TRANSMISSION ELECTRON BACKSCATTER DIFFRACTION IN THE SEM: SPECIMEN THICKNESS EFFECTS

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Introduction

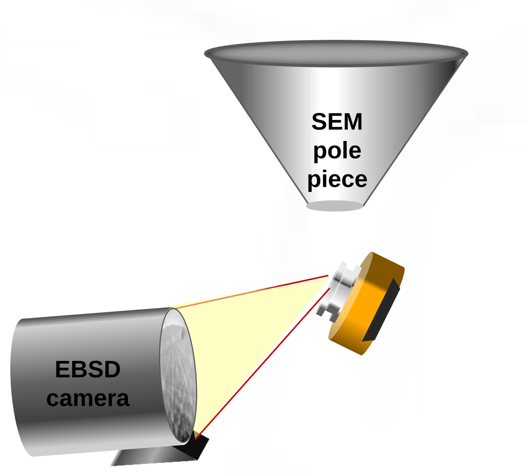
Transmission electron backscatter diffraction (t-EBSD) is a new SEM-based electron diffraction technique that provides a significant improvement in spatial resolution over conventional EBSD for crystallographic analysis of materials[1]. The electron-specimen interaction volume associated with t-EBSD is significantly smaller than that of conventional EBSD because the signal is formed by collecting Kikuchi-scattered transmitted electrons in the SEM rather than backscattered electrons. The smaller volume results from the experimental conditions needed to detect transmitted electrons:

- Specimen tilt is ~ 10° to 20°, drastically reducing the projected incident beam diameter in the specimen surface direction lying normal to the tilt axis;
- Electron-transparent specimens are analyzed, which reduces the maximum total scattering length before electrons are detected.

We describe in this poster the potential of t-EBSD for characterization of films relevant to the nanoelectronics industry. Included are examples of ultrathin films (< 10 nm thick) as well as thick foils (> 1 µm thick). The phenomenon is discussed in terms of electron scattering through materials of varying specimen mass-thickness, ρ * t, where ρ is density and t is thickness.

