-4V and Inconel 718 Fabricated by Electron Beam Melting

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ABSTRACT

In this project, Avure and ORNL evaluated the influence of hot isostatic pressing (HIP) and thermal cycling as standalone post processing techniques on the microstructure of electron beam powder bed deposited Ti-6Al-4V and Inconel 718 alloys. Electron beam powder bed deposition is an effective technology for fabricating complex net shape components that cannot be manufactured with conventional processes. However, material deposited by this technology results in columnar grain growth which is detrimental for many applications. For Ti-6Al-4V, it has been found that thermal cycling alone is not sufficient to breakdown the columnar microstructure that is typical of electron beam powder bed technology. HIP, on the other hand, has the potential to be an effective technique to break down the columnar microstructure of Ti-6Al-4V into a more equiaxed and refined β grain structure, and provide a more homogeneous microstructure compared to the thermally cycled samples. Overall, the project showed that hot isostatic pressing reduced/eliminated porosity in both Ti-6Al-4V and Inconel 718 However, based on the unique thermal cycle and the application of pressure in the HIP vessel, Ti-6Al-4V e-beam deposited microstructures were modified from columnar grain growth to equiaxed microstructures; a significant outcome to this collaboration.

Inconel 718, on the other hand, shows no change in the macrostructure as a result of the current HIP cycle based on the thermal history, and would require further investigation. Though the results of HIP cycle were very good at changing the microstructure, further development in optimizing the post heat treatments and HIP cycles is required to improve mechanical properties.

1. UNDERSTANDING THE ROLE OF HOT ISOSTATIC PRESSING PARAMETERS ON THE MICROSTRUCTURAL EVOLUTION OF TI-6AL-4V AND INCONEL 718 FABRICATED BY ELECTRON BEAM MELTING

This phase one technical collaboration project (MDF-UP-2012-014) began on December 12, 2013 and was completed on December 31, 2014. The collaboration partner Avure Technologies, Inc.is a global leader in hot isostatic press fabrication and installation. The project was successful in demonstrating that hot isostatic pressing of electron beam powder bed deposited Ti-6Al-4V is an effective technique to break down the columnar microstructure into a more equiaxed grain structure which will broaden the potential fields of use for these additively manufactured parts. In addition, this project provided insights towards the role of currently existing HIP parameters on the microstructure and mechanical behavior of Inconel 718 and the need to modify these parameters for additively manufactured Inconel 718.

1.1 BACKGROUND

Powder bed based additive manufacturing has the potential to revolutionize transportation, energy production, and biomedical industries by producing complex near net shape parts, deposit material in a site specific manner and reducing the associated machining costs. However, the biggest challenge in powder bed based additive manufacturing technologies such as electron beam melting is the formation of columnar grains, along with a complex microstructure resulting from directional solidification and multiple heat cycles on subsequent layers as powder is deposited layer by layer. Figure 1 shows an optical micrograph of Ti-6Al-4V alloy fabricated using the ARCAM Q10 electron beam melting system. It can be seen that the β grains are columnar in nature and are parallel to the build direction. As a result of this directional nature of solidification, these parts often have slightly inferior mechanical properties compared to their wrought counterparts. The properties of these parts in the build direction (z) can be quite different than the properties in the x and y directions.

