

PROBING HOT SPOT AND COLD SPOT REGIONS OF SRF CAVITIES WITH TUNNELING AND RAMAN SPECTROSCOPIES

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Abstract

Point contact tunneling (PCT) and Raman spectroscopies are presented on high purity Nb samples, including pieces from hot spot and cold spot regions of tested SRF cavities and Nb coupons subject to similar treatment. Near ideal tunneling spectra with sharp coherence peaks and low zero bias conductance were observed on cold spot samples (Fermilab and Jlab), revealing the bulk Nb gap at the surface, with minimal smearing of the density of states (DOS). Hot spot samples exhibit high smearing suggestive of pair breaking along with generally lower superconducting gap. In addition, pronounced zero bias conductance peaks were frequently observed in hot spot regions indicative of spin-flip tunneling from magnetic impurities in the oxide layer. Optical microscopy reveals higher density of surface blemishes on hot spot samples. Raman spectra inside those blemishes show clear difference from surrounding areas, exhibiting enhanced intensity peaks identified as either amorphous carbon, hydrocarbons or the ordered NbC phase. The presence of surface NbC is consistent with TEM studies. Regions with excess C and O have been found by SEM/EDX spectroscopy in the hot spot samples, corroborating the Raman results. Such regions with high concentrations of impurities are expected to suppress the local superconductivity which may explain the observation of reduced gaps and the formation of hot spot regions at high accelerating field. We have found the development of high surface carbon regions to be a general consequence of strain in all Nb samples.

INTRODUCTION

Superconducting radio frequency (SRF) cavities, having Q values orders of magnitude higher than normal Cu are essential for high energy particle accelerators. SRF cavities made from high purity Nb plates involve numerous processing steps including deep drawing, acid etching and annealing. The connection between processing and the surface superconductivity relevant for cavity operations is still not well understood [1]. Analytical techniques that can easily measure the surface superconductivity and material properties are needed. Here we present PCT spectroscopy which probes the surface superconductivity directly and Raman spectroscopy that probes molecular vibrational modes as well as bulk phonons via inelastic scattering.

EXPERIMENTAL

Various Nb samples were investigated including cold spot (C8) and hot spot (H2) samples of a high field Q slope cavity from Fermilab, hot spot (Jlab #3,4,9,10,11, and 12) [2] samples from Jlab and sample A from Jlab which underwent 4 min BCP, 62 micron EP, 600 C annealing for 10 hours and 30 micron EP. A 40% strained Nb foil was also tested.

Tunneling junctions were formed by using a mechanical contact between the Nb sample and a Au tip. Raman spectroscopy was performed using a Renishaw, inVia Raman microscope with a 785 nm laser source, 10s exposure time, 100% laser power (50mW), and a 50X objective lens. The estimated probing depth for Nb is 20 nm. SEM/EDX measurements were performed using Hitachi S-4700 FESEM and EDXS acquisition system.

RESULTS AND DISCUSSION

Hot Spots versus Cold Spots

PCT spectra show clear difference between hot and cold spots. On cold spots we observe near ideal tunneling spectra with sharp coherence peaks and low zero bias conductance. Data fits using Blonder-Tinkham-Klapwijk (BTK) model [3] show gap values expected for bulk Nb

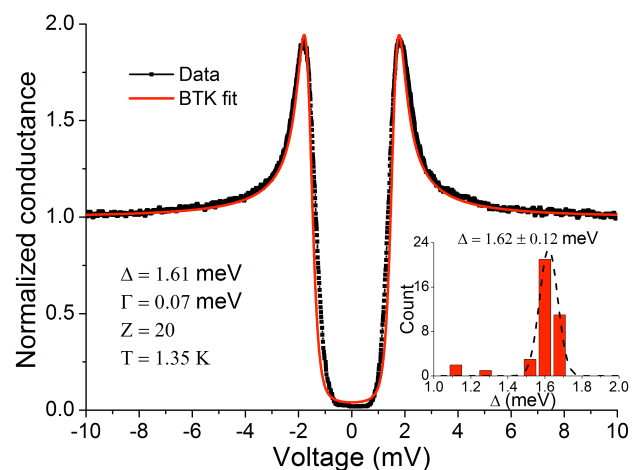


Figure 1: A spectrum taken from Fermilab cold spot C8. BTK fit shows bulk Nb gap and very small smearing. Inset shows the distribution of Δ found in 35 junctions.

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and Dynes Γ values (pairbreaking energy [3]) that are low, less than 5% of Δ . A typical junction and its BTK fit measured on Fermilab cold spot C8 are shown in Fig. 1. Such data reveal a high quality Nb superconducting surface, consistent with the region being a cold spot.