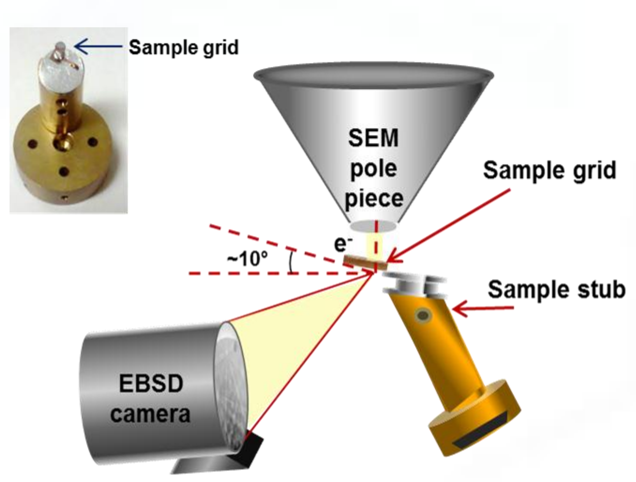
Experimental

Conventional EBSD geometry

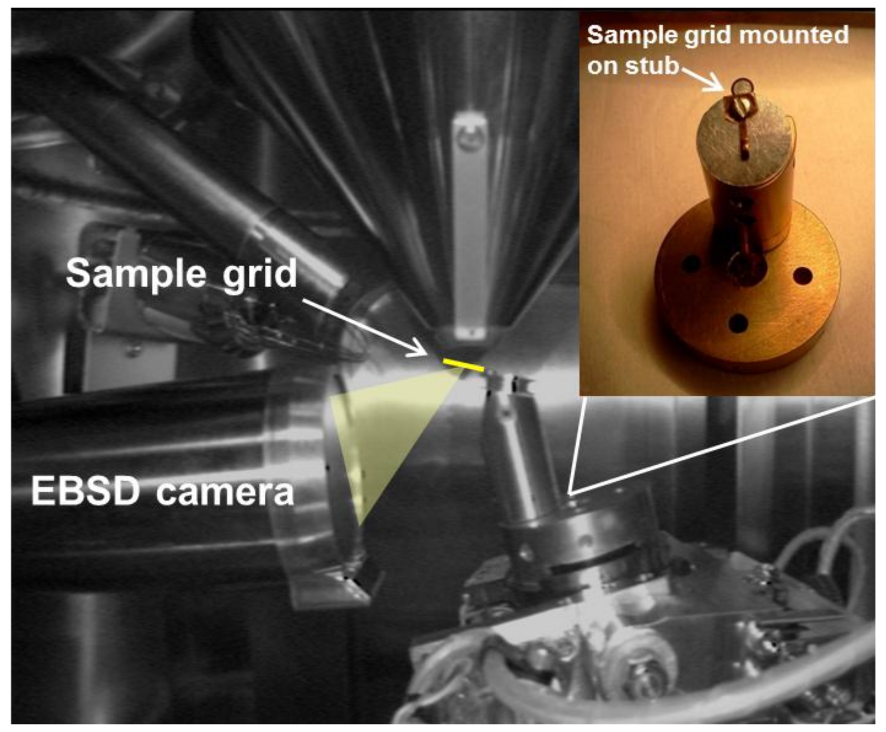


Conventional EBSD specimen-detector configuration, showing a sample tilted to 70° from horizontal.

Transmission EBSD geometry



t-EBSD specimen-detector configuration. A simple custom specimen holder is used to hold a sample at 10° to 20° from horizontal.



Infrared camera image of mounted sample and EBSD detector within SEM specimen chamber.

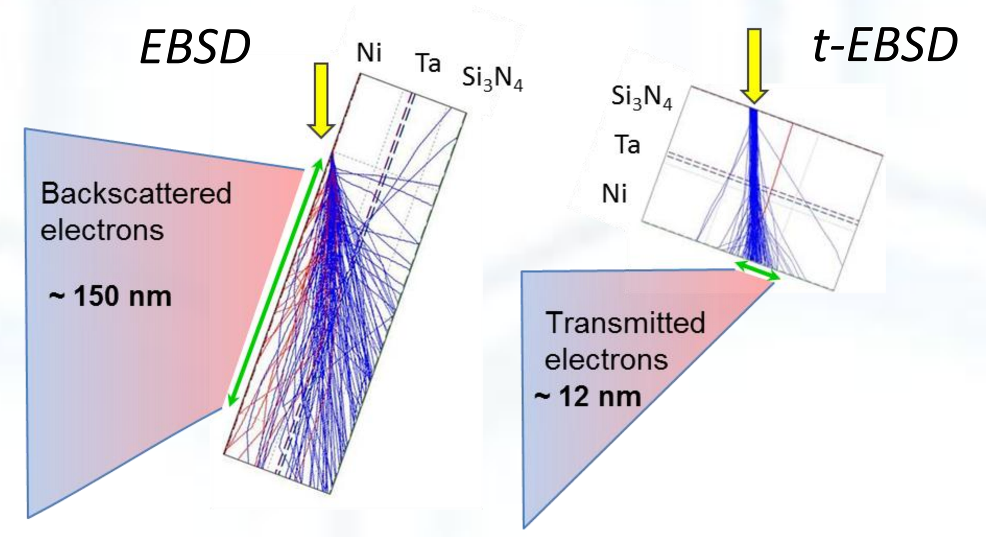
t-EBSD can be performed on any sample that is acceptable for conventional TEM:

- thinned specimens,
- free-standing films,
- sectioned film stacks,
- nanoparticles, or
- other material placed onto TEM windows or grids.

Samples are mounted on SEM stubs, and tilted between 10° and 20° from horizontal. Accelerating voltages are typically between 20 kV and 30 kV, and probe currents are typically in the range 300 pA to 1.2 nA. Commercial EBSD hardware and software are used to acquire and analyze data.

