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INTERFACE SCIENCE

Grain Boundary Plane Distributions in Modified 316 LN Steel Exposed at Elevated and Cryogenic Temperatures

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Abstract A stereological analysis of electron backscatter diffraction data has been used to measure the five-parameter grain boundary character distribution of chemically modified 316LN stainless steel exposed to both elevated and cryogenic temperature. The results were analyzed to determine if the thermal treatments induced any significant changes in the overall grain boundary character distribution and fractional twin density. The results of this study show that the grain boundary character distribution of this steel is very similar to other FCC polycrystals and not affected by typical thermal treatments used in processing or the cryogenic temperatures employed during service.

Background

316LN-type stainless steels have been heavily utilized as structural materials for high field magnets. The chemically modified 316LN steel examined in this study is currently used as a conduit for Nb_3Sn superconductors in the Superconducting Outsert of the 45T Hybrid Magnet System at the National High Magnetic Field Laboratory. As a conduit, the material is exposed to the same high temperature, long duration reaction heat treatment schedule (700 °C for 100 h) that produces Nb_3Sn superconducting wire within the coil. Furthermore, the conduit material is expected to remain stable throughout the heat treatment as well as maintain high strength and toughness during service at liquid helium (L-He; ~4 K) temperature.

A preliminary study (Downey et al. submitted) was carried out to characterize the microstructural stability of modified 316LN exposed to the Nb_3Sn heat treatment schedule. The work used orientation imaging microscopy (OIM) to illustrate the frequent occurrence of twin boundaries in the microstructure and their contribution to the strength of the material. It was concluded that the fractional density of twin boundaries remained at a limit of about 50% of all grain boundaries even after 100 h heat treatment at 700 °C. In addition, the study showed that about 90% of the twins found were $\Sigma 3$ and coherent in nature. The two-dimensional OIM approach utilized has been found to be valid in assessing the contribution of twin boundaries with a high probability of success [1]. However, since grains are three-dimensional entities, characterizing the grain boundary types (tilt, twist) and coherency of the twin present requires analysis in three dimensions for validation.

It is known that a grain boundary has five independent crystallographic parameters that specify its character. These include three parameters necessary to describe the lattice misorientation and two for the boundary plane orientation. Of the five parameters, four are readily observed from single planar sections. However, to determine the inclination angle between the observation plane and grain boundary plane, serial sectioning was traditionally required. Saylor et al. [2] have developed a stereological

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procedure to determine the distribution of boundary planes from orientation maps on single planar sections. This technique, known as five-parameter analysis, works as an extension to OIM and offers an automated, cost effective approach to the characterization of boundary plane orientations comparable to conventional three-dimensional methods. Since its inception, this method has been utilized in assessing the distribution of grain boundaries in numerous materials including magnesia [3] grain boundary engineered brass [4–7], aluminum [8], nickel and copper (Randle et al. submitted). The present report employs the five-parameter stereology to investigate the distribution of grain boundary plane orientations in the modified 316LN steel exposed to both the elevated and cryogenic temperature. It must be recalled that the hybrid magnet although processed at elevated temperature is operated at cryogenic temperature. By characterizing the conduit material exposed to these extreme temperatures, one is able to assess if the integrity of the material is maintained during heat treatment and service.

Experimental

For this research, rectangular test specimens of the as received (cold rolled and annealed) material were annealed at 700 °C for 50 and 100 h in an argon atmosphere. Samples prepared for 4 K quenching were annealed at 700 °C for 100 h and cooled to room temperature. Due to the extremely low boiling point of L-He, quenching the specimen to 4 K was a two-step process. The first phase involved a quench in liquid nitrogen, which cooled the sample from room temperature to 77 K. Once cooled to 77 K, the specimen was then transferred to the L-He dewar for quenching down to 4 K. The data from these samples provided the microstructural representation of the conduit material during service.

Each sample was cold mounted, and finally polished on a vibro-polisher for 4 h in preparation for OIM/EBSD. The electron backscatter patterns (EBSPs) were examined with TexSEM laboratories (TSL) OIM data collection and analysis software. All planar sections were mapped utilizing a 0.5 μm step size to insure a suitable number of indexed points within each grain given the mean grain diameter (~5 μm) found in the alloy. All resultant scan files were then modified using the grain confidence index (CI) standardization *(grain tolerance angle: 5°, minimum grain size: 2 μm)* and Neighbor CI Correlation *(minimum confidence index: 0.3)* cleanup procedures. After the cleanup procedure, the OIM map should display clearly defined grains while remaining an accurate representation of the originally scanned microstructure, as shown in the comparison of the OIM image quality (IQ) and grain maps of Fig. 1. The IQ map is an accurate representation of the microstructure based on the quality of the indexed backscatter diffraction patterns and not the orientation. The color shading is such that it is bright or dark at points with high or low quality patterns, respectively. For example, the IQ maps are dark at grain boundaries, shear bands, scratches, pits, voids, etc., because the patterns are distorted and therefore difficult to resolve.

