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RESULTS ON BULK NIOBIUM SURFACE RESISTANCE MEASUREMENT WITH PILLBOX CAVITY ON TE011/TE012 MODES

G. Martinet, Institut de Physique Nucléaire d'Orsay, CNRS/IN2P3, Université Paris-Sud, France
Université Paris-Saclay, 91406 Orsay, France

Abstract

Surface measurement of superconducting sample is required to characterize processes of bulk niobium preparation for SRF resonators. In order to reduce characterization cost and improve measurement performances, a pill-box cavity has been developed at IPN Orsay. Using TE011 and TE012 modes, we describe the latest results based on calorimetric method.

INTRODUCTION

Nowadays a major effort on research and development on superconducting materials are pushed forward for SRF accelerating technologies development. It includes surface preparation of bulk niobium (chemical and mechanical treatment, baking, infusion,), alternative superconducting matérials on copper and/or on niobium (NbN, Nb3Sn, MgB2, …), or alternative processes and structure as multilayer on bulk niobium. Because the amount of characterization, it is less expensive and more affordable to use samples instead of accelerating cavities. Several apparatus already exist with their own specificities. One can cite CERN [1] and HZB [2] QPR resonators, Cornell devices [3] and Jefferson Laboratory cavity [4]. Both take the benefit of calorimetric method measurement which allows accessing directly the power dissipated on the sample with high accuracy. For this purpose, we have developed a pill-box cavity [5] based on previous instrument [6, 7]. We present here last results on bulk niobium sample with the apparatus which has been developed in our laboratory.

EXPERIMENTAL DEVICE

The experimental device is a modified pill-box cavity which is working on TE011 and TE012 modes. The top of the cavity has been modified with a curved shape in order push away the degenerated modes TM111 and TM112. This modification does not change the magnetic field map and the sample surface. Figure 1 shows a cutting view of the device. On the top is the RF cavity which is made with bulk niobium of 4 mm thick. A Nb/Ti flange with a Ra 0.1 surface state allows clamping the sample with the thermometric chamber. Indium gasket is used on both sides of the sample to seal the cavity vacuum and chamber vacuum from helium bath. Three pipes with 12 mm inner diameter on the top of the cavity allow pumping and RF coupling (Input and transmitted coupling). The stainless steel chamber is closed with standard CF-100 flange with five 16 mm pipes. One is used for dedicated pumping, the four others are closed with CF-16 glass-metal feedthrough (Deloy) to connect thermometers to the acquisition system at room temperature. The thermometric chamber contains 30 Allen Bradley sensors which are encapsulated in copper part. 3 Sectors are disposed at 120° which are composed of 10 sensors: 2 paired sensors are disposed to measure thermal gradient near the border, and 6 other along the radius with a step of 7 mm (12.4 mm – 47.4 mm from the center). The thermal contact of thermometers on sample is made with Apiezon grease. Bronze-beryllium springs allow a contact force in the range of 1 DaN. The pumping system dedicated to the cavity is composed of membrane dry pump, turbomolecular pump and ionic pump whereas the system dedicated to measurement cell is composed of dry scroll pump and turbomolecular pump. All the apparatus is cooled in liquid helium bath and the sample is cooled down by the flange all around the disk as showed on Figure 1. A static heater is fixed on the center of the sample with Stycast epoxy to perform calibration of the temperature variation.