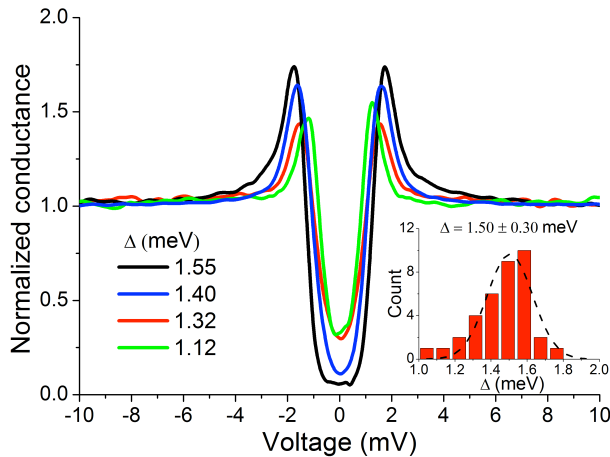


Figure 2: Spectra taken from Fermilab hot spot sample H2. Inset shows the distribution of gap values found in 35 junctions.

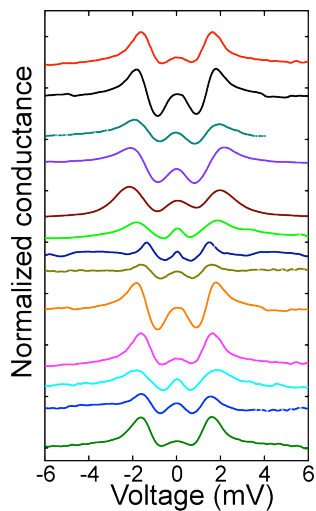


Figure 3: Spectra with pronounced zero bias conductance from Jlab hot spots, indicative of Kondo tunneling.

Hot spots show a higher density of surface blemishes than cold spots under a microscope and PCT measurements reveal suppressed surface superconductivity. Figure 2 shows a collection of junctions measured on Fermilab hot spot H2. BTK fits yield smaller gap values indicative of regions with lower T_c . The inset shows a distribution of Δ to as low as 1 meV. In addition we observed spectra with pronounced zero bias conductance peak as shown in Fig.3. This is evidence of Kondo spin-flip tunneling, indicating magnetic moments in the niobium oxide layer. [4] Such spectra are observed much more frequently on hot spots compared to cold spots.

Chemical Composition of Surface Blemishes

Hot spots are correlated with large density of surface blemishes, including etch pits [2] and other rough patches typically $\sim 10\text{-}20\ \mu\text{m}$ on edge. The chemical composition of those blemishes was studied utilizing Raman and EDX spectroscopy. In addition to hydrocarbon and amorphous carbon reported in Ref. [5], we also observed Raman

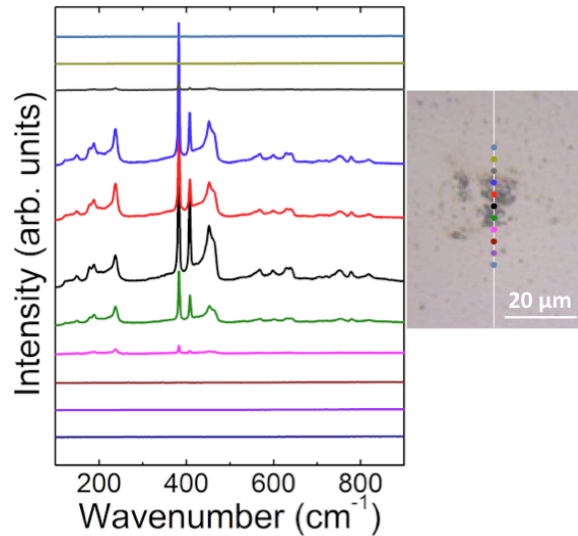


Figure 4: Line scan of a blemish on Jlab sample A showing ordered NbC modes. Color of the spectra corresponds to colored dots on the microscopic image.

spectra that resemble ordered NbC. In Fig. 4 is shown a line scan across a blemish on sample A. Colored dots on the right image indicate the location where the same colored spectra on the left were taken. Regions outside the blemish show low, flat Raman spectra, indicating no impurity complexes. However, inside the blemish the Raman spectra show striking differences, with pronounced peaks. Such spectra were also observed on Jlab hot spots and the strained foil.