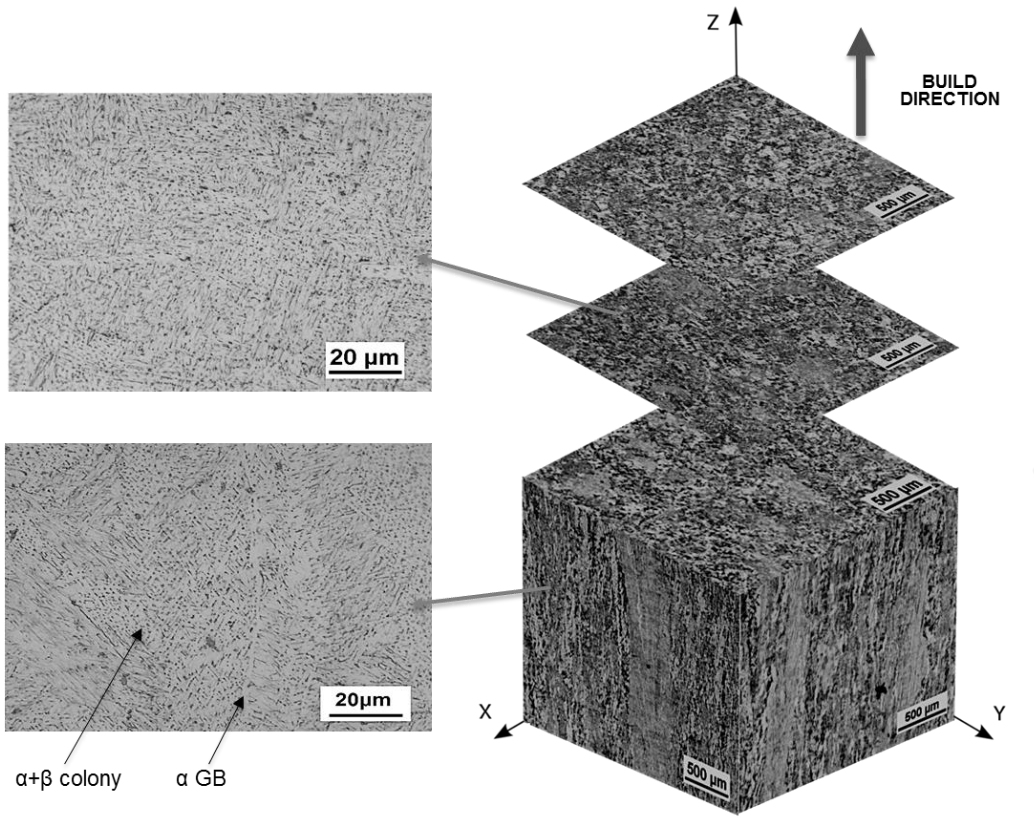

Fig. 1. Optical micrographs showing the α+β microstructure along with columnar b grain structure along the build (z) direction.

In order to increase the potential applications that can benefit from the complex geometries possible with additive manufacturing, it is important to develop a post processing schedule that would help breakdown the coarse columnar grain structure into a more refined and equiaxed grain structure. A thermal cycling regime along with a four step HIP cycle developed at ORNL has been used with the rapid quench HIP cycle technology developed by Avure Inc. to understand the potential influence of changing the microstructure of the as fabricated sample.

The post processing techniques resulting from this CRADA between UT-Battelle LLC and Avure Inc. will help enhance properties of additively manufactured components. Additive manufacturing has the ability to decrease weight of components over conventional processing, improve heat exchange in heat management applications, and improve the energy efficiency of other systems. However, the technology requires further development to improve and/or modify resulting microstructures inherent to the process.

1.2 TECHNICAL RESULTS

Oak Ridge National Laboratory performed the electron beam deposition of samples used in this project, performed thermal only post processing, and performed the mechanical and microstructural evaluations. Avure performed all hot isostatic press processing, and worked with the ORNL team to develop the thermal and pressure treatments. The following sections will provide the results found by this team.

1.2.1 Deposition, Microstructure Analysis and Mechanical Testing (ORNL)

Cylindrical and prismatic bars of Ti-6Al-4V were fabricated using the ARCAM Q10 electron beam melting system at the ORNL Manufacturing Demonstration Facility (MDF). Standard E-Beam deposition parameters provided by ARCAM were used with the powder lifts deposited at 50um. Samples were then either retained for analysis at ORNL on as fabricated samples and to carry out thermal cycling experiments, or were sent to Avure Inc. for rapid quench and cycle HIP processing. The ORNL samples were thermally cycled at ORNL using a Gleeble system (HT1). The samples retained by Avure Inc. were subjected to two different HIP cycles. The first cycle involved subjecting the samples to the same thermal cycle as the Gleeble samples at a constant holding pressure of 100MPa (HT2). The second and third HIP cycles involved holding the samples at 920^{o}C for two hours under a pressure of 100MPa (HT3 and HT4). However, some samples (HT3) were rapidly quenched using a proprietary technique developed by Avure Inc. instead of the standard practice of a natural cool-down (HT4). The samples from ORNL as well as Avure Inc. were then subjected to microstructural analysis, the details of which will be discussed subsequently.

The Inconel samples were deposited at the ORNL MDF using the ARCAM A^{2} EBM system. The HIP processing for Inconel 718 samples were carried out by Avure Technologies Inc., the details of which are provided in the following section. ORNL carried out the microstructural and mechanical characterization of the as deposited and HIP'd samples.

1.2.2 Hot Isostatic Pressing (HIP) (Avure Inc.)

Avure Inc. carried out hot isostatic pressing on the Ti-6Al-4V samples using three different thermal cycles (HT2, HT3, and HT4). Figure 2 presents the thermal cycle that was common to both Gleeble HT1 and HIP processed samples HT2. These two sets of samples have been evaluated for in-depth microstructural analysis. The other two HIP (HT3 and HT4) cycles involved subjecting the samples to the standard HIP cycle recommended by ARCAM Ab, which is 920^{o}C for 2 hours at 100MPa. The difference between these two sets is only the cooling rate with one set being subjected to normal cooling (HT4) and the other set being rapidly quenched using a proprietary technique developed by Avure Inc. (HT3). The thermal history of Ti-6Al-4V samples is summarized in Table 1 with appropriate nomenclature that would be used henceforth.