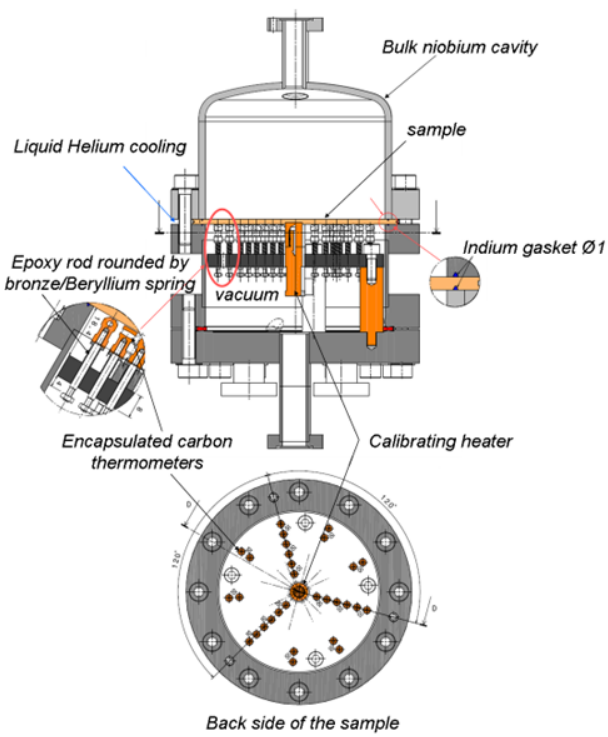

Figure 1: Pill-box cavity equipped with sample and calorimetric chamber. A bottom view shows the disposition of the 30 thermometers on the backside of the sample.

The cavity with all instrumentation is then fixed on the insert of the cryostat of 350 mm diameter as shown on Figure 2. The liquid helium volume capacity is roughly 80 L. The static losses on the bath are in the range 1 W to 2 W depending on the level of liquid helium which allows

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measurement during 12 hours without refilling. A movable coupler has been developed to adapt critical coupling with surface resistance variation of the cavity and the sample. There, a wide range of surface resistance measurement can be explored. Typically, a loaded Q of 10^6 up to 10^9 can be achieved.

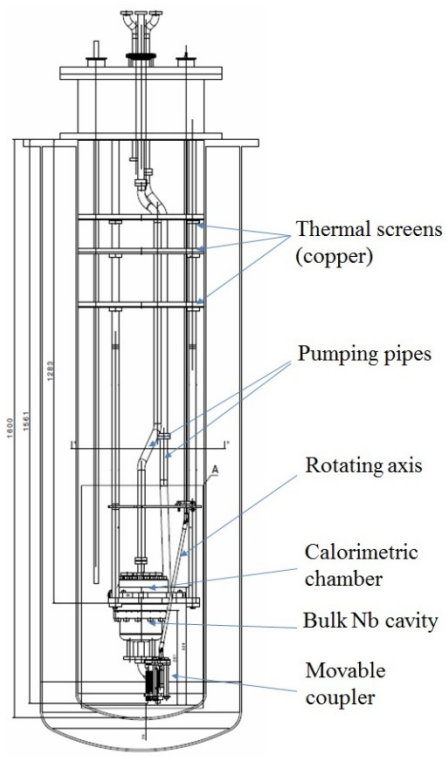

Figure 2: Drawing of the cavity mounted inside the cryostat with movable coupler.

MEASUREMENT METHOD

The principle of the method has been described previously [5, 7]. The Helium bath temperature is fixed by controlling the pressure by the way of roots pump on the cryostat and a MKS regulated butterfly valve. This fixes the temperature of the entire system. Then, it has been shown that the temperature variation on the outer circle composed by the last temperature sensors is the same whether the power is applied by static heater or by RF dissipation. Thus, the calibration curve is obtained by applying several steps of static power. This is described by the curve $\Delta T(P_{stat})$ which is bath temperature dependent because of the effective thermal exchange coefficient. A typical calibration set is shown on Figure 3. Another asset here is to measure the thermal conductivity of the sample. Assuming the thermal conductivity is constant between two consecutive thermometers ($\Delta T<100$ mK), because of the axis-symmetry configuration, the thermal conductivity is calculated from the Fourier's Law:

$$k_\lambda = \frac{P_{stat}}{2\pi e(T_i - T_{i+1})} \ln\left({}^{R_{i+1}}/_{R_i}\right) \quad (1)$$

Where k_λ is the thermal conductivity, P_{stat} the applied static power, e the thickness of the sample, T_i the temperature at the position R_i and T_{i+1} the temperature at the position R_{i+1}.