Insuring the OIM map is comparable to the as scanned microstructure is important to obtain an accurate account of the grain boundary types present. When extracting reconstructed boundaries, incorrectly indexed points can be taken as individual grains, and these will increase the random fraction of the grain boundary character distribution. Also, too much cleanup may combine dissimilar grains, consuming real boundaries present in the microstructure. Using data processed as described above, the TSL software was used to extract the reconstructed boundary segments using the methods developed by Wright and Larsen [9].

Previous work [2] on the five-parameter technique found that the error associated with the method does not change substantially after 5×10^4 observations, thus a minimum of 50,000 reconstructed boundary segments, or an average of 15,000–20,000 scanned grains, are used for each specimen. For this work, the distribution of grain boundary planes was analyzed for the [100], [110], and [111] misorientation axes. Stereographic projections showing the grain boundary plane distributions at fixed misorientations about the [111] and [110] axes, in multiples of random distribution (MRD) units, are compared in this study.

Results and Discussion

The grain boundary plane distributions that are most relevant to twin boundaries in this work are the Σ3, Σ9, and Σ27 types. The Σ9 and Σ27 arise from intersection of Σ3 grain boundaries, which make up approximately 50 % of all the grain boundaries in the sample. Because the Σ3 grain boundaries encompass such a large fraction of the population, it is expected that the plane distributions, together with those of Σ9 and Σ27, will be the most sensitive to changes in the population.

Grain Boundary Plane Distribution and Characterization of Twin Boundaries

Analysis of results from the exposure to the prescribed elevated and cryogenic temperatures shows that the distribution of boundary planes about the ⟨111⟩ misorientation axis favor pure twist boundaries with [111] normals (see Fig. 2). The distribution of grain boundary planes for misorientations of 60° about ⟨111⟩ has the highest peak in

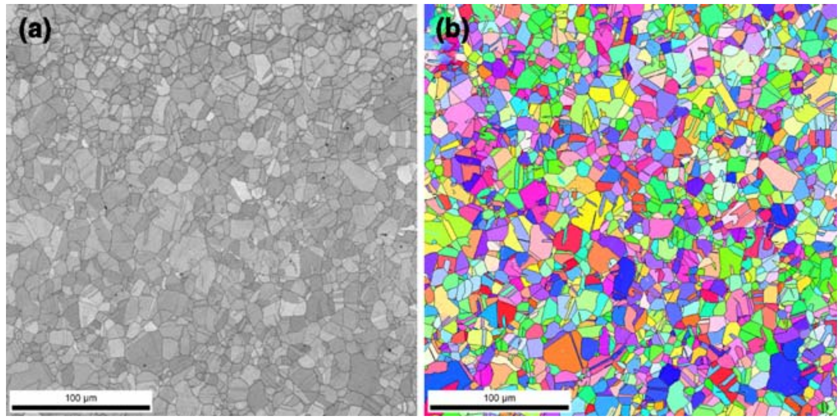

Fig. 1 (**a**) Image quality (IQ) Map and (**b**) OIM grain map of as-received M316LN used for five-parameter analysis

