Fermilab

Apparatus for Measuring RRR

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1) Introduction

The evaluation of purity and thermal conductivity at liquid Helium temperatures of the Niobium to be used in the fabrication of superconducting RF cavities is normally done by measuring the Residual Resistivity Ratio (RRR) of a sample of the material. The relationship between the thermal and the electrical conductivity (Wiedemann-Franz Law) simplifies the task by leading to the measurement of electrical instead of thermal resistance. The RRR is the ratio between the resistances of the sample at room temperature and at the operating temperature of the cavity. A more precise definition is discussed later. The conductivity at low temperatures depends on lattice defects and impurities. Impurities are also important for cavities in a direct way as affecting the RF properties of its surface when exposed by chemical etching. The following describes the experimental apparatus for RRR measurements developed at Fermilab's Beams Division. Part 2 contains a description of the sample-holder and measurement hardware. Part 3 contains a discussion on definition, measurements and errors. Part 4 gives a step-by-step description of the measurement procedure. Finally, Part 5 gives an example of results obtained recently on a Niobium sample for CKM cavities.

2) Experimental Apparatus Description

The RRR measurement system discussed here was designed by M. Kuchnir in 1998. In order to simplify the cryogenic hardware it takes advantage of the availability of liquid Helium 500 l storage dewars into which the sample is introduced using a dipstick. Advantage is taken of the natural stratification of the Helium gas above the liquid surface in the dewar. The insertion depth of the dipstick determines the sample temperature. A carbon-glass RTD (Resistive Temperature Device) next to the sample serves as thermometer. A Copper foil surrounding the sample holder inside the dipstick promotes a lower temperature gradient along the sample.

The dipstick is a 60 inches long stainless tube (0.500" OD by 0.035" wall) on which outside length marks every inch were scribed. A tiny hole 6 inches from the open bottom end facilitates venting with Helium the sample holder upon insertion. It also serves as an indicator of the state of withdrawal for the closing of the ball valve on the storage dewar. The upper end of the dipstick has a u-shaped pipefitting leading to a sealed feed-through socket connector from which hangs the cable to the electronics. The wiring consists of the sample voltage tap and current lead wires (two pairs) and the temperature sensor voltage tap wires and current leads (2 pairs).

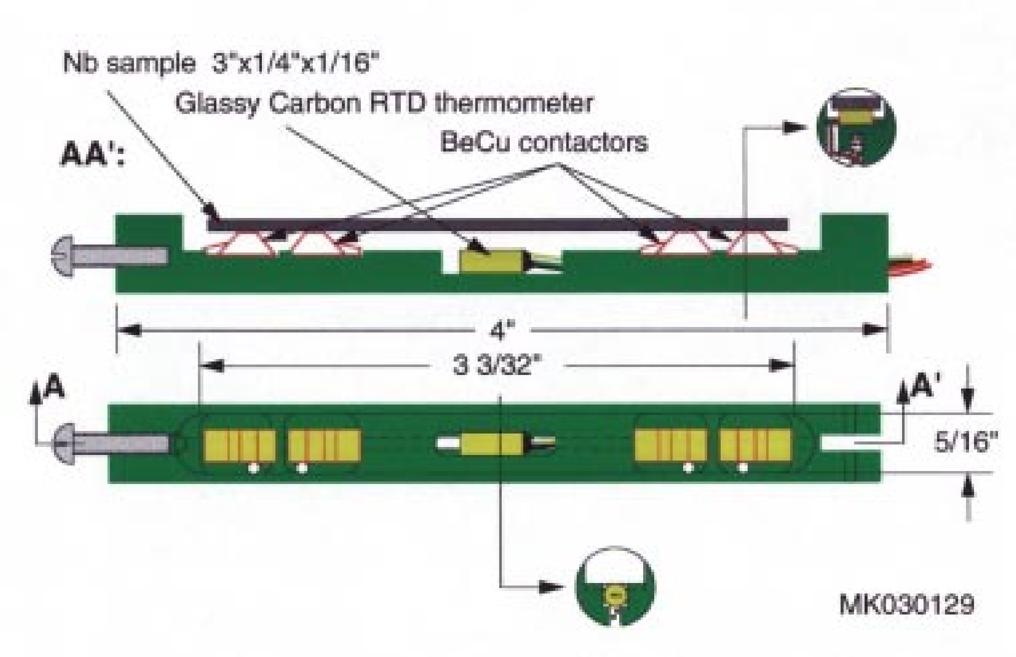

Figure 1: Mechanical sketch of sample holder

The sample holder (see Figure 1) is machined out of fiberglass epoxy composite (G-11) and contains four Beryllium-Copper springs for contact electrodes. The sample is pressed against the copper springs in order to provide electrical contacts for the resistance measurement. A screw attached to the lower end of the holder is used for withdrawing and inserting it in the bottom end of the dipstick.