Then, the static heater is switched off and the RF field is applied on the sample. The resulting temperature variation is coming from RF power dissipated on the sample which is determined by the calibration curve. Knowing the RF magnetic field H_S applied on the sample from the transmitted pickup, the surface resistance is calculated from the relation:

$$P_{RF} = \frac{1}{2}\oiint R_S H_S^2 dS \quad (2)$$

The solution R_S is then found regardless of the thermal exchange condition with the Helium bath and the temperature range. One can obtain the residual component and the BCS part of the total surface resistance.

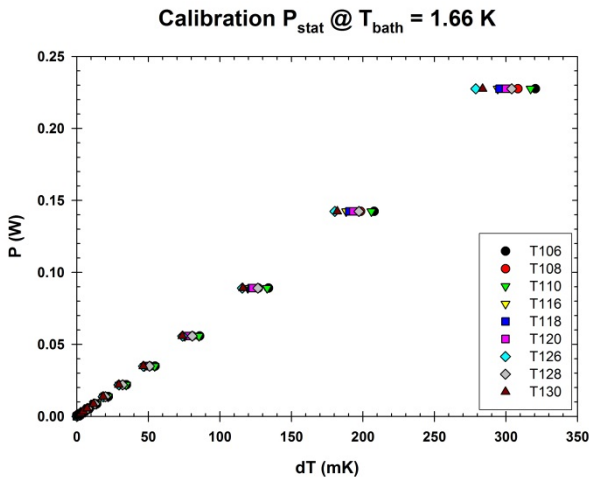

Figure 3: Calibration curves for the 9 outer thermometers. The temperature variation is strongly dependent of the exchange coefficient (surface roughness and Kapitza conductance à Nb/He(II) interface).

EXPERIMENTAL RESULTS

For the commissioning of the cavity, a bulk niobium sample has been used from the same grade of the niobium cavity. This RRR300 polycrystalline niobium is provided by Tokyo Denkai industry. The cavity and the sample were treated with standard BCP used at IPN and cleaned with high pressure rinsing with high purity water. All parts (except thermometric flange) were mounted in ISO-100 clean room.

Thermal Conductivity

During calibration of the temperature elevation with the static power applied on the backside of the sample, the radial temperature gradient is measured by the way of three temperature arms (6 Allen Bradley thermometers) disposed with an angle of 120° from each other. Assuming a surface S for heat exchange with He bath at 4.235 K, a parietal temperature T_p, one obtains the heat exchange coefficient h_k with the relation:

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$$h_k = \frac{P_{stat}}{S(T_p - T_6)} \quad (3)$$

Depending on S which is not really known in experimental condition (a part is in contact with cavity and thermometric chamber, see Fig. 1), the exchange coefficient is found at 240 W/m²K if all the surface external to indium gaskets is taken, or 900 W/m²K if only the outer surface is taken. In both case, we obtain a good agreement with temperature distribution as it is shown on Figure 4. The thickness is fixed à 2.2 mm which is in agreement with the removed thickness after several BCP.

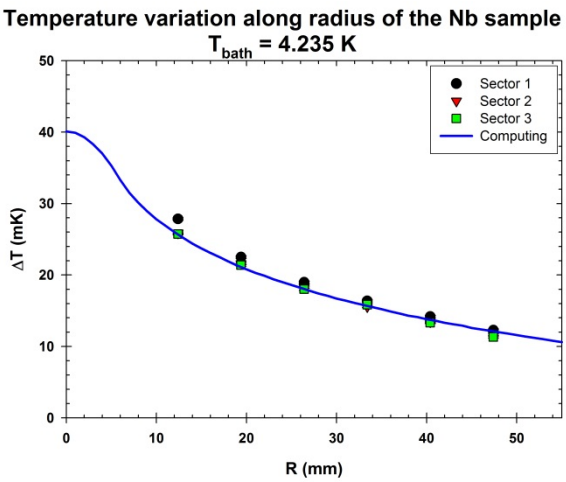

Figure 4: Temperature variation profile along radius of sample. The theoretical curve has been obtained from ANSYS calculation with following parameters: Pstat=12.5 mW, e=2.2 mm, h_k= 900 W/m²K. The thermal conductivity has taken from experimental measurements.

