Lee PJ, Larbalestier DC (2000) Position normalization as a tool to extract compositional and microstructural profiles from backscatter and secondary electron images. Microscopy and Microanalysis, 6, Supplement 2:1026–1027. https://doi.org/10.1017/S1431927600037624

POSITION NORMALIZATION AS A TOOL TO EXTRACT COMPOSITIONAL AND MICROSTRUCTURAL PROFILES FROM BACKSCATTER AND SECONDARY ELECTRON IMAGES

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Position normalization is a method of manipulating data from any image source so that the effective information to noise ratio is dramatically increased. In this paper we present two very different examples of the application of position normalization used in the study of high current density superconductors. In the first example we use FESEM fractography to analyze grain boundary density and grain shape in micron-diameter filaments and then normalize the data obtained from individual grains to their positions with respect to the filament-matrix interface. In this way we extract the change of grain structure with position in apparently inhomogeneous microstructure. In the second example we analyze the backscattered electron intensity, BEI, from the cross-section of a superconductor filament with respect to its proximity to phase boundaries. In this way we extract small trends in the mean atomic number with a spatial resolution of better than 100 nm.

The primary commercial superconductor for applications in the 10-20 T field range is Nb_3Sn. The key property for application in high field magnets is the critical current density, the maximum current that can be carried per unit cross-sectional area. Increasing the grain boundary density can increase critical current density. We have been using FESEM images of fractured Nb_3Sn strands to view the grains and to determine the grain boundary density, grain morphology, and grain aspect ratio.[1] Figure 1 presents a fracture cross-section of a 4 μm-diameter Nb_3Sn filament produced by reaction between a Nb filament and surrounding Cu-Sn matrix. The external filament surface after etching away the matrix is shown in Figure 2. As the reaction progresses through the filament, the Sn content of the Cu-Sn phase is reduced and Sn must diffuse over greater distances, resulting in a change in Nb_3Sn grain size, morphology, and composition. We here analyze the 1200 inhomogeneous Nb_3Sn grains in Figure 1 with respect to distance from the original Nb-bronze reaction interface. Figure 3 shows that the local grain boundary density and grain aspect ratio increase significantly with distance from the original bronze-Nb interface.

The potential for applying backscattered electron spectroscopy, BES, to reveal chemical composition changes with submicron spatial resolution has been explored by a number of authors.[2,3,4] Practical resolution appears to be only limited by the signal to noise ratio.[5] By breaking a conventionally obtained Backscattered Electron Image, BEI, into individual pixels and normalizing the positions of the pixels to the interfaces under study, we can combine signals from the entire image. For Nb_3Sn filaments with < 200 nm grain diameters, this approach also averages over multiple grain orientations, thus eliminating back scatter yield variations from consideration. The superconducting properties are sensitive to composition so it is important to know if there is a compositional gradient and if so how steep and how uniform it is. With superconducting layers only 1-4 μm in thickness, EDS only has sufficient resolution to provide an average composition. In Figure 4 we show a cross-section of a partially reacted Nb_3Sn filament, where the Nb_3Sn has formed from diffusion a Nb filament and Sn from the surrounding bronze matrix. Figure 5. is a profile compiled from four analyses of the image and over 110,000 data points (from a 512×384 pixel image). First the BEI of the Cu(Sn) area around the filament was analyzed with respect to the distance from the Nb_3Sn filament surface, second the outer Nb_3Sn filament area was analyzed with respect to the filament surface, third the inner Nb_3Sn area was analyzed with respect to the interface between the Nb_3Sn and the unreacted Nb core and finally the Nb area was analyzed with respect to the Nb_3Sn-Nb interface. As the profile approaches the center of the core the noise level increases because of the reduced number a pixels available for analysis. Key features are revealed that have not been previously observed: that the Cu(Sn)-Nb_3Sn and Nb_3Sn-Nb interfaces are sharp (within the confines of the 5 kV accelerating voltage and the filament surface irregularity) to within ~50 nm, and most importantly for the superconducting properties that the compositional gradient is small but continuous across the superconducting layer.