Electron Scattering: modeling & experiments

Understanding electron scattering during t-EBSD tells us about operating conditions as well as its applicability to specimens of different thickness. Monte Carlo (MC) simulations provide insight into the phenomenon by modeling interaction volumes and electron energy distributions within the specimen. The following MC results assume elastic single scattering via a screened Rutherford cross-section, coupled with the Bethe-Joy-Luo[2] continuous energy loss approximation.



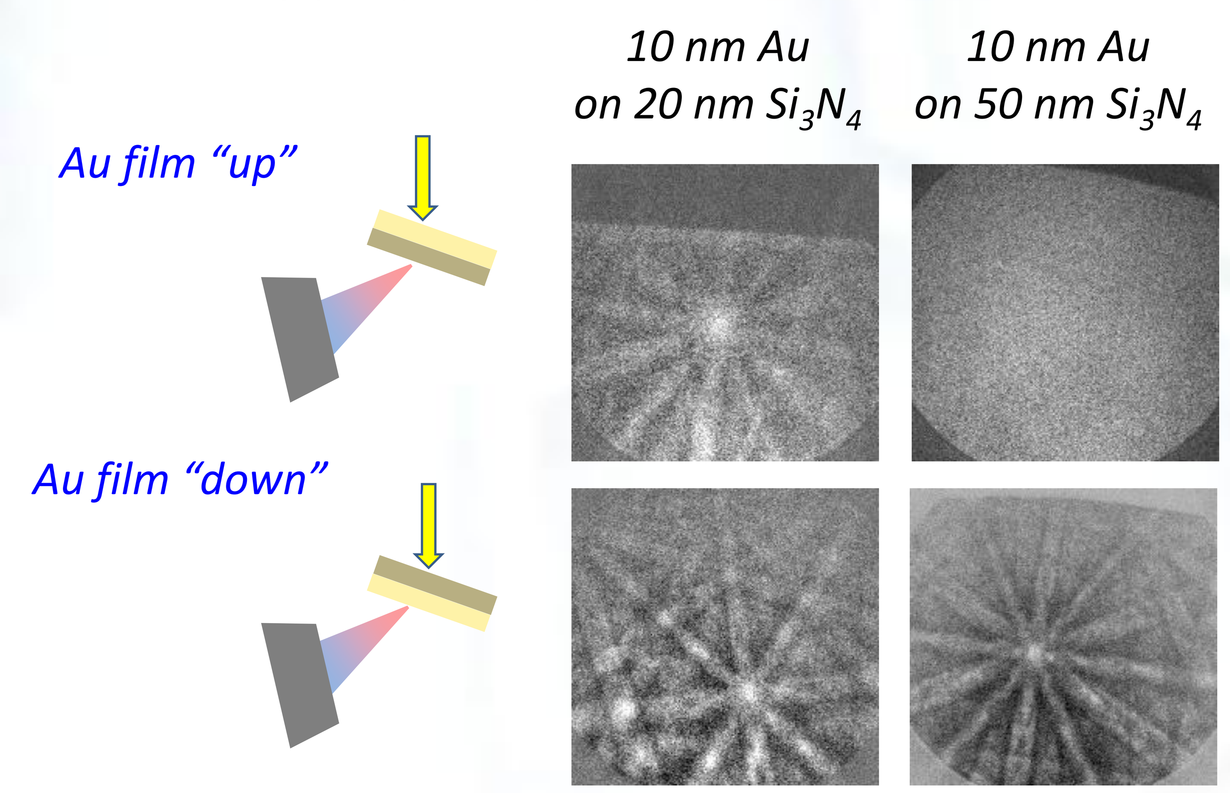
Interaction Volumes: conventional EBSD vs t-EBSD:
Electrons with energy 28 keV easily traverse a specimen stack consisting of 40 nm Ni /2.5 nm Ta/40 nm Si_3N_4, in the t-EBSD case. In fact, the ease with which electrons penetrate leads to the large interaction volume for the highly-tilted, conventional EBSD case from the same specimen: ***the lateral spatial resolution in this example improves by over one order of magnitude!***

