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# 15 Years of Characterizing Titanium Alloys' Microstructure by DBFIB

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Over the past 15 years, transformative advances have been realized in the field of three-dimensional microscopy for micro- and nano- meter scale microstructural morphology and phase characterization. This is in no small part due to the development and maturation of the small, dual-beam focused ion beam (DBFIB). OSU accepted delivery of FEI company's first commercially available small DB-FIB platform in 1999 and immediately began TEM sample preparation by ex-situ lift-out. Shortly after delivery of the instrument, the technique of serial sectioning characterization was attempted and some of the initial serial sectioning data sets were collected during 2000 and 2001 by researchers at The Ohio State University (OSU) and Wright-Patterson Air Force Base with FEI company as shown in Figure 1[1].

Initial attempts at data collection were limited to only SEM imaging, using an everhart-thornly detector, to produce secondary electron (SE) images of the freshly milled surface. After acquiring serial stacks of SE images, tedious and time consuming post-processing was necessary in order to three dimensionally reconstruct a physical representation of desired features from the resultant 2D images. Quite often, inadequate contrast was present in the SE images and made automatic feature identification impossible, thus leaving room for significant variation in subjective thresholding[2]. In an attempt to increase signal to noise for desired microstructural features, OSU installed a custom back-scattered detector to collect SEM images based on compositional variation as. This provided for significant increase in signal to noise, but had only begun to approach the multi-faceted tool that is available today with multi signal analysis to assist researchers' in fully characterizing phase in 3D. Today, one of the more powerful technique afforded researchers' by commercially available DBFIBs is the ability to section serially through small volumes of materials while collecting various signals from freshly milled surface.

Concurrent to the DBFIBs technological advancements, OSU developed and matured post-processing and analytical software to handle the large, robust data sets that are generated when using a DBFIB. The development and application of this software is critical to enable users to process data sets in realistic time frames as well as for producing quantitative 3D data sets of desired microstructural features[3].

Titanium alloys have a wide range of applications in industry, and thermo-mechanical processing of these alloys produces complex microstructures containing inter-connected features spanning a wide range of length scales(nm-mm). Due to the complex nature of titanium microstructure, two-dimensional images provide very limited information regarding the three-dimensional nature of the microstructure. The range of length scales of microstructural features found in α/β and β Ti alloys is ideal for characterization with the DBFIB and further development and expansion of these types of techniques is important, as many details of the microstructures are either misinterpreted or not observed when only considering classical two-dimensional projections

Thus, this talk will discuss briefly, the previous research and development attempted at OSU to help reach today's current level of 3D characterization for titanium alloys, but more specifically, show how improvement in DBFIB technology, as well as improvement in software to handle the data, has allowed for collection of rich three-dimensional data sets consisting of multiple electron images, electron backscattered diffraction patterns (EBSD), and energy dispersive x-ray spectroscopy (EDS). Indeed, with this number of characterization signals, it is possible to determine phase, microstructural morphology, interconnectivities and a host of other metrics that are not otherwise measureable from 2D images. Additionally, 2D stereographic calculations will be compared against three-dimensional quantification for comparison and refinement of stereological factors. It will be shown that software development has provided the ability to iterate thorough data sets multiple times, within a reasonable time frame, allowing for significant improvements in final, overall data quality for both old and new data sets collected with various level of DBFIB technology. For the most recent data collection, with the addition of multiple signals, more robust and quantitative analysis will be presented as shown in Figure 2.

References:
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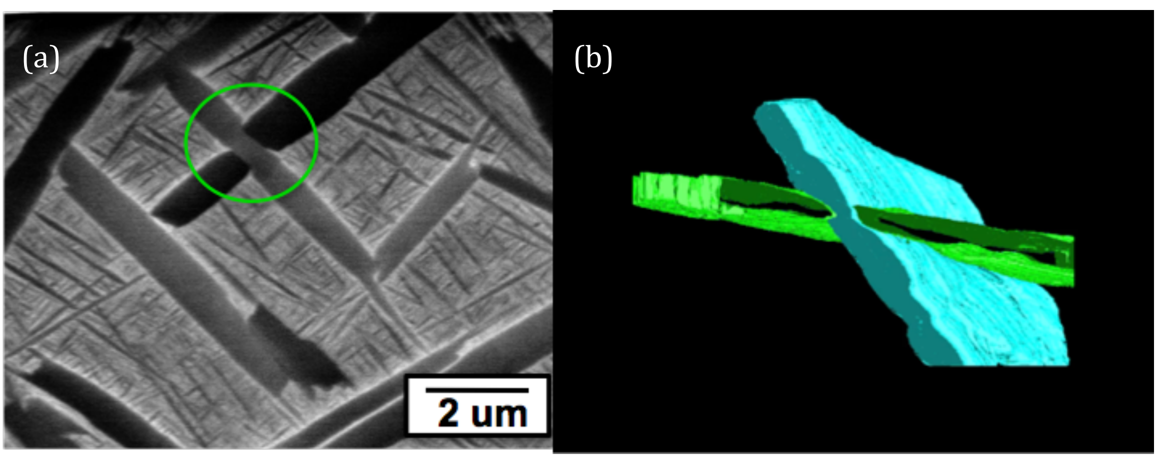


Figure 1: (a) backscattered electron image of alpha lath intersection, (b) hand segmented 3D reconstruction from green circle region of (a) circa 2002.

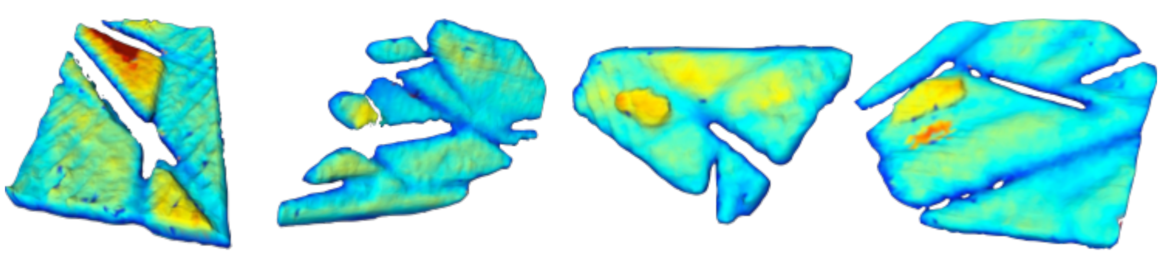

Figure 2: 3D representations of 4 alpha laths found in same volume as Figure 1(a), reconstructions were isolated with objective automation and reconstructed with custom software. Color represents variation in minimum lath thickness. circa 2012.

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