A COMPARISON OF GRAIN SIZE MEASUREMENTS IN AL-CU THIN FILMS: IMAGING VERSES DIFFRACTION TECHNIQUES

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Numerous microscopy techniques, based on both imaging and diffraction, exist for the measurement of grain size distributions in polycrystalline thin-film samples. The accuracy of each technique is affected by three major factors: the effective resolution of the instrument relative to the characteristic grain size, the detection of the grain size through the thickness of the film, and the recognition of boundaries between adjacent grains during post-processing. When the instrument resolution is primarily considered, the measurement technique has a practical grain size measurement range, see Fig. 1 for a comparison of ranges for several measurement techniques. In bulk metallurgy grain size analysis, methodology has been developed to represent 3-dimensional grain structures from measurements taken on 2-dimensional images.[1],[2] In thin films, these procedures are often simplified because many films have columnar microstructures with single grains spanning the film thickness and the electron mean free path through the material. For example, the interaction volume can be less than 200 nm deep in Al films due to the oblique incidence angle (approximately 20° from the surface) employed for EBSP measurements.[3] The ability to accurately determine the location of grain boundaries in a micrograph can be a source of measurement error. Most measurement techniques rely on manual or automated recognition of grain boundaries through differential contrast between grains (STEM and FIB) or at grain boundaries (optical and AFM). Presently, commercially available software that automatically finds grain boundaries in micrographs typically renders grain boundaries inaccurately so grain structure analyses are often manual and tedious.[4] EBSP delineates grains by calculating the relative misorientation of adjacent data points and therefore can be more easily automated. However, EBSP measurements are limited by the precision of determining orientations within a single grain or single crystal. Typical orientation noise of 1° has been reported which can be problematic when studying low angle grain boundaries.[5]

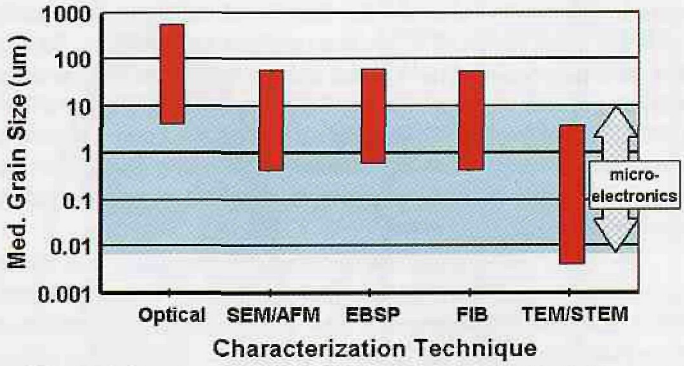

Figure 1: A comparison of grain size measurement techniques based on the practical grain size measurement range.

Due to the aforementioned factors that effect grain size measurement techniques, we studied the effectiveness of various measurement methods on a series of three 0.5 μm thick Al-0.5wt.% Cu blanket thin films sputter deposited on either Si/SiN_x (N) or Si/phosphorus-silicate glass (G) substrates. The three films were labeled 1N, 2G, and 2N and had median grain sizes of 0.6, 1.2, and 2.2 μm, respectively. The grain size measurements were initially performed by recording the thin film microstructure using various grain imaging techniques including dark field optical microscopy, AFM, FIB, and STEM. From these images, the grain boundaries were hand traced and digitized, the grain areas were measured from the digital images using Scion Image image analysis software[6], and log-normal distributions of the grain areas were plotted. Median grain diameters were determined from the median grain areas assuming circular grains. The grain area distributions derived from these imaging techniques were compared to area distributions determined from EBSP orientation imaging using a HKL Technologies EBSP system with Channel 4.0 software on a LEO 1530 field emission SEM. The EBSP maps were collected on the three thin film samples tilted to 70° using step sizes of 0.1 μm by 0.3 μm for samples 2G and 2N and 0.02 μm by 0.06 μm for sample 1N. A grain boundary was defined as two adjacent pixels having greater than 10 degrees of misorientation. The EBSP patterns were indexed to an Al *m3m* face centered cubic crystal structure with an indexing success rate that ranged from 75 to 90% of all of the pixels. Orientation images obtained from the EBSP data were filtered using a noise reduction routine where unindexed pixels were replaced with indexed neighbors. Though the Channel 4.0 software allows direct grain area measurements, typically a large number of small grains