Lateral resolution was estimated by observing trajectories of electrons that have retained 90+ % of their incident energies (low-loss electrons), consistent with energy filtering experiments that suggest such electrons contribute most significantly to EBSD pattern contrast.[3]

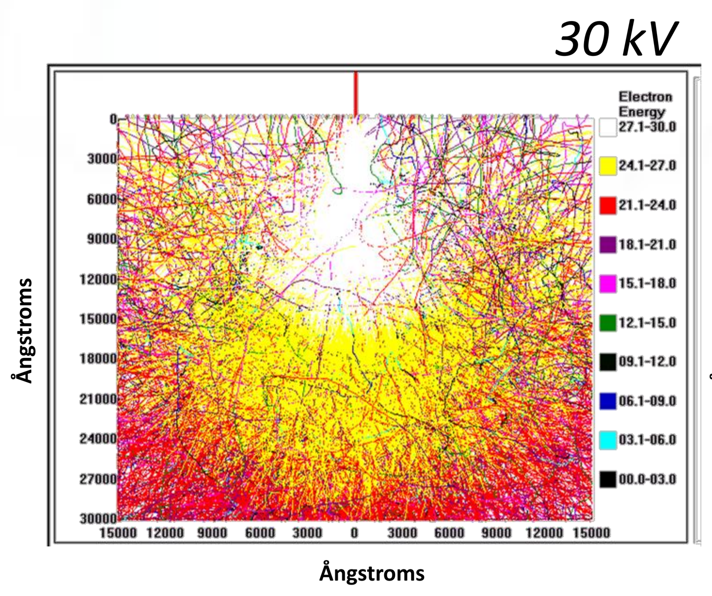
Kikuchi scattering during t-EBSD:
Diffraction pattern contrast arises from Kikuchi scattering, so it is paramount to understand how such scattering can occur during t-EBSD. Potential Kikuchi sources are distributed throughout the specimen. However, to form a pattern, we must detect electrons that maintain their post-Kikuchi coherence. The experiment to the right attempts to identify the primary location of Kikuchi sources.

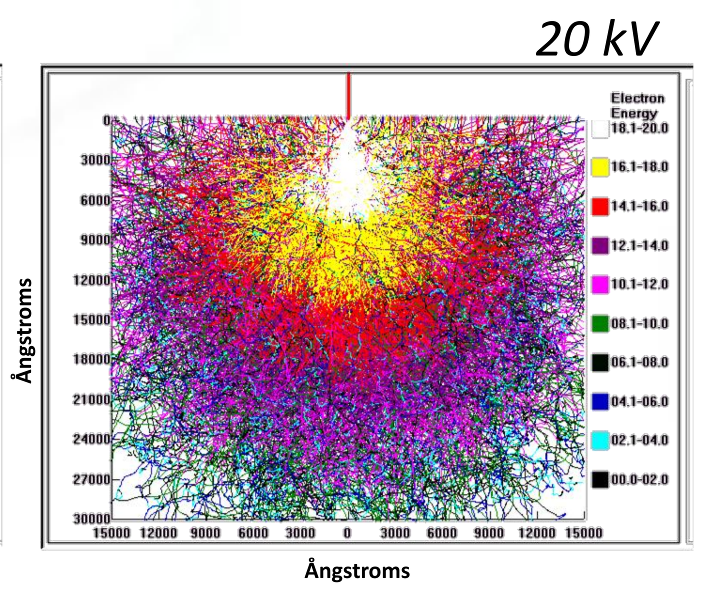
The result suggests that it is more difficult for electrons that have diffracted near the top, entrance surface to maintain coherence for a significant distance in the material. The most important Kikuchi scattering likely occurs near the exit surface.