Table 1. Processing conditions for Ti-6Al-4V

Cycle nomenclature	Thermal Profile	Pressure and Cooling Rate
HT1(Gleeble)	25C→1020C-10min→700C→1020C-10min→700C→1020C-10min→700C→1020C-10min→700C→25C	-N/A- and 285C/min
HT2	25C→1020C-10min→700C→1020C-10min→700C→1020C-10min→700C→1020C-10min→700C→25C	100MPa and rapid cool
HT3	25C→920C-2 hours→25C	100MPa and rapid quenching
HT4	25C→920C-2 hours→25C	100MPa and standard cool

The Inconel 718 samples were subjected to HIP at 1120^{o}C for 2 hours under a pressure of 100MPa.

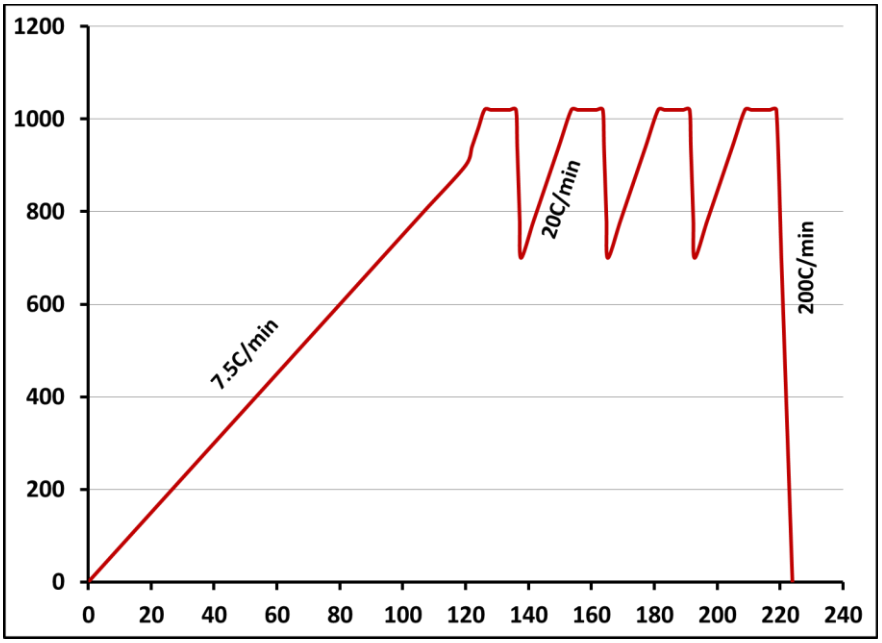

Fig. 2. Temperature profile of Ti-6Al-4V samples subjected to thermal cycling in HT1 and HT2.

1.2.3 Results and Discussion Ti-6Al-4V

Figure 3 (a) shows a schematic cross section of a typical sample after processing. The arrows indicate the positions on the sample where optical microscopy was carried out. Figure 3 (b) shows the micrograph of the sample in the as-built condition and the resulting process porosity is evident as black spots in the micrograph. Figure 3 (c) presents the micrograph of a sample subjected to the HT3. The HIP process eliminates porosity. However the β grain structure remains columnar and similar to that of the as built sample. The samples subjected to HT1 in the Gleeble also display the columnar structure as shown in Figure 3 (d). On the other hand Figure 3 (e) shows the micrographs for samples subjected to HT2, a significant change in the microstructure from samples in as-fabricated condition and also samples subjected to HT3 and HT1. The α laths help identify the prior β grain boundaries and it can be seen that the β grains are now more equiaxed and the microstructure is homogeneous. This result demonstrates that the HT2 developed in this project is an effective post processing route for changing the as build structure of electron beam deposited Ti-6Al-4V, accomplishing the primary objective of the project which aimed at breaking down the columnar microstructure of β grains into more equiaxed grains using the HIP process.