These calculations allow fixing the boundaries conditions especially for the heat exchange coefficient to calculate temperature distribution coming from RF losses on the sample.

Each temperature profile is measured for various P_{stat} steps from 0.5 mW up to 500 mW at a fixed temperature bath in the range of 1.6 – 4.25 K. Using equation (1) for each step of applied static power, the thermal conductivity of the sample is measured. Figure 5 presents the thermal conductivity of RRR300 grade bulk niobium sample which has been supplied from Tokyo Denkaï. The results are compared to the parametrization of the thermal conductivity which has been calculated in [8] using the following expression:

$$\kappa_\lambda = R(y)\left[\frac{\rho}{LT} + AT^2\right]^{-1} + \left[\frac{1}{Dexp(y)T^2} + \frac{1}{BlT^3}\right]^{-1} \quad (4)$$

Where $y=\Delta(T)/K_BT$, $\Delta(T)$ the BCS superconducting gap, R is the ratio of the electronic conduction of superconducting state to the normal state, ρ is the residual resistivity, L the Lorentz constant (Wiedeman & Franz), A the coefficient of moment exchange with the lattice vibrations, D related to phonon scattering by electrons, l the mean free path of phonons and B is related to the phonon scattering at crystal boundaries.

The theoretical thermal conductivity has been taken from [8]. The thermal conductivity at 4.2 K is in agreement with the RRR of sample which has been stated to roughly in a factor 4. The RRR has been measured with 4-wires method at 380.

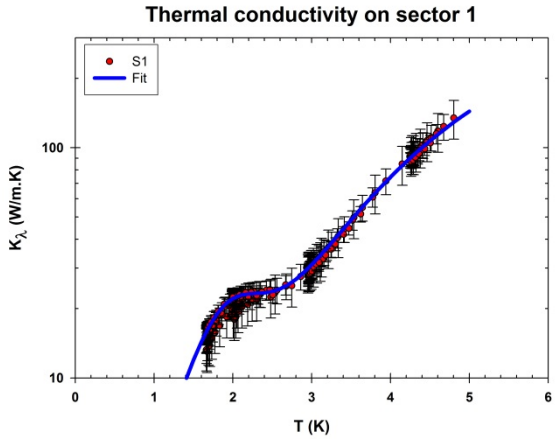

Figure 5: Thermal conductivity measured on sector 1 with bulk niobium. The fit curve is obtained according given expressions in eq. (4) [8].

Surface Resistance

The main RF parameters of the cavity have been calculated with Ansys/HFSS and are presented in table 1 and are closed to the analytical calculations.

Table 1: RF Parameters of Cavity

Parameter	TE011	TE012
Frequency (GHz)	3.87	5.16
$H_p/\sqrt{\omega U}$ $(A/m/\sqrt{J/s})$	0.451	0.598
G (Ω)	763	913

The movable coupler allows reaching critical coupling for both modes and for the temperature range of 1.5 – 4.3 K. The critical coupling is applied for RF measurement to minimize errors.

Regardless the origin of the dissipated power on the sample, the exchange coefficient is considered as constant for a fixed applied power. Equation (3) shows the direct relation of the temperature variation of the outer thermometers and dissipated power. Using calibration curve, the RF power dissipated on the sample is calculated directly from the temperature variation. The surface resistance is calculated from eq. (2). Indeed, the surface magnetic field on the sample is described with Bessel's functions as a result of solving Maxwell's equations. In this case, assuming the surface resistance as constant along the radius for small RF field and small temperature variation, one can have analytical solution by using Lommel's integrals:

$$P_{RF} = 2.2761 \times 10^{-3} R_s H_s^2 \quad (5)$$

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Where R_s is the surface resistance and H_s the peak magnetic field on the sample

Figures 6 and 7 show respectively the surface resistance for the TE011 and TE012 modes at 1.66 K using eq. (5). High values of surface resistance are measured with the thermometer T120 for both frequencies. The origin if this defect must be investigated (Indium gasket or bad BCP treatment of sample and cavity).

