doi:10.1017/S1431927622004214
Microsc. Microanal. 28 (Suppl 1), 2022

Development and Validation of In-Situ Specimen Orientation Method for Quantitative SEM/EDS Analysis

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Quantitative analysis of the elemental composition of specimens with scanning electron microscopy/energy dispersive x-ray spectroscopy (SEM/EDS) requires specimens to meet a set of conditions to ensure specimen geometry does not induce errors. In particular, for accurate quantitative EDS analysis, the specimen surface must be sufficiently flat and orthogonal to the SEM's electron beam [1, 2]. In this presentation, we report the development and validation of a method for orienting sufficiently flat microscale surfaces, invisible to the naked eye, in the SEM such that the surface is orthogonal to the incoming electron beam. The method is based on using multiple SEM images to measure the change in distance between two points, the line between which is perpendicular to the SEM tilting axis, on images of different tilt angles. As the method utilizes multiple SEM images and measurements, it provides a good testing ground for tools being used in our current efforts to develop and statistically analyze a specimen orientation procedure more efficient and precise than previous methods [3].

The SEM has two operations which enable in-situ manipulation of specimens: rotation and tilt. The rotation operation rotates the specimen through an angle θ about an axis parallel to the incoming electron beam (defined as the z axis), and the tilt operation tilts the specimen through an angle ϕ about an axis (the y axis) perpendicular to the rotation axis. For a plane inclined at some arbitrary angle, we define the proper angles as the coordinates $(\theta_0,\ \phi_0)$ in the θ-ϕ parameter space such that the plane's surface is orthogonal to the electron beam. Once a sufficiently flat plane is identified, we may determine the proper angles with the following procedure: (1) take a series of SEM images at incremental rotation angles, (2) tilt the specimen by some angle $\Delta\phi$, (3) repeat (1), and (4) measure, for each rotation angle, the x distance between two features in the tilted and untilted images. The proper angles may be calculated by forming the ratio of the tilted and untilted measurements at each rotation angle and fitting a theoretically determined curve to the data.

To apply the method, we used a macroscopic flat specimen oriented at a random rotation and tilt angle. With a 50μm field of view, a series of SEM images were taken every 10° rotation at tilt angles of 0°, 20°, and -20°. Measurements were made on the SEM images as shown in Fig. 1 to form the ratio of the distance between two points. The curve that results from these measurements is show in Fig. 2. Using a least squares curve fitting program, the optimal θ_0 and ϕ_0 values were determined. A picture of the specimen oriented at the proper angles is also shown in Fig. 2; we see that the surface appears to be orthogonal to the direction of the electron beam. [4].

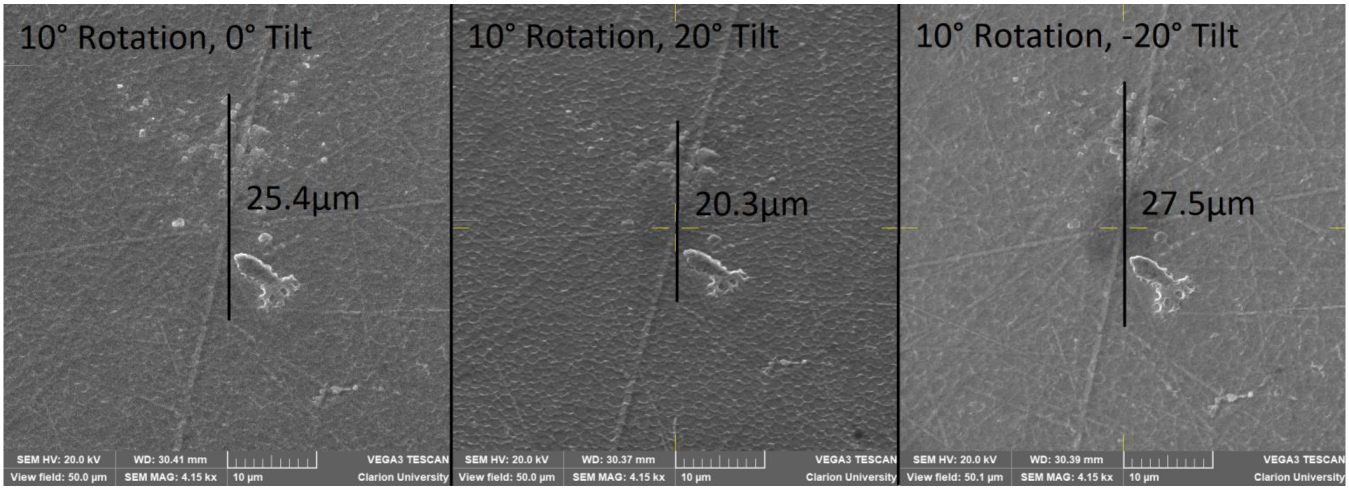

Figure 1. Measurements made on SEM images at 10˚ rotation.

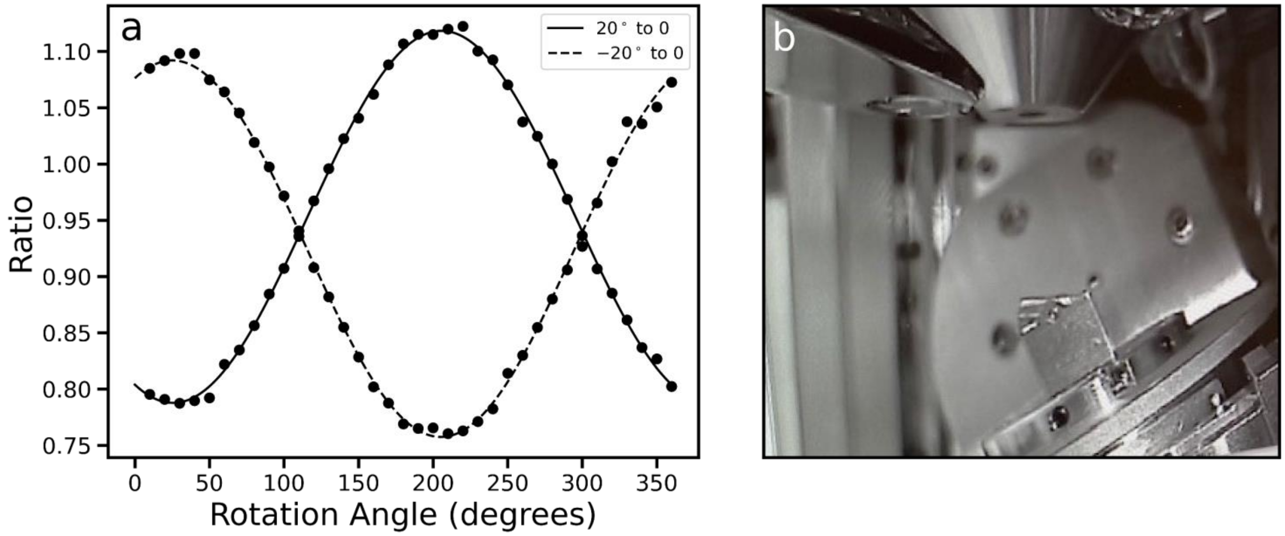

Figure 2. (a) a plot of the experimental data at each rotation angle along with the curve fit to the data, and (b) the specimen oriented at approximately the calculated proper angles.

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[4] Financial support by National Science Foundation (DMR-1900077) is acknowledged.