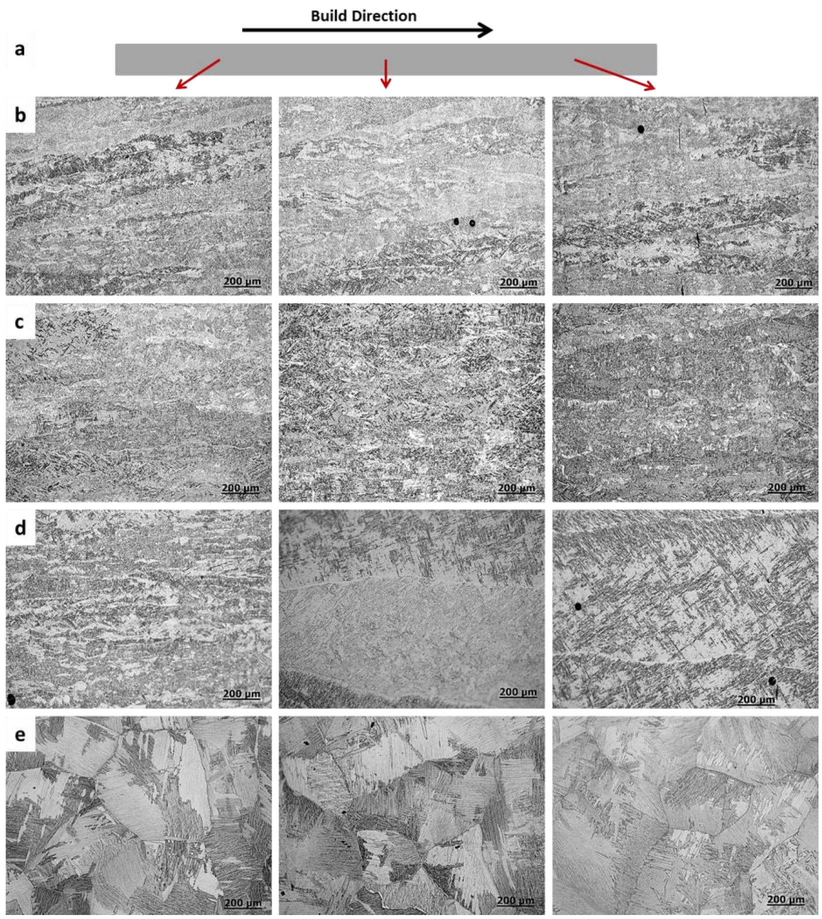

Fig. 3. (a) Build direction and marks the regions where the optical microscopy was carried out, (b) micrographs for the as built samples, (c) micrographs for samples subjected to standard HIP cycle with rapid quenching HT3, (d) samples thermally treated in the Gleeble (HT1) and (e) samples subjected to 4 HIP cycle regime with rapid quenching HT2.

A theory behind the resulting equiaxed βgrains when HIP'd above the β transus is that the stored plastic energy is sufficient to cause new grain growth and recrystallization at local defects, whereas when HIP'd below the transus temperature, this phenomenon may be hindered by the presence of α phase.

Figure 4 (a) illustrates the microstructures throughout the cross section of the samples subjected to HT1 (left) andHT2 (right) samples. It can be seen from Figure 4 (b) that the samples subjected to HT1 result in an inhomogeneous microstructure on edges vs. the center of the sample, with the edges showing alpha casing based on exposure to oxygen during the Gleeble process. On the other hand, the HIP processed (HT2) microstructure is largely homogeneous across the specimen as evident from Figure 4 (c).

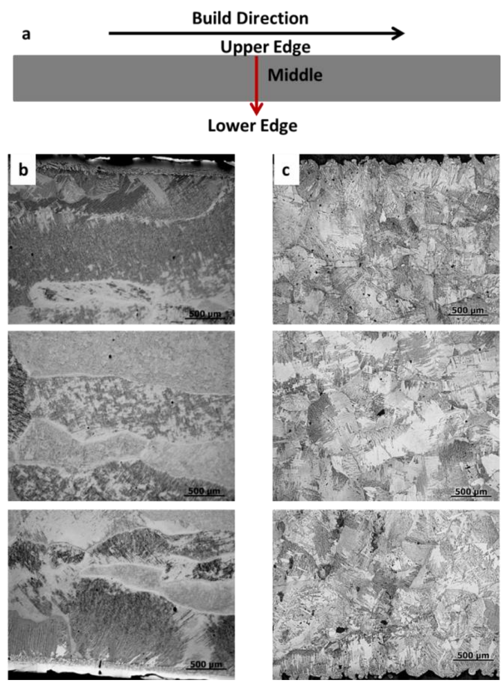

Fig. 4. Change in microstructure from one edge of the sample for (b) thermally cycled samples HT1 and (c) HIP processed samples HT2.

Electron Back Scattered Diffraction (EBSD) analysis was carried out on samples subjected to HT1 and HT2 to further establish the grain structures. Figure 5 shows the columnar microstructure from HT1 sample, whereas Figure 6 shows the more equiaxed grain structure from the HT2 processed sample. The grain boundaries can be distinguished by the colony structure that originates at the grain boundary.

