# RF SURFACE IMPEDANCE MEASUREMENT OF POLYCRYSTALLINE AND LARGE GRAIN NB DISK SAMPLE AT 7.5 GHz \*

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## Abstract

A Surface Impedance Characterization (SIC) system has been proposed at the 2005 SRF workshop [1] and its commissioning was detailed at the 2009 PAC conference [2]. Currently, the SIC system can make direct calorimetric surface impedance measurements on 50 mm samples in the temperature range from 2–20K exposed to a 7.5 GHz RF magnetic flux density of less than 3 mT.

We report on new results of a BCP etched large grain Nb sample measured with this system as compared with previous results of a BCP etched polycrystalline Nb sample. The design of an upgraded SIC system for use at higher magnetic flux densities is on the way to more efficiently investigate correlations between local material characteristics and associated SRF properties, both for preparation studies of bulk niobium and also new thin film SRF developments.

### **DESCRIPTION OF APPARATUS**

The SIC system is designed to measure the SRF properties of samples small enough to be accommodated in commercial surface characterization instruments, surface treatment facilities and laboratory-based thin film deposition equipment.

### RF Portion of SIC System

A sample is mounted on a holder located at the open end of a  $TE_{011}$  cylindrical niobium cavity, shown in Figure 1. A sapphire rod is inserted into the cavity to lower the resonant frequency of this size cavity to 7.5 GHz. Two adjustable couplers are located above the cavity. The sample is thermally isolated from the cavity and there is a gap between the cavity and the sample. Two rf choke joints are used at the bottom of the cavity to minimize the rf power leaking out of the cavity from the gap. This system provides controlled rf fields onto ~0.7 cm<sup>2</sup> area on the 5 cm diameter sample.

### Thermometer Portion of SIC System

The sample is thermally bonded to its holder. Heat generated or applied on the sample can be conducted away to the bath only via a stainless steel insulator, a copper ring, followed by another stainless steel insulator. The sample temperature is feedback-controlled by a heater and thermo sensors mounted on the back of sample holder. The cavity interior and calorimeter are evacuated. The cavity is otherwise typically immersed in 2 K liquid helium during normal operation. This configuration is also convenient for future investigation of higher- $T_c$  materials.

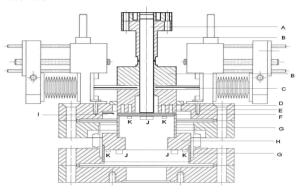


Figure 1: SIC system overview (Designed by J. Delayen, H. L. Phillips, H. Wang and B. Xiao) A. Sapphire rod, B. Nb, C. Coupler, D.  $TE_{011}$  cavity, E. Choke joint, F. Nb sample on copper plate, G. Stainless steel thermal insulator, H. Copper ring, I. Pickup coupler near the choke joint to monitor the rf leaking from the open gap, J. Heater, K. Thermal sensor.

### **EXPERIMENT**

#### **Parameters**

The surface impedance can be calculated from formula (1):

$$Z_s = \frac{P_{rf}}{kH_{pk}^2} + i\omega\mu_0(\lambda_{ref} + \frac{f - f_{ref}}{M})$$
(1)

The real part is the surface resistance and imaginary part is the surface reactance.  $P_{rf}$  is the rf induced heat, kand M are geometry dependent coefficients and  $\omega$  is the resonant circular frequency. The RF induced heat is calculated from the difference between the power from the heater required to keep a constant sample temperature without rf fields in the cavity and the power from the heater required to keep the sample's equilibrium temperature unchanged when rf fields are present, the so called calorimetric technique [3-6]. Formula (2) is used to derive the surface resistance:

$$P_{rf} = \frac{1}{2} \int R_s H^2(S) dS$$
 (2)

H(S) is the magnetic field distribution on the sample.  $k = \frac{1}{2} \int H^2(S) dS / H_{gk}^2$  is geometry dependent, so

$$P_{rf} = kR_s H_{pk}^{2}$$
(3)

