

# CORNELL SAMPLE HOST CAVITY: RECENT RESULTS \*

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## Abstract

The Cornell sample host cavity is a 3.9 GHz testing system for RF analysis of novel superconducting surfaces. The cavity applies fields up to 100 mT on a removable and replaceable 5-inch sample plate in order to measure the surface resistance of the material under investigation. The cavity also includes a temperature-mapping system for localization of quench events and surface defects. In this paper, we present recent experimental results from the host cavity of niobium deposited onto molybdenum and copper substrates using chemical vapor deposition, in collaboration with industry partner Ultramet. The results indicate low BCS resistance and good adhesion but also areas of high residual resistance due to chemical and morphological defects.

## INTRODUCTION

When developing new materials and surfaces for superconducting radio-frequency (SRF) accelerator physics, it is useful to study their behavior under RF power early in their development, often before it is possible or economical to produce an entire cavity with the material. At Cornell, our sample host cavity [1–7] serves to bridge this gap, offering a test bed for novel materials with a simplified geometry that allows for early and rapid investigations of material performance. The cavity features a removable 5-inch disk, the “sample plate”, that can be replaced with a sample of the material under investigation. We perform traditional “RF-off” measurements of the intrinsic quality factor  $Q_0$  and use calibration measurements to extract the surface resistance of the sample plate.

We also equip the cavity with a temperature-mapping system on the back side of the plate in order to search for areas of localized heating. These areas indicate regions on the sample plate with higher surface resistance, suggesting the presence of defects on the surface. These defects can be morphological or chemical in nature, creating higher losses due to decreased superconductivity or field enhancement.

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Figure 1: The Cornell sample host cavity, shown here installed on the test insert with the temperature-mapping system mounted on the sample plate, laying horizontally on the top of the cavity.

Figure 1 depicts the sample host cavity mounted for RF testing and equipped with the temperature-mapping system.

In our work published here, we have collaborated with industry partner Ultramet, a research and development company focusing on chemical vapor deposition (CVD) methods, to investigate niobium surfaces deposited on molybdenum and copper substrates using CVD. CVD niobium on copper is a promising SRF technology which offers significantly reduced manufacturing costs (due to lower Nb content) and better thermal performance (due to the increased thermal conductivity of the copper substrate). The niobium films considered in this work had nominal thicknesses of 150-200  $\mu\text{m}$ ; furthermore, the robust CVD coatings show exceptional adhesion and as such are compatible with centrifugal barrel polishing.

Our work at Cornell includes measurements of the surface resistance of many of these samples, temperature mapping results for those samples, and some additional microscopy analysis of sample coupons cut from high-loss regions on the sample plates (as revealed by the temperature-mapping system).

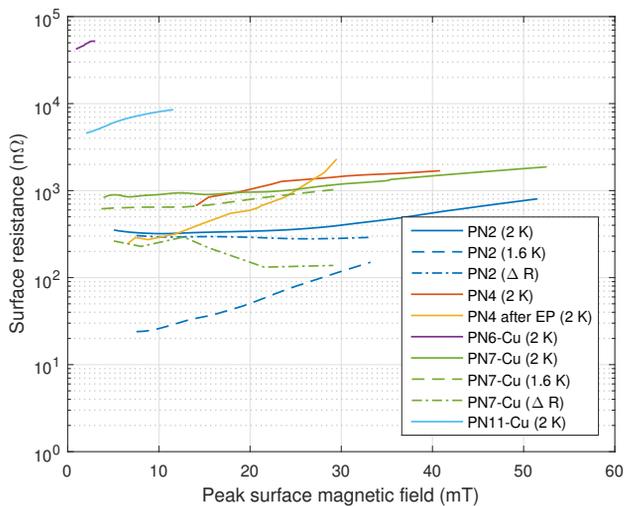


Figure 2: Surface resistance measurements at 2 K and 1.6 K for the CVD plates tested.  $\Delta R$  is the difference in resistance between the two temperatures.

### SURFACE RESISTANCE MEASUREMENTS

As mentioned above, we performed measurements of the surface resistance of many samples prepared with CVD, followed by electrochemical and/or mechanical polishing. The samples and their preparations are summarized in Table 1. The results of the RF tests are summarized in Fig. 2.

