Ex-Situ Investigation of the Effects of Heating Rate on the Recrystallization in Rolled Polycrystals of High-Purity Niobium

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MOTIVATION

Performance variability of Nb cavities arises from microstructural defects such as dislocations and grain boundaries. There is significant interest in the effect of trapped flux associated with dislocations [1]. The working hypothesis of this study is that an increased heating rate will allow more dislocations to remain within the material by the time that the heat treatment temperature is reached, thus increasing the recrystallization fraction. This work focuses on assessing multiple metrics for recrystallization based on EBSD data as well as the effect on the geometrically necessary dislocations (GNDs), which will allow the identification of production specifications for improving high purity niobium SRF cavities.

SAMPLE PREPARATION

Excess high-purity niobium, produced by Tokyo Denki, was saved from the production of end-caps for low beta cavities at the FRIB. Sections of the pieces were cold rolled to ~30% reduction parallel to the original rolling direction and then coupon samples were cut using electrical discharge machining (EDM). All samples were then chemically polished using a buffered chemical polishing (BCP) solution to remove 10-15um of material (including the EDM recast surface), and then given a final polish using a vibratory polisher with a colloidal silica suspension to ensure the surface was suitable for electron backscatter diffraction (EBSD). For the final polish a mount was 3D printed using a polymer in order to secure the samples.

HEAT TREATMENTS

Four different heat treatments were chosen for this investigation.

- 3 hour ramp from 25°C - 900°C with a 3 hour hold at 900°C
- 3 hour ramp from 25°C - 1000°C with a 3 hour hold at 1000°C
- Placement in furnace at 810°C, 20 min to 900°C + 3 hour hold
- Placement in furnace at 900°C, 20 min to 1000°C + 3 hour hold

Each heat treatment was followed by a ramp to cool in 1 hour, and samples were retrieved after 10+ hour furnace cooldown.

RESULTS AND DISCUSSION

The density of GNDs is systematically reduced with a faster heating rate and, the percent which falls below the classical annealed metal dislocation density of 10^12/m^2 also follows the recrystallization fraction identified through traditional EBSD methods.

From the as-received condition to the rolling and the subsequent heat treatments it can be seen that the higher temperature heat treatments resulted in more grain growth, but the fraction which is recrystallized is quantified using several methods.

ACKNOWLEDGEMENT

Research supported by DOE/OHEP contract DE-SC0009960.