Beam Broadening in Transmission EBSD

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Introduction

Transmission electron backscatter diffraction (t-EBSD) [1] also known as transmission electron forward scatter diffraction (t-EFSD) [2] or transmission Kikuchi diffraction in the SEM (TKD-SEM) [3], can provide significant improvements in spatial resolution in many cases over conventional EBSD performed in reflection. Over the last few years, truly remarkable results have been demonstrated, including phase identification of nanoparticles as small as 5 nm [2], effective orientation mapping resolutions of 2 nm to 3 nm for thin metal films [3], and high-contrast patterns from films as thin as 5 nm in plan-view [4]. Perhaps the most exciting thing about this technique, however, is that it requires no special equipment beyond the conventional EBSD setup. The sample simply requires mounting such that the beam can pass through and the diffraction pattern can be collected by the camera, as shown in Figure 1 [4]. Some users mount the sample horizontally, and some tilt it at a shallow angle from horizontal, mainly to prevent shadowing effects on the camera.

The increase in spatial resolution achieved with this technique has important implications outside the area of nanomaterials and even can provide more information about the microstructure of traditionally prepared thin specimens or geological samples. In fact, most samples that are well suited for transmission electron microscopy (TEM) generally provide good results for transmission EBSD, including nanoparticles deposited from solution, electrolytically thinned bulk materials, and focused ion beam (FIB)-prepared lamellae.

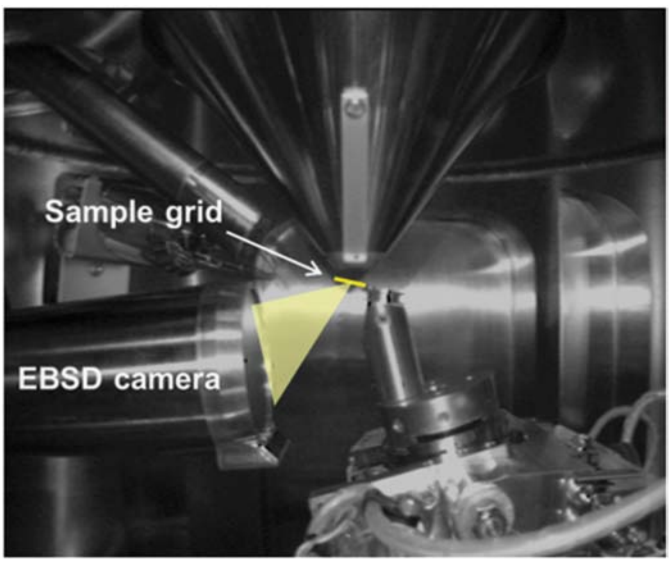

Figure 1: Experimental setup showing sample position for transmission EBSD.

Materials and Methods

One of the primary factors to consider while discussing spatial resolution in the transmission configuration is where the detectable diffraction occurs within the specimen thickness; this affects the achievable lateral resolution and determines which regions of the specimen give rise to diffraction patterns. We have demonstrated through use of bilayer samples that the detectable diffraction occurs within the bottom few nanometers of the crystalline region of a specimen [4], that is, the surface that is closest to the EBSD camera. Figure 2 (reproduced in part from [4]), shows a crystalline Au film deposited on two different thicknesses of amorphous Si_3N_4 (a- Si_3N_4). When the sample is mounted such that the beam passes through the Au first, then exits the a-Si_3N_4, patterns can be seen for the 20 nm a- Si_3N_4 layer, but not for the 50 nm a- Si_3N_4 film. When the sample is reversed such that the beam enters the a- Si_3N_4 layer first and then exits the crystalline Au, patterns can be seen for both thicknesses of the amorphous layer. Diffraction is occurring everywhere throughout the sample, but electrons that diffract above the bottom surface generally do not maintain coherence because they have to travel through more material where they have the potential to re-diffract or incoherently scatter. This experiment therefore suggests the patterns that are detected likely come from the bottom few nanometers of the sample. Armed with this information, we can guide our sample preparation techniques such that the region of interest is face-down toward the detector. Another practical note that should also be considered is that FIB-prepared samples can have a significant amount of ion-beam-induced damage (essentially an amorphous layer) at the surface of interest, and thus a low-kV cleanup may be necessary to maintain crystallinity in that region.

Because the incident beam must traverse nearly the full thickness before undergoing detectable Kikuchi scattering, transmission EBSD is unlike conventional EBSD, where the detectable diffraction occurs near the top surface [5]. As the beam travels within the sample, electron energies and trajectories change as a result of both elastic and inelastic scattering. These effects lead to broadening of the incident beam and can be estimated to a first approximation by Monte Carlo scattering simulations [6]. Beam broadening has for some time also been a consideration in TEM, but the order-of-magnitude higher energies used in TEM imaging, and the specimen thicknesses typically used, make it less of a concern compared to STEM-in-SEM techniques.

