Simultaneous Indexing of Spot and Kikuchi Patterns in Scanning Electron Microscopy (SEM)

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Background incl. aims

Introduced in 2011, Transmission Kikuchi Diffraction (TKD) overcame the challenge in characterizing isolated nanoparticles or ultrafine grains in thin film structure, which was a limiting factor in electron backscattered diffraction (EBSD) due to the small interaction volume and insufficient scatterings [1]. Traditionally, TKD relies on indexing the Kikuchi patterns of the forward-scattered electrons from an electron transparent sample, and it can be performed using a standard EBSD detector in either an off-axis or on-axis configuration with a spatial resolution down to a few nm in on-axis configuration [2]. However, the formation of Kikuchi pattern requires sufficient sample thickness, meaning very thin (e.g. 2D materials) or very light (e.g. organic) materials cannot be investigated via the traditional TKD method in SEM. Hence, a different approach has been developed recently to acquire and analyze the spot diffraction pattern in transmission mode in SEM instead, and it has successfully been used to index 2D materials such as graphene at nm resolution across mm range [3]. Since both spot patterns and Kikuchi patterns have been individually indexed in SEM, the natural next-step is to combine the two techniques so that the on-axis TKD configuration can be applied to a wider range of sample thickness. However, when acquiring both spot and Kikuchi patterns, the intense direct beam often drowns out the spot patterns, and the drastic difference in intensity between the spots and Kikuchi patterns (up to 10^3) makes the detection of both patterns with a conventional CCD detector challenging. Here we present our methods for acquiring both patterns without detector saturation and highlight the advantage by simultaneously indexing spot and Kikuchi patterns in a multi-layered Au thin film structure.

Methods

Two methods have been experimented to address the intensity issue: (1) By applying a filter to the CCD detector screen, the intensity near the optical axis can be reduced hence revealing the geometry of the spot patterns. (2) By acquiring diffraction patterns at different exposure time at each scanned location and combining/stacking the patterns via image fusion algorithm, the dynamic range can be greatly increased and both Kikuchi and spot patterns can be revealed.

Results

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We will illustrate the simultaneous acquisition spot and Kikuchi patterns using both methodologies and provide a comprehensive analysis of their respective advantages and disadvantages on single and multi-layered thin film with various thicknesses. Finally, we will introduce and evaluate the two approaches for obtaining orientation mapping with both spot and Kikuchi diffraction patterns, as well as discussing their strengths and limitations concerning accuracy, throughput, and computation power requirement.

Conclusion

Both CCD detector filtering and multi-exposure image acquisition are valid strategies to acquire both spot and Kikuchi patterns without detector saturation in on-axis TKD configuration. The simultaneous indexing of both spot and Kikuchi patterns makes up for the thickness limitation of the individual spot/Kikuchi pattern indexing, and expands the range of samples investigated with a single technique. With this approach, we aim to expand TKD from an exit-surface characterization technique into a volume information extraction technique with the diffraction information of both spot and Kikuchi signals.

Keywords:

TKD, simultaneous indexing, detector saturation.

Reference:

[1]: Transmission EBSD from 10 nm domains in a scanning electron microscope, Journal of Microscopy, 245 (2012), 245–251, R.R. Keller & R.H. Geiss
[2]: Orientation Mapping by Transmission-SEM with an on-axis Detector, Ultramicroscopy, 161 (2016), 17-22, J. J. Fundenberger, E. Bouzy, D. Goran, J. Guyon, H. Yuan, A. Morawiec
[3]: Orientation Mapping of Graphene using 4D STEM-in-SEM, Ultramicroscopy 219 (2020) 113137
Benjamin W. Caplins, Jason D. Holm, Ryan M. White, Robert R. Keller