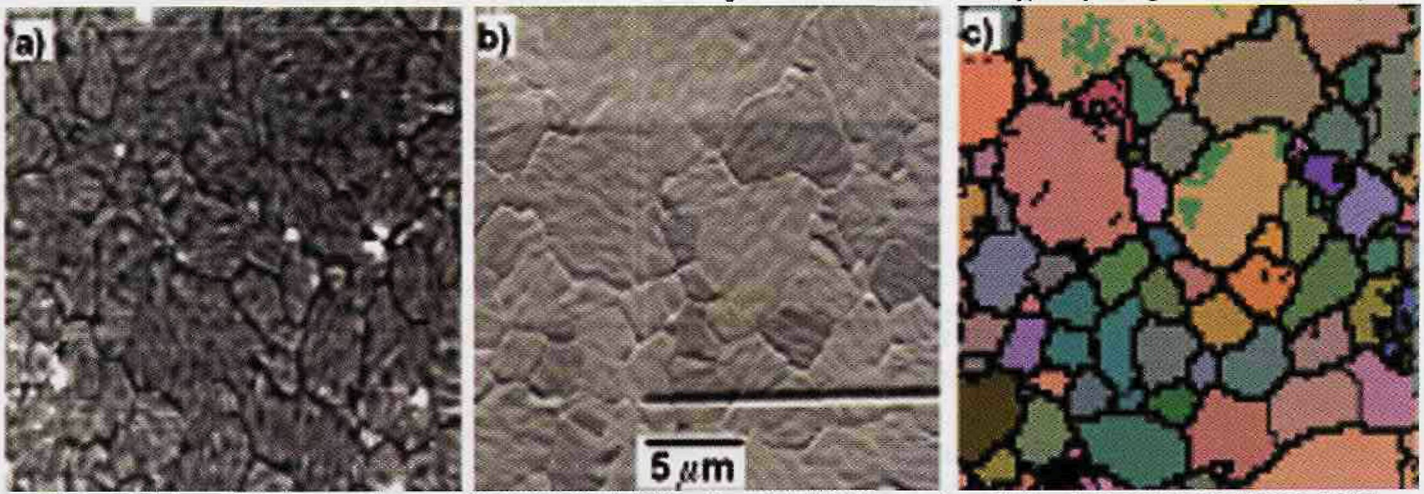

Figure 2: Various microstructural images of sample 2N: a) AFM surface plot, b) FIB secondary electron image generated from a Ga ion beam, and c) EBSP grain orientation image.

