

RESISTIVITY RATIO OF NIOBIUM SUPERCONDUCTING CAVITIES\*

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Abstract

Resistivity measurements have been made on Nb cavities, as well as on Pb and Cu, at 296° K, 77° K, and 4.2° K by means of a contactless induced-current method. For superconductors, a constant magnetic field drives the material normal below the transition temperature. These measurements provide a simple means for initial material evaluation as well as a direct means of monitoring the effects of material parameters (purity, heat treatment, gas incorporation, etc.) on the electron-mean-free path. Approximate determinations of  $H_c$ ,  $H_{c1}$ , and  $H_{c2}$  can also be derived from these measurements. Normal state thermal conductivity and the Ginzburg-Landau parameter,  $\kappa$ , are calculated from the resistivity measurements.

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# RESISTIVITY RATIO OF NIOBIUM SUPERCONDUCTING CAVITIES

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Measurement of the resistivity ratio (between  $296^{\circ}$  K and  $4.2^{\circ}$  K) of Nb rf superconducting cavities is desirable for many reasons. Resistivity ratio measurements before and after vacuum firing can indicate the decrease in impurity content and other crystalline defects, as well as measure the increase in electron mean-free-path. The latter parameter is important in designing superconducting accelerators and in analyzing the results from superconducting cavities. The effects on electron mean-free-path due to irradiation, incorporation of gases, etc., can also be determined. The difficulty in measuring resistivity by ordinary means lies in the size and unwieldy geometry of a cavity. To date, no resistivity ratio measurements have been reported on Nb cavities or even on representative samples, though many speculations have been made regarding electron mean-free-path in cavities.

We have used a contactless, induced-current method for measuring resistivity similar to that used by others<sup>1,2</sup> for normal metals, with the addition of a constant magnetic field to drive the superconductor normal. As far as we know, our measurements are the first using this method on superconductors below their transition temperature, and on actual superconducting cavities.

With a constant magnetic field parallel to the pulsing field, the induced currents in the sample are perpendicular to the constant field, and the radially directed Lorentz force has no net force on samples having origin-reflection symmetry cross sections. However, there is an electrical coupling through the mutual inductance between the constant field coil and the pulsing field coil, which affects the decay time of the induced currents. We found that for practical purposes this cannot be made negligible.

A constant magnetic field applied perpendicular to the pulsing field eliminates the mutual inductance coupling. However, now the Lorentz force produces

a torque on the sample, tending to rotate the axis of the sample into parallelism with the constant field. Mechanical motion caused by this torque produces a time rate of change of flux through the pickup coil, causing a noise signal to be superimposed on the decaying induced current signal. Because of mechanical support constraints, the sample will oscillate about its equilibrium position, producing an oscillatory superimposed noise signal. The mechanical constraints can be made sufficiently rigid to minimize the oscillation amplitude, and hence the noise.

A small secondary pickup coil of  $N$  turns is placed around the sample conductor in the region where the resistivity,  $\rho$ , is to be determined. A pulsing solenoid encompasses both the pickup coil and the sample. If the solenoid is suddenly de-energized, the time rate of change of magnetic flux induces currents to flow in the sample to oppose this change in flux. From Maxwell's equations, the voltage induced in the pickup coil is:

$$V(t) = N \int \frac{\partial B}{\partial t} dA = N \int D \nabla^2 B dA, \quad (1)$$

where  $B$  is the magnetic flux density in the sample and  $D$  is the magnetic diffusivity. Solutions to Eq. (1) exist for various geometries.<sup>1</sup> The result they have in common and which should also be true for any geometry, is that at large times:

$$V(t) \propto N \exp(-t/\tau), \quad (2)$$

where the decay time  $\tau \propto 1/\rho$ . Where analytical solutions exist,  $\rho$  may be determined directly from  $\tau$ . In the case of complicated shapes, such as rf cavities, the resistivity ratio between room temperature and temperature  $T$  is simply:

$$\frac{\rho(296^\circ \text{K})}{\rho(T)} = \frac{\tau(T)}{\tau(296^\circ \text{K})} \quad (3)$$

