

RRR MEASUREMENTS ON NIOBIUM FOR SUPERCONDUCTING RF CAVITIES AT FERMILAB *

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Abstract

Fermilab is involved in the development of superconducting RF cavities of the bulk Niobium (Nb) type for several projects, with the most advanced being those for the CKM separated kaon beam experiment.

The following reports on RRR measurements on samples cut from Nb sheets for the CKM prototype cavities. The RRR was measured upon receipt and after the 800°C heat treatment that is used to outgas the cavity parts before assembly. Then, the effect of electron-beam welding on the Nb RRR was investigated by using samples cut from plates produced by joining sheets by e-beam welding. These measurements allowed to verify if the Nb sheets, as delivered by industry, had the specified RRR and if it could be upheld throughout the cavity fabrication process. The RRR measurement systems used are also described.

This work was performed in collaboration with DESY.

INTRODUCTION

The evaluation of purity and thermal conductivity of the Nb to be used in the fabrication of superconducting RF cavities at liquid Helium temperatures is normally done by measuring the Residual Resistivity Ratio (RRR) of a sample of the material. The RRR is the ratio between the resistances of the sample at room temperature and at the operating temperature of the cavity.

Fermilab is involved in the fabrication of superconducting RF cavities for the separated kaon beam experiment (CKM) and the third harmonic cavities for FNAL-NICADD photo-injector and TTF-DESY. While both cavity types are fabricated with the same technology using the bulk Nb approach (developed at Cornell University) and operated at 1.8 K and 3.9 GHz, the CKM cavities are designed for a transverse deflecting mode. The RRR plays a particularly important role in these cavities because of the high frequency where the BCS surface resistance and hence the RF losses are higher. Due to the strong dependence of the BCS resistance on temperature the surface resistance further increases if the thermal conductivity of the Nb is low and the RF losses cannot be removed efficiently from the inner surfaces of the cavity to the superfluid helium bath on the outside of the cavity. It is therefore crucial that the thermal conductivity and therefore the RRR parameter be as high as possible.

The following discusses RRR measurements, which were performed on the material for the CKM cavities. First the RRR measurement systems will be described. Two systems, using 4-point, DC-resistance measurements were developed. Comparative measurements recorded on

both systems are reported. Finally the results of detailed investigations of the effect on RRR of some major cavity fabrication steps, such as welding and high temperature outgassing are discussed.

RRR MEASUREMENT SYSTEMS

The following describes the experimental apparatus for RRR measurements developed at Fermilab. Two different systems were developed, one, optimized for fast measurements of a small quantity of samples, and the other for series measurements allowing the simultaneous cool-down of up to 12 samples. We define the RRR as the ratio of the sample resistances at ~10 K (just above the transition point) and at room temperature. The resistance measurements are performed using the 4-point method with the current being injected at the ends of the sample and the voltage taps being located further inwards such as to minimize the effect of non-uniform current distributions. The 4-point resistance measurements are typically performed with 100 nV precision after low-pass filtering. Thermo-voltages are eliminated by averaging over voltage readings obtained with alternating current polarities. The typical measurement current is 100 mA. Samples are typically 50-100 mm long and 2-4 mm wide. They are usually obtained by electro-erosion cutting. Studies at DESY have shown that this method does not affect the RRR of the material, [1].

Description of the fast RRR measurement system

The RRR measurement system discussed here is documented in further detail in [2]. It allows fast measurement but can accommodate only one sample per cool-down. The cryogenic hardware consists of a standard liquid helium 500 l storage Dewar into which the sample is introduced using a long dipstick. Advantage is taken of the natural stratification of the He gas above the liquid surface in the Dewar. The insertion depth of the dipstick determines the sample temperature. Fig. 1 shows the sample-holder which contains a ~75 mm long, up to ~6 mm wide and several mm thick sample, which is pressed against flexible Be-Cu contactors for current transfer (outer) and voltage measurement (inner). A carbon-glass RTD (Resistive Temperature Device) next to the sample serves as thermometer. A Cu foil surrounding the sample holder inside the dipstick promotes a lower temperature gradient along the sample. The sample-holder is introduced into the bottom end of the 1.5 m long, stainless steel dipstick tube. A hole, 15 cm from the open bottom end, facilitates venting with He the sample holder upon insertion. The current leads (one pair) and voltage taps (one pair) as well as the temperature sensor wires are routed to the outside of the Dewar through the core of the dipstick.