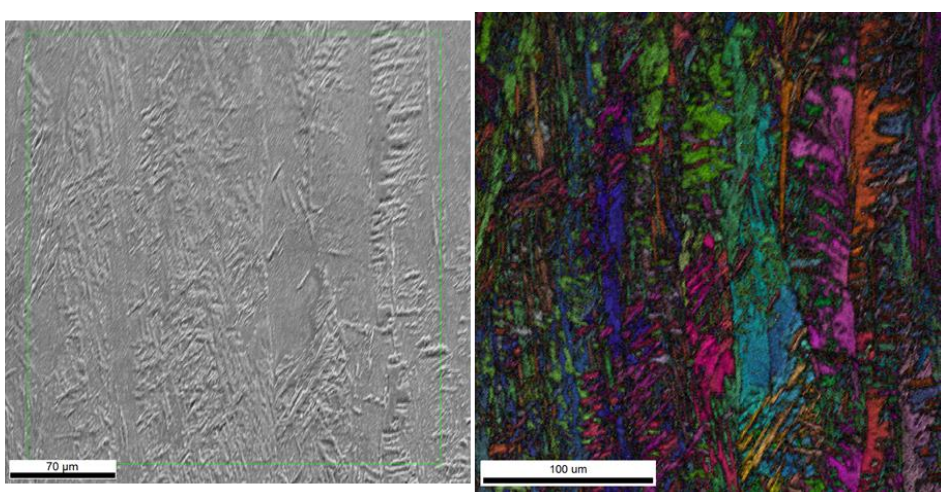

Fig. 5. EBSD map showing the columnar grain structure obtained from the thermal cycling HT1.

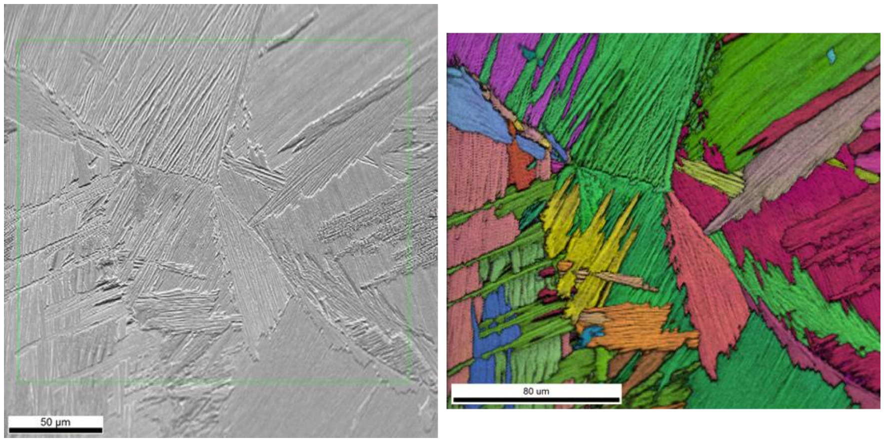

Fig. 6. EBSD map showing the more equiaxed structure obtained by HIP processing HT2.

It can be seen from figures 5 and 6 that the thermally cycled sample (HT1) along with HIP standard cycle and as built samples have fine scale α laths whereas the 4 cycle HIP (HT2) sample develops large α colonies inside individual β grains. These α colonies may have a deleterious effect on the fatigue properties as discussed below.

