COMPARATIVE SURFACE STUDIES ON FINE-GRAIN AND SINGLE CRYSTAL NIOBIUM USING XPS, AES, EBSD AND PROFILOMETRY*

A. Romanenko†, H. Padamsee, G. Eremeev, CLASSE, Cornell University, Ithaca, NY 14853, USA
J. Shu, CCMR, Cornell University, Ithaca, NY 14853, USA

Abstract

As the surface magnetic field in niobium cavities approaches the theoretical critical field, RF losses grow sensitive to increasingly subtle features of the material and the surface. A striking example is the familiar onset of the high field Q-slope, where RF losses increase exponentially with field. A surprising feature of the high field Q-slope is its positive response to a mild baking at 100-120 °C. But the Q-slope returns after the first 20 nm of the niobium metal surface is converted to loss-less pentoxide via anodization. The latter result suggests that the cause of the fast growing losses resides in the first 20 nm of the RF surface. Although there are several propositions, the exact mechanism for the high field Q-slope is not yet fully understood and demands further research. We are conducting surface analytic studies with optical profilometry, EBSD, XPS, Auger and SIMS to shed light on the mechanism of the high field Q-slope. We are comparing the behavior of fine-grain niobium with single crystal niobium, buffered chemical polishing (BCP) treatments with electropolishing (EP) treatments and properties before and after 110 °C bake. Our approach is based on identifying lossy regions, dissecting of these regions and range of analysis.

INTRODUCTION

Performance of superconducting niobium cavities at high surface magnetic fields is characterized by the appearance of the high field Q-slope, which is a drastic drop in the cavity quality factor at peak surface magnetic fields higher than approximately 80 mT. Several mechanisms have been suggested as possible explanations of the effect but appear to be either not relevant to the high field behavior or can not explain some of the established experimental facts. Since RF field penetration depth into superconducting niobium is of order 50 nm, cavity surface preparation plays a major role in the observed behavior. Baking at temperatures of 100-120°C for about 48 hours was shown to consistently remove the high field Q-slope in EP cavities and improve it in BCP cavities, with rare cases of complete removal.

In previous surface studies [1, 2, 3] the approach was to prepare samples in the same way as cavities and apply analytical techniques on them. In our work we utilize a different approach, which is:

- Prepare several 1.5 GHz single cell cavities, large or fine grain niobium with BCP or EP

- Test the cavities with the temperature mapping system

- Identify regions, which exhibit high field Q-slope or variations and cut samples from the cavity

- Analyze the samples with various surface analytical technqiues

This method provides us the unique opportunity to directly correlate sample properties with the high field Q-slope behavior. In this work we present results on the first samples cut from BCP small grain cavity, while EP and large grain cavities experiments are planned in the near future. Q-slope characterization of large grain Nb cavities has been started.

PREPARATION

A small (0.5-1 mm) grain niobium cavity was treated with BCP for 100 μm material removal and tested with the temperature mapping system attached. In Fig. 1 the Q_0 vs. E_{peak} curve obtained is shown. The cavity was not baked to preserve the behavior of the the high field Q-slope.
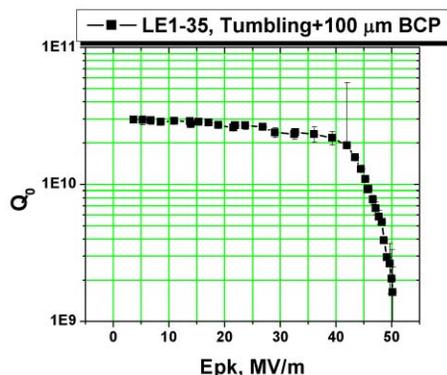

Figure 1: Q_0 vs. E_{peak} curve for BCP-treated cavity used in the experiment.

Fig. 1 shows a high field Q-slope present with the onset field E_{peak} of about 40 MV/m. Temperature map (Fig. 2) was obtained at the highest field of E_{peak} = 50 MV/m, which corresponds to peak surface magnetic field of H_{peak} = 123 mT. Maximum field reached was limited by the available power. An interpolated contour plot of the temperature distribution over the cavity walls was used to identify the regions to cut. The regions with the strongest Q-slope are labeled H1-H10 and with weak Q-slope C1-C10.