The sample itself should have for dimensions a length of 3 inches, a maximum width of 1/4 inches and a thickness less than 1/8 inches. It is usually cut from a Niobium sheet by EDM (Electric Discharge Machining).

The sample is kept in place by wrapping it with a thin (non-adhesive) Kapton tape. An additional filler rod might be needed to help compress the sample against the electrodes. In case the filler rod used is made from a high conductivity material, the temperature uniformity along the sample can be improved. As discussed in section 4.5 of this document it might be of advantage to abstain from using such a highly conductive rod.

The wires leading to the sample-holder have sufficient slack to allow pulling the sample-holder out of the dipstick for exchanging samples.

3) Measuring Schematic

Figure 2 shows the main hardware components required in the measurement: the LHe dewar, the dipstick with the sample-holder and two “4-lead” resistance measurement circuits one for the sample and one for the RTD as well as a recorder.

The RTD circuit is designed for Ω to 100 kΩ resistances and low currents (to avoid self-heating) and that for the sample is selected for mΩ to nΩ resistances and relatively high currents (~0.1 A) for sensitivity reasons. Automatic current reversal to eliminate thermal emf is an essential feature of these circuits. The recorder is used for determining whether temperature equilibrium has been achieved and future reexamination of the data. Originally, it used to be a paper chart recorder. In the present implementation it is an Apple computer running Parampage[1] and accessing via Ethernet two channels of an IRM (Internet Rack Monitor)[2] in which the analog outputs of the circuits voltage tap measuring instruments are fed. This allows several computers (in different locations) to access and record the data as well as take advantage of the Parampage zooming capabilities for close data examination.

The four-lead-resistance measuring circuit for the sample is implemented with a Keithley 2400 Series SourceMeter coupled to a Keithley Model 2182 Nanovoltmeter via a trigger-link cable (Model 8501) to allow for Delta mode operation. In this mode, the thermal emf is automatically cancelled.

The four-lead-resistance measuring circuit for the RTD uses the DC current source and amplifiers of the IRM based thermometry system of the A0 South Cave. This current