Transmission EBSD has the potential to become an effective method of characterization for bilayer samples or other samples where the material of interest is buried in a stack of amorphous material, or with adhesion or barrier layers.



Electron scattering in 3 µm Al:





Electron energy distributions in a thick foil:
The ability of electrons with SEM energies (≤ 30 keV) to sufficiently penetrate a material is critical for generating near-exit-surface Kikuchi scattering, which is responsible for t-EBSD. We showed the case for 40 nm Ni/2.5 nm Ta/40 nm Si_3N_4. But is t-EBSD applicable to thicker structures?

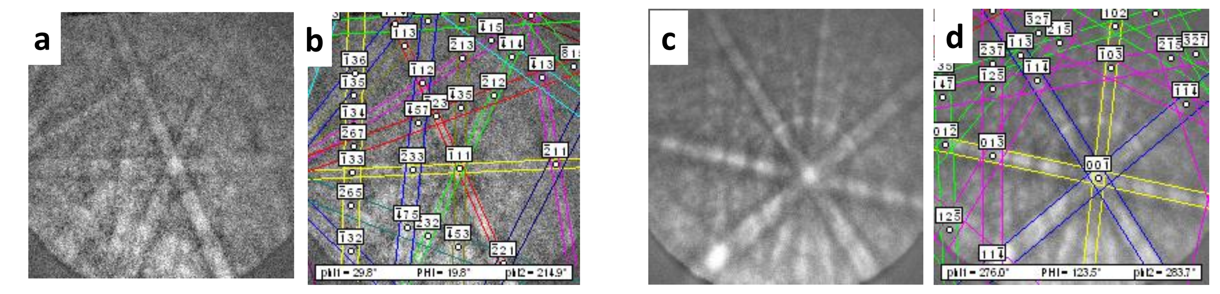
The figures show MC results from the thickest film measured to date – a 3 µm Al foil. At 30 kV (far left), there are many low-loss electrons (in red), some exiting the specimen. There are significantly fewer at 20 kV (near left) resulting in few, if any, electrons exiting the specimen. Experimental results on such a foil are shown below.

Incident beam energy can be tuned for thicker samples to provide enough high energy electrons for pattern formation. MC simulations are valuable for determining if a sample with given composition and thickness will be able to produce Kikuchi patterns.

Ultrathin ALD-deposited HfO_2 and thick foil samples

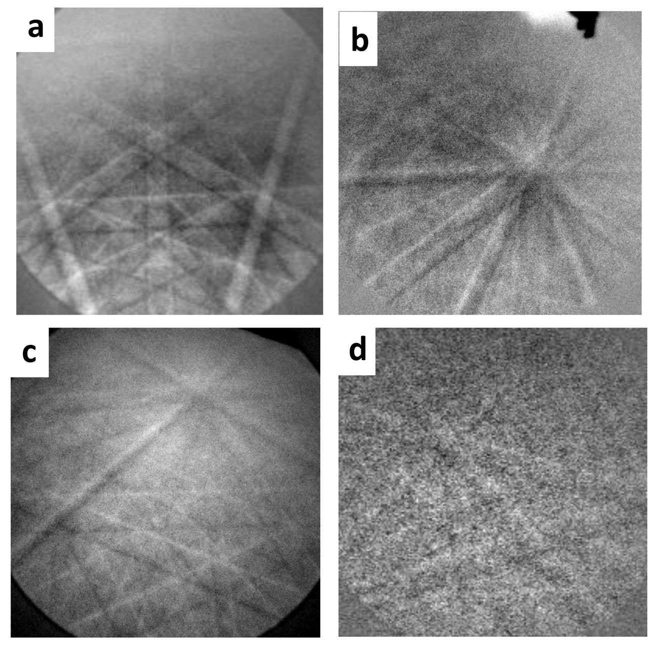
Ultrathin hafnium oxide is used as a high-k dielectric layer in nanoscale electronics. The orientation and crystal phase of the material affect its electronic properties, but measurement of these material characteristics is extremely difficult with established electron microscopy methods due to the extreme thinness.