The resistivity ratio  $r \equiv \rho(296^\circ \text{K})/\rho_0$  is one of the most sensitive determinations of impurities and other lattice defects, particularly for highly pure samples. (The residual resistivity,  $\rho_0$ , occurs for  $T \geq 4.2^\circ \text{K}$ , unless the lattice is extremely free of defects.) However, for our purposes, it is not necessary to obtain the absolute value of the resistivity precisely. When this is desired, any one of the following methods may be used to determine  $\rho(T)$  from Eq. (3) for complicated geometries. Since the resistivity at  $296^\circ \text{K}$  does not vary greatly from sample to sample of a given metal, one may (1) use either a literature value of  $\rho(296^\circ \text{K})$ , or (2) make a d. c. measurement on a representative rod; (3) when neither is feasible,  $\rho(296^\circ \text{K})$  may be obtained by comparison to measurements on a sample of a different material but identical geometry and known resistivity, such as OFHC copper. This experimentally gives the geometrical factor relating  $\rho$  and  $\tau$ . Using these three methods, a typical value of Nb resistivity at  $296^\circ \text{K}$  is  $1.6 \times 10^{-7} \Omega \text{m}$ . Even for simple geometries, such as cylinders, if the length-to-diameter ratio is less than 4 or so, the one-dimensional analytic solutions do not give the correct relation between  $\rho$  and  $\tau$ .

The shortest  $\tau$  measured with no sample in the coil was  $2.6 \times 10^{-5}$  sec., whereas the longest was 7.5 sec. with Pb at  $4.2^\circ \text{K}$ . Fast and reliable switching of the pulsing coil was made possible by construction of a liquid Ga vacuum switch.  $\tau$  was obtained from either a semi-log plot of the  $V(t)$  data from the oscilloscope trace or by matching the  $V(t)$  curve on a storage oscilloscope with a variable RC pulser which we made for this purpose. There was  $\pm 8\%$  agreement between  $\tau = RC$  and  $\tau$  from the semi-log plot, as well as between measurements.

Stock material in the form of solid Nb cylinders from which cavities are machined was measured. The resistivity ratio varied from 26 to 83, depending on impurity content and lattice structure, whereas the ratio to  $77^\circ \text{K}$  was ca. 5

for all these samples. A solid cylinder, 4.75 cm in diameter, 7.6 cm long, having  $r = 26$  was vacuum-fired at  $1800^{\circ}$  C for 16 hours and for another 31 hours at about  $2000^{\circ}$  C at  $10^{-9}$  Torr. This increased  $r$  to only 36. The initial impurity content for this sample is  $\sim 1000$  ppm with  $\sim 300$  ppm of Ta and Zr as the major impurities. This is typical of the as-received Nb stock. Since the resistivity measurement senses the entire bulk of the sample, and the decrease in impurity content is governed both by diffusion and surface evaporation, a much larger increase in resistivity ratio would be expected for a hollow cylinder or cavity.

A  $TE_{011}$  X-band cavity was made from this very same stock. It was vacuum-fired five times at pressures  $\sim 10^{-9}$  to  $10^{-8}$  Torr under conditions similar to those of the solid Nb cylinder. At this stage, the cavity was found to have  $r = 143$ , residual  $Q$  of  $Q_0 \sim 10^9$ , with a breakdown field,  $H'_p \sim 150$  Oe. It was subsequently refired at  $2200^{\circ}$  C for 50 hours with a final pressure of  $1 \times 10^{-9}$  Torr at  $2200^{\circ}$  C. After this treatment,  $r$  increased to 500, with  $Q_0 = 1.5 \times 10^9$  and  $H'_p = 260$  Oe. However, after high power operation,  $Q_0$  deteriorated to  $5 \times 10^8$ . The resistivity ratio may be higher near the surface within the penetration depth, due to a decrease in the concentration of impurities near the surface. However, for this cavity, having a 4.37-mm wall thickness,  $r$  near the surface may not differ substantially from the measured bulk value, considering the amount of high temperature vacuum-firing it received.

As has been shown analytically,<sup>3-5</sup> the  $Q$  and the breakdown field of a cavity may be dominated by trapped flux and/or other local irregularities. This is why there may be no simple correlation between  $r$  and  $Q_0$  and  $H'_p$ . Nevertheless, the resistivity measurements may be used as a simple means for initial material evaluation wherein substandard material with too high an impurity content can be

rejected. As has been pointed out,<sup>6</sup> there may even be an ideal normal resistivity, below or above which power losses would be increased in the cavity. Additionally, the normal resistivity is an important parameter in determining magnetic breakdown.<sup>4</sup>

Not only have resistivity measurements not been reported for a cavity or a representative sample, but neither have thermal conductivity measurements. The normal thermal conductivity

$$K_n = k_{1n} T \quad (4)$$

is derivable from the Wiedeman-Franz law

$$K_n = (2.45 \times 10^{-8} \text{ W-}\Omega/\text{K}^2) T/\rho \quad (5)$$

at least for the less pure samples. For the previously-described solid Nb cylinder, after its final firing, Eq. (5) gives  $K_n = (5.5 \text{ W/m}^2 \text{K}^2) T$ , which is reasonable.

