doi:10.1017/S1431927619002460
Microsc. Microanal. 25 (Suppl 2), 2019

Combining EBSD with Serial Sectioning for the 3D Analysis of Materials

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Recent decades have shown significant progress in the analysis of 3D microstructures across many length scales, with Atom Probe Tomography (ATP) and Transmission Electron Tomography at nanometer length scales, and various X-ray computed tomography (microCT) techniques at length scales that span sub-micron to millimeter resolutions. While not as common, serial sectioning has some distinct advantages over these more traditional techniques, specifically the ability to collect large analysis volumes that contain thousands of objects at high resolutions thereby allowing for large statistical analysis of 3D objects. There are a number of material removal methods for serial sectioning, including focused ion beams, plasma beams, laser machining, and traditional mechanical polishing. One factor of 3D characterization that is often underestimated is the amount of time and effort needed to segment the domains of interest within the 3D volume, often requiring the processing of thousands of 2D images. The time associated with this task is a function of the imaging technique, the quality of contrast between features and the ability of the processing algorithm to translate that contrast into clearly defined domains. For multi-phase systems where density differences (as in the case of traditional microCT) are large, or where compositional differences are large (as in ATP), this task can be relatively easy. Even in serial sectioning, combining an appropriate etchant that attacks one phase preferentially to another with optical microscopy can make this a relatively easy task. However, most single phase polycrystalline systems show very poor and inconstant contrast at the grain interfaces, making the 3D analysis of these structures extremely difficult.

These limitations can be overcome by incorporating advanced imaging methods that are suited for the differentiation of polycrystalline structures, such as Electron Backscattered Diffraction (EBSD). While EBSD is not a new methodology, the ability to map out large areas at high resolutions has become much more feasible as collection rates have increased in recent years. With EBSD data collected for every section, then the segmentation of the structure into separate grains is easily accomplished by selecting a misorientation threshold to denote a grain boundary interface.

To this end we have developed the Robotic Serial Sectioning System for 3D (RS3D), which incorporates a fully automated polishing station -- which performs the material removal between sections -- with an automated scanning electron microscope equipped with EBSD, and a robotic interface between the SEM and polishing station. Here we show the 3D structure of an Additively Manufactured (AM) 316L stainless steel from powder-bed fusion, with a build layer thickness of 30μm. Figure 1 shows the 3D reconstruction of the AM316L from 300 sections, with a spacing of 1.44μm and an in-plane step size of 0.75μm. The cycle time for one section is approximately 2.5hrs, with approximately 2hrs for imaging. Thus the entire dataset required 30 days to collect. While this is not a trivial amount of time, very little user interaction was required with only approximately 1 day used to stack, align and segment the data, thus the total data collection to analysis time is relatively short compared to most 3D data collection. From this reconstruction it is obvious that the microstructure formed by the rapid solidification is highly complex, with grains that span multiple AM layers. Figure 2 shows a reconstruction of one large grain in the volume, which demonstrates that the grains are not simply columnar, but that they demonstrate a large amount of branching. These branches are associated with local variations in the raster direction changing the local temperature gradients, and thus the local preferred growth direction [1].

References:

[1] This work was funded by the Naval Research Laboratory under the auspices of the Office of Naval Research and from the Structural Metallics program of ONR.

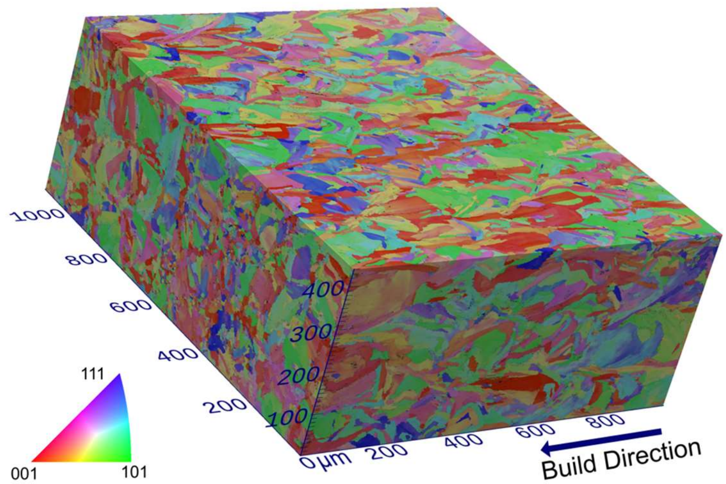

Figure 1. 3D EBSD reconstruction of additively manufactured with powder-bed fusion 316L. Colors are determined from the legend and the crystallographic direction aligned with the build direction.

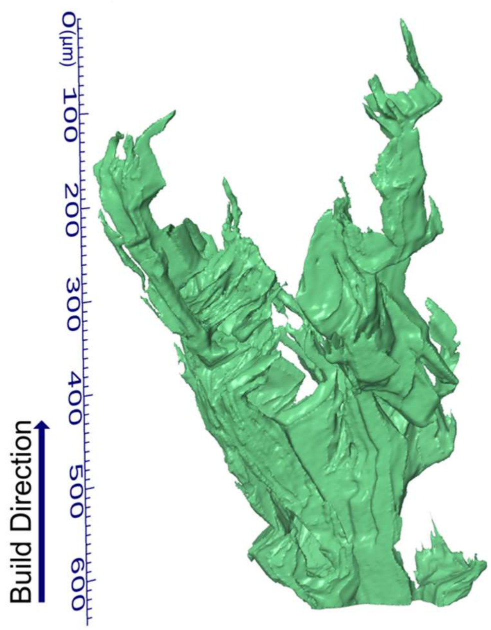

Figure 2. 3D Reconstruction of a single grain from the AM 316L dataset. Color is correlated with the average crystallographic direction aligned with the build direction.