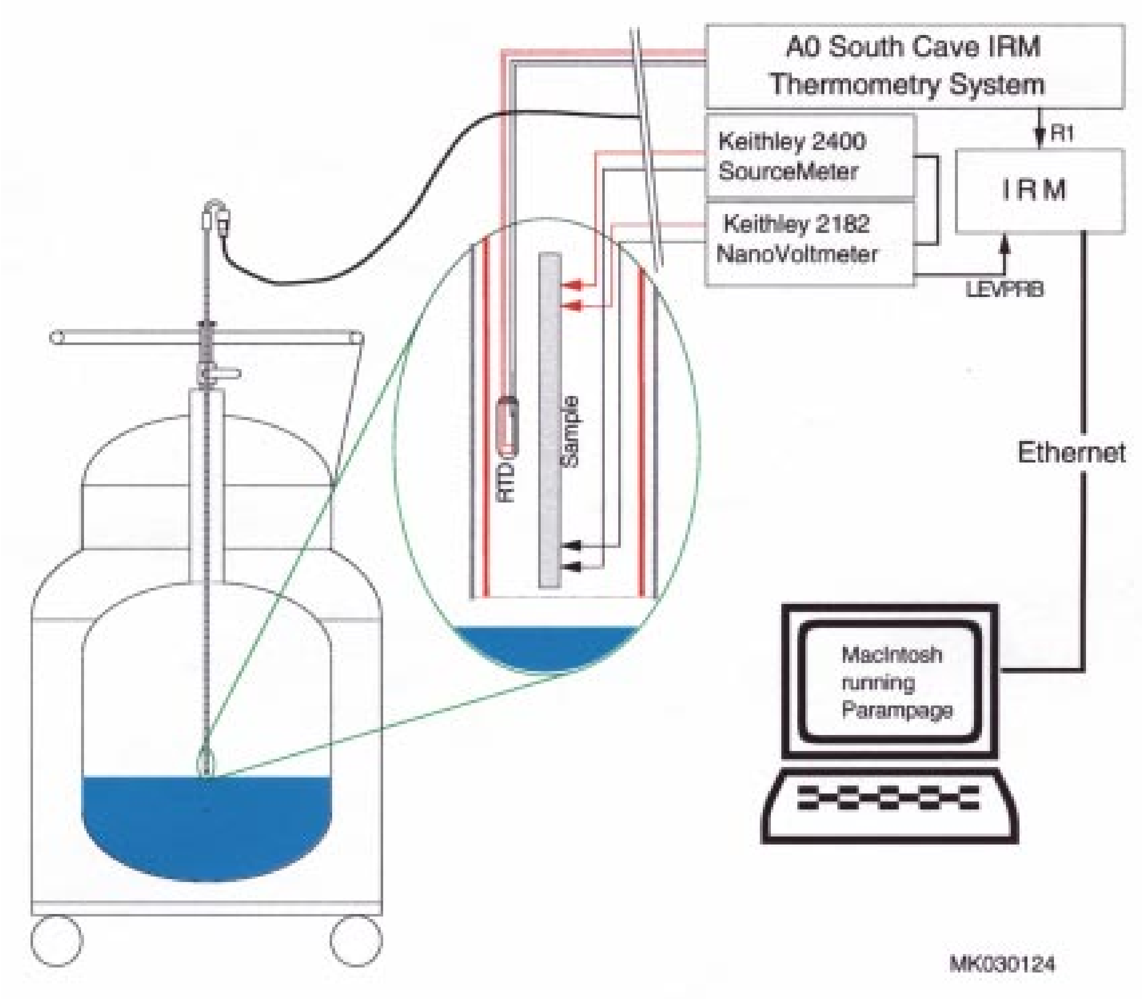

Figure 2: Schematic of RRR setup.

source has three settings: RT (Room Temperature 0.1 mA), HeI (5.0 µA) and HeII (0.5 µA). The current is reversed every ~20 minutes and the thermal emf measured and used in the calculation of the RTD resistance by the IRM program. Although accurate low temperatures are not required the RTD in the sample holder is a calibrated glassy carbon (Lake Shore Cryotronics model CGR-1-1000 serial number C16325). A plot of its calibration is shown in Appendix A (Figure 5).

The sample-holder is lowered through a standard UCRL brass fitting into a commercial 500 l liquid helium dewar in order to cool the sample to near the transition temperature. This cooling is monitored with the RTD and the voltage between the sample voltage taps.

The temperatures involved in the definition of RRR vary. Padamsee et al[3] set the higher temperature at 300 K and do not specify the low temperature. NIST[4] sets them as 273.16 K and 4.2 K, the resistance at the latter to be obtained by extrapolation from values measured above T_c under zero magnetic field. Some workers take the low temperature as the one just above T_c (the superconducting to normal transition temperature that for Niobium is 9.2 K). We define RRR as the ratio between resistance measurements at 300 K and at a temperature just above $T_{c.}$

In the absence of a 300 K oven we actually measure the resistance of the sample at room temperature, R_{RT}, and the value T_{RT} of this temperature (in degrees Celsius) and correct for the difference using the expression:

$$R_{300} = R_{RT} + 4.54 \cdot 10^{-10} \cdot (l/s) \cdot (26.8 - T_{RT})$$

where l/s is the length over cross-section ratio in m^{-1} of the part of the sample under measurement. This expression was derived from a fit of published Nb resistivity data[5] (shown in Appendix B).

For l = 0.048 m (distance between voltage taps in sample holder) and s = $5.767 \cdot 10^{-6}$ m^2 (typical cross section in a series of samples) this becomes:

$$R_{300} = R_{RT} + 3.78 \cdot 10^{-6} \cdot (26.8 - T_{RT})$$

The Nb transition temperature occurs for the sample situated above the liquid surface (4.2 K). The near transition point resistance is usually inferred from voltage measurements with different currents and verifying that they fall onto a linear voltage-current characteristics for currents above a certain value. Below the transition, the sample is superconducting and the voltages are essentially zero.