References

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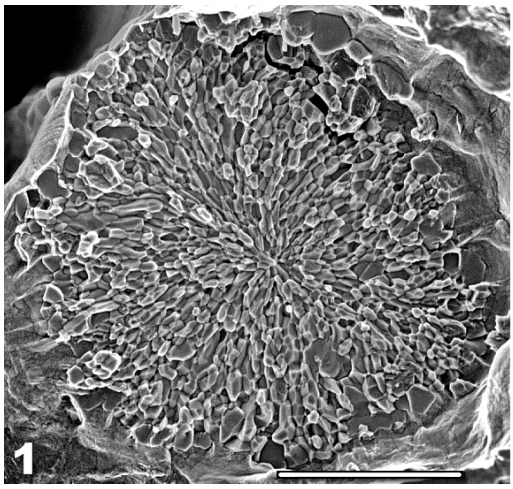
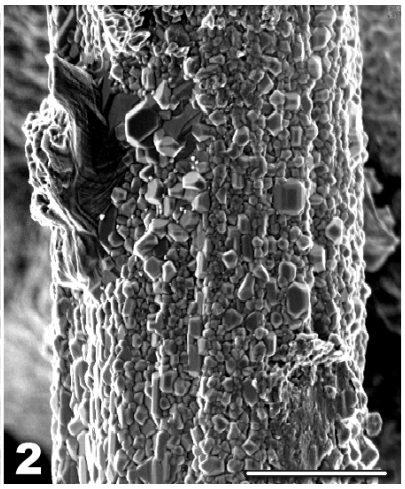
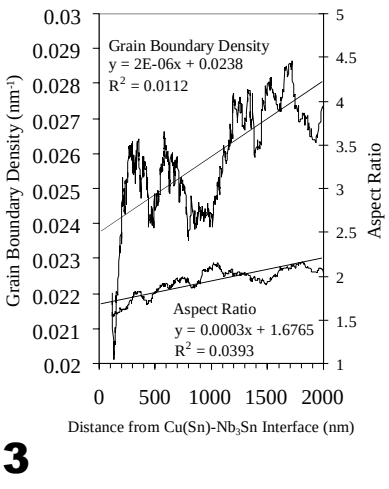
6. This work supported by the U.S. Dept. of Energy, Office of Fusion Energy Sciences, DE-FG02-86ER52131 and Division of High Energy Physics DE-FG02-91ER40643, it also benefited from NSF-MRSEC DMR-9632427 supported facilities.







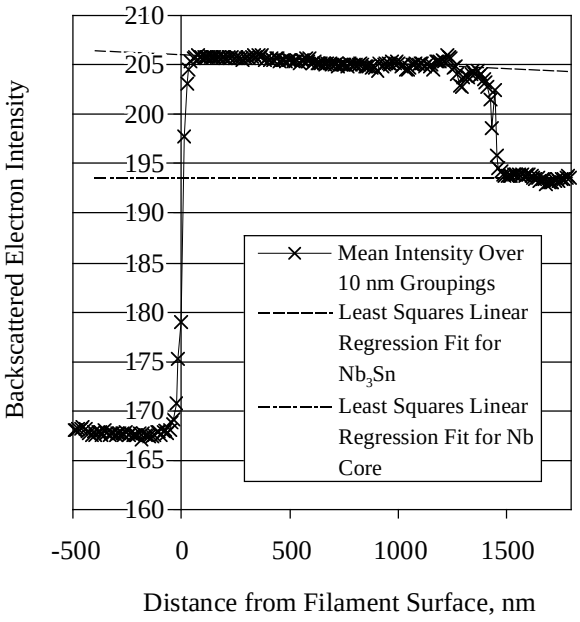


FIG 1. FESEM image of a fracture cross-section of a Nb_3Sn superconductor filament showing irregular grain size across filament. Scale bar is 2 µm.

FIG 2. FESEM image of extracted Nb_3Sn filament showing irregular surface on filament. The irregularity of the surface means that in order to obtain sharp profiles of the filament interface in transverse cross-section, the accelerating voltage must be as low as possible without loosing to much signal to noise ratio. Scale bar is 2 µm.

FIG 3. 100 point moving averages for local grain boundary density and aspect ratios of grains in Figure 1 are shown with corresponding least squares linear regression fits and the resulting parameters.

FIG. 4. BEI (5 kV) of a partial cross-section of a composite Nb_3Sn superconductor where the Nb_3Sn has been formed from the reaction between a Nb filament and Sn from the surrounding bronze matrix. Key features in this image are the central Nb_3Sn superconducting filament with an unreacted Nb core, the surrounding Cu(Sn) matrix and a void adjacent to the filament. The image intensity curve has been adjusted to emphasize the filament. Scale bar is 1 µm.

FIG. 5. Composite BE intensity profile obtained from Figure 4, assembled in four sections as described in the text. The data has been grouped in 10 nm increments and has not been deconvoluted.