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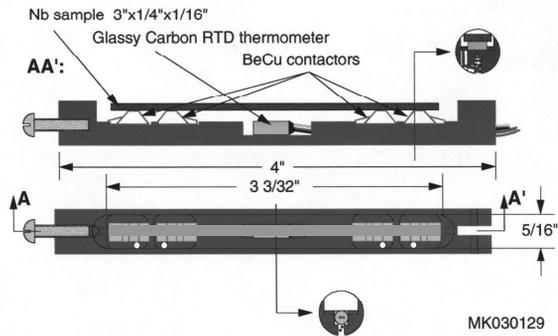


Figure 1: One-sample-holder for RRR measurement.

Description of the 12-sample RRR system

In order to allow measurement of larger quantities of samples during one cool-down a 12-sample-holder was fabricated, that can be inserted into an existing cryogenic temperature sensor calibration facility at Fermilab. This test station features a 25 cm high, 6 cm diameter sample volume in which ambient to 2 K temperatures can be imposed with mK precision using liquid/gaseous helium in conjunction with electrical heaters. The temperatures in the test volume are measured with calibrated Cernox™ temperature sensors. Fig. 2 shows the sample-holder, which consists of a G10 plate to which the samples are affixed with Cu clamps and brass screws. All samples are connected in series. The voltage is recorded using voltage taps, that consist of flexible Cu blades underneath the sample (see schematic in Fig. 2) placed ~5 mm inwards from the clamps to minimize current distribution effects. Wires connect the flexible Cu contactors to conducting strips at the edge of the G10 plate, that are positioned such as to connect to the proper channels when the G10 plate is inserted into the spring-loaded connector slot.

The system is cooled down to 4.2 K by flooding the measurement volume with liquid helium, which is then evaporated with heaters to generate the temperatures in the range 4-300 K. The data-acquisition is performed through a Labview data acquisition program. Measurements of the sample resistance are typically performed between 9 K and 10 K in steps of ~300 mK as well at some select temperatures during the warm up to room temperature.

Results of Comparative RRR Measurements

The following RRR data were obtained from the ratio of the resistances between room temperature and ~10 K. Thus the data were not extrapolated from 10 K to 4.2 K and the sample-to-sample variations of the ambient temperature conditions were not taken into account.

Fig. 3 shows the resistivity as a function of temperature measured with the 12-sample system on two samples of Nb cut from 1.6 mm sheets as delivered by Wah Chang for the CKM cavity fabrication. The resistance data were corrected for the geometrical parameters of each sample to convert the data to a resistivity. The 300 K resistivity

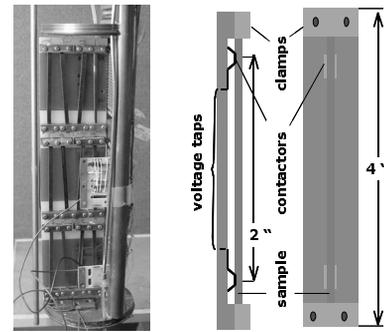


Figure 2: 12-sample-holder for RRR measurement.

found was consistent with the published value of $14 \times 10^{-8} \Omega\text{m}$, [3]. Table 1 gives a comparison of the RRR measured on the above two samples with both measurement systems. The results agree to ~5%, which is consistent with the variation found between two runs using the 12 sample system.

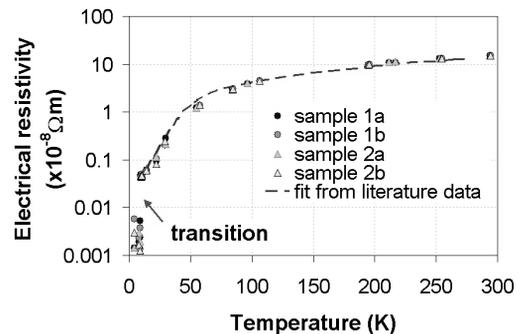


Figure 3: Resistivity of 2 CKM Nb samples (a, b refer to separate cool-downs). The fit represents data from [3].

Table 1: RRR comparison on CKM Nb samples.