In general, the samples exhibited temperature-dependent BCS resistances of 100-200 n $\Omega$  at 2 K, near the expected range for niobium at 3.9 GHz. Their residual resistances were higher, ranging from  $\sim$  200 n $\Omega$  up to  $>$  10<sup>4</sup> n $\Omega$  varying from plate to plate, and dominating the total resistance for most of the samples. These results indicated that the niobium on the surface is of high quality but that there were numerous surface defects, either morphological (*i.e.* rough and bumpy regions) or chemical (*i.e.* regions of non-niobium inclusions).

In addition, several samples were tested multiple times with additional electrochemical polishing between tests. In general, this extra polishing improved the residual resistance, though the extent of the improvement was limited.

### THERMOMETRY

During the RF tests described above, we used the sample host cavity's thermometry system to map areas of localized heating on the sample plate, which would indicate areas of interest on the surface. The performance of the plates was qualitatively similar for all preparations; the plates showed some general heating everywhere, indicating a high baseline residual resistance on the plate, with several "hot spots" of increased heating, indicating more serious localized defects. Figure 3 shows a representative example of these temperature maps. Here, the strong heating in the WNW area of the plate suggests the presence of a significant defect at that location.

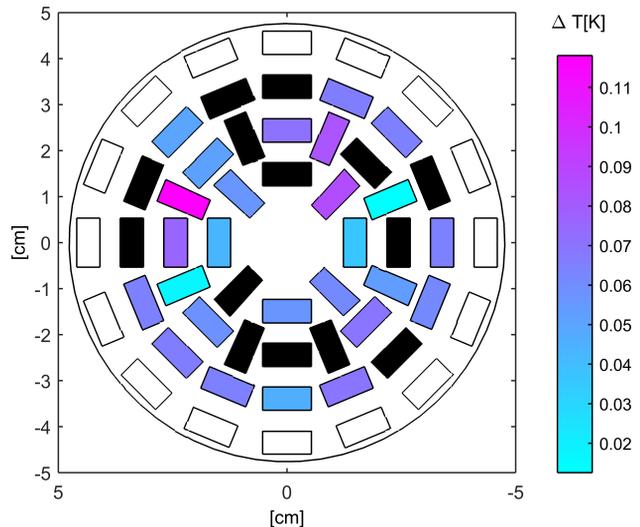


Figure 3: Temperature map for sample PN7-Cu at a peak surface field of 52 mT, with strong heating in the WNW area of the plate and some milder heating in the NNE area.

Much like the surface resistance measurements above and the microscopy results to be discussed in the next section, these findings indicate the presence of surface defects, either chemical or morphological, which create local areas of strong heating due to higher surface resistance.

### MICROSCOPY

After isolating areas of interest on the plates using the temperature-mapping system, we performed surface analysis using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX) on coupons cut from the sample plates. The analysis revealed both morphological and chemical defects on the surface, either of which could have contributed to the high resistance and localized heating measured during the RF tests.

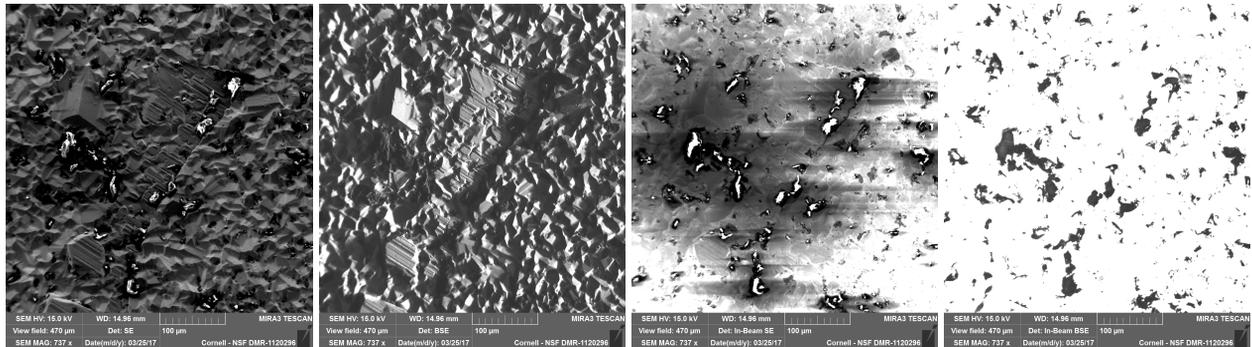
The images in Fig. 4 show a selection of SEM images used in this analysis, from sample PN11-Cu. These images are representative of defects found in areas of high heating on the plates.

