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# Investigation of the Multiscale Microstructure of an Age-Hardenable Metal Matrix Composite using Correlative Microscopy

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High strength aluminum alloys are essential materials for modern aerospace applications. Aluminum's low density, castability, and precipitation hardening capability through alloying allows fabrication of complex geometries with very high specific strengths. Strength, ductility, toughness, creep and fatigue resistance depend on controlling microstructural features from the sub-nm to mm scales, i.e. solute atoms, defects (dislocations to voids), precipitates (nano- to micro-meter scale) and polycrystals (size, shape, orientation, boundary type). Materials scientists work to understand the interplay of local microstructure and the intrinsic mechanisms at each length scale that control the mechanical properties, but too often focus on one component, i.e. nano-precipitates on yield strength, because of the range of instruments required and loss of fidelity from one instrument to another at the intersection of their resolutions.

In many fields of study, it is imperative to understand the behavior of a material system across several length scales, and in 3D, in order to properly address the structural parameters that govern its performance. As characterization techniques have progressed individually, a clear challenge that has emerged has been how to intelligently navigate to and acquire 3D volumes of interest (from cm-scale to nm-scale in three dimensions), and, subsequently, to efficiently fuse multi-scale, multi-modal datasets across length scales so as to enable navigation to and investigation of regions of interest (ROIs) based on multi-modality information collected from a range of microscopes. While TEM and SEM provide high resolution to probe the microstructure down to the atomic scale, they enable investigation of smaller volumes requiring destructive preparation. This gap can be bridged by using non-destructive X-ray microscopy covering the nano- to micro-scale while providing information from a much larger probe volume and enabling better statistics and sample representivity. Recently, adaptations of diffraction contrast tomography on lab-based X-ray microscopy (termed as LabDCT) allow determining the crystallographic information of large volume of grains non-destructively. The large 3D grain data, complements high-resolution grain orientation data obtained from EBSD. Additionally, EDS mapping delivers elemental information of the sample. The most important aspect of such a correlative approach is the unison of software and hardware solutions to be able to seamlessly integrate data from a variety of modalities and resolution scales.

In this work, we present correlative microscopy results from different imaging modalities allowing us to visualize and explore the rich multiscale microstructural landscape of a recently developed age-hardenable Al-Cu-Mg-Ag-$TiB_2$ in-situ composite alloy [1] which can be sand-cast into near net-shape geometries with grain size controlled by cooling rates [2] and features a non-dendritic, isotropic cellular aluminum grain matrix with interfaces decorated by $TiB_2$ particles. Further, through specific heat treatments the matrix can be strengthened by controlling the dispersion of copper segregated during the solidification process [3]. In the as-cast state the sample has a grain size of 50 micrometers and after heat treatment can develop a rich hierarchical microstructure with features ranging in size from few nanometers to several micrometers. In this work we compare the microstructure of as-cast and heat treated conditions. A typical workflow begins with non-destructive 3D X-ray absorption and LabDCT, to obtain 3D morphological and grain orientation data, enabling investigation of the microstructure at the macroscale (grain shapes, orientation and grain boundary characteristics, void and other defect distribution). A specific region of interest within the large volume is accessed using a combination of the laser and FIB milling. Deeper ROIs that are more than a few hundred micrometers below the surface can be rapidly excavated using a femto-second laser integrated into the Crossbeam (FIB-SEM) load lock. “Atlas 3D” FIB-SEM tomography is performed on the exposed ROI, allowing a wide variety of imaging modalities as well as analytical techniques such as EBSD or EDS to be performed over 3D volumes with nm voxels across thousands of cubic-microns. Resulting images from the correlative workflow demonstrate a fused multimodal, multiscale dataset for easy analysis and investigation of the in-situ cast alloy composite.

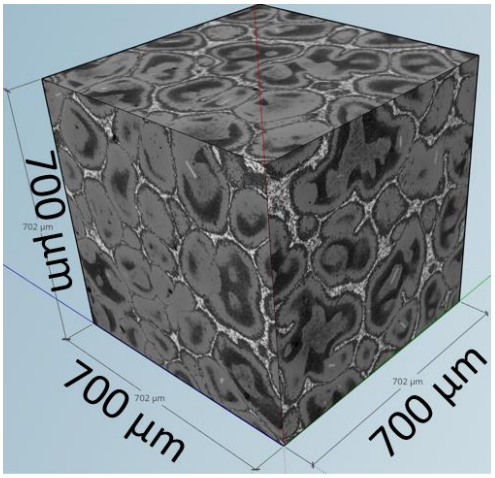

**Figure 1.** Composite 3D reconstruction from orthogonal optical microscopy images. X-ray microscopy can obtain similar resolution non-destructively throughout the volume.