Transmission EBSD provides the capability to measure composition, phase, and orientation of films as thin as 5 nm to 10 nm (and perhaps thinner), as shown below. Measurement of Kikuchi patterns at this size scale in the electron microscope is unprecedented.

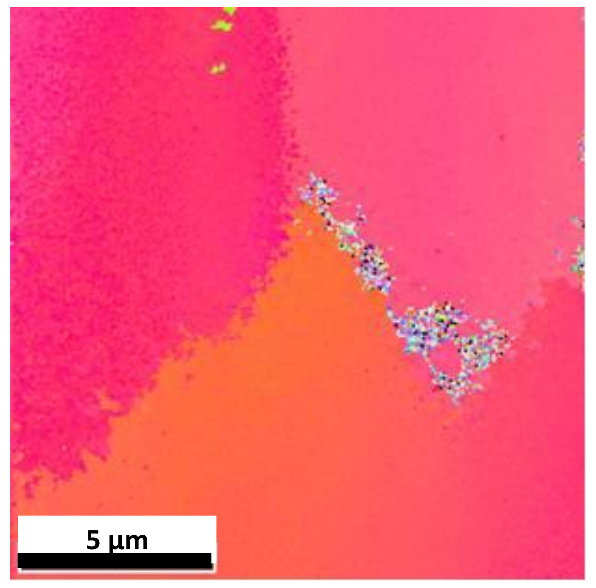


Kikuchi patterns from (a,b) 10 nm HfO_2; and (c,d) 5 nm HfO_2. Samples were deposited by atomic layer deposition (ALD) and annealed at 500 °C for 3 min to induce crystallinity. All patterns were indexed to the ***monoclinic*** *phase of HfO_2*

While t-EBSD is well suited to nanoparticles and ultrathin films, it is also suitable for very thick samples. Orientation maps have been collected from foils up to 3 µm thick, making this technique a solid choice for a wide variety of materials requiring crystallographic characterization.