EDX analysis of these defects showed that the morphological defects did not correspond with chemical impurities, but that there were numerous chemical impurities located across the surfaces. For the sample pictured, Fig. 4d highlights areas of high-purity niobium (white) and areas of chemical defects (black). These chemical defects showed high concentrations of carbon and oxygen, as well as trace amounts of several alkalis and alkaline earth metals. Future CVD niobium surfaces will need a decreased number of these impurities on the RF surface. However, our belief is that these chemical defects are created by CVD reactor condensates forming on overhanging cold surfaces inside the reactor and then falling onto the flat and upward-facing sample plate; in the case of coating full cavities, no cold overhanging surfaces will be present during the CVD operation effectively

Table 1: Summary of CVD Niobium Samples Tested

Sample	Substrate	Treatment
PN2	Molybdenum	High-temperature CVD, mechanical polishing
PN4	Molybdenum	Low-temperature CVD, 5 $\mu\text{m}$ EP <sup>1</sup> for second test
PN6-Cu	Copper	Low-temperature CVD, mechanical polishing, 5 $\mu\text{m}$ EP, addl. 11 $\mu\text{m}$ EP after first test
PN7-Cu	Copper	Nb diffusion coating, Low-temperature CVD, 5 $\mu\text{m}$ EP, addl. 5 $\mu\text{m}$ EP after first test
PN11-Cu	Copper	Low-temperature CVD, mechanical polishing, 5 $\mu\text{m}$ EP
PN4-Cu	Copper	Nb diffusion coating, Low-temperature CVD, 1.7 $\mu\text{m}$ EP (Nb coating removed)
PN8-Cu	Copper	Nb diffusion coating, Low-temperature CVD, 3 $\mu\text{m}$ EP (Nb completely removed)

<sup>1</sup> Electropolishing.



(a) Secondary electron image.

(b) Back-scattered electron image.

(c) In-beam secondary electron image.

(d) In-beam back-scattered electron image.

Figure 4: Scanning electron microscope images of samples cut from plate PN11-Cu. In-beam images highlight chemical defects, while off-beam images highlight morphological defects.

eliminating the risk of this type of cold-condensate surface contamination.

## CONCLUSIONS

CVD niobium is a novel SRF surface that offers some exciting advantages over traditional bulk niobium, including reduced manufacturing costs and better thermal performance. In collaboration with Ultramet, we have studied a number of samples of CVD niobium for use as an SRF material, using Cornell's 3.9 GHz sample host cavity. We measured the surface resistance of these samples and determined that they exhibited high residual resistance in the range of 200 n $\Omega$  to > 10<sup>4</sup> n $\Omega$ , but BCS resistance near the 100-200 n $\Omega$  expected for niobium at this frequency. Thermometry mapping results revealed that the plates showed general heating with several "hot spots", areas of strong localized heating. Further SEM/EDX analysis of coupons cut to investigate these hot spots indicated morphological and chemical defects that may have caused the heating and poor residual resistance. Future work will need to improve deposition and polishing techniques to reduce the presence of the morphological defects. However, we believe that the difference in reactor geometry between coating the flat plates (the current phase of research) and coating cavities (the next phase) will eliminate the issue of chemical impurities, which are suspected to arise due to condensation on cold reactor

surfaces. In all, these are exciting early results for CVD niobium in SRF which have illuminated a path for improvement moving forward.

## REFERENCES

- [1] Yi Xie and Matthias Liepe, "TE Sample Host Cavities Development at Cornell", in *Proc. SRF 2011*, Chicago, Illinois, July 2011.
- [2] Yi Xie and Matthias Liepe, "Coupler Design for a Sample Host TE Cavity", in *Proc. SRF 2011*, Chicago, Illinois, July 2011.
- [3] Yi Xie, "Development of Superconducting RF Sample Host Cavities and Study of Pit-Induced Cavity Quench", PhD thesis, Cornell University, 2013.
- [4] D.L. Hall *et al.*, "Development and Performance of a High Field TE-Mode Sample Host Cavity", in *Proc. SRF 2013*, Paris, September 2013.
- [5] D.L. Hall, M. Liepe, D.A. Gonnella and I.S. Madjarov, "SRF Material Performance Studies using a Sample Host Cavity", in *Proc. IPAC 2014*, Dresden, Germany, June 2014.
- [6] J.T. Maniscalco *et al.*, "Recent Results from the Cornell Sample Host Cavity", in *Proc. SRF 2015*, Whistler, BC, Canada, September 2015.
- [7] J.T. Maniscalco *et al.*, "New Material Studies in the Cornell Sample Host Cavity", in *Proc. IPAC 2016*, Busan, Korea, May 2016.