Scanning Electron Microscopy (SEM) of Twin Bands

INTRODUCTION:
The research goal is to identify twin bands in order to accurately determine grain size. Electron Backscatter Diffraction Patterns (EBSP) are used to obtain crystallographic orientation which can allow for separation of grains and identify twin bands. Using this method in an SEM, nano-sized bands may even be distinguished from a regular grain boundary.

PROCEDURE:
SEM samples must be vacuum compatible, conductive, and resistant to the electron beam. Standard metallographic procedures were used to prepare samples for the SEM:
- Samples were mounted in thermo-set conductive Bakelite
- Mounted samples were ground with successively smaller grit silicon carbide paper
- Ground samples were polished with micron diamond solution
- Polished samples were exposed to the vibratory polisher with a grit of 0.05 microns
- Samples were etched with a concentrated acid solution for fifteen seconds.

- Polishing induces a strain that contorts grain structure.
- Etching eliminates strained structure in order to expose true orientation of grains.

TheFocused Ion Beam (FIB) in the SEM was used to smooth the samples for orientation imaging investigation. The FIB is most effective when initial sample surfaces are level. The FIB uses gallium ions to remove one atom at a time to reveal undisturbed atoms.

Microscopic Orientation Maps
Stainless Steel Conduit for Cable-in-Conduit
High-Field Superconducting Magnet

Inverse Pole Figure Map
indicating crystallographic direction by color (above orientation triangle).

Grain Shade Map indicating unique grains by an angular difference of a minimum of 15 degrees.

Grain Shade Map with representative cubes indicating the crystallographic orientation given by the IPP.

STAINLESS STEEL SUMMARY RESULTS:
- Grain Size Average: 14.8 microns
- Standard Deviation: 8.4

DISCUSSION:
Conduit constrains the wire under the stress of high-field magnets. Therefore the conduit needs to be strong enough to withstand wire expansion. This means the grain sizes have to be within a specific size range to not be brittle (small grains) or soft, pliant (large grains). For our purposes, the grains must be less than 30 microns in diameter on average.

Twinning occurs at low temperatures and high strain rates. Elimination of twins is crystallographically valid because twins are part of the same grain.

The orientation imaging method is more precise than optical microscopy because it is empirical rather than subjective. It can also determine twin width down to the nanometer scale which is not possible using optical microscopy. The smallest twin bands observed were 130 nanometers.

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Charts courtesy of: info.lu.tamingdale.edu/dept
SEM maps created with EDAX/OM software