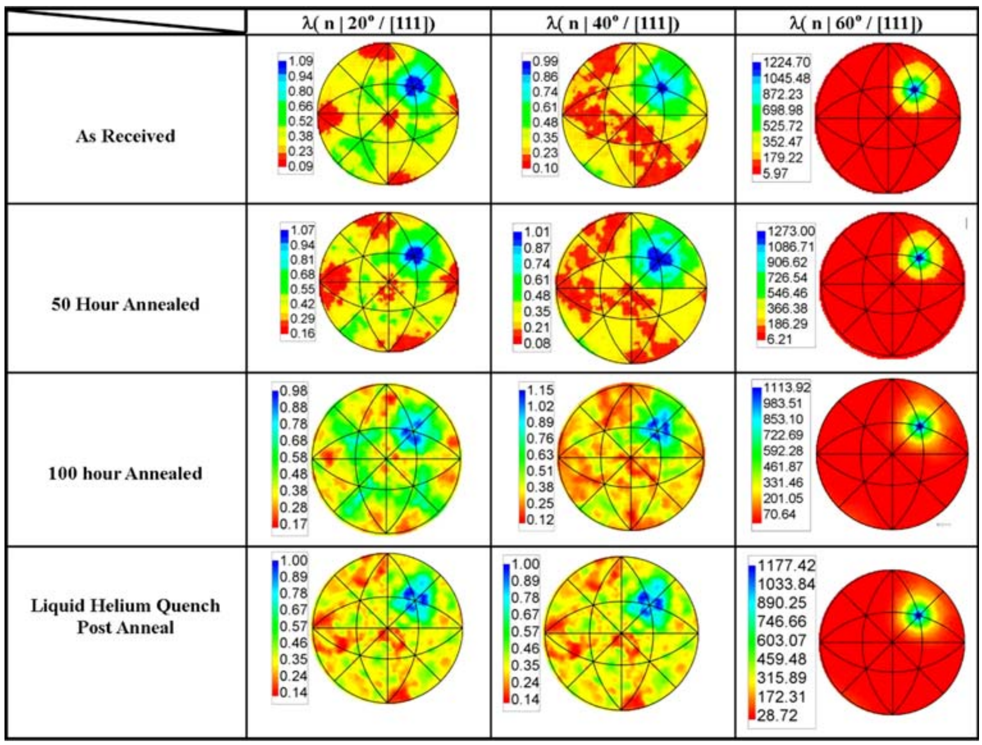

Fig. 2 Distribution of boundary plane normals in M316LN with misorientations corresponding to rotations about [111] of (**a**) 20° (**b**) 40° (**c**) 60°

the entire five-parameter distribution, showing that Σ3 boundaries comprise the majority of boundaries present. Given the high percentage of twin boundaries observed in prior work (Downey et al. submitted), this result is not surprising. The intensity spread around the (111) plane normal position illustrates that some incoherent twins exist although the vast majority are coherent in nature. Although some qualitative differences exist up to 40° around ⟨111⟩, their maximums all coincide with [111] boundary plane normals. However, considering how frequently these grain boundaries occur, these grain boundaries do not make up a significant fraction of the population.

As illustrated in Fig. 3, the distribution of boundary planes at 38.9° and 31.6° misorientations around ⟨111⟩, representing Σ9 and Σ27a boundary types respectively, reveal peaks with normals between [001] and [1–11]. These planes are all in the [110] zone and are, therefore, pure tilt grain boundaries. However, rather than peaking at the orientations of the symmetric tilt, the maxima are spread across asymmetric grain boundary plane orientations. It is of note that the relative intensities of the Σ9 and Σ27a plots at a given measurement point are nearly equal with MRD values ranging from 5 to 6.2 over the thermal treatment period. Observations of the grain boundary distribution at 35.4° about ⟨210⟩, corresponding to the Σ27b coincident site lattice, show plane normals between (–111) and (1–11). However, these grain boundaries do not make up a significant fraction of the population. The intensity

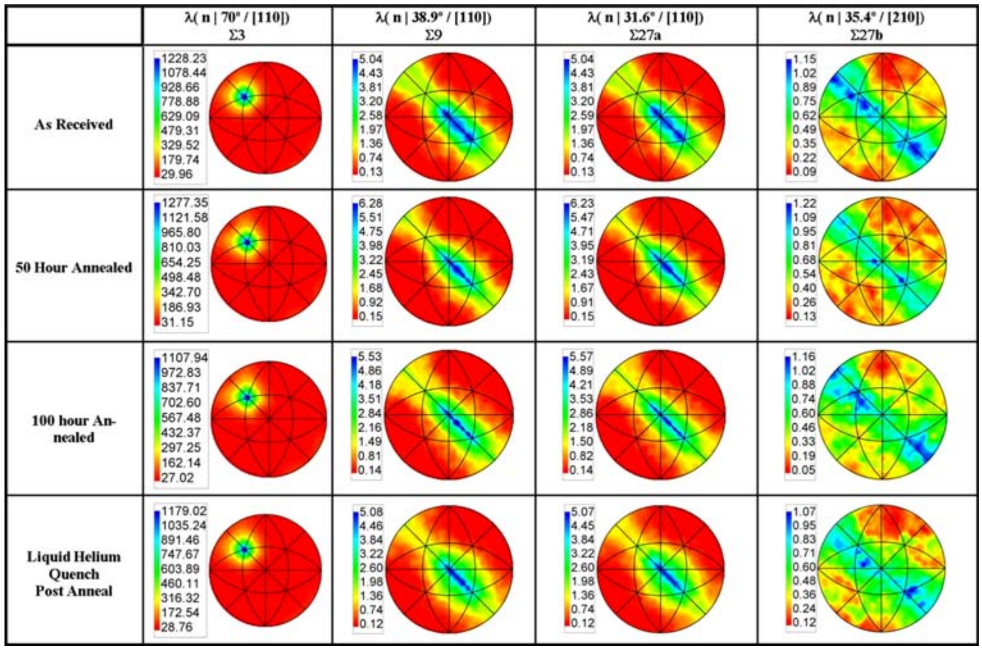

Fig. 3 Distribution of boundary plane normals for misorientations corresponding to Σ3 symmetric tilt, Σ9, Σ27a, and Σ27b

