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RECENT RRR MEASUREMENTS ON NIOBIUM FOR SUPERCONDUCTING RF CAVITIES AT FERMILAB *

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Abstract

Fermilab is developing superconducting RF cavities of the bulk Niobium (Nb) type. Several prototypes of a 3rd-harmonic cavity and a transverse deflecting mode (CKM-type) cavity were already built. The first three-cell third harmonic model recently achieved the expected performance limit [1].

The following reports on RRR measurements on samples cut from Nb sheets for the 3rd-harmonic and CKM prototype cavities. The RRR was measured upon receipt and after the chemical polishing and heat treatment steps used in the cavity fabrication. These measurements not only serve the purpose of quality control of the pre-cursor material but also as a check of the cavity processing. We also measured the RRR of the electron-beam welds using samples cut from plates produced by joining sheets by e-beam welding in the same device used for welding cavity parts. Finally we will discuss our next generation RRR measurement system, currently in the design stage.

INTRODUCTION

Fermilab is involved in the fabrication of transverse deflecting and third harmonic 3.9 GHz superconducting RF cavities. Both cavity types are fabricated through deep-drawing of half-cells from ~3 mm thick Nb sheet into half-cells and subsequent electron beam-welding in vacuum into multi-cell cavities. The cavity operational parameters are 1.8 K and 3.9 GHz. The purity of the niobium (Nb) in superconducting RF cavities is usually evaluated via the Residual Resistivity Ratio (RRR). The RRR is the ratio between the resistances of the sample at room temperature and 4.2 K. The RRR plays a particularly important role in the 3.9 GHz cavities because it correlates with the thermal conductivity of the cavity wall. At 3.9 GHz the BCS resistance heating on the RF exposed surface is strong enough that these cavities are limited by the thermal conductivity through the bulk and the heat transfer properties of their surface.

The following discusses RRR measurements, which were performed on the material for the Fermilab SRF cavities in the Technical Division temperature sensor calibration test facility. This facility features a 25 cm high, 6 cm diameter insulated sample volume in which ambient to 2K temperatures can be provided via in situ evaporation of liquid helium. The sample-holder consists of a G10 plate to which twelve ~75 mm long (~2x2mm^2 cross-section) samples (cut from sheets with electro-erosion machining) are affixed with copper clamps and brass screws. The clamps also conduct the 100 mA current to the samples. Flexible copper blade voltage taps record the voltage-drop over the length of the samples. The 4-point resistance measurements are typically performed with 100 nV precision after low-pass filtering. Thermal emfs are eliminated by averaging over voltage readings obtained with both current polarities. 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 up to room temperature. The measurement reproducibility of the test station was found to be ±5%. Benchmarking measurements on 2 samples with the university of Wisconsin yielded agreement at the 3% level [2].

The RRR as defined in this paper is the ratio of the resistances at room temperature and ~10 K (just above the super- to normal-conducting transition). This definition is different from the NIST standard [3], which specifies the lower measurement temperature to 4.2K[1]. Our measurement system presently does not have a magnet and therefore we used the simpler RRR definition. Some trials using the temperature extrapolation method indicate that our RRR definition generates RRR values that are ~10% lower than those obtained with the NIST definition. The RRR measurement system discussed here is documented in further detail in [5]. Fig. 1 shows an example of the temperature dependent resistivity measured in 12 Nb samples. A simple formula obtained from [6] (Eq. 1), fits the data surprisingly well (neglecting RRR related differences at the low temperature end).

$$\boldsymbol{\rho(T)} = 0.05 \times \boldsymbol{T} - 0.7 \quad (\mu\Omega - \boldsymbol{cm}) \tag{1}$$

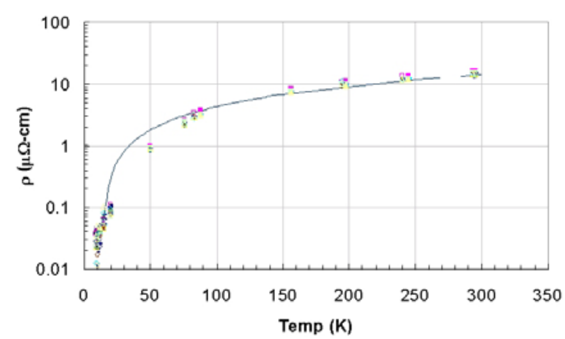

Figure 1: Electrical resistivity vs temperature as measured on 12 samples. Also shown is a simple fit (Eq. 1).

[1] Two methods are usually used to satisfy the NIST standard: -1- the normal state resistivities from 9.5K to ~15K are extrapolated back to 4.2 K or –2- the superconducting state is suppressed applying different magnetic fields above ~0.4 T and extrapolation to zero magnetic field such as to eliminate the magneto-resistive component [4]

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RRR MEASUREMENT RESULTS

The following discusses results of RRR measurements obtained on a series of samples cut from 3 and 2.3 mm thick high purity Nb sheets from Wah-Chang.