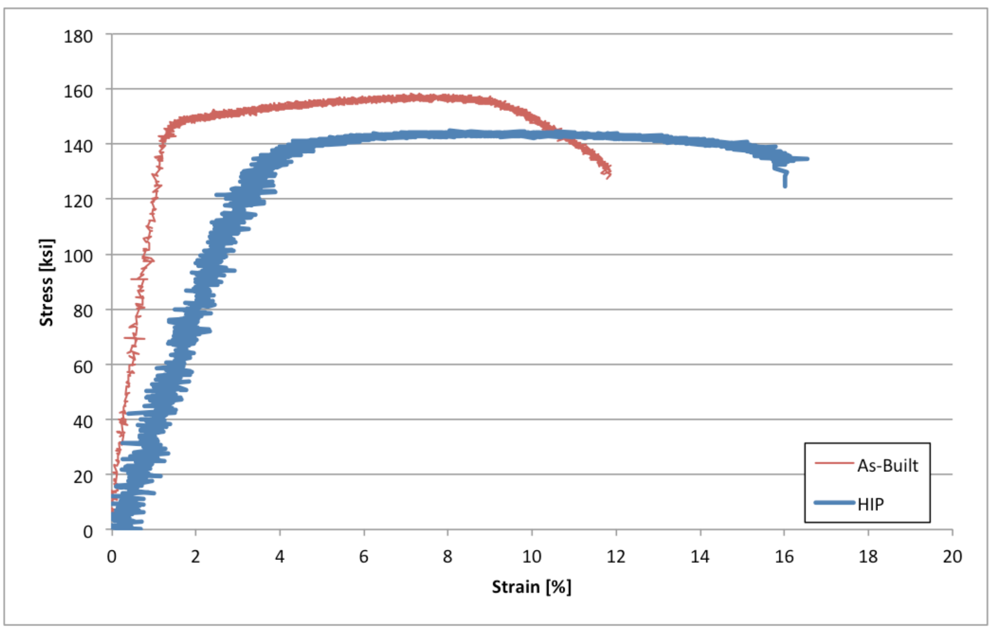

Fig. 7. Monotonic tensile data for the as built and 4 cycle HIP samples HT2.

Figure 7 shows the monotonic tensile curves for the as-built and 4 cycle HIP (HT2) samples. Both tensile curves look good with high strength values and good elongation. Strength values above 130 ksi and 10% elongation exceed ASTM Ti-6Al-4V specifications. The HIP cycle did not improve the strength of the electron deposited material since values were already very good with little impact from the limited porosity found in the as-deposited. In fact, it seems to have slightly lowered the tensile strength due to the reduction in the average alpha lath (grain) width and the long time exposure to high temperature. However, the HIP process had a significant effect on the total elongation of the test specimen.

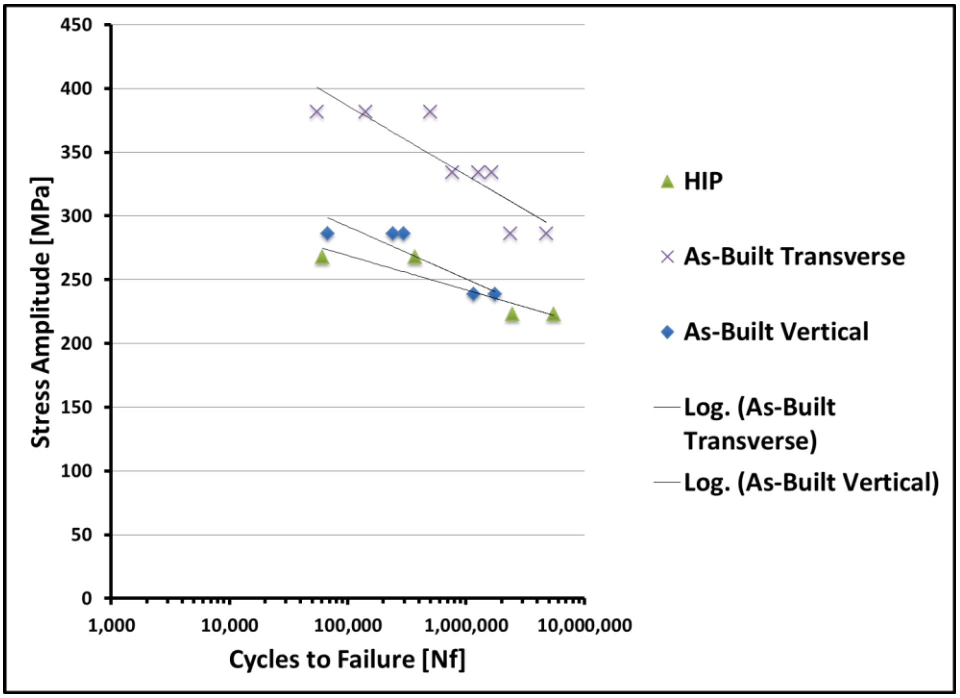

Fig. 8. The S-N curve for the as built and 4 cycle HIP samples HT2.

From figure 8 it can be seen that the as-built fatigue strength is higher in the transverse direction than in the build direction, illustrating the non-isometric properties of the e-beam deposited samples. The HIP'd sample (HT2) results are very similar to the As-Built vertical samples. This may be due to the fact that any improvement observed by closing porosity during HIP is offset by the increase in α lath size as well as the size of the colonies. The increase in the α lath width and the colony size reduces the effective slip length and a crack once initiated can travel unhindered through the colony until it meets the colony of a different orientation, thus lowering the fatigue life as shown in Figure 8.

Future research should be geared towards further modifying the thermal cycle of the HIP and/or developing a post heat treatment for the material now that equiaxed microstructures are known to be achievable. The end result of further developing the post heat treatment or modifying the thermal cycle of the HIP is anticipated to improve the fatigue result, balancing the results of good elongation and fracture toughness with high strength and a good fatigue endurance limit.