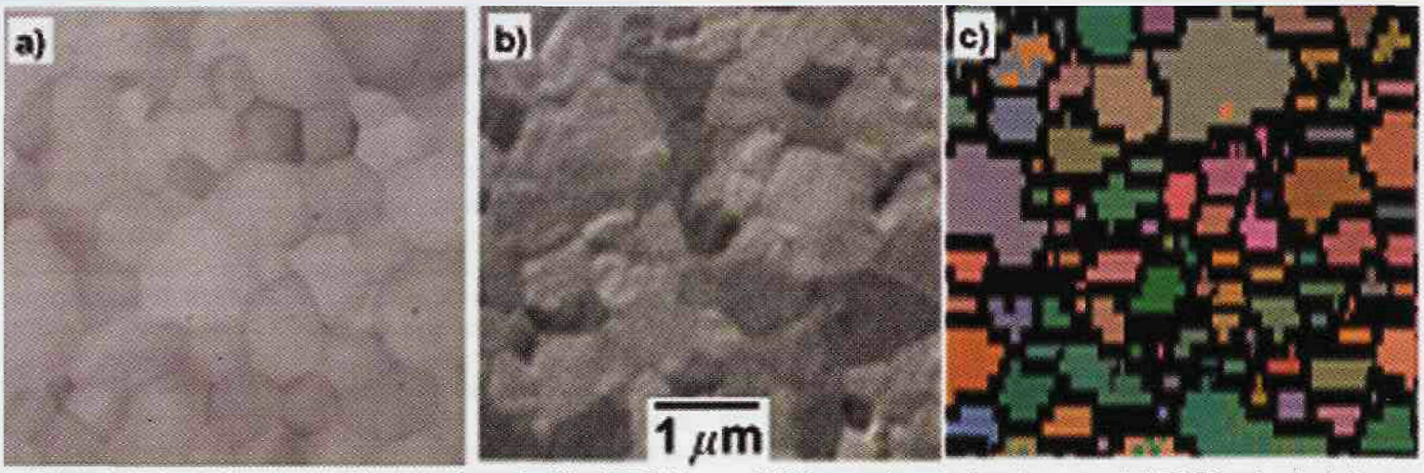

Figure 3: Various microstructural images of sample 1N: a) STEM image, b) FIB secondary electron image, and c) EBSP grain orientation image.

(e.g. single pixels = one grain) were erroneously determined with this software. Instead, black and white orientation maps were used to measure the grain areas in a manner similar to the digitized grain tracings of FIB, AFM or STEM images. Grain area distributions and median grain sizes were compared for all measurement techniques. To further understand the accuracy of EBSP in representing the grain morphology, a region of sample 2N was marked and imaged in the FIB and then orientation imaged using EBSP. A comparison of the grain images for these two techniques was determined by overlaying the grain images.

Fig. 2a-c shows a comparison of grain structure images obtained by AFM, FIB and EBSP orientation imaging, respectively, taken at similar magnifications on sample 2N. For films with grain sizes that are relatively large for typical thin films (> 1 µm), all three methods imaged the grains similarly. For the finer grained sample, 1N, STEM imaging produced the best grain images, see Fig. 3a-c. The FIB images showed indistinct grain structures suggesting that the resolution limit was nearly reached for this technique. The EBSP orientation maps imaged the larger grains well but the smaller ones were represented by single pixels. Also, the EBSP grain morphology was not similar to the STEM and FIB imaged grains and this disparity was probably due to the step size and resolution limit of the EBSP technique. Fig. 4 shows a comparison of the median grain areas and corresponding median grain diameters for all three samples. This figure shows that there is good agreement between all the measurement techniques for the two samples with median grain diameters greater than 1 µm but there is disparity between these methods in the finer grained sample, 1N. As with the qualitative assessment of the grain images, the finer grained sample appeared to push the resolution limit of both FIB and EBSP grain imaging methods and therefore could have been a source of measurement error. This is particularly true for the grains from the lower end of the 1N grain area distribution where grain diameters are less than 0.2 µm.

To better understand the accuracy of grain size and shape determination using EBSP orientation imaging, sample 2N was FIB marked with fiducials and then grain images were obtained from identical regions using both FIB and EBSP. Fig. 5a-b shows the FIB and EBSP images, respectively. Fig. 5c is an overlay image of the grains traced from the FIB image (shown in red) on the EBSP image. These images show that for large grain areas, the EBSP orientation images determine similar grain morphology as FIB imaging but some of the small grains shown in the FIB images were not imaged as distinct grains in the EBSP image. The missing small grains in the EBSP images have been attributed to choosing too large of a step size during analysis, the inability to control the x and y step size independently during analysis, errors in pattern recognition, and grains boundaries with < 10° of misorientation.

In summary, we found that AFM, FIB, STEM and EBSP grain size analysis techniques produced grain area distributions and median grain diameter measurements that are in good agreement for films with median grain diameters greater than 1 µm for Al-0.5wt.% Cu blanket films. These techniques also

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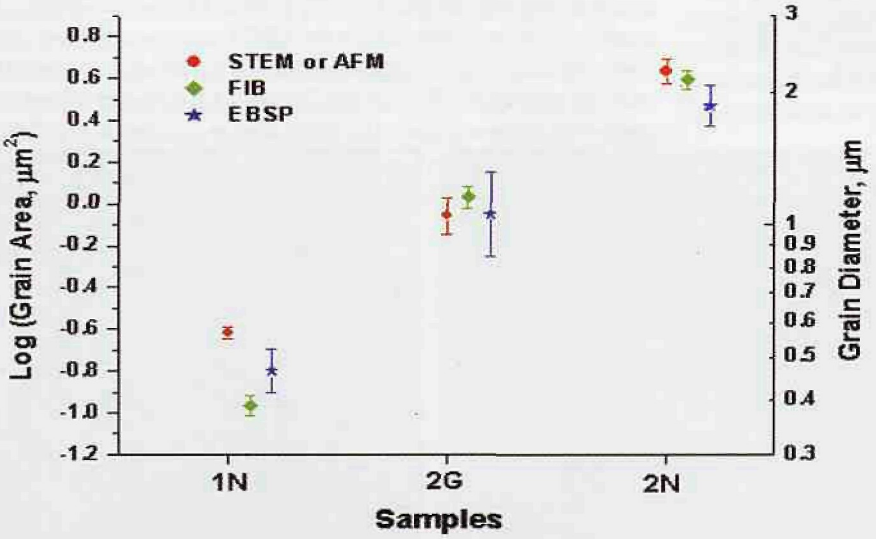