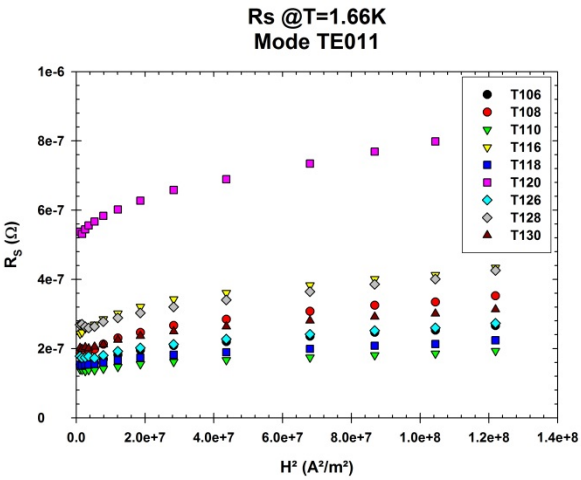

Figure 6: Surface resistance at 1.66 K for the TE011 mode (3.87 GHz) measured with the 9 outer thermometers.

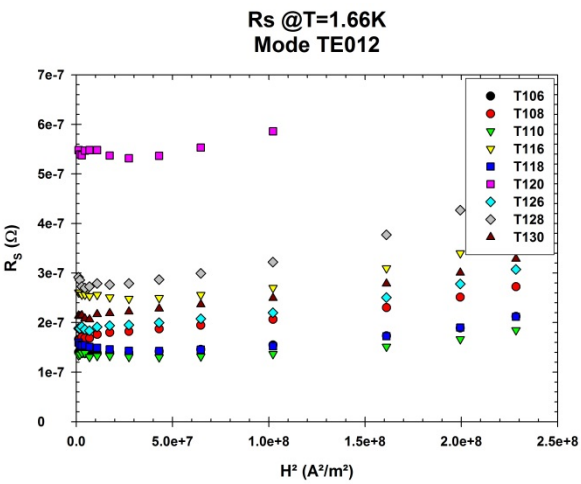

Figure 7: Surface resistance at 1.66 K for the TE012 mode (5.16 GHz) measured with the 9 outer thermometers.

Using a better area of the sample, the measured surface resistance dependence with temperature at 1 mT has been represented on Figure 8. Results show the residual surface resistance which is measured at ≃140 nΩ. Investigation must be carried out to understand the origin of this high value. As magnetic measurements have not demonstrate high residual magnetic field (<10 mG), this could be due to RF components, especially from a defect in the feedthrough and coupling ports.

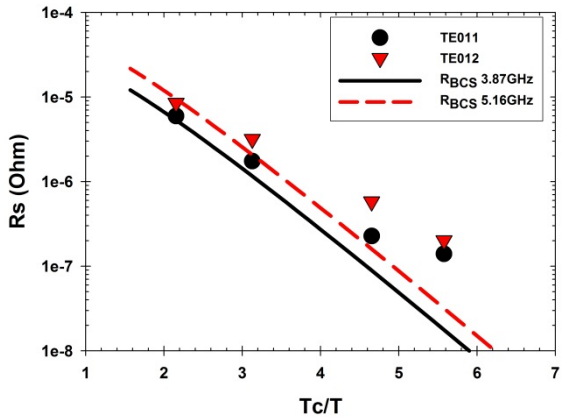

Figure 8: Surface resistance measured with the thermometer T110 at B≃1mT for 4.3 K, 3.0 K, 2.0 K and 1.65 K. Straight lines are the theoretical R_{BCS}.

CONCLUSION

The pillbox cavity has been successfully operated to characterize bulk niobium sample. The measured thermal conductivity is in agreement with RRR=300 grade niobium. RF measurements highlight defect on the sample which can be localized. These results show the efficiency of the method despite issue on RF coupling. Indeed, the cold transmitted Q value is much higher than expected. Further investigations are carried out to determine how the adaptation of feedthrough to the antennas must be fixed to enhance RF magnetic field measurement on the sample

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