

## Advancing Materials Characterization in the FIB-SEM with Transmission Kikuchi Diffraction

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SEM-based Transmission Kikuchi Diffraction (SEM-TKD) [1,2] is a technique variation of EBSD, applied here using an unmodified Oxford Instruments AZtec EBSD system. The advantage of SEM-TKD is the collection of point and mapping data, from particles and electron transparent samples, at spatial resolutions which exceed those of traditional EBSD. This improvement is primarily a consequence of examining thin, electron transparent TEM samples in transmission, which reduces the effective diffraction and escape volume, as well as the use of zero to low sample tilts, reducing anisotropic beam spreading effects. Like EBSD, TKD may be used in conjunction with EDS for single-point, chemistry and crystallography-based phase identification. This is readily applicable to freestanding particles since the high spatial resolution is combined with a large solid angle of diffraction pattern capture, enabling automated indexing for any crystal orientation, for particle sizes down to the deep submicron scale. The technique is also used to produce 2D datasets from flat, thin sample areas, for microstructural analysis including crystallographic orientation, grain size, phase distribution, and grain boundary character and distribution

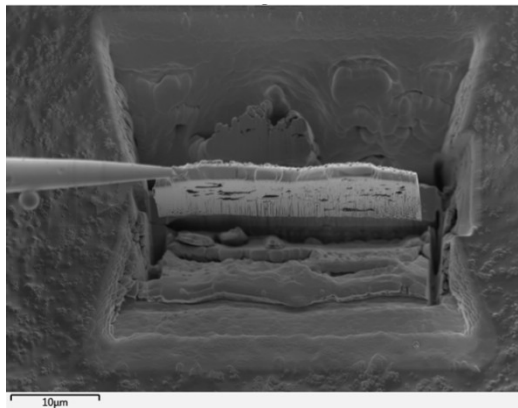
The application of TKD in FIB-SEMs delivers additional benefits. FIB-SEMs can be equipped with the same range of analytical instrumentation as standard SEMs, but add the ability to mill, deposit, and manipulate the sample, which offers an additional dimension for characterization. For example, imaging and EDS/EBSD map data from an 'as-provided' sample can serve as location references for specifically targeted cross-sectional sample extraction for TEM and TKD. Also, the use of advanced, high stability nanomanipulation devices brings the ability to prepare and characterize newly extracted samples *in situ* ('on-tip') during the same FIB-SEM session, eliminating the requirement in some cases to mount and remove the sample before characterizing separately. This can include characterisation by EDS, EBSD, and when the sample is appropriately thinned, TKD. Unlike some alternative preparation techniques, 'lift out' sections can be made as foils with relatively large areas of useful thickness, from SEM imaging/EDS/EBSD-targeted locations on a variety of material types.

The use of *in situ* TKD on FIB-SEMs has diverse potential applications. For example, in gunshot residue (GSR) analysis, thin slices through selected particles may be prepared and EDS & TKD used for combined chemical and crystallographic phase identification (Fig. 1a - e) in determining the type and origin of the ammunition used, in a single session.

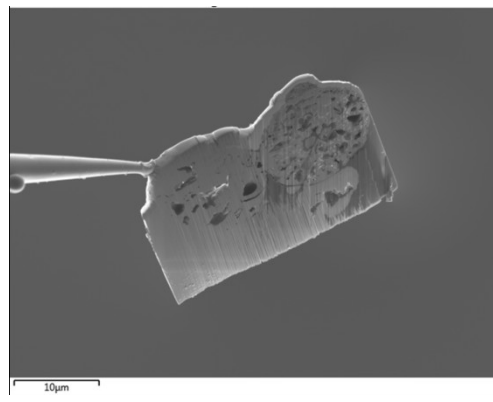
References:

[1] R.R. Keller, R.H. Geiss, Transmission EBSD from 10nm domains in a scanning electron microscope, *Journal of Microscopy* 245 (2012) 245-251

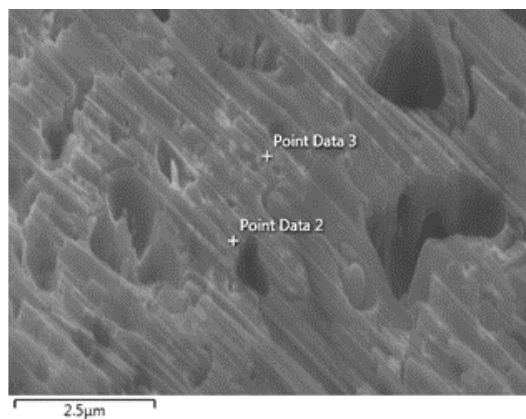
[2] Trimby, P.W., Orientation mapping of nanostructured materials using transmission Kikuchi diffraction in the scanning electron microscope, *Ultramicroscopy* 120 (2012) 16-24



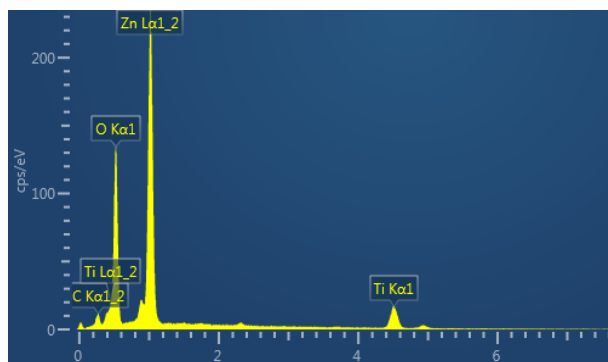
**Fig 1a** FIB-SEM preparation and lift out of a lamella from a selected GSR particle



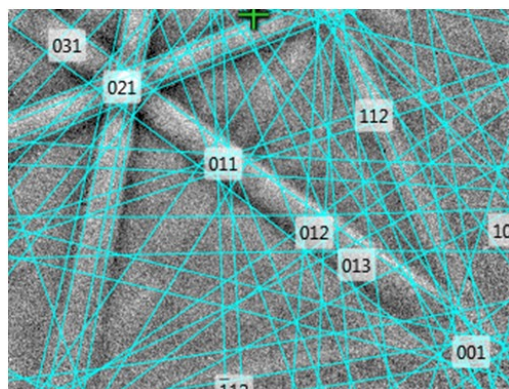
**Fig 1b** Manipulating the lamella for final preparation and analysis by TKD and EDS with an OmniProbe 400 nanomanipulator



**Fig 1c** Upper surface image used as reference for EDS & TKD point analysis



**Fig 1d** EDS spectrum from point 2



**Fig 1e** TKD pattern from point 2, solved as  $(\text{Ti}_{0.92} \text{Zn}_{0.08})(\text{Zn}_{1.92} \text{Ti}_{0.08}) \text{O}_4$  phase, residue consistent with “sintox” gunpowder primer