4) Measurement Procedure

The following is a step-by-step outline of the RRR measurement procedure. The DAQ system is presently not automated requiring manual setting of the measuring hardware. The Keithleys have GPIB and automating part of the procedure with LabView is an interesting project. The following describes the hardware settings that were successfully used in recent tests. Keithley commands are written in italic. The procedure does not address possible problems or malfunctions. A measurement takes typically 2 hours.

4.1) Sample Installation

The first step consists of inserting the sample into the sample-holder. The sample-holder is attached to the voltage tap wires and current leads running within the SS sample-holder tube. The wires are strain relieved and have sufficient slack to allow for pulling the G10 sample holder out of the tube. After pulling out of the sample-holder, the copper shield is removed together with the Al or G10 pressure bar (which has a shape similar to the sample). Then, the non-adhesive Kapton wrap is removed and the sample taken out of the holder (see Figure 1). The beginning and the end of this tape is affixed to the sample holder with short lengths of white electric adhesive tape. The new sample is inserted and the Kapton tape is wrapped with 1-2 mm of overlap. The Kapton wrap is affixed to the sample-holder with adhesive tape at the ends, as mentioned. The pressure bar is placed on top of the sample and the copper shield is slid over the assembly. Finally the sample can be pushed back into the dipstick.

4.2) Setting up the Keithley 2182 Nanovoltmeter

The following options for the voltage reading are activated:

To eliminate thermal emfs the measurements should proceed using the DELTA mode, in which the average of readings taken with opposite current polarity are produced. To activate the DELTA-mode:

SHIFT V1-V2

Note that the DELTA mode uses only input channel 1 and that it requires the trigger link cable (in which case *TRIG* is displayed on the LCD screen). Note that the measurement speed determines the number of readings that are averaged for each measurement current. It should be set to *SLO*W to increase the filtering due to averaging. In the *SLOW* setting the DVM averages over 5 line-cycle periods (*PLC)*, that is, it averages over 83 ms for each polarity.

Voltage results can be further refined by using the default filter settings of the Nanovoltmeter. Pressing *FLT* (*FLT* appears on the display) activates the two stage filtering. Stage1 is an analog low-pass filter for line power noise and stage 2 is a digital (moving or repeating) filter averaging over a window of 1-100 data-points and 1-100% range.

To amplify the signal for the representation in the data recorder, set amplification in the analog output to 10.

SHIFT TEMP1=AOUT, STATE ON, M=10, B=0

Note that the range setting (V or mV) affects the output gain. The settings above refer to the V range. The maximum Analog Output voltage is 1.2 V. It will be necessary to further increase the amplification to 10,000 for the low temperature measurements.

4.3) Setting up the Keithley 2400 SourceMeter

Setting the +/- current mode and current amplitude is done by the command sequence:

CONFIG SWEEP-TYPE-CUSTOM-ADJUST-POINTS
Point 0 = 100 mA, Point 1 = -100 mA

This will produce a +/- 100 mA current square waveform. Since the current is the main measurement variable this step is performed very often.

4.4) Setting up the Data Recording System

Data can be recorded by many different devices, from analog chart recorders to PC based Labview programs. Presently used is a data acquisition based on IRM and Parampage (see references 1 and 2). Typically, the following two IRM channels are activated:
BETA (if voltage from the temperature sensor is not going through the IRM based A0 South Cave thermometry system) or R1 (if the thermometry system is being used and the RTD signal fed to the R1 channel) and LEVPRB (for the voltage from the sample).

4.5) Cold Resistance Measuring Activity

The dipstick is partially inserted into the dewar through the O-ring based UCRL brass fittings. The insertion into the dewar should proceed slowly (especially during the pre-cooling phase at the beginning) to prevent disturbing the equilibrium in the dewar and/or pressurizing it. The insertion is done while measurement currents are being injected such that the normal to superconducting transition can be seen in the voltage signal. Typically the samples start with ~1 mΩ and the measurement current during cool-down is 100 mA.