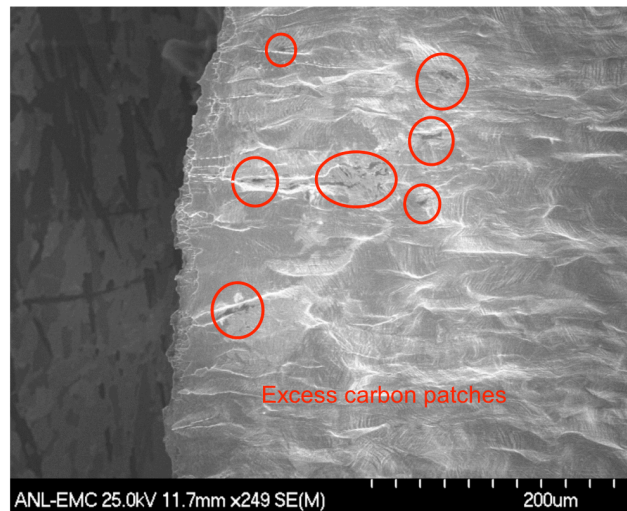


Figure 5: SEM image taken from a 40% strained foil. Red circles indicate regions with excess carbon.

A comparison of a spectrum found on Jlab hot spot and that of NbC_{0.98} reported in Ref. [6] shows strong agreement, indicating that these spectra are from NbC inclusions near the surface. Recent TEM measurements of strained, etched and annealed Nb rods reveal NbC inclusions at the surface. [7]

A high purity Nb foil from Alpha Aesar was strained by 40% to simulate deep drawing done on SRF cavities. SEM image of the strained foil show high density of dark patches indicated by red circles in Fig. 5. EDX mapping on the dark patches reveals excess carbon. Figure 6 shows an example. Such impurities are expected to suppress local superconductivity and increase dissipation. This may explain the formation of hot spots.

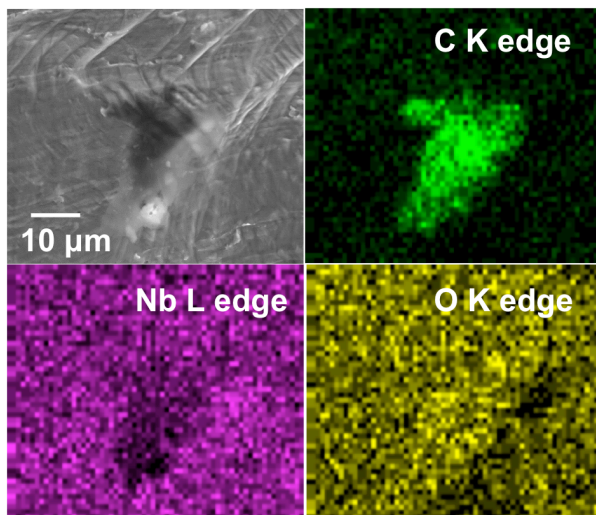


Figure 6: EDX mapping of a blemish on the strained foil shows excess carbon.

CONCLUSIONS

PCT spectroscopy can be used as a predictive tool of the performance of SRF cavities. Cold spots show near ideal spectra, close to that of bulk Nb, and hot spots show spread of gap values and increased broadening. Hot spots also display Kondo tunneling indicative of magnetic moments in the oxide layer. Surface blemishes are observed on both cold and hot spots, however the areal density of such blemishes is much higher on hot spot regions. Raman spectroscopy reveals that these blemishes are excess C regions in various forms, including hydrocarbons, amorphous carbon [5] and ordered NbC. SEM and EDX spectra confirm the presence of excess carbon. Regions with high concentration of carbon are also indicating the presence of other impurities such as hydrogen (via hydrocarbon formation) and oxygen. The correlation of these excess C regions with the observations of magnetic moments in the oxide is not yet understood. The hot spot regions are revealing an array of complex defects, such as impurities, inclusions and magnetism, each of which may be playing a role as a cause of dissipation (hot spots) at high accelerating field.

ACKNOWLEDGMENTS

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REFERENCES

- [1] H. Padamsee, RF Superconductivity vol II Science, Technology and Applications (Weinheim: Wiley-VCH, 2009).
- [2] X. Zhao, G. Ciovati, and T. R. Bieler, Phys. Rev. STAB 13, 124701 (2010).
- [3] T. Proslir, J. F. Zasadzinski, L. Cooley et al, Appl. Phys. Lett. 92, 212505 (2008).
- [4] T. Proslir, M. Kharitonov, M. Pellin, J. Zasadzinski, G. Ciovati, IEEE Trans. Appl. Sup. 21, 2619 (2011)
- [5] C. Cao, D. Ford, S. Bishnoi et. al, Phys. Rev. STAB 16, 064701 (2013).
- [6] H. Wipf, M. V. Klein, and W. S. Williams, Phys. Stat. Sol. (b) 108, 489 (1981).
- [7] Runzhe Tao et al, unpublished.