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Application of Electron Back Scattering Diffraction in Facet Crystalline Orientation Analysis

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The surface of a crystal is often enclosed with facets, which are planes smooth down to atomic level [1]. Overall, it is known that the formation is to minimize surface energy, which means that a crystal will take a shape that maximizes the portion of crystalline planes of low surface energy. Therefore, information about the crystalline orientation of a facet gives us an insight about a specific crystal structure. Further, facets affect the performance of some materials, such as the chemical activity of catalysts [2]. This is especially true for nano- and micro- particles, where the increased surface area means stronger driving force in forming crystals with shapes corresponding to equilibrium.

The crystalline orientation of facets can be determined by simple morphology observation and by relating the orientation of facets with crystal structure. The first approach tends to be a speculation based on previous knowledge. This approach is widely used in examining crystals of macroscale. As examples for the second approach, there are reports about the characterization of nanoparticles using Transmission Electron Microscope (TEM) [2]. TEM is an excellent tool to study nanoparticles, when specimen preparation can be accomplished by simple dispersion. When a particle is in micrometer order, there is challenge in specimen preparation. The combination of Scanning Electron Microscope (SEM) and Electron Back Scattering Diffraction (EBSD) is believed to be an effective tool in this case. This paper reports our results in expanding the application of SEM-EBSD in this field.

The facet that has been studied is on spherical particles found in alloy of $Al_{65}Cu_{25}Fe_{15}$ composition prepared by arc melting. The spherical particle can be considered as cubic β AlFe(Cu) phase [3]. A TESCAN Vega-3 XMU SEM equipped with Oxford Aztec symmetry EBSD system was used for characterization.

A typical SEM image of the interested spherical particle is shown in Fig. 1. (a). On the SEM image, the facets appear as different patterns, based on which they can be classified to three types and have been marked with markers of elliptical, triangular, and square shapes. The focus of the present paper is on those marked with squares. This type of facets appears to be multiple concentric circles on the SEM image. The circles define multiple parallel planes. Based on the β crystal structure consideration and a quick examination of the layout of different facets, we proposed that the planes of the circles are parallel to the (1 0 0) crystalline plane or perpendicular to the [1 0 0] crystalline direction of the cubic phase. In other words, this facet corresponds to the four-fold rotational symmetry. Similar consideration and the choice of markers are adopted for other two types. Facets marked with elliptical and triangular shaped markers correspond to two- and three- fold rotational symmetries, respectively. This model is confirmed by two independent approaches.

In the first approach, the orientation of the spherical particle is adjusted so that the facet of interest is edge-on. Here, edge-on orientation is defined as the case when electron beam is perpendicular to the planes of a facet, which is achieved by adjusting the rotation and tilting functions of the SEM stage. An

example is shown in Figs. 1(a), where the central facet is in random orientation. In Fig. 1(b), the facet is edge-on. To examine the arrangement of facets, two lines connecting the centers of surrounding facets have been drawn. The two lines are expected to form angle 90^{o} and the measured one is 86^{o}. We consider this difference comes from the uncertainty of edge-on adjustment and the inaccuracy in considering the particle to be spherical shape. With these factors considered, it is concluded that the arrangement of facets is consistent with our expectation, validating the proposed model.

This conclusion is verified by EBSD analysis. For the analysis by EBSD, the specimen is tilted by 70^{o} from the setting shown in Fig. 1(b). A typical SEM stage cannot accommodate this large angle tilting. To solve this problem, a special specimen holder has been assembled as shown in Fig. 1(c), which has its own unlimited rotation and tilting ranges. An SEM image of the same sphere after such tilting is shown in Fig. 1(d), in which the facet of interest can be found on the top of the sphere. This setting allows us to take high quality EBSD pattern, which is shown in Fig. 2(a). The pattern is in good agreement with the simulated one by considering the cubic β phase. Further, as shown in Fig. 2(b), analysis of the pattern revealed that the specimen surface, which is the facet surface in our case, is 2^{o} off from the (1 0 0) crystalline plane. Within the experimental uncertainty, the two can be considered as parallel with each other, again supporting our proposed model.

It should be noted that, while both approaches support our model, the facet of interest is parallel to the (1 0 0) plane. The first approach involves significant speculation; therefore, it has fewer applications. In contrast, there are no limitations in applying the second approach, demonstrating the effectiveness of SEM-EBSD combination in facet orientation analysis [4].

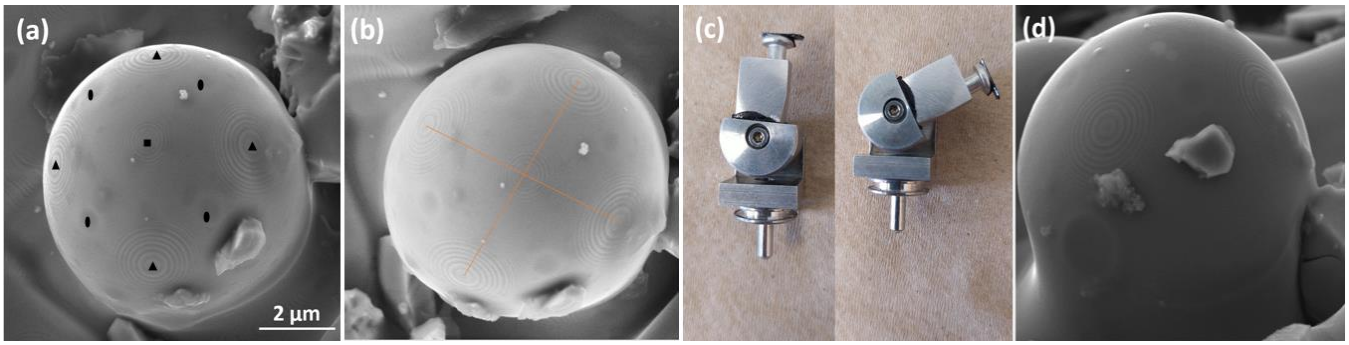

Figure 1 (a) and (b) are SEM images of a spherical particle at random and edge-on orientations. (c) shows the un-tilted (left) and tilted (right) images of the assembled special specimen holder. By using this holder, the sphere can be tilted 70o from the edge-on orientation and the corresponding SEM image is shown in (d).

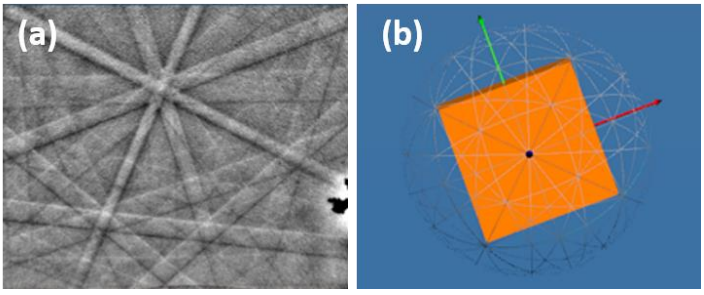

Figure 2 EBSD pattern (a) and the three-dimensional view of the cubic unit cell relative to specimen.

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