At low temperature, first find the superconducting condition (voltage < 1nV) and reduce current to 8 mA. Pull sample up until voltage increases with measurement current (raise measurement current to 10, 20, …, 60 mA), that is find the probe position in which voltages are still smaller than expected from a "reasonable" RRR but larger than zero (the "transition zone" region). Reinsert sample slightly to reduce temperature slightly below transition. Inject currents between 10-150 mA. The measurement current heating partially drives the sample into the normal state when there is a temperature gradient along the sample. The voltage rises steeply until it reaches a stable resistance. Vary the measurement current between 10-150 mA and take voltage readings. Plot voltage vs current and determine the low temperature resistance.

4.6) Warm Resistance Measuring Activity

The determination of the sample temperature near room temperature, T_{RT}, is not trivial since its heat capacity is highest and thermal equilibrium involves long time constants. Usually two measurements are made. One before cooling, after the sample is installed in the holder. At this stage, the sample (warmed up by the handling) is let stabilize to room temperature and this temperature is measured with a device like a Fluke 73III portable multimeter with a Fluke 80T-150U temperature probe. The second measurement is carried out after the cold measurements, again after waiting for thermal stabilization.

These two measurements are then extrapolated to the expected value at 300 K and used in the RRR determination.

5) Measurement Example

The following shows an example of a measurement obtained on a Niobium sample for CKM (sample S1, data taken on 02/11/27). Figure 3 shows a Parampage record of the voltage response of the sample to different sample currents. The IRM channel LEVPRB represents the amplified voltage received from the Nanovoltmeter analog output.

