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Developments in Large Volume 3D Analysis via P-FIB: EBSD & EDS

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The integration of electron backscattered diffraction (EBSD) and energy dispersive spectroscopy (EDS) on scanning electron microscopes (SEM) is an increasingly common method of characterising materials. EDS offers chemical quantification and spatial distribution of the elements whilst EBSD enables microstructural characterisation. The integration of these two techniques with simultaneous acquisition, as in the AZtec platform, enables full material characterisation and data correlation within a single user interface.

With the integration of these analytics onto the focused ion beam (FIB) SEM, there is the potential to conduct this analysis in 3D [1]. When transitioning from 2D analysis to 3D, it is important to retain the benefits delivered through the recent developments in 2D analysis. It is also advantageous to the operator if the method of acquisition in 3D is comparable to that of 2D. The integration of FIB-SEM's with the AZtec system has the advantage that the set up for 3D is almost exactly the same as for 2D and as such all of the innovative functions are retained.

The AZtec 3D option will work in combination with FEI's Auto Slice and View 4 (ASNV4) software. FEI ASN4 manages the milling and imaging of the specimen before handing over control to the Oxford Instruments AZtec, for the collection of EDS, EBSD or simultaneous data. By integrating both EBSD and EDS together it is possible to fully interrogate a sample, despite this being a destructive technique. This method has the additional advantage of not been ion source specific, as such is compatible with both FEI Ga^+ ion and Xe^+ Plasma FIB-SEM's (PFIB-SEM). PFIB-SEM has the benefit over conventional FIB-SEM of achieving significantly high milling currents enabling greater volumes to be processed [2].

Use of a FEI Helios PFIB-SEM with AZtec 3D has enabled the collection of large volume datasets. For example Figure 1 shows EBSD data from a volume of 300 x 300 x 200 µm (300 x 300 µm is the size of the cross section and 200 µm the depth of the block). The slices in this set are 500 nm thick and the set consisted of ~400 slices. The milling current was 180 nA and milling took ~2 minutes. EBSD maps were acquired at 20 kV, with a step size of 750 nm. In addition by lift out and mounting of a sample onto an TEM Lift-Out grid, simultaneous EDS and EBSD data can be collected without the potential shadowing from the FIB trench [3,4]. For example a 7xxx series aluminium alloy of size 250 x 250 x 70 µm was serially sectioned by this method. The sample was milled at 180 nA with 500 nm slice thickness. EBSD data acquisition was at 20 kV with 500 nm step size and ~7 minutes per slice (Figure 2a). EDS data was acquired simultaneously (Figure 2b), the detector position and acquisition settings were matched to the requirements for EBSD.

The combination of high speed acquisition and analysis in AZtec 3D with the large usable currents in the FEI Helios PFIB has enabled fast and precise EBSD and EDS data collection on previously unattainable scales. Data volumes of several 100's of microns, combined with full set of analytical data are possible whilst retaining all of the detail and ease of setup expected from conventional 2D analysis.

This powerful new solution will be discussed, with case studies from a range of materials demonstrating the expanding versatility of this approach.

References

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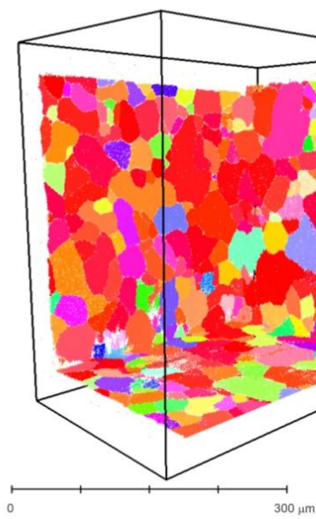

Figure 1. Large volume 3D EBSD reconstruction from a Zircaloy-4 fuel cladding, obtained via AZtec 3D in conjunction with FEI's ASNV4.

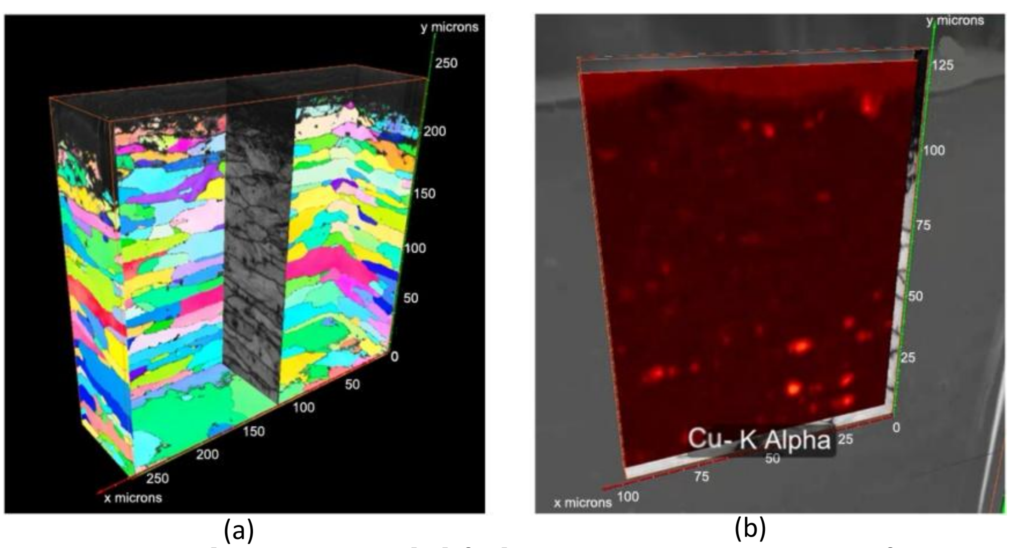

Figure 2. Large 7xxx series aluminium sample lifted out in PFIB onto an TEM Lift-Out Grid allowing for 3D EBSD (a) and EDS (b) collection without signal shadowing associated with a FIB trench.

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