1.2.4 Results and Discussion Inconel 718

The Inconel 718 sample was subjected to the HIP standard cycle followed by rapid quench. The sample was subjected to a pressure of 100MPa at 1120^{o}C and held for 2 hours before being rapidly cooled to room temperature. Figure 9 (a, and b) shows the microstructures of the as-built and HIP'd samples respectively. The grain boundary decorations seen in the as-built sample are not visible in the HIP'd sample. However, the grain structure remains largely columnar in both cases and HIP has no influence on changing the grain structure.

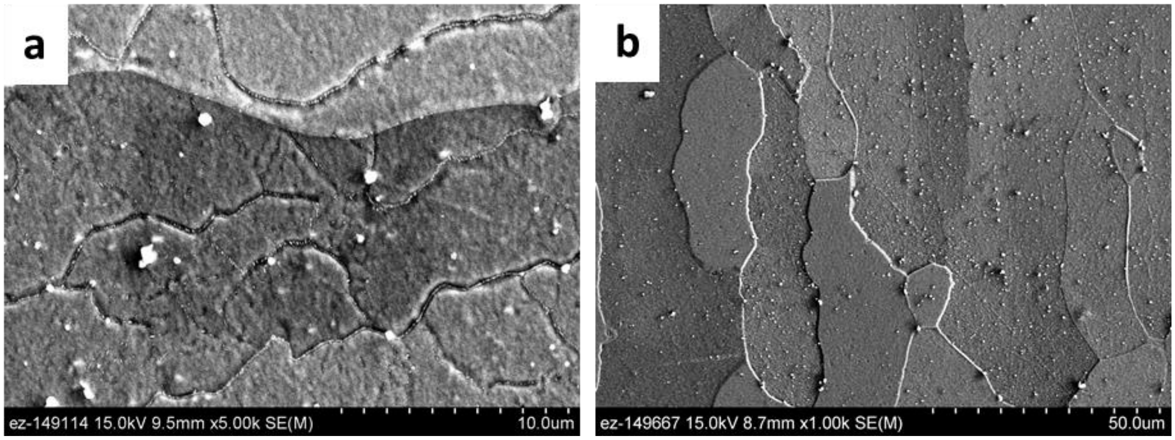

Fig. 9. Scanning electron micrograph of Inconel 718 (a) as-built sample, and (b) HIP'd sample.

Figure 10 shows the influence of HIPing on the monotonic tensile curves for the Inconel 718 samples. It can be seen that the as-built sample, despite the porosity, has a higher tensile strength than the HIP'd sample. However, elongation has been greatly improved for the HIP'd sample. The drop in tensile strength can be attributed to the fact that at the HIP temperatures the precipitates dissolve in the matrix and do not precipitate out during cooling due to the high cooling rates. Thus, a drop in the tensile strength is observed since Inconel 718 is a precipitation-strengthened system.

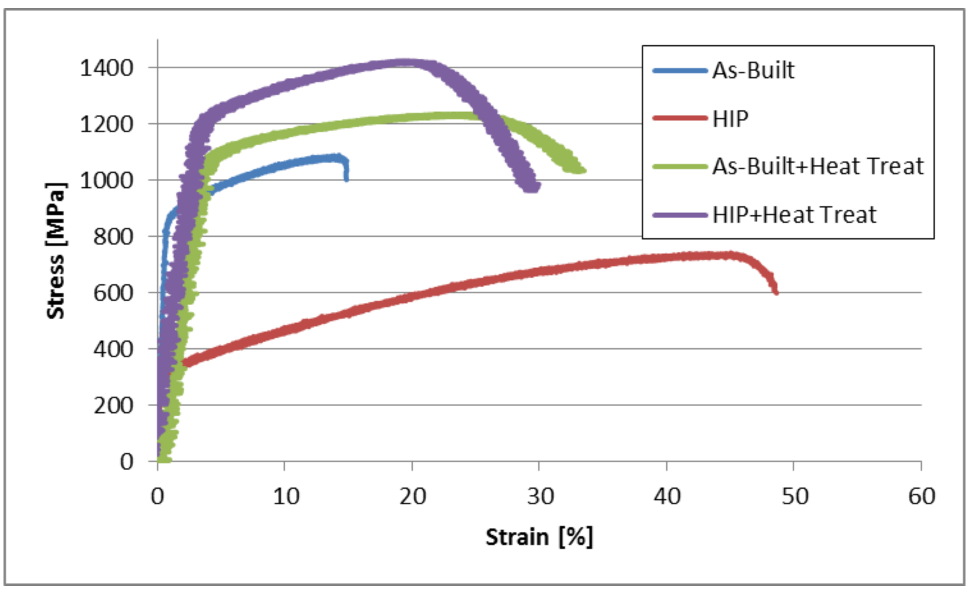

Fig. 10. Monotonic tensile curves for as-built , HIP only, as-built plus heat treated, and HIP plus heat treated Inconel 718 samples.

