



Crystalline Microstructure Role in a High Field Q-slope

Alexander Romanenko Cornell University CLASSE



- Background
- Samples used
 - "Hot" and "cold" areas from small and large grain EP and BCP cavities

Outline

- Further baking at 100-120C applied to samples
- EBSD data analysis
 - Local misorientation
 - Calculation of dislocation density from raw data
- Possible cause of the HFQS
- Model for baking mechanism
 - Vacancies-assisted dislocation climb
- FIB/TEM and EELS analysis of cavity samples
 - No oxygen diffusion observed
 - Interface structure similar
- Conclusions



Typical small grain EP cavity test

results



September 24, 2009

Alexander Romanenko



EBSD characterization of samples

EBSD raw data: crystalline orientation map



101



Local misorientation = $1/8 * \Sigma(\Delta \alpha_i)$

001



- Lattice curvature tensor directly measured by EBSD (change of orientation from pixel to pixel)
- From curvature tensor under assumption of no elastic stresses => dislocation density tensor
- More details:
 - [W. Pantleon, Scripta Materialia 58 (2008) 994– 997]
 - [D.P. Field et al., Ultramicroscopy 103 (2005) 33– 39]



Dislocation density and local misorientation – extreme case

Local misorientation



۔

Alexander Romanenko



Hot/cold EBSD summary





- Hot areas on average have misorientations shifted toward higher angles
- Difference hot/cold is largest in the case of large grain BCP, which is consistent with thermometry
- Motivated annealing of the samples at 100-120C and subsequent EBSD analysis



Small/large grain BCP - baking





Small grain EP - baking



- Strong shift due to baking is observed in local misorientation distributions of both hot and cold samples
- Dislocation density maps were obtained to confirm and to get more info on distribution



Average screw dislocation density







Average screw dislocation density







- Significantly reduced dislocation density in small grain EP and large grain BCP samples after 120C baking
 - Consistent with the strong baking benefit on EP and large grain BCP cavities
- Almost no change in the small grain BCP sample due to baking
 - Consistent with the lack of baking effect on the small grain BCP cavities

Baking mechanism – dislocation climb



- Dislocation climb
 - Vacancies are necessary for the climb to occur
 - Vacancies in Nb become mobile around 100C due to Vacancy-H complexes dissociation see next slide for positron annihilation studies [*Phys. Rev. B*, 32(7):4326–4331, 1985]
- Possibly reduction in the dislocation density near the surface due to climb and annihilation of dislocations



Vacancy-H complexes dissociation



• Vac-H complexes in niobium containing some H (Nb2 in plots) dissociate at ~380K (107C) – compare to baking temperatures • Mobile vacancies => dislocation climb becomes possible

[P. Hautojarvi et al., Phys. Rev. B., Vol.35, Num.7, 1985]



- Dislocation tangles, dislocation cell walls might serve as sites for magnetic flux penetration at lower fields due to
 - Lower surface barrier
 - Higher $\kappa => \text{lower H}_{c1}$
- After flux penetrates losses due to oscillatory flux motion



Estimates from Rabinowitz's model

- Assumptions for the fluxoid:
 - Image force negligible
 - Effective mass negligible
- Results
 - Effective surface resistance
 R_{eff}= 4 mOhm
 - Interfluxoid spacing for Q to decrease by a factor of 2 at 100 mT is $d_{flux} = 45$ um – comparable to the size of the "cells" seen in dislocation density maps







- A. Romanenko, J. Mundy, P. Ercius, J. Grazul details eslewhere
- FIB prepared samples from "hot" spot before and after 120C baking
- EELS elemental analysis with atomic scale resolution
- No oxygen-enriched layer
- No oxygen diffussion
- No oxide modification
- No cracks or suboxide clusters very nice and uniform oxide





[nm]





September 24, 2009



- Lattice defects (i.e. dislocations) may be the cause of the HFQS based on:
 - Difference between hot and cold samples
 - Change with 100-120C baking
- Dislocation climb as a possible mechanism
 - Vacations immobilized by H up to ~ 100C
- FIB/TEM + EELS disqualify oxygen diffusionrelated and/or oxide modification baking mechanisms





Thank you!