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 $H_{pk}$  can be obtained from the stored energy since  $H_{pk}/\sqrt{U}$  is also geometry dependent. The parameters k and  $H_{pk}/\sqrt{U}$  are derived from the RF field simulation using code MWS with a 0.2 mm gap between the cavity and the sample surface. Result is given in Table 1. A closed gap analytical calculation using code MathCAD gives a similar result on k and different  $H_{pk}/\sqrt{U}$ , which is close to a closed gap simulation using code MWS. One can expect with a closed gap, the stored energy shouldn't change much and the peak flux density on the sample should be bigger comparing that with 0.2 mm gap, thus a bigger  $H_{pk}/\sqrt{U}$  is expected.

Table 1: Key Parameters to Derive Surface Impedance

Simulation	Tuning Sensitivity [Hz/nm]	$k\left[\frac{W}{\Omega T^2}\right]$	$\frac{H_{pk}}{\sqrt{U}} \left[ \frac{T}{\sqrt{J}} \right]$
Closed gap MAFIA	-30		
Closed gap MathCAD		3.70×10 <sup>7</sup>	0.530
Closed gap MWS			0.503
0.2 mm gap MWS	-30	3.62×10 <sup>7</sup>	0.336
7.51	-3	4Hz/nm, measured a	ł 4K

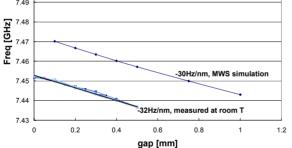


Figure 2: Calculated and measured tuning sensitivity of the  $TE_{011}$  mode with a 0.2 mm gap.

The change of surface reactance is proportional to the change of penetration depth. It can be derived from changes of the resonant frequency of the TE<sub>011</sub> mode versus sample temperature. Simulation result of the coefficient *M* with 0.2 mm gap is listed in Table 1 using code MWS. A closed gap simulation using code MAFIA has also been done to cross check with 0.2 mm gap MWS simulation. The resonant frequency of the TE<sub>011</sub> mode of the cavity can be tuned by adjusting the gap, the ratio of the frequency and gap length is the mechanical tuning sensitivity. The coefficient *M* is mathematically equal to the tuning sensitivity. The tuning sensitivity has been measured at room temperature and 4K, resulting in - 32 Hz/nm and -34 Hz/nm respectively as shown in Figure 2.

# Sample Preparation

The SIC system relies on a good thermal contact between the sample and sample holder. The present SIC calorimeter system uses a Cu sample holder which thermally bonds well to a Cu substrate. We have tested two different Nb samples, i.e. a 0.2 mm thick polycrystalline and a large grain Nb coupon brazed onto a 2 mm thick Cu piece and then chemically or electrically treated. Each of the Nb-on-Cu sample was mounted onto the Cu sample holder using Ga:In:Sn 1:1:1 in volume as the thermo-bonder. To verify the achieved thermal bond required for a precise calorimetric measurement, he temperature difference between the sample surface and the back of the sample holder (where the temperature sensor is located, see Figure 1) has been measured under thermal equilibrium. The result shows a less than 1% temperature difference below 7 K and a less than 2% temperature difference below 10 K.

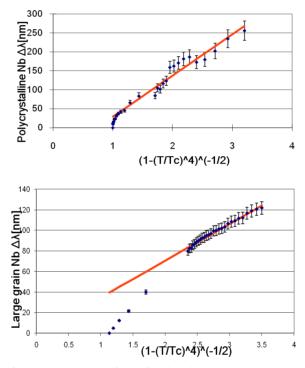


Figure 3: Penetration depth change versus sample temperature for polycrystalline and large grain Nb brazed on Cu substrate, red line shows the two-fluid model fit.

### Transition Temperature

A vector network analyzer was used to measure the sample temperature dependence on the resonance frequency of the cavity, as well as its loaded Q. A transition temperature of  $9.25\pm0.05$  K has been measured for polycrystalline Nb, and  $9.26\pm0.01$  K for large grain Nb.

