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Application of Rocking Beam Tableau DF Imaging on Crystal Size Mapping

Shixin Wang

Micron Technology, Inc., 8000 S. Federal Way, Boise, ID 83707, USA

Crystal size characterization is important in the investigation of polycrystalline materials. TEM-based orientation mapping methods are powerful techniques to study crystal grains qualitatively [1]. In our search of fast and easily accessible alternatives to the diffraction-pattern-indexing based mapping techniques, the traditional dark field TEM (DF-TEM) imaging became our first choice.

A complete survey of crystals of different orientations requires a series of DF-TEM images taken by tilting either the specimen or the electron beam. An effective procedure for this is the hollow-cone dark field (HCDF) imaging, where a tilted electron beam steps to different azimuth angles. The HCDF has been used to reveal crystal grains [2, 3] or dislocation densities [4]. With computer control, this HCDF collection can be automated as demonstrated by Watanabe et al. [3]. For the purpose of creating orientation mapping, we coded a similar control program using DigitalMicrograph® scripting language. The electron beam geometry and the experimental parameters are shown schematically in Fig. 1. The electron beam is tilted stepwise to a certain combination of zenith angle, θ, and azimuth angle, ϕ, according to a predefined pattern. A TEM image is collected at each step.

Depending on the relationship between θ and the angle subtended to objective aperture radius (δ), we might get dark field image (DF, $\theta > \delta$), bright field image (BF, $\theta < \delta$), or high resolution image (HREM, $\theta > \delta$ and θ is larger than a certain Bragg angle θB). This imaging technique uses a predefined pattern of beam tilt and it usually generates a stack of images from different beam tilts, we call this image mode “tableau TEM”. The above listed condition gives the tableau DF, tableau BF, and tableau HREM, respectively.

Observed in back focal plane, a typical tableau pattern has evenly spaced spots on a set of concentric circles, with θ controlling radius and ϕ controlling the rotation steps. For a good survey of crystal grains, tableau DF with multiple θ values might be used in order to record the DF images of any possible diffraction spot. To correct sample drift, the shifting amount can be calculated from a set of companion zero-tilt BF images taken alternatively along the tilted-beam images.

Dark field image contrast is sensitive to the crystal orientation variation. A precession beam has been shown to be effective in reducing the strong dynamic effect and to average out small orientation variations [5]. To reduce the contrast variation due to small crystal orientation, we introduce a beam precession during each DF image collection. The electron beam rocks at a precession angle, α. One or more rotations of precession are completed during a single exposure time (e.g. 4 seconds). Thus, the resulting dark field image is averaged from a range of crystal orientation and the definition of individual is enhanced. The tableau DF is tested on a polycrystalline Si specimen and the result is shown in Fig. 2. In this test, 36 sequential DF images (for 12 ϕ and 3 θ values) are collected. Fig. 2 (A) – (E) are some example DF images from the stack. When the DF images are displaced in an animated movie, the result is a dynamic grain mapping. We can assign a different color to some selected DF images to form a color-coded grain mapping, such as Fig. (F). The result shows the tableau DF can be used to get a qualitative (i.e., without explicit orientation information) orientation mapping.

