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Microstructure Change of an Additively Manufactured High-Strength Titanium Alloy Over Large Areas Using Mapping and EBSD

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Titanium alloys are heavily used due to their high-performance characteristics and near crack-free processing using traditional methods [1]. Machining of titanium parts leads to large amounts of scrap which may not be able to be reused. Additive manufacturing (AM) is an attractive alternative to traditional methods as it can be used to create uniquely shaped parts with little waste compared to traditional machining or casting methods [2]. However, during the additive manufacturing process, titanium generally forms large columnar grains parallel to the build direction resulting in anisotropic properties. It has been shown that by alloying with Fe or Ni the grains will transition to an equiaxed grain structure [3]. Characterization of large AM builds has typically been done over small regions due to time and equipment limitations. Recent advances in imaging and analytical technology have made it possible to collect large area SE, BSE and EBSD maps in a reasonable time to fully understand non-homogeneous samples.

In this work, an Optomec LENS Directed Energy Deposition AM system was used to deposit a compositionally graded specimen from Ti-64 (base layer) to Ti-64 + Fe (top layer) as seen in Fig. 1a and 1b. A full description of the process can be found in Welk, et al. 2021. Imaging and EBSD of the sample were performed in the ThermoFisher Scientific Quattro SEM at The Center for Electron Microscopy and Analysis. Mapping of a cross-section parallel to the build direction was performed using secondary and backscattered imaging with automated MAPS software (step size 337 nm). Large area EBSD mapping of a sample subsection was performed using an EDAX Velocity detector (step size 4 µm) in 4 total montage collections. The prior beta grains were reconstructed from the EBSD alpha phase orientation dataset. The prior beta grain reconstruction process is described further in Pilchak et al. 2011.

Initial characterization of the entire build cross-section was performed using large area mapping in the SEM. The large area imaging software (MAPS) allows for interpolated focus, so large areas are easily imaged even if the sample is not perfectly parallel to the stage. The microstructure variation across the LENS deposited sample can be observed with backscattered electron imaging. As seen in Fig. 1c, the microstructure changes along the build direction (approximate area is 12 mm x 28 mm) but does not change substantially across each layer. Imaging a data set this large can be problematic for subsequent data analysis. For quantitative analysis, it can often be beneficial to segment out smaller sections from the large map or collect many smaller maps across the length of the specimen (either along build direction or across the build layers). While the grains can be observed in the BSE large area map, they are difficult to quantify, so additional analysis of the sample was performed using EBSD.

After using BSE mapping to confirm the structure was similar across the build layers, a smaller area (approximately 4mm x 26mm) was characterized by EBSD. Figure 2 shows the grain boundary map overlaid with the field of view, the reconstructed prior beta grain for unique grains, and the inverse pole

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figure. From EBSD analysis the grain size and morphology are clearly observed with coarse columnar grains predominating at the start of the build for the base alloy with structures up to 10 mm in length. Full analysis of small, selected areas of the sample can be found in Welk et al. 2021. With the addition of Fe to Ti-64, the grain structure significantly changes from course columnar grains to an equiaxed grain structure with grains on the order of 100s of microns. To successfully acquire a dataset like the one seen in Fig. 2, extra care must be taken to ensure data quality is maintained across the sample along with project size for data management and processing solutions. Pattern quality across a large area scan in this highly tilted geometry needs to be optimized by ensuring the sample is completely parallel to the stage surface prior to tilting. Large area scans may also require montage mapping if pattern quality is lowered by artifacts near the extremes of the scan. Slight errors in the x/y stage movement and misalignment to the beam scan can cause large discrepancies, which may add the need for overlap depending on accuracy needed. This analysis has shown the benefits of using large area SE, BSE and EBSD mapping to fully characterize a sample. Analysis of isolated smaller regions across a compositionally graded specimen would be time consuming and could potentially miss important transitions. Being able to see the entire grain structure along the build permits a better understanding of the microstructure as a function of composition.