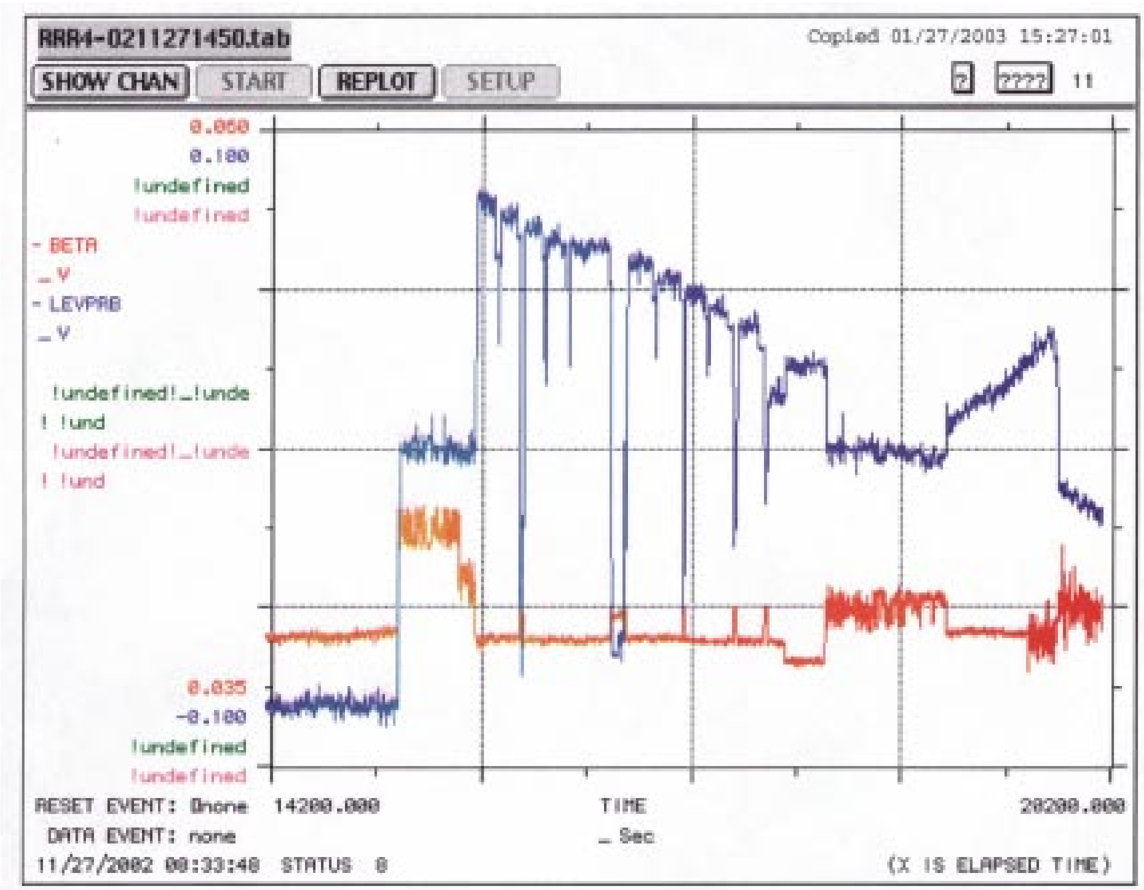

Figure 3: Parampage record during measurement of CKM sample S1 (02/11/27). The LEVPRB trace shows the (amplified) voltage response of the sample to decreasing measurement currents. The measurements shown here were taken at low temperature (~10 K). The V-I points extracted from the above page are then collected in the spreadsheet (see Figure 4) to derive the sample resistance.

The V-I points extracted from the lab book recording of the current and the corresponding voltage in this record are collected in a spreadsheet (see Figure 4) to visualize the derivation of the sample resistance. Note that the IRM channel BETA shown here represents the (amplified) voltage response from the RTD sensor. Usually the channel of interest is the R1 channel, showing the resistance of the RTD sensor.

Since, however, absolute knowledge of the sample temperature is not paramount, little attention was given to the RTD channel in this particular measurement. Figure 5 shows the V-I plot for room temperature and at low temperature. The slope of the V-I characteristic is the resistance (it is also indicated in the plot). The low temperature measurement in Figure 4 clearly shows the superconducting transition zone discussed before.

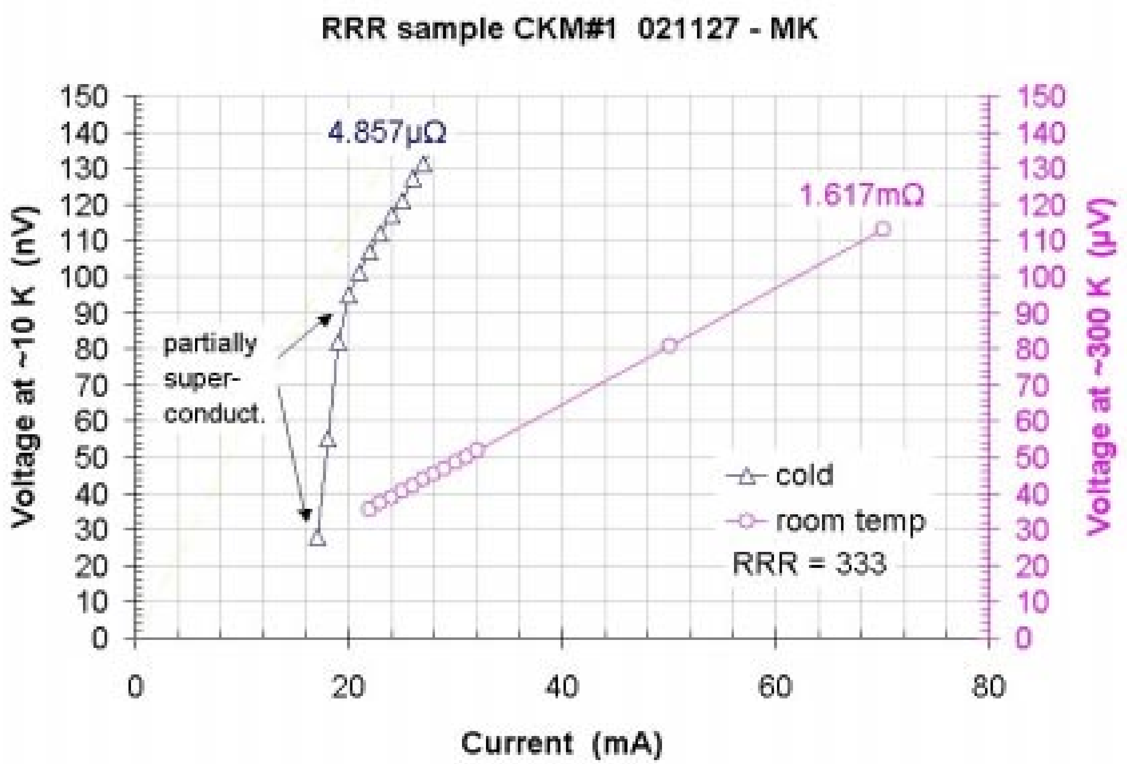

Figure 4: Example of RRR measurement of sample CKM-S1 (data taken on 02/11/27). The plot shows the measured voltage-current characteristic at room temperature and at ~10K. This particular sample has a RRR=1.617mΩ / 0.004857mΩ=333.