Results

Figure 3 shows an example of the electron trajectories as they scatter throughout a copper film. The exit trajectories of the transmitted electrons are color-coded for the energy they have at each point, which leads to estimates about effective

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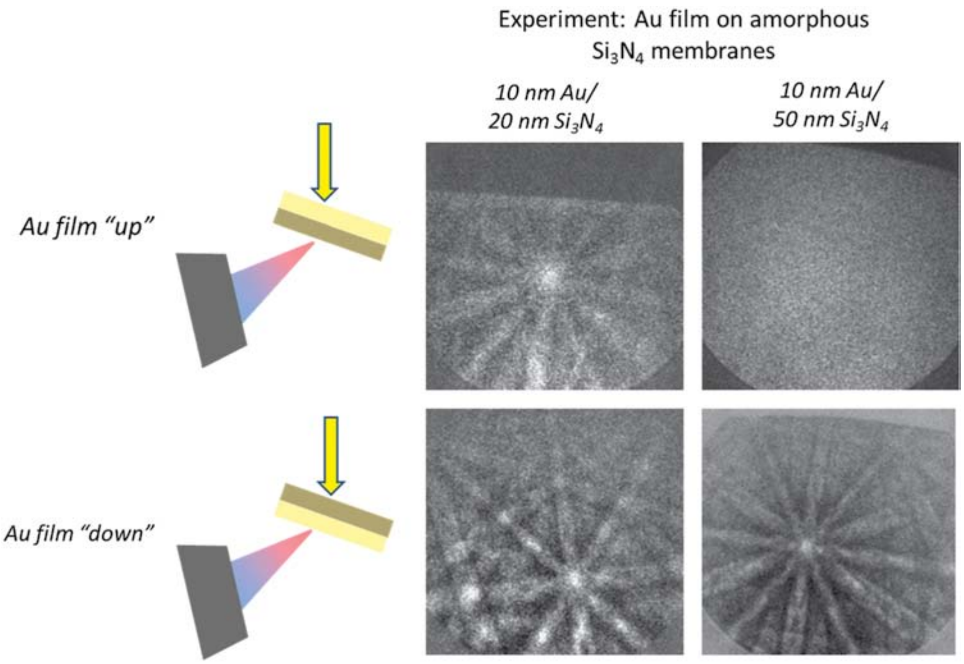

Figure 2: Bilayer crystalline Au/amorphous Si_3N_4 films demonstrate likely area of sampling in transmission EBSD, reproduced in part from [4], copyright of Wiley and Sons. The top row demonstrates the patterns obtained for 20 nm (left) and 50 nm (right) Si_3N_4 layers with the crystalline Au layer facing away from the detector. The bottom set of patterns shows Kikuchi patterns from both Si_3N_4 thicknesses when the crystalline Au layer is facing toward the detector.

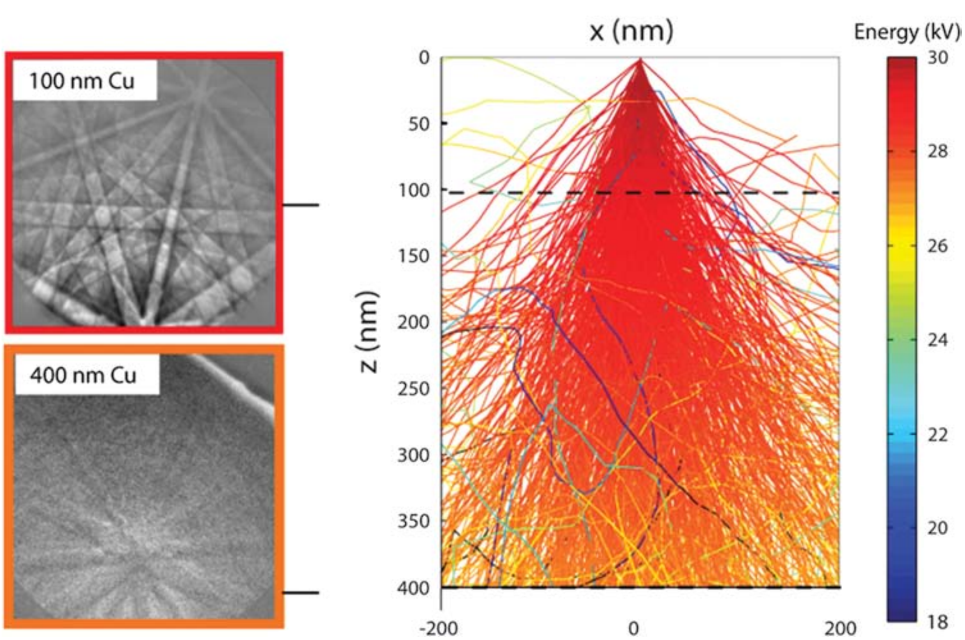

Figure 3: Monte Carlo simulations of electron trajectories through a sample of copper, where colors represent energy. Thumbnail images show patterns obtained with a 100 nm Cu film (top) and a 400 nm Cu film (below) at 30 kV.