Typical temperature vs. field curves for hot and cold regions are shown in Fig. 3. It should be emphasized that

07 Accelerator Technology Main Systems
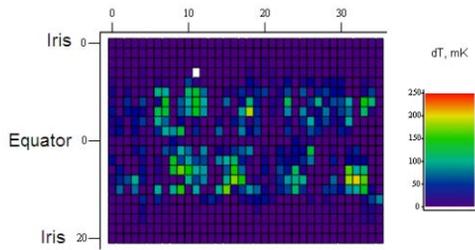
T07 Superconducting RF

Figure 2: Temperature map at H_{peak} = 123 mT.

both strong Q-slope and weak Q-slope regions have a high field Q-slope. In fact *all* thermometers in the high magnetic field region indicate a high field Q-slope present, but the Q-slope is different.
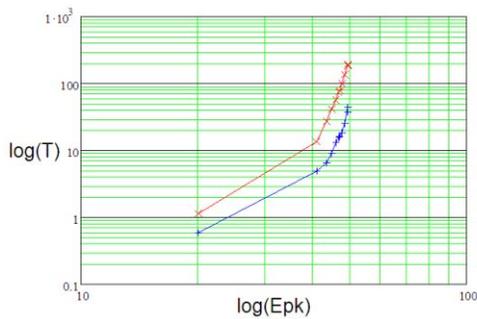

Figure 3: Typical log(T) vs. log(E_{peak}) dependence for hot (red) and cold (blue) regions.

RESULTS

Optical Profilometry

In order to compare the roughness of hot and cold regions an optical profilometer was used. We distinguish between the roughness observed on the micro scale over a region smaller than a grain size and the macro-roughness at the scale of a few grain sizes. In Fig. 4 the typical 3D profiles obtained for a "hot" and a "cold" sample are shown. From statistical analysis of the data obtained it was found that the micro-roughness for both hot and cold samples was of order S_q = 1.5-2 μm.

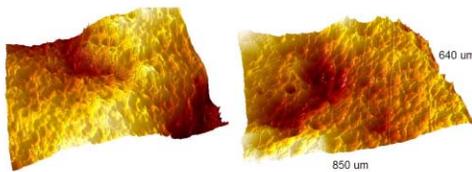Figure 4: Optical profilometer 3-D images (850 μm × 640 μm) of the hot (left) and cold (right) samples.

The number and height of the relatively large steps due to surface irregularities such as grain boundaries is also im-

portant since they result in local magnetic field enhancement as shown in [4]. Several line profiles were taken across a few mm of each sample surface and the histogram of a step height distribution, which is shown in Fig. 5 was constructed. The step height distributions for hot and cold samples look strikingly similar.
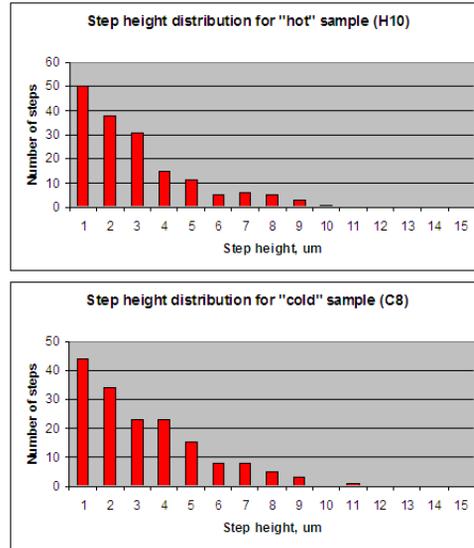

Figure 5: Step height distributions for a "hot" and a "cold" sample.

Electron Back-scattered Diffraction

The EBSD technique was applied on the samples to obtain crystal orientation maps of the surface. The goal was to see if crystal orientation plays any role in the high field Q-slope behavior of niobium.

From the analysis of the data no difference was found between the two types of samples, but further studies on large and single grain material will be able to give more information on the subject.

X-ray Photoelectron Spectroscopy

An SSX-100 system, with an Al Kα source (1486.6 eV) and photoelectron detector at 55-degrees from sample normal, was used for XPS measurements. Electrons emitted from niobium (202 eV) correspond to an information depth of about 7 nm, which includes the niobium/oxide interface.

The main difference, discovered between the hot and cold samples, was that 3 hottest out of 10 hot samples had a nitrogen signal at the level of 3-4 atomic percent present in the photoemission spectra as compared to only one spot with nitrogen signal on one of the cold samples. Fig. 6 shows typical XPS survey spectra of the samples.