T-EBSD from thick foils. (a) 800 nm, (b) 1.5 µm, (c) 2 µm, and (d) 3 µm

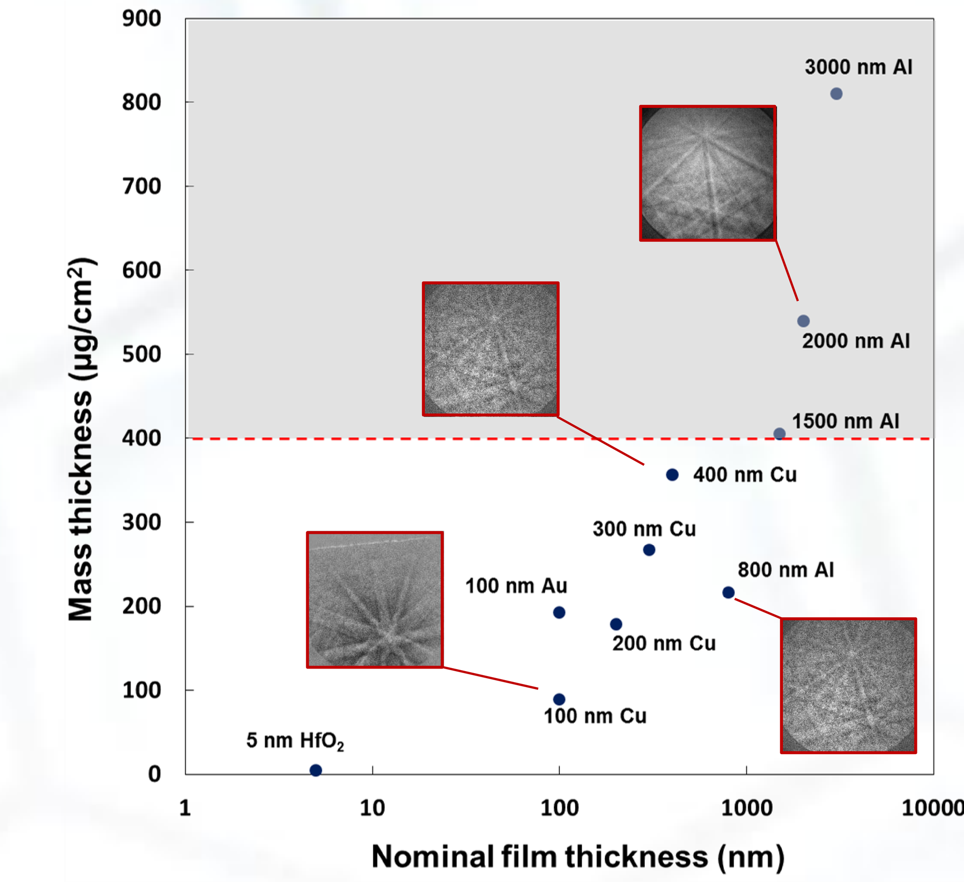


Orientation map from 2 µm Al foil. ND + TD direction shown to highlight grain structure.

Mass-thickness effects

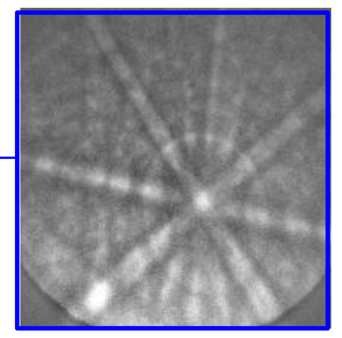
t-EBSD can be applied to a wide variety of crystalline films and particles. An important factor determining suitability for a particular sample is the *mass-thickness* (*i.e.*, density*thickness). The figure below shows mass-thicknesses of samples that gave clear, indexable diffraction patterns, plotted against the nominal film thickness on a log scale.

t-EBSD has so far been successfully applied to samples with mass-thickness spanning over two orders of magnitude (< 5 µg/cm² to > 800 µg/cm²). The shaded region indicates samples where diffraction patterns could be indexed on portions of the film, but not the entire film, either indicating the approach of an upper limit to mass-thickness or being due to microstructural effects such as multiple grains in the thickness direction.

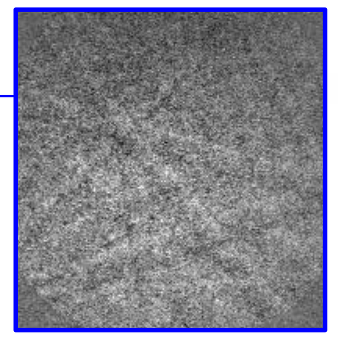


Conclusions

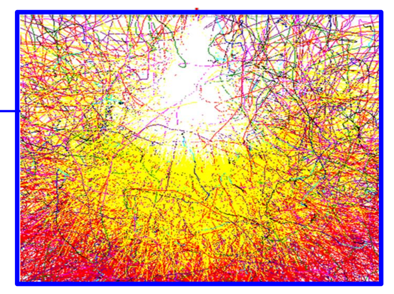
- *Transmission EBSD is a breakthrough technology in materials characterization, enabling measurements of crystallographic properties from films of thickness 5 nm, with sub-10 nm lateral spatial resolution.*



- *The t-EBSD signal arises from the bottom few nanometers of crystalline films, and this signal can maintain coherence through a few tens of nm of amorphous material.*
- *Crystallographic data can be obtained from very thick films, exceeding 3 µm.*



- *Mass-thickness is a key factor in determining whether t-EBSD may be effective on a particular sample. Generally, any sample suitable for TEM studies will be suitable for t-EBSD.*
- *Monte Carlo simulations can be used as a guide to determine whether a sample will have enough high energy electrons to produce a Kikuchi pattern.*



- *The ability to perform t-EBSD in an SEM with commercial EBSD hardware/software means the technology infrastructure is already widely in place.*

Acknowledgements/References

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