Acceptance Tests

The first test consisted in verifying whether the procured material had the specified RRR. Fig. 2 shows that the Wah-Chang material largely exceeds the RRR300 specification. To verify if the various processing steps applied to the Nb material during cavity fabrication affect the RRR, the samples underwent a full sequence of treatment steps, including a ~100 min BCP (1:1:2) etch, a high temperature heat treatment (800°C, 5 hrs) in vacuum ($<10^{-6}$ Torr) followed by another 20 min BCP polish. The results of the RRR measurements on these samples is shown in Figure 1. The figure clearly shows that within the measurement reproducibility there is no discernible effect of the surface treatment on the RRR. This indicates that the processing only affects the surface and not the bulk. Data are discussed in further detail in [2].

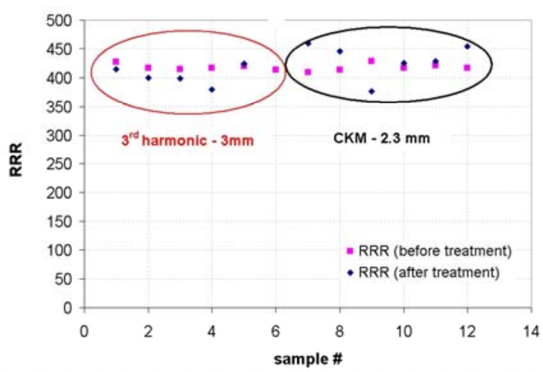

Figure 2: RRR test results on Nb for the FNAL 3rd harmonic and transverse deflecting (CKM) cavities, as received and after a complete standard surface treatment for cavities, including several cleaning, rinsing and etching steps as well as a high temperature anneal.

Weld-tests

The superconducting cavities for CKM will be fabricated by electron-beam (EB) welding of deep-drawn half-cells. The weld region is melted during the process, possibly leading to contamination of the Nb during the welding process with residual gas from the weld chamber. Extensive measurements at DESY in the context of the TESLA R&D, [7], have shown that the welding does not deteriorate the RRR if the vacuum in the EB welding machine is better than 10^{-5} mbar. To test the effect of the EB welding, performed for Fermilab at the Sciaky company, several samples of butt-welded 10 × 2.5 cm^2 Nb sheets were prepared. The sheets were welded in a special Al fixture (see Fig. 3) according to the procedures adopted for the welding of the cavity half-cells (50 kV, 44 mA, 0.5 m feed/min, 4×10^{-5} mbar vacuum) and electro-erosion cut into 2.5 mm wide, 75

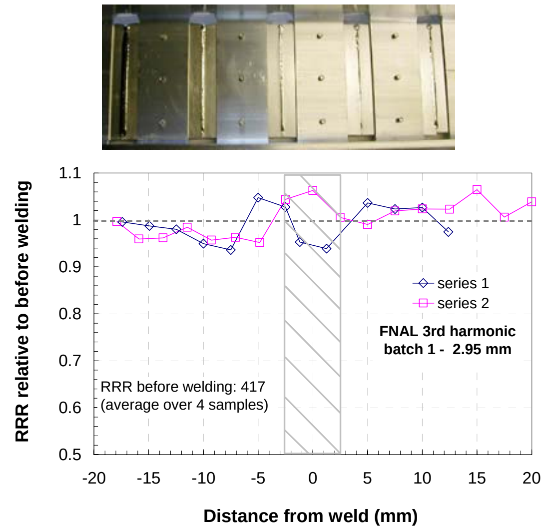

Figure 3: Top: Welding test fixture top after welding. The samples plates are clamped between Al plates Bottom: 3rd harmonic batch-1 weld series results (normalized to average RRR before welding, ~417). The grey region represents the weld.

mm long strips parallel to the weld seam. Fig. 3 shows the results of RRR measurements after welding versus the position of the stick with respect to the weld (each point represents a sample). The average RRR measured on four samples cut from the edges of the plates of weld sample 1 was used as the reference measurement on which the data after welding were normalized (RRR=417). Note that this measurement result is consistent with the RRR measured on six other samples of similar material discussed in Fig. 2. A detailed discussion of the measurement data is given in [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 for vacuum pressures below 3-4 $\times10^{-5}$ mbar (such as in the cases discussed here) [7].