It is only when the voltage-current data fall into a straight line through the origin that it can be assumed that the sample is fully in the normal state. As mentioned before the superconducting transition is a reliable indication of the fact that the measurement was performed at a temperature just above transition, thus increasing the correctness of the measured data.

Acknowledgment

We appreciate T. Berenc contribution in acquiring the data presented here and H. Edwards support for this project.

Appendix A

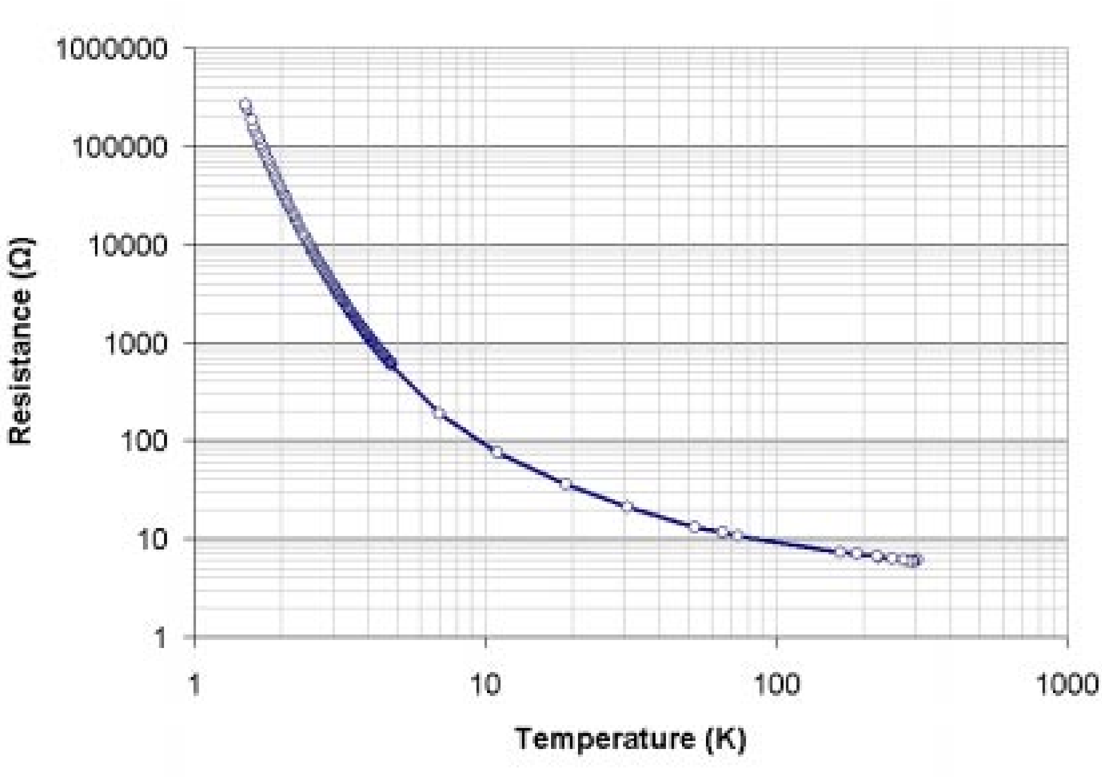

Figure 5: Resistance-temperature calibration of Lakeshore Cryotronics sensor for sample temperature measurement on RRR sample-holder (Lake Shore Cryotronics model CGR-1-1000 serial number C16325).