Based on the microstructural observation of dissolution of precipitates during the HIP cycle, additional research was performed on the proper heat treatments for HIP'd samples and compared with as-built and as-built plus heat treatment. The additional heat treatments and mechanical testing was supported by the Manufacturing Demonstration Facility's metals additive manufacturing task, but is provided here to provide additional information as to the impact of proper heat treatment. A five step heat treatment was used to strengthen the HIP'd samples. The samples were first held at 1066°C for one hour and then air cooled to room temperature. Then the samples were held at 760°C for ten hours followed by furnace cooling to 649°C where they were held for twenty hours before being finally air cooled to room temperature.

As Figure 10 illustrates, the heat treatment has a significant impact on the final strength values. The HIP'd plus heat treated sample had the highest strength value, and best overall mechanical behavior. However, the role of cooling rates and control of cooling rates during the HIP process needs to be investigated in greater detail, thereby allowing to attain the desired mechanical properties during a single HIP cycle, thus reducing the time and cost associated with additional heat treatments that are currently required.

1.3 IMPACTS

The post processing techniques resulting from the current CRADA between UT-Battelle LLC and Avure Inc. will help enhance properties of additively manufactured components. Additive manufacturing has the ability to decrease weight of components over conventional processing, improve heat exchange in heat management applications, and improve the energy efficiency of other systems. However, the technology requires further development to improve and/or modify resulting microstructures inherent to the process.

Commercialization Possibilities: The preliminary microstructural analyses on Ti-6Al-4V are promising and have a potential for commercializing the technique resulting from the current CRADA. In addition, these results further the ability for hot isostatic pressing to reduce porosity and can be used to alter microstructures; these results can lead to the increased sale of hot isostatic presses. Avure has interest in continuing this research with ORNL to improve mechanical properties of additively manufactured components.

Plans for Future Collaboration: Future collaboration involves developing appropriate heat treatment cycles for Ti-6Al-4V and Inconel 718 systems to improve their mechanical properties. One possible way to improve the fatigue life of Ti-6Al-4V would be to β solutionize the sample followed by a rapid cooling that will result in the basket weave structure instead of the colony microstructure thus producing finer lamellae and smaller colonies. Also, samples should be subjected to just a single HIP cycle above the β transus to understand the influence of the number of cycles on transforming the grain structure from columnar to equiaxed. For Inconel 718, post processing thermal treatments that could be performed in the HIP vessel as part of the HIP processing would be advantageous, could save energy, cost, and allow for the precipitation of the desired phases.

1.4 CONCLUSIONS

The key results from this CRADA can be summarized as follows:

a) Thermal cycling or pressure alone is not sufficient to change the β grain structure from columnar to equiaxed for electron beam deposited Ti-6Al-4V. A correct combination of pressure and HIP temperature (above β transus) as displayed in the 4 Cycle HIP (HT2) can result in equiaxed grain formation in Ti-6Al-4V.
b) The temperature profiles are closely controlled in HIP as compared to Gleeble which is reflected in the microstructures across the sample. The thermally cycled microstructure is inhomogeneous and is oxygen enriched whereas the HIP processed microstructure is more homogeneous across the width of the Ti-6Al-4V sample.
c) Preliminary HIP processing of Inconel 718 without post heat treatments causes a decrease in the monotonic tensile strength due to dissolving the strengthening phases. However, a proper post heat treatment greatly improved the overall tensile strength, providing higher strength

values as compared to the as-fabricated sample.

d) Further development is needed for developing a HIP cycle that eliminates subsequent heat treatment. Avure's rapid quenching technology may be able to provide the correct cooling rates for both Ti-6Al-4V and Inconel 718 to attain desirable mechanical properties.

Based on the above results it is evident that HIP processing stands out in terms of attaining a homogeneous and equiaxed microstructure as compared to thermal cycling alone and hence is a viable option to modify the microstructure of the as fabricated EBM processed Ti-6Al-4V components.

2. PARTNER BACKGROUND

For more than 50 years, Avure has been the global expert in applying high pressure technology to industrial and consumer goods manufacturing. Avure has a global installed base of over 1,200 isostatic presses. Avure Technologies offers a total and sustainable solution as a long term partner to help advance the knowledge and capabilities in critical advanced alloy and ceramic manufacturing areas.