Figure 4: Comparison of median grain area and grain diameter data obtained from STEM/AFM, FIB and EBSP measurement methods.

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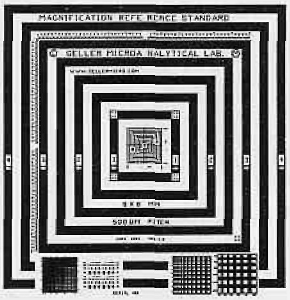

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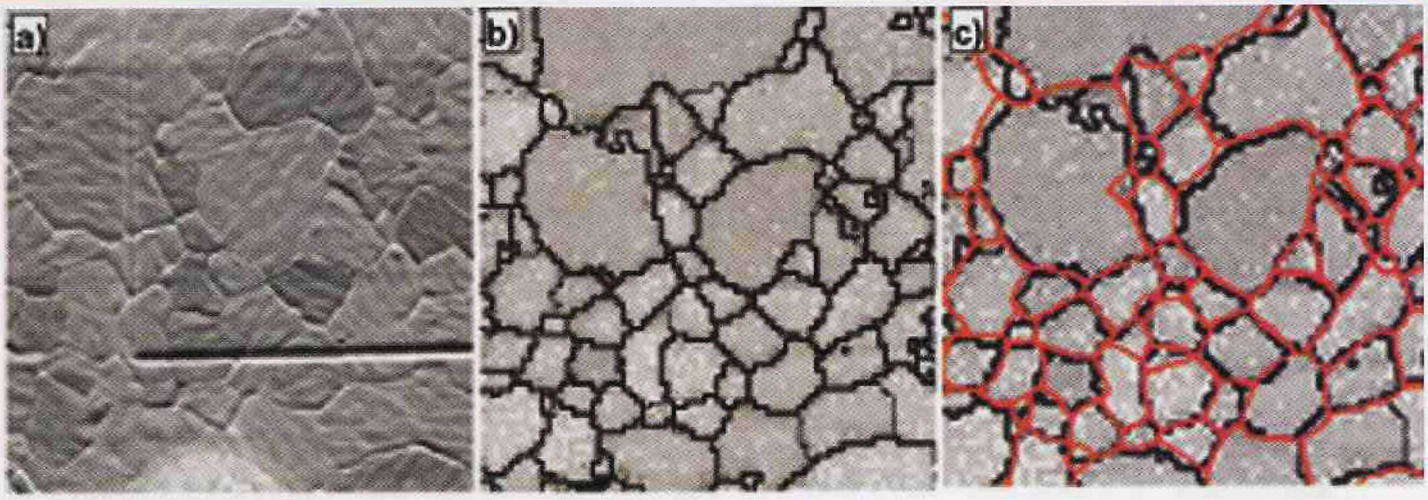

Figure 5: Direct comparison of FIB imaging and EBSP orientation imaging taken from the same region on sample 2N: a) FIB image of the box corner, b) EBSP orientation image of the same corner, and c) overlay image of FIB traced grains on EBSP image.

worked reasonably well for sample 1N with a median diameter of 0.6 μm but error was probably introduced when attempting to image grains from the lower end of this distribution. Direct correlation of the grain morphology determined by both FIB and EBSP was found for sample 2N, except that some of the very fine grains were not always detected by EBSP. Comparing all of the grain size measurement methods, EBSP is the measurement technique of choice since it is automated, relatively rapid, and orientation information is also obtained during the analysis but there is a finite grain size limit below which this technique is less accurate. ■

References:

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