### Surface Reactance

From the measurement of the sample temperature dependence on the resonance frequency of the cavity, one can derive the change of surface reactance versus temperature.

Figure 3 shows the penetration depth change with sample temperature (blue diamonds) in comparison to an analytical estimate based on the two-fluid model (red line) with  $\lambda(0) = 110$  nm for polycrystalline Nb and  $\lambda(0) = 36$ nm for large grain Nb [7]. One can derive surface reactance change with sample temperature from the penetration depth change with sample temperature using equation (1).

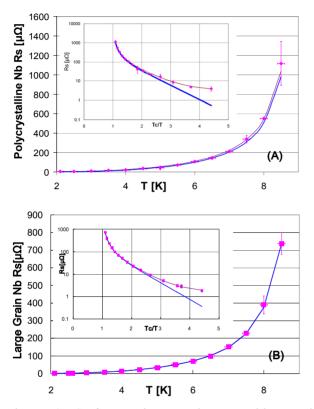


Figure 4: Surface resistance changes with sample temperature for polycrystalline (A) and large grain (B) Nb brazed on Cu substrate. • Measured Rs, — Theoretical value with (A)  $\Delta/kT_c = 1.85$ ,  $T_c = 9.25$  K, London penetration depth = 43.8 nm, Coherence length = 22.4 nm, Mean free path = 99.3 nm and residual resistance = 3.27  $\mu\Omega$  for polycrystalline Nb and (B)  $\Delta/kT_c = 1.87$ ,  $T_c = 9.26$  K, London penetration depth = 39.8 nm, Coherence length = 39.8 nm, Coherence length = 89.7 nm, Mean free path = 826.4 nm and residual resistance.

### Surface Resistance

The surface resistance of these polycrystalline and large grain Nb samples have been measured using the method described above. The corresponding results are shown in Figure 4. The surface resistance measured here includes the BCS resistance and residual resistance. The least-square multi-parameter fits [8] with  $T_c$  fixed at 9.25 K for polycrystalline Nb and at 9.26 K for large grain Nb, and the BCS resistance portion of this fit are also shown in Figure 4.

One expects the surface resistance at 7.5 GHz of typical bulk niobium to be dominated by BCS to temperatures as low as 2 K. The fit for this data suggests a residual resistance as high as 3.27  $\mu\Omega$  for polycrystalline Nb and 1.54  $\mu\Omega$  for large grain Nb, higher than expected. This high residual resistance may come from the deformation and contamination during the brazing procedure.

### PATHS TO HIGHER FIELD

Currently the SIC system uses stainless steel as the thermal insulator in the calorimeter, (see Figure 1). The high thermal impedance of stainless steel constrains the stable heat load and thus limits the magnitude of magnetic fields supportable in CW measurements. A secondgeneration thermal system of the apparatus employing copper with good thermal conductivity as a thermal insulator is under fabrication. A pulse mode operation with a VCO control system has also been developed to overcome the limitation of overheating in CW mode of the present thermal system. 12 mT peak magnetic field has been achieved in a preliminary test using pulse mode.

### SUMMARY

RF surface impedance measurements have been completed at flux densities < 3 mT under different sample temperature conditions for two polycrystalline and large grain Nb samples brazed on Cu with the current SIC system. Least-square multi-parameter fits have been performed for polycrystalline and large grain Nb surface resistance. Large grain Nb has smaller  $\lambda(0)$ , larger coherence length and a significantly large mean free path comparing with polycrystalline Nb. 12mT peak magnetic field has been achieved in a preliminary pulse mode test. A second-generation calorimeter will be ready soon to achieve 20 mT fields in CW mode. It is anticipated that use of the SIC system will enable valuable and efficient correlation of local material characteristics with associated SRF properties, both for preparation studies of bulk niobium and also new thin film SRF developments.

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