System	Measurement #	RRR (sample 1 / 2)
fast	-	333 / 298
12-sample	a	320 / 307
12-sample	b	337 / 331

RRR MEASUREMENT RESULTS

Weld-series

The superconducting cavities for CKM will be fabricated by electron-beam (EB) welding of deep-drawn half-cells. The weld region is strongly heated during the process, raising the concern of a possible contamination of the Nb during the welding process. Extensive measurements at DESY in the context of the TESLA R&D, [4], have shown that the welding does not deteriorate the Nb RRR if the vacuum in the EB welding machine is better than 10^{-5} mbar. To test the effect of the EB welding, performed at the Sciaky company, several samples of butt-welded $10 \times 2.5 \text{ cm}^2$ Nb sheets (1.6 mm thick) were prepared. The sheets were welded in a special Al fixture according to the procedures adopted for the

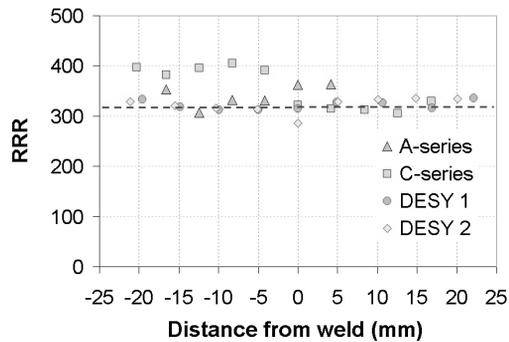


Figure 4: Weld-series RRR for CKM Nb vs. distance from weld. The dashed line indicates the RRR before welding. A&C/DESY1&2 were measured at FNAL/DESY.

welding of the cavity half-cells (50 kV, 44 mA, 0.5 m feed/min, 4×10^{-5} mbar vacuum) and cut into 3 mm wide, 75 mm long strips parallel to the weld seam.

Fig. 4 shows the results of RRR measurements after welding. Two sets of samples obtained from the same weld were measured at Fermilab (A&C). A second set of sheets was welded to produce a second set of samples, which were measured at DESY (DESY 1&2).

The results indicate no drastic change of RRR across the weld region. This is consistent with the results found in the DESY study of Nb for TESLA, [4]. In fact the DESY group observed that the RRR is increased in the weld region and reduced in the so-called heat affected region adjacent to the weld. For vacuum pressures above $3\text{-}4 \times 10^{-5}$ mbar (such as in the cases discussed here), however, this effect is suppressed and the RRR more-or-less unchanged by the welding. Another aspect of the measurement results, namely the non-symmetric increase of RRR on one side of the weld in the C-series, confirms similar occasional observations at DESY, [1]. This could possibly be related to a stronger heating of this side during the welding process as a result of a reduced thermal contact with the welding fixture (which also serves as a heat drain).

Fig. 5 shows a micrograph of the weld-seam of weld series C over the full (~3mm) width of the sample. As seen previously, the grains are (partially) aligned along the weld seam in an “arrow-pattern”, [5], with the long side of the grains being up to a 1 mm.

Other Measurements

A set of samples was surface treated according to the procedure developed for the CKM cavity fabrication, that is: etched by $\sim 100 \mu\text{m}$ with BCP (1:1:2), followed by a 5 hrs bake at 800°C in a UHV furnace, followed by a $20 \mu\text{m}$ BCP etch. The RRR of a sample that followed the entire procedure was found to be unchanged from before. Interestingly, however, a strong RRR degradation was found when the sample had not been etched prior to the 800°C bake. This indicates that surface pollution that could not be removed by pre-cleaning (such as ultrasonic

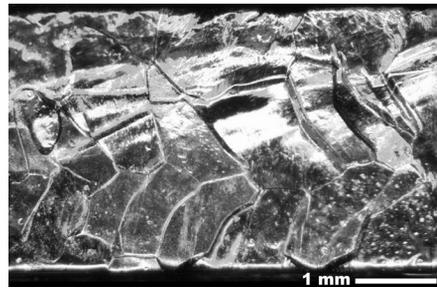


Figure 5: Micrograph of weld-seam of sample after full surface preparation procedure as for CKM cavities. Courtesy of P. Lee, University of Wisconsin.

rinsing in ultra-pure water), possibly introduced during electrical erosion cutting, was diffused into the sample bulk during the heat treatment. Two samples were produced as part of this series, revealing a RRR reduction from 300 to 150/110.

SUMMARY

DC RRR measurement systems were developed at Fermilab for the characterization of Nb sheets to support the superconducting cavity development. First measurements of the RRR on samples revealed that

- the RRR of the received sheets is >300 , as specified,
- the RRR is not reduced during EB-welding,
- the RRR is unchanged following the final surface treatment (BCP etching and 800°C bake),
- the RRR is degraded when the high temperature baking is performed without prior surface etching.

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