Appendix B

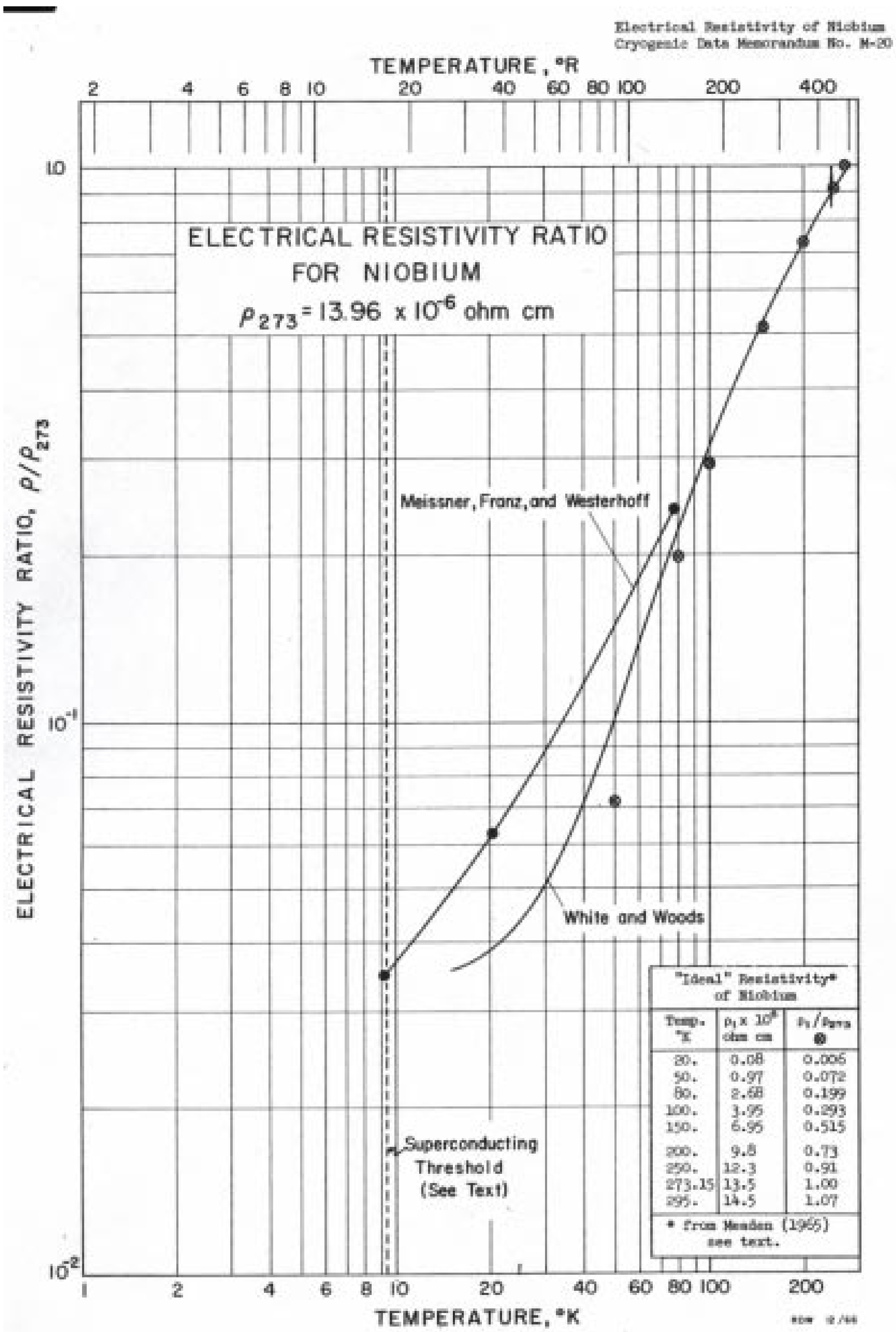

Figure 6: Nb electrical resistivity, as published in [3].

References:

[1] Parampage is a Fermilab program for Macintosh computers developed by R. E. Peters for displaying of IRM collected data. It has excellent data examination capabilities that do not interfere with the data acquisition activity.

[2] IRM is a Fermilab data acquisition system accessible via Internet Protocol.

[3] Hasan Padamsee, Jens Knobloch, Tom Hays "RF Superconductivity for Accelerators", John Wiley & Sons Inc, New York 1998, p.62.

[4] National Institute of Standards end Technology, http://www.boulder.nist.gov/div816/2002/StandardsForSuperconCharacter/

[5] NBS Technical Note 365, "Survey of Electrical Resistivity Measurements on 16 Pure Metals In The Temperature Range 0 to 273 K". page 85.