Corresponding high resolution spectra around nitrogen peak are shown in Fig. 7. The N 1s peak positioned at 401 eV instead of 399 eV for free nitrogen indicates that

07 Accelerator Technology Main Systems

T07 Superconducting RF

nitrogen is in the chemically bound state, most likely in the NO_3 group [5].
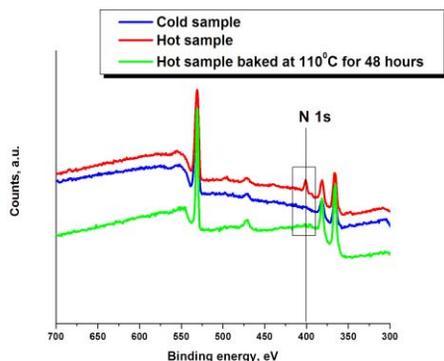

Figure 6: XPS surveys for the hot and cold samples. Hot sample was baked at $110°C$ for 48 hours to see the effect of baking on nitrogen.
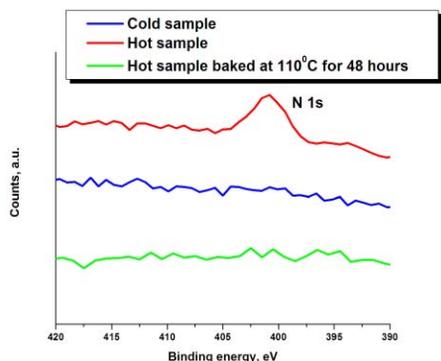

Figure 7: High resolution XPS N 1s peak for hot and cold samples.

The Nb 3d XPS peak, which reveals information about the oxide and oxide/metal interface was found to be almost exactly same for all hot and cold samples analyzed as shown in Fig. 8.
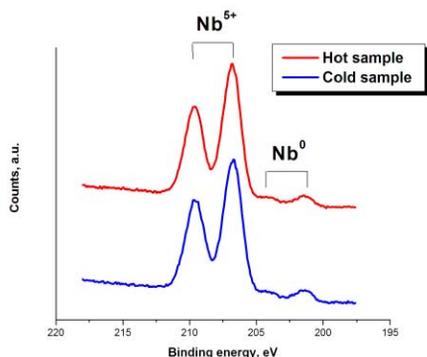

Figure 8: High resolution XPS Nb 3d peak for hot and cold samples.

Auger Electron Spectroscopy

AES was used as a complimentary technique to confirm the presence of nitrogen. Information depth of AES for niobium is about 1 nm thus making it more sensitive to the very surface contamination and inferior to XPS for the investigation of samples. Nevertheless AES was able to detect a higher nitrogen content in the samples where nitrogen was previously found with XPS.

DISCUSSION

One of the main mechanisms for the high field Q-slope suggested recently was related to the possible existence of a "bad" superconducting layer underneath the natural niobium oxide (Nb_2O_5), which was thought to consist of niobium suboxides NbO and NbO_2 or a very high content of interstitial oxygen. In our XPS studies we did not find any difference between the oxide and oxide/niobium interface in hot and cold regions before and after baking. SIMS studies will be performed in order to check that conclusion.

Another theory of the high field Q-slope origin is based on the magnetic field enhancement at surface topographical irregularities (i.e. grain boundaries). Our optical profilometry studies showed that the stronger Q-slope regions do not have higher surface macro- or micro-roughness. However, our studies do *not* exclude the overall role of roughness in the Q-slope, since it is present everywhere.

Correlation found between the presence of nitrogen and the high field Q-slope severity suggests that nitrogen might play a role in the effect.

CONCLUSION

The first attempt to directly correlate results of surface studies with the behavior of niobium at high surface magnetic fields via cutting the cavity proved to be successful. We found N or NO_3 to play a role in the Q-slope. Further experiments based on the same approach will be carried out in the near future on EP, large and single grain cavities.

REFERENCES

[1] Qing Ma et al., "Thermal effect on the oxides on Nb(100) studied by synchrotron-radiation x-ray photoelectron spectroscopy", J. App. Phys 96 (2004) 12.

[2] H. Tian et al., Proc. of the 12th Workshop on RF Superconductivity, 2005, Cornell University, Ithaca, NY, USA.

[3] K. Kowalski et al., Proc. of the 11th Workshop on RF Superconductivity, 2003, DESY, Travemunde, Germany.

[4] J.Knobloch et al., "High field Q slope in superconducting cavities due to magnetic field enhancement at grain boundaries", Proc. of the 9th Workshop on RF Superconductivity, 1999, Santa Fe, USA, pp.77-91.

[5] V. S. Chathapuram et al., "Role of oxidizer in the chemical mechanical planarization of the Ti/TiN barrier layer", Microelectronic Engineering, Volume 65, Issue 4, May 2003, pp. 478-488.