and shape of the Σ9, Σ27a, and Σ27b plots throughout the thermal processing are in line with the presence of a relatively small amount of second and third order twin boundary types present.

It is also interesting to note that plotting the 70° misorientation about ⟨110⟩ reveals a very large distribution of (111)-type boundary plane normals, which is of equal intensity as that of 60° about ⟨111⟩. Schematics of the two Σ3 misorientations are presented in Fig. 4a,b, for 70° about ⟨110⟩ and 60° about ⟨111⟩, respectively. It is obvious from the representation of the crystal structure that the two types of twins are the same boundary viewed from different (perpendicular) directions. Therefore, it is not surprising that the intensities of the two types of twins are similar, except that the distribution is located at 90° apart (see Figs. 2 and 3). The negligible qualitative and quantitative variations in the distributions of boundary planes over the

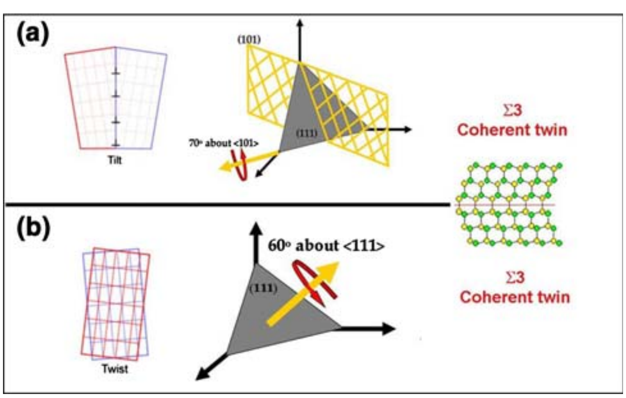

Fig. 4 Schematic of ∑3 (**a**) symmetric tilt and (**b**) twist boundaries

heat treatment and subsequent L-He quench shows that the overall grain boundary character and density of twins is not affected by typical thermal treatments used in processing and service.

Overall, some basic similarities exist in the distribution of grain boundaries found in M316LN as compared to other fcc polycrystals. As found in the stereological analysis of aluminum [8], nickel and copper (Randle et al. submitted), the most common grain boundary planes identified in the distribution are {111}. Also, for ⟨111⟩ misorientations, most boundaries have a twist character while in the case of ⟨111⟩ misorientations, most boundaries are tilt and had asymmetric grain boundary planes.

Comparison of M316LN Microstructure to Random Grain Assemble

Given the fact that precise values of the densities of the various twins were determined in this work, it was necessary to compare the contribution of the twins in this alloy to that of a material with random grain assemble. Earlier work by Mykura [10] used Brandon's criterion [11] to determine the frequency of occurrence of coincident site lattice boundary types in random grain assembles. The results showed that in a random grain assemble, Σ3 (60° about ⟨111⟩) boundaries will contribute only 1.76% of the total boundaries while Σ9 (38.94° about ⟨110⟩) makes up only about 1.02%. Therefore, the ratio of Σ3 to Σ9 in a random grain assemble is 1.73. Table 1 presents the ratios of Σ3–Σ9 for the various conditions that the M316LN alloy was subjected to. An average ratio of about 19 was obtained, which confirms the dominance of Σ3 twin

Table 1 Comparison of the ratio of Σ3–Σ9 twins in a random assemble to that in M316LN exposed to various thermal treatments

Microstructure	$f\ \Sigma3/f\ \Sigma9$
Random grain assemble[9]	1.73
As-received M316LN	20.21
50 h annealed M316LN	18.21
100 h annealed M316LN	19.56
Liquid helium quenched M316LN	19.08

boundary in the microstructure. It is evident that the alloy was nothing close to a random assemble.

Conclusions

The microstructure of modified 316LN has been found to be dominated by twin boundaries. Based on the five-parameter grain boundary distribution it is concluded that the population and coherency of the twins is unaffected by the prescribed thermal processing schedule.

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