Goodman<sup>7</sup> has derived an expression relating the Ginzburg-Landau parameter  $\kappa$  to  $\rho$ :

$$\kappa = \kappa_0 + 2.37 \times 10^6 \gamma^{1/2} \rho, \quad (6)$$

where  $\kappa_0 = 1.22$  is the value of  $\kappa$  for pure Nb<sup>8</sup> at 4.2° K,  $\gamma = 7.4 \times 10^2 \text{ j/m}^3 \text{K}^2$  is the electronic specific heat for Nb,<sup>8</sup> and  $\rho$  is the resistivity in  $\Omega\text{-m}$ . Using Eq. (6), we find that  $\kappa$  varies from 1.62 to 1.24 as tabulated in Table I.

Rosenblum et al.<sup>9</sup> relate  $H_{c2}$  to  $\rho$  on the basis of an empirically modified theoretical expression:

$$H_{c2} = 0.0321 T_c^2 (1 - t^2) (1.59 - 0.72t + 0.097 T_c \rho), \quad (7)$$

where they have  $\rho$  in  $\mu\Omega\text{-cm}$ ,  $H_{c2}$  in kOe, and  $t = T/T_c$ . Taking  $T = 4.2^\circ \text{K}$  and  $T_c = 9.1^\circ \text{K}$ , we obtain the values given in Table I.

These calculated values for  $H_{c2}$  agree well with the values obtained directly from the resistivity measurements on the solid Nb cylinder, as shown in Table I. ( $H_{c2}$  measurements were not made on samples Nb3 and Nb cavity A.) In the mixed state for Nb, as in the intermediate state for Pb, the decay time  $\tau$  varies as a function of the perpendicular magnetic field,  $H$ . When  $H_{c2}$  is exceeded for Nb,  $\tau$  is approximately independent of  $H$ , neglecting a slight magnetoresistive effect. Thus the threshold for constant  $\tau$  determines  $H_{c2}$ .

An experimental determination of the demagnetizing factor allowed a determination of  $H_{c1}$ . When the sample is totally in the superconducting state, the decay curve is very steep and constant up to  $H_{c1}$ —corresponding to the small amount of flux between the pickup coil and a penetration depth into the specimen. As soon as flux begins to penetrate the specimen, the decay curve alters and does not break quite as sharply, for both type I and II superconductors. Pb served as a calibration to determine the demagnetization factor, by using its known  $H_c$  value to calculate the field enhancement, and thus determine  $H_{c1} = 1.1$  kOe for the solid Nb cylinder, Nb1B. This agrees well with the value of 1040 Oe obtained from  $\kappa$  determined in our experiments, and the graph of  $\kappa$  vs  $H_{c1}$  in Harden and Arp's paper.<sup>10</sup>  $H_{c2}$  has been determined previously by others,<sup>9</sup> using ordinary d. c. resistivity measurements. This is the first time that either  $H_{c1}$  or  $H_{c2}$  has been determined by induced current resistivity measurements.

The electron mean-free-path,  $l_e$ , can be obtained from the resistivity measurements. Niobium has a complicated Fermi surface, and  $l_e$  varies with position on this surface. Goodman and Kuhn<sup>11</sup> have derived the following expression for the average value of  $l_e$  taken over the Fermi surface of Nb:

$$l_e = 3.7 \times 10^{-12} \Omega\text{-cm}^2/\rho \quad (8)$$

with  $\rho$  in  $\Omega\text{-cm}$ . Values of  $l_e$  are tabulated in Table I.

The induced current method for resistivity measurements offers exciting possibilities which might otherwise not be feasible. In addition to resistivity measurements on cavities and other unwieldy specimens, superconducting parameters such as  $\kappa$ ,  $H_{c1}$ , and  $H_{c2}$  may also be obtained. After vacuum firing, the material near the (nearly identical) inner and outer cavity surfaces may be relatively more pure than in the bulk, and