Noise in the System

A special test was performed on some samples, which consisted of not only measuring with the usual measurement current of 0.1 A, but also with 0.01 A. Differences in the results obtained with different currents are indicative of non uniform current distributions or heating effects, especially important at temperatures close to critical. The resistivities obtained from the measurements with two currents agree reasonably well, in general (see Fig. 1 for instance). Closer inspection, however, shows that there is quite some difference between the results obtained from different current measurements at the low temperature end (9-10 K). Fig. 4 clearly shows these large differences, very much in contrast to the small difference at higher temperatures (the absolute difference decreases exponentially with temperature). These differences originate to a large extent

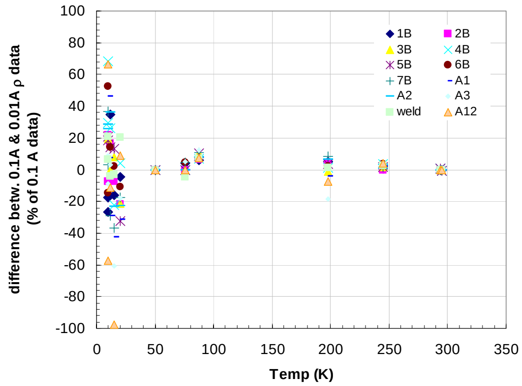

Figure 4: Difference in electrical resistivity between measurements with 0.1 A and 0.01 A currents (in % of the 0.1 A value) for all samples measured on 10/08/2004.

from variations of the low current (0.01A) data. This, together with the fact that the sign of the difference scatters, make us believe that the issue is voltage noise. With typical sample resistances of the order of 1 μΩ, the voltage at 0.01 A becomes ~10 nV, hard against the resolution limit of the Nano-voltmeter as well as below the "noise" level in the system.

DESIGN OF A NEW RRR TEST SYSTEM

To double the 3R measurement turn-around time, cut noise (by ~10) and allow for normal state resistivity measurements at 4.2 K, we launched the design of a new and improved system. It incorporates many features of the existing system, as well as new features. The basic principle for the temperature control, for example, remains the same: liquid helium is dropped into the measurement volume at a rate controlled by a supply valve, while heaters bring the helium gas and the samples to the set temperature. RRR data are obtained through 4-point electrical resistance measurement.

Greater ease of disassembly and assembly will result from having only one indium seal between the support flange, from which the system hangs, and the sample-holder (Fig. 5). The sample-holder outer layer is one piece, containing a vacuum, instead of two vessels as before. Once the holder cover is removed, the connectors with voltage tap wires (4 per sample, 10 samples, + 2 current leads) and heater wires (+ temperature sensors) is disconnected and the sample insert un-screwed from the central support rod. The heater and voltage tap wires run through separate connectors, and more space is available for better shielding, factors that should reduce electrical noise. The samples can be removed by hand, since they are just clamped into the sample-holder, with no need for tools, further reducing mounting time. Unlike the old system, the new system will feature an automatic feedback loop to control the temperature. In the 5 to 40 K regime the helium gas will be pumped through the sample insert at a significant rate such as to allow for rapid

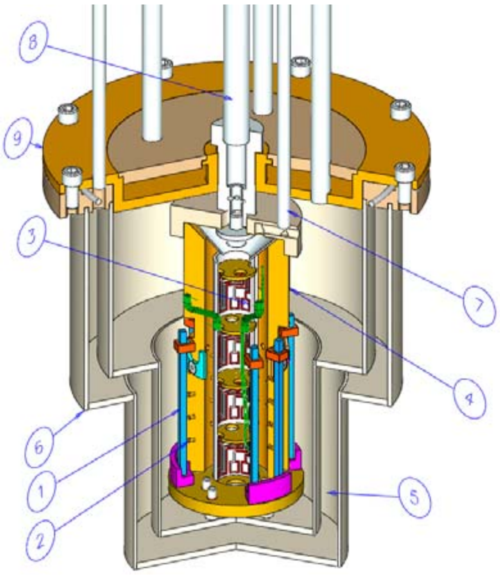

Figure 5: CAD model of new Fermilab 3R measurement insert. –1- sample, -2- pogo-pin, -3- helium heating channel, -4- G10 sample-holder, -5- sample insert insulating vacuum, -6- removable sample insert cover, -7- liquid helium supply, -8- support, -9- top flange;

thermalization of the samples. At set-point temperatures above 40 K the heater power will not suffice to warm the needed helium throughput so that we need to revert to the "old" system (with the addition of the feedback system). Finally the sample-holder (as shown in Fig. 5) will fit into a superconducting solenoid, cooled by the liquid helium bath surrounding the system. The magnet will allow resistivity measurement in the normal state at 4.2 K.

SUMMARY

RRR measurements conducted at Fermilab in support of several cavity development projects have shown that –1- the RRR of the received sheets is >300, as specified, -2- the RRR is not reduced during EB-welding, -3- the RRR is unchanged following the final surface treatment (BCP etchings and 800°C bake). A new station will be built in the near future to improve accuracy and turn-around time.

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