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New Measurements on the Minimum and Maximum Sample Sizes in t-EBSD

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The technique of acquiring transmission electron diffraction patterns in the scanning electron microscope, SEM, using components of commercially available electron backscattered diffraction equipment, EBSD, normally used in a reflection geometry, was first introduced in 2010 [1]. Since then it has been pursued by a number of laboratories. In particular, during the past year a number of publications have appeared demonstrating various applications of the technique, [2- 4]. The technique, which we call transmission electron backscattered diffraction, t-EBSD, provides a completely new capability for obtaining diffraction patterns from thin, electron transparent crystalline samples in the SEM. In the publications referenced above, transmission Kikuchi patterns have been presented from a variety of TEM like samples including nanoparticles, nanowires and thin films and foils. In all the results, the spatial resolution obtained has been on the order of 5 nm or less, far smaller than has been shown from reflection EBSD. To realize t-EBSD using standard EBSD hardware, the only requirement is that an electron transparent sample be positioned in such a way as to allow the transmitted diffraction pattern to fall on the phosphor of the EBSD camera. If the sample normal is tilted 20 degrees relative to the incident beam, the standard indexing software can be employed after considering that this sample normal is 90 degrees relative to the sample normal in standard reflection geometry.

A number of questions are often asked relating to various applications of the technique. (1) What is the smallest particle from which an indexed pattern has been obtained? (2) What is the thinnest film from which an indexed pattern has been obtained? (3) What is the thickest film, or foil, from which an indexed pattern has been obtained? Here we present results from data obtained in our laboratory as preliminary answers to these questions. All patterns are raw data.

(1) We obtained and indexed patterns from Ag and Au nanoparticles with diameters on the order of 5 nm. An example from a 5 nm Ag particle is shown in Figure 1.
(2) For very thin films, we obtained patterns from 5 nm thick films of HfO_2 deposited on C-substrate TEM grids, annealed for 3 min @ 500 deg C in 50% O_2, as shown Figure 2.
(3) With t-EBSD we have obtained patterns from a variety of metallic thin films and foils. A pattern obtained from the thickest foil thus far, a 2 μm thick Al foil, is shown in Figure 3.

Not surprising, there is a correlation between the maximum thickness and the mass-thickness of the material and the pattern quality. Figure 4, which is a plot of thickness versus mass-thickness from different materials, annotated with thumbnails of t-EBSD patterns, shows our results. Pattern quality is observed to be lower for very thin and very thick films, as might be expected.

References:

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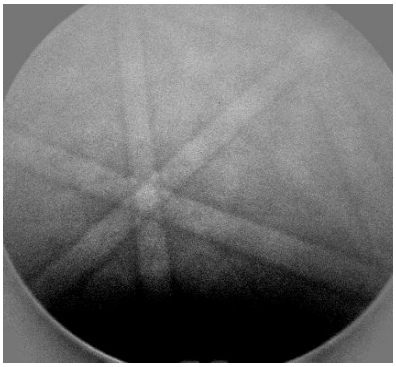

Figure 1. t-EBSD pattern from an approximately 5 nm diameter particle of Ag. 20 kV.

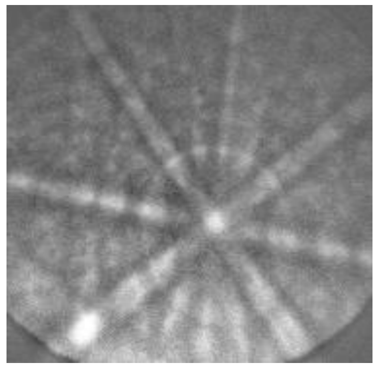

Figure 2. t-EBSD pattern from 5 nm thick annealed film of HfO_2 on C. 20 kV.

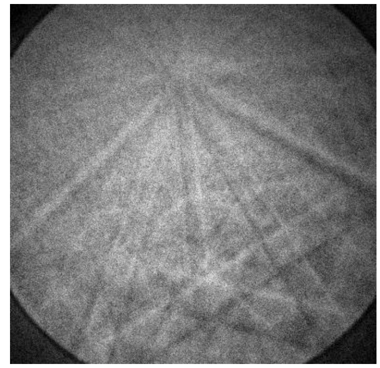

Figure 3. t-EBSD pattern from 2 µm thick Al foil. 30 kV.

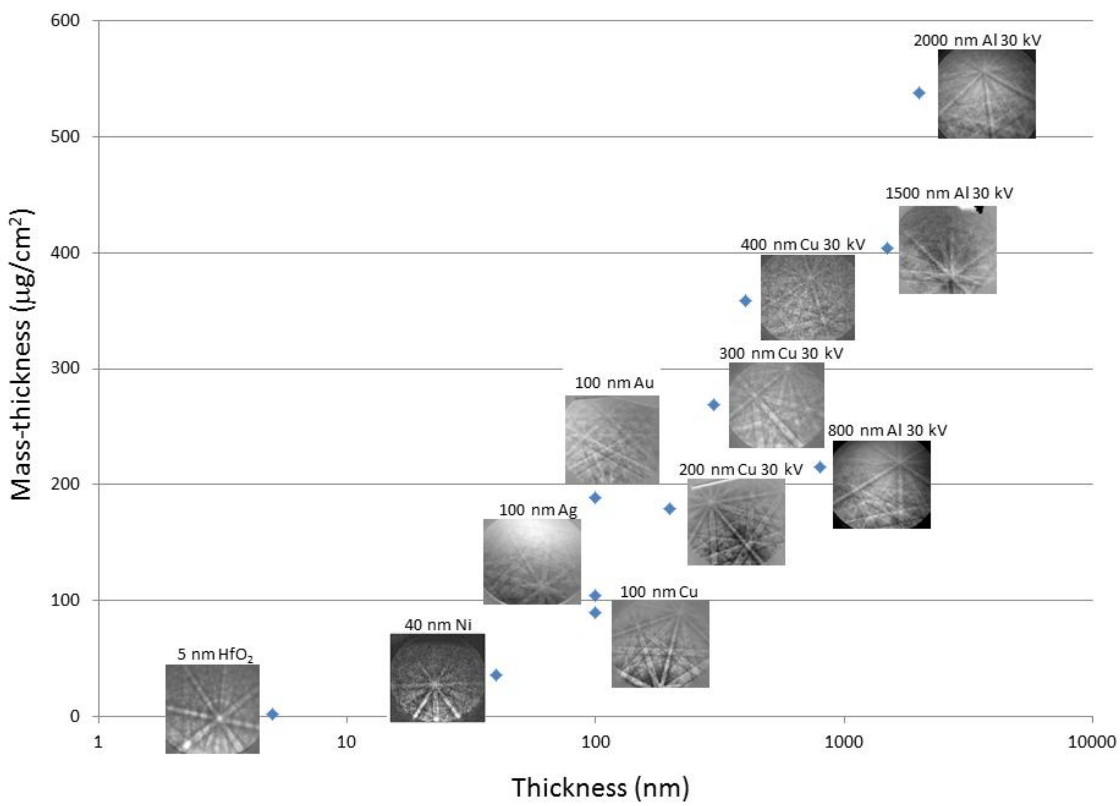

Figure 4. Sample thickness v. mass-thickness, annotated with thumbnails. 20 kV, unless otherwise noted.

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