doi:10.1017/S1431927620020735
Microsc. Microanal. 26 (Suppl 2), 2020

EDS Analysis of Icosahedral Quasicrystalline Thin Film in $Al_{65}Cu_{25}Fe_{15}$ Alloy Prepared by Arc-melting

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It has been well accepted that for quantitative EDS elemental analysis on SEM, the surface of specimen must be flat and perpendicular to primary electron beam. In our recent study of $Al_{65}Cu_{25}Fe_{15}$ alloy prepared by arc melting, we have found icosahedral quasicrystalline (IQC) thin film on the surface of slab-shaped λ phase, which has not been reported previously. The elemental composition need to be determined, which can be accomplished by using SEM-EDS technique. The slab is in the several ten micrometer order and its surface is typically not perpendicular to primary electron beam in the as prepared SEM specimen. Stage adjustment has to be carried out to satisfy such requirement. The result of our attempt toward this goal is reported here.

The as-prepared $Al_{65}Cu_{25}Fe_{15}$ alloy was mechanically crushed to small pieces to expose the interesting slabs. The obtained small pieces are mounted on SEM stub with double-sided carbon tape. An extensive survey was carried out to locate pieces that contain slabs of interest and are suitable for EDS analysis. The criteria used are: (a) the surface of the slabs should have IQC thin film, and (b) the piece should be separated from others so that signals for EDS are not blocked. The challenge is that while the surface of the slab is locally flat, it is typically not perpendicular to the primary electron beam.

The following procedure has been suggested to make the slab surface perpendicular to the primary electron beam. It is accomplished by using stage rotation and tilting function, which are typical for modern SEM. The flat surface of the slab forms an angle with a plane perpendicular to the primary electron beam. As shown in the schematic drawing of Fig.1, the direction of the interception line of the two planes can be found out experimentally and plays important role in bringing the slab to the proper orientation for EDS analysis. To determine the direction of the interception line, we rotate the stage at a step size of 10^{o}. At each rotation angles, three images are taken with tilting angles of -10^{o}, 0^{o}, and 10^{o}, respectively. Then, the distance of two features on the image in a direction perpendicular to the tilting axis were measured and are represented by dT-10, dT0, and dT10 for the cases of tilting angles of -10^{o}, 0^{o}, and 10^{o}, respectively. An example of such images and measurement is shown in Fig. 2 part 'a'. The ratio of [(dT-10)-(dT0)]/(dT0) was calculated and graphed as a function of rotation angle. In a similar way, a graph corresponding to 10^{o} tilt was generated, as shown in Fig. 2 part 'b'. When the tilting axis is perpendicular to the interception line of the two planes, both ratios are close to zero while at other angles the ratios deviates from zero significantly. When the interception line is parallel to the tilting axis, the ratio has maximum values. In our particular case, the ratios are approximately zero at a rotation angle of 50^{o}. This implies that, at a stage rotation angle of 140^{o}, the interception line is parallel to tilting axis and tilting alone could bring the slab surface to the desired perpendicular orientation to the primary electron beam. When the slab is perpendicular to the primary electron beam, the distance of two features in a direction perpendicular to the tilting axis has maximum value.

A more accurate procedure, such as fitting the experimental data to mathematical model, to determine the optimum rotation and tilt angle will be established. The composition of the IQC thin film will be determined and will be compared to that of the quasicrystalline particles with distinct pentagonal facet,

which will lead to better understanding about the formation process of IQC phase. Results from SEM-EDS will also be compared to that obtained from TEM-EDS study.

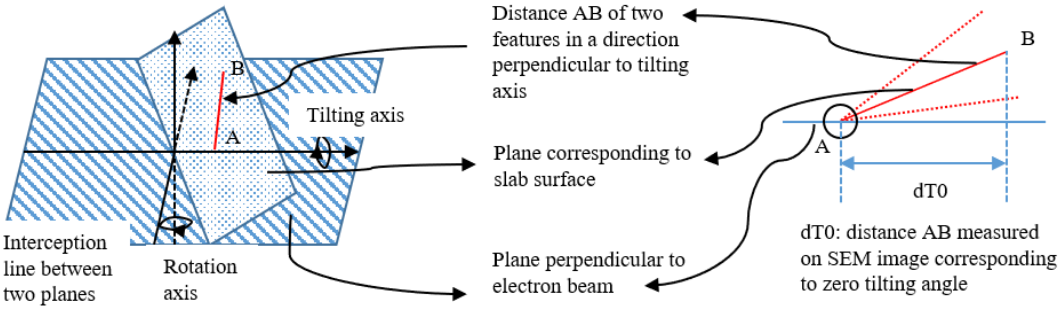

Figure 1. Schematic Drawing

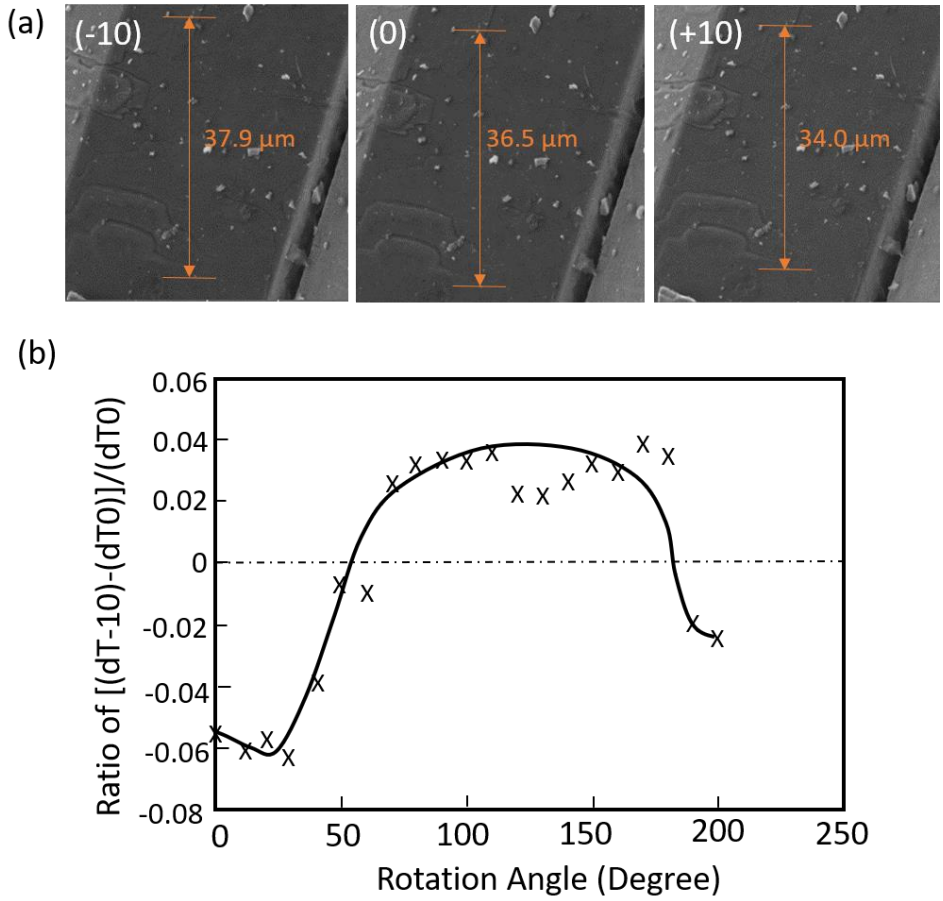

Figure 2. Tilts and Ratio of [(dT-10)-(dT0)]/(dT0)

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https://doi.org/10.1017/S1431927620020735 Published online by Cambridge University Press