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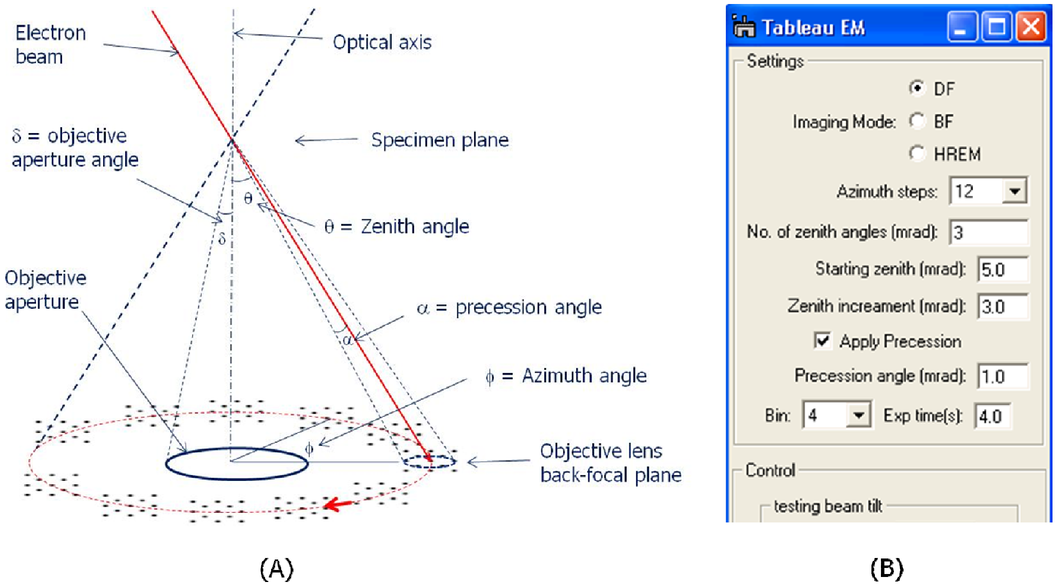

Settings

Imaging Mode: ● DF ○ BF ○ HREM

Azimuth steps: 12

No. of zenith angles (mrad): 3

Starting zenith (mrad): 5.0

Zenith increament (mrad): 3.0

☑ Apply Precession

Precession angle (mrad): 1.0

Bin: 4 Exp time(s): 4.0

Control

testing beam tilt

Fig. 1. (A) A schematic of electron beam geometry for tableau TEM. (B) A partial view of the control interface.

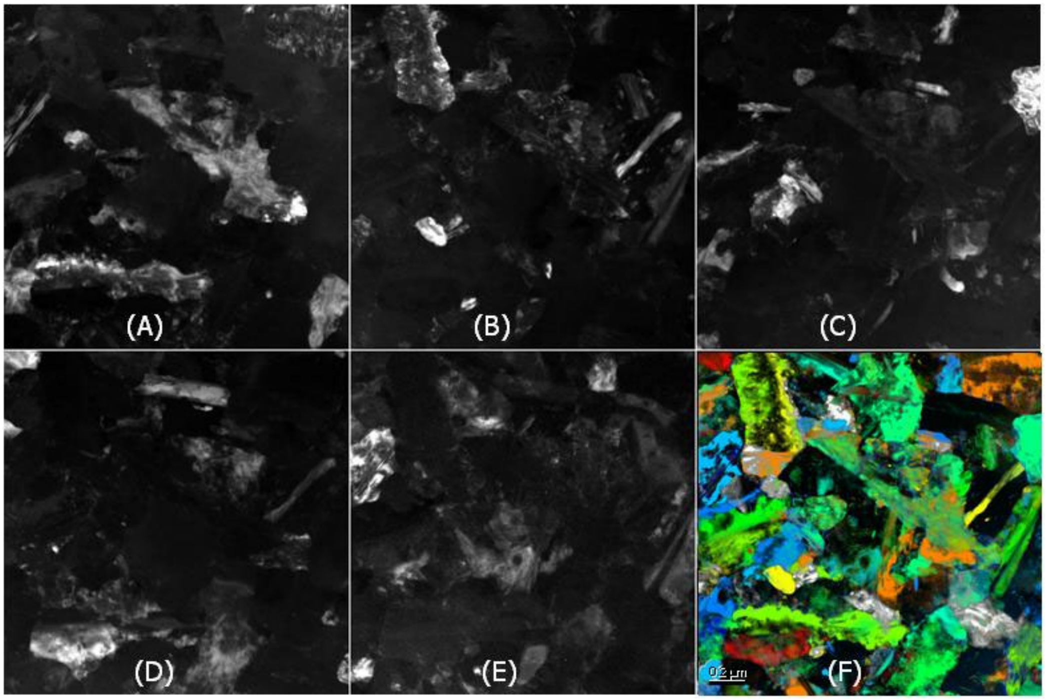

Fig. 2. (A) – (E) Some dark field images from a tableau DF stack. (F) A color-coded grain map generated from the tableau DF stack.