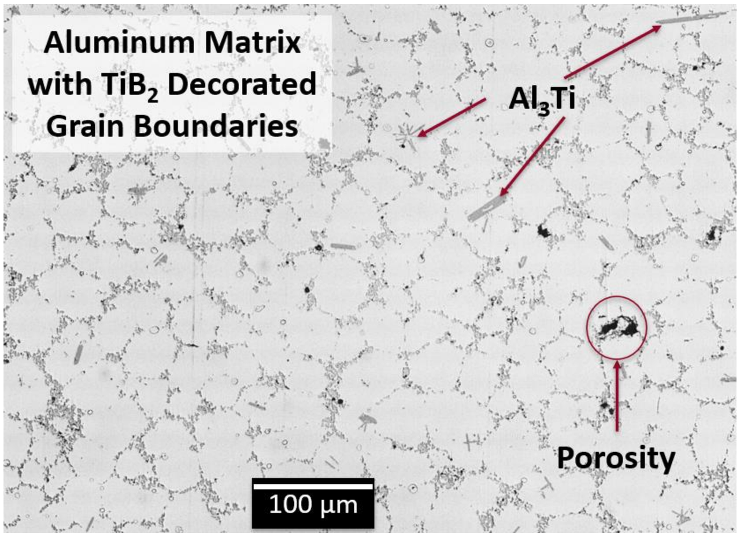

**Figure 2.** Optical metallography of the as-cast aluminum alloy.

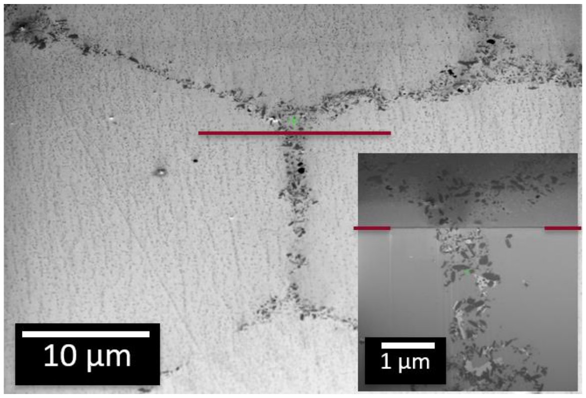

**Figure 3.** SEM SE2 image of the as-cast structure; a red line indicates the FIB cross-section target approaching a triple point; at inset a 2kV Inlens SE image of the FIB cross-section showing precipitates on two surfaces 90° apart

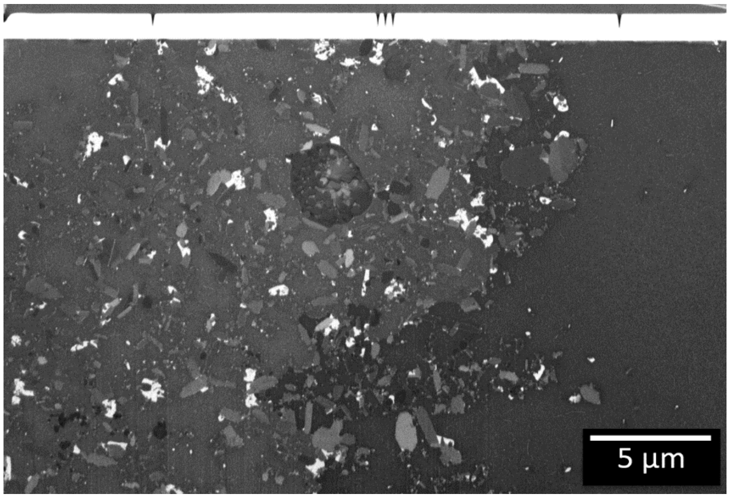

**Figure 4.** A slice from an Atlas 3D FIB-SEM tomography of the heat treated alloy showing Inlens SE and Backscattered modalities

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