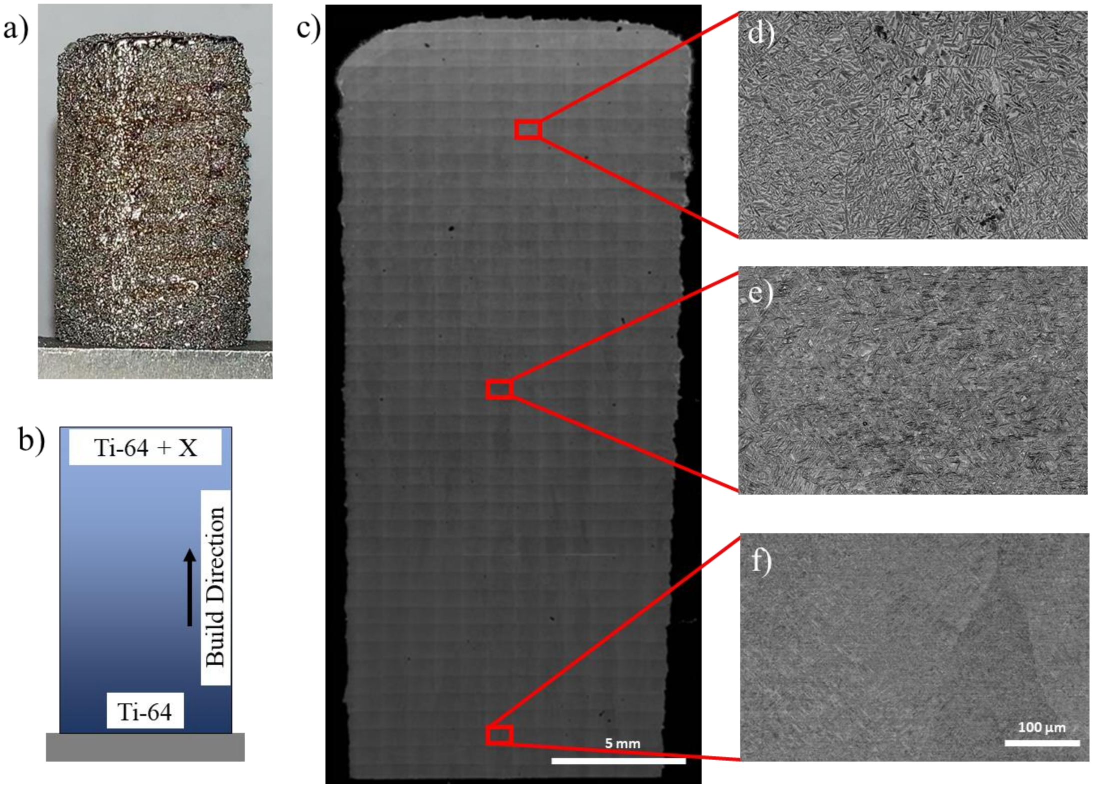

Figure 1. Large area cross-section imaging of the compositionally graded AM specimen. a) As-deposited specimen and b) schematic showing the composition and build direction. c) BSE image of total mapped area and full resolution images from the d) top, e) middle, and f) bottom.

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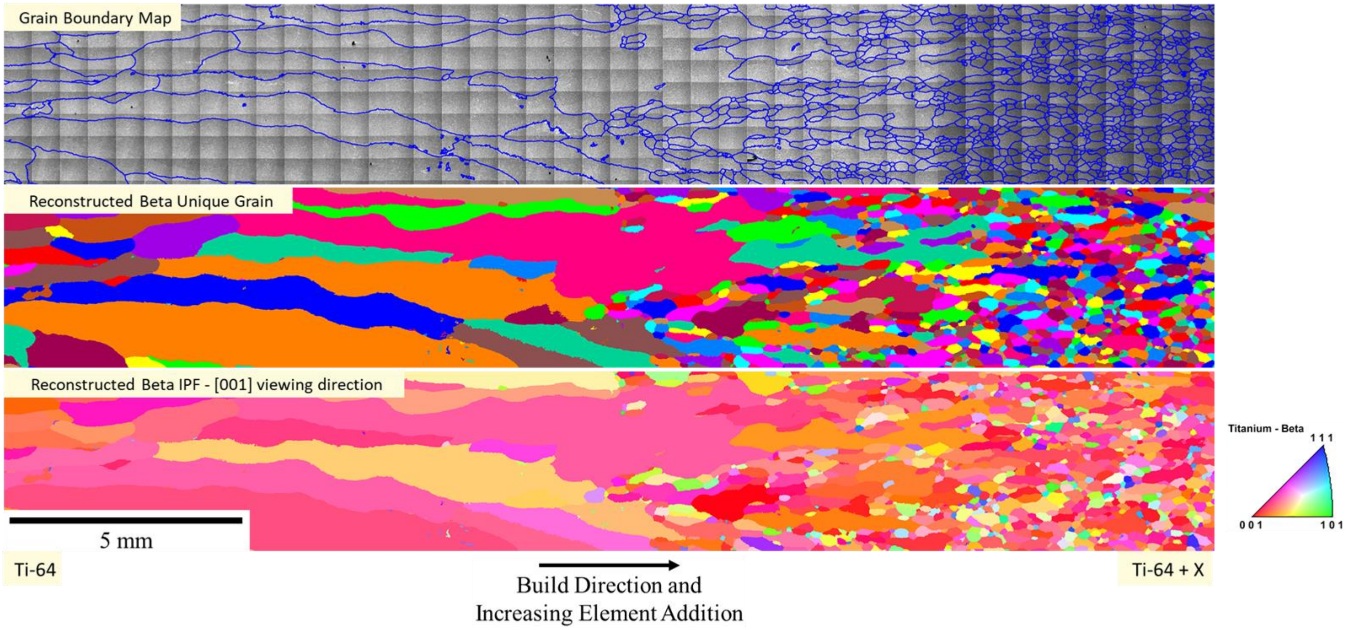

Figure 2. EBSD analysis of the compositionally graded sample along the build direction with Ti-64 on the bottom (left side) and Ti-64 + Fe on top (right side). a) Grain boundary map overlaid on the SE image, b) reconstructed beta unique grain map, and c) reconstructed beta grain inverse pole figure map in the [001] viewing direction.

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