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Particle Orientation Adjustment Inside Scanning Electron Microscope: Side View Approach

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Because of the ease of operation and straight-forward explanation of the images, SEM is widely used in variety of applications. Many attachments have been developed, collecting different signals and enabling the insight of the material from different perspectives. Two well-known examples are Energy Dispersive X-ray Spectroscopy and Electron Backscattering Diffraction. In such analysis, for reliable result, it is assumed that the specimen has a flat surface, and the primary electron beam forms a specific angle with the specimen surface [1, 2]. When the specimen can be resolved with unaided eyes, the specimen can be directly placed into the necessary orientation [3, 4]. However, if the surface of interest is not visible outside the SEM because of small size, an in-situ method to orient the specimen must be employed.

For a typical modern SEM, the only information and degrees of freedom available during an in-situ specimen orientation are SEM images and the rotation and tilt operations of the SEM stage, respectively. The goal of in-situ specimen orientation adjustment is to establish a reference point of the orientation, followed by stage rotation and tilt manipulation to bring the specimen to desired orientation relative to the electron beam and the detector. There are two approaches to establish the reference point: making the surface perpendicular or parallel to electron beam. The first approach is based on the consideration that a planer shape has maximum projected area in a top-down view. This approach works well when the area can be measured with accuracy, especially with computer aid [5]. When computer program fails and the area has to be measured manually, it has been found that the accuracy is less satisfactory and is in the order of 10°, which is not sufficient in many applications [6]. This paper reports our attempt to set up specimen orientation reference point by a side view approach.

Fig. 1(a) is a SEM image of a spherical particle. For convenience, we adopt a coordinate system with x pointing toward the right, y pointing up, and z pointing out of the paper relative to the SEM image. It should be pointed out that this coordinate system is fixed relative to the SEM instrument frame while the specimen moves with the SEM stage relative to the frame. The rotation function of SEM corresponds to rotation along z-axis when the stage tilt angle is zero. The tilting always corresponds to rotation along negative x-axis. Our chosen reference point for a specimen with flat surface is when the surface is parallel to the x-z plane or perpendicular to the y-axis. In this orientation, the area of the specimen surface is zero or the specimen surface appears to be a straight line in a SEM image.

The specimen studied is a spherical particle found in alloy of $Al_{65}Cu_{25}Fe_{15}$ composition prepared by arc melting. For this study, the crystalline structure can be considered as TiNi type cubic system [7]. As marked on Fig. 1(a), facets are observed on the surface. Two examples are marked as 3A and 3B, respectively, where 3 is used because these facets are related to 3-fold rotational symmetry. The goal of the present study is to bring a facet to the reference point by manipulating the stage tilt and rotation functions. Then, we are able to calculate the surface normal vector of the facet at zero tilt and rotation angles relative to the adopted coordinate system. Once the surface normal of two facets are determined, the angle between them can be calculated. Such experimental results can be compared with the predicted one, providing a measure about the accuracy of the method. The SEM used is a Tescan Vega-3.

An example of such adjustment for facet 3A is shown in Fig. 1(b) and (c). This facet is of terrace-like structure, consisting multiple parallel planes. Fig. 1(b) is the SEM image at zero stage rotation and tilt angles, where the facet appears to be multiple concentric ellipses. When the facet is in reference orientation, the surface of terrace-like structure is parallel to the electron beam or chosen z-axis and is normal to the y-axis. As shown in Fig. 1(c), the facet appears to be multiple parallel lines in the horizontal direction. Each line corresponds to one plane parallel to the electron beam and is normal to the chosen y-axis. This reference point is achieved through stage tilt of 26° and rotation of 359°, which allows us to calculate the surface normal unit vector of the facet relative to the chosen coordinate system when the stage is at zero tilt and rotation angles. In a similar way, surface normal unit vector of facet 3B in Fig. 1(a) can be calculated. Then, the angles between the surface normal of facet 3A and 3B is determined to be 68°. This is consistent with the predicted 70.5° based on crystalline structure consideration, demonstrating the effectiveness of the orientation adjustment approach [8].

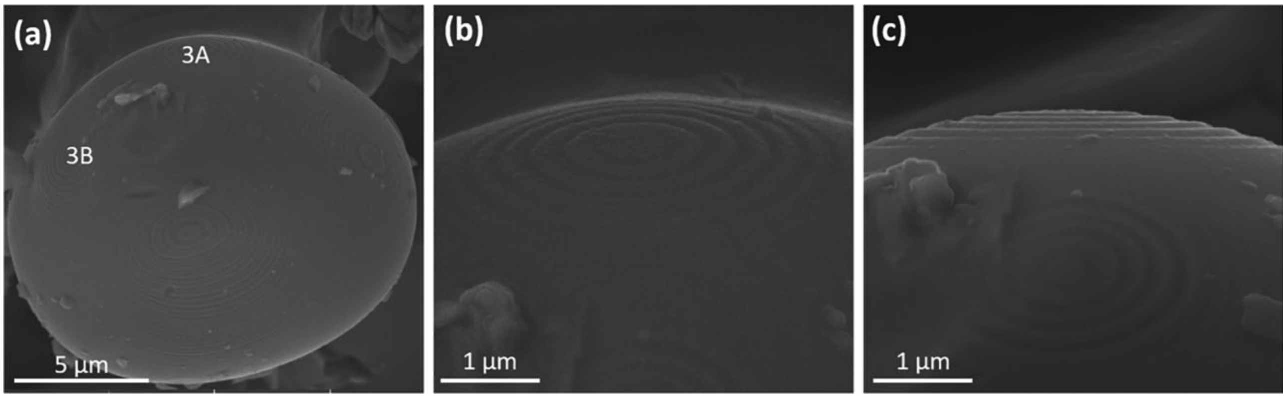

Fig. 1. SEM images showing (a) the whole particle, (b) the enlarged image of facet 3A, and (c) the facet in reference orientation.

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