beam size and energy at the surface where detectable patterns are generated. Qualitatively, the figure shows beam broadening has a significant impact on the experimentally demonstrated pattern quality, as seen in the difference between the patterns obtained from the 100 nm film (top left) and the 400 nm film (bottom left). Both films were prepared by thermal evaporation onto a lacy carbon grid and interrogated with a beam of 30 keV electrons.

The term "beam diameter" means different things to different microscopists because it is difficult to say with any certainty which electrons can be considered part of the beam and how much of the beam is contributing to the output. In the EBSD community, electrons retaining at least 90 % of their incident energy are thought to be the primary contributors to pattern contrast, whereas lower-energy electrons contribute to the background, which may adversely affect the pattern [7]. These "low-loss" electrons comprise only a small portion of all backscattered electrons, so the effective beam diameter is something that will need to be determined by clever experimentation. We can, however, make some estimates of how the beam changes by comparing one measure of beam size over a range of thicknesses or by visualizing the positions of electrons as they exit the film.

Several criteria have been used to describe beam diameter, including full width at half maximum value (FWHM), the Gaussian beam diameter, 10/90 or 20/80 ratios, and likely many more [8–9]. Here we will avoid choosing one specific method but rather demonstrate what happens to the beam at the surface of interest with progressively thicker films.

Figure 4 shows the positions of 10,000 incident 30 keV electrons that have reached the exit surface for three different aluminum film thicknesses: 100 nm, 750 nm, and 1500 nm. Each dot represents an electron exiting the film surface closest to the detector, and the dot color represents the percentage of the incident energy retained at a particular exit location. These are somewhat extreme cases, but the figure illustrates that for a typical TEM sample thickness of 150 nm, our transmitted beam is compact and still retains an energy of nearly 30 keV. The beam maintains a high proportion of the incident energy at 750 nm yet has spatially broadened. Finally, at 1500 nm thickness the beam has spread significantly while continuing to lose energy, especially at the points of greatest broadening. These simulations demonstrate in a visual way that the gains in spatial resolution made in transmission EBSD are very much thickness-dependent, and only by preparing the sample to appropriate thicknesses will those resolution gains be achieved.

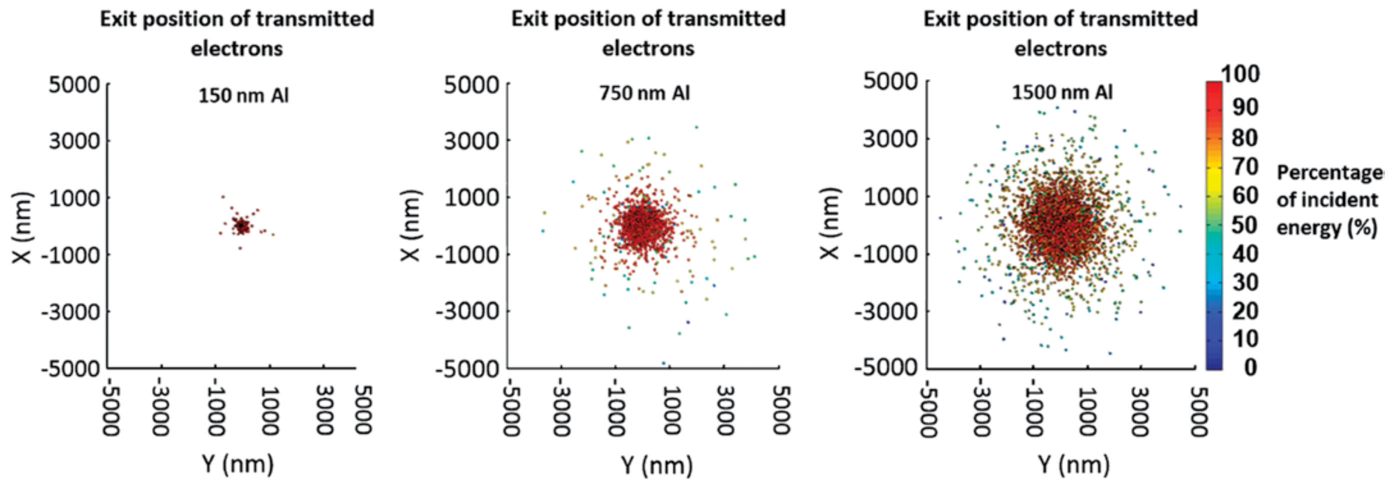

Figure 4: Electron positions for the transmission case, colored by energy retained at the exit surface for three different film thicknesses of aluminum: from left, (a) 150 nm, (b) 750 nm, and (c) 1500 nm. Each simulation contains 10,000 electrons at 30 keV incident beam energy. The directions *x* and *y* represent the planar surface of the film, the out-of-page direction toward the reader is the exit surface of the film.

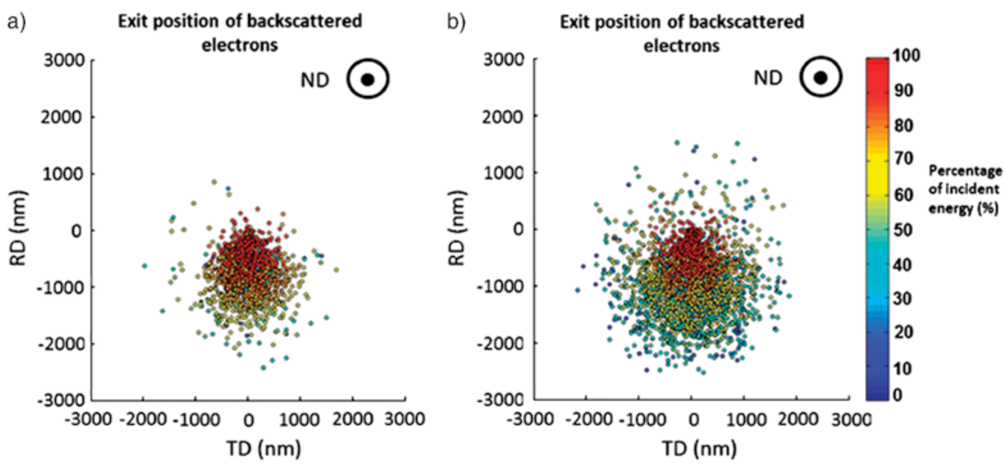

Figure 5: Electron positions for the backscattering case, colored by energy retained at the exit surface for (a) 200 nm Al film and (b) 5,000 nm Al film. The sample is tilted toward the detector at 70 degrees from normal, and the incident electron energy is 20 kV. The sample normal direction is in the out-of-page direction, and the transverse and rolling directions are indicated.

Electrons of very low energy have been shown to create Kikuchi patterns [10], but these may lead to significant Kikuchi band broadening, which may make rapid automated indexing difficult. However, the widening electron energy distribution with thickness can be even more detrimental to pattern indexing because band edges will become more diffuse. Here we have ignored the effects of changing the incident beam energy, but one might expect more drastic changes versus thickness with a lower-energy incident beam.

The simulations presented here also can reveal some things about how our electrons interact with a bulk material in reflection. Much of the reason that resolution gains have been made in the transmission setup is the type of sample used in transmission compared to reflection EBSD, where samples are typically polished bulk materials. When in "transmission mode" electrons have only a thin film or nanoparticle to travel through; little scattering occurs leading to a compact probe. In reflection mode, there are always a number of high-energy electrons that have entered the specimen and were backscattered without losing much energy. But the flipside of this is also true, where electrons are able to enter the material and scatter many times, losing a great deal of their incident energy and still exit the film to contribute to the pattern. Most of the time, these low-energy electrons are unwanted contributors to the pattern, and energy filtering strategies have been employed to remove them to improve pattern contrast [7]. Figure 5a shows the energy and position distribution for 10,000 20 kV incident electrons traveling through a 200 nm film of aluminum, and Figure 5b shows the same for a 5000 nm Al film, or close to what might be expected from a bulk sample. Many EBSD users don't have access to an energy filter to eliminate some of these low-energy electrons and consequently improve their Kikuchi patterns. One strategy that may help in conventional EBSD is thinning and mounting the sample such that electrons can be absorbed by an underlying substrate or transmitted through the thinned sample [11]. This can prevent multiple scattering events within the sample that lead to contrast-blurring background effects and poor pattern quality. For some cases, the extra effort in sample preparation may be truly worth the hassle to obtain a high-quality orientation map.

Conclusion

Transmission EBSD or TKD is an excellent tool for crystallographic orientation mapping with increased spatial resolution compared to traditional EBSD. The downside of this technique is that the ultimate achievable resolution is dependent on the thickness of the sample. Particularly because with lower electron energies compared to TEM, broadening of the beam throughout the sample can be more of a concern. Thinning samples to typical TEM thicknesses of 100 nm to 200 nm should limit broadening enough to maintain higher spatial resolution compared to conventional EBSD methods. Thinning the sample and mounting it such that electrons can pass through provides an advantage even in conventional EBSD and may be an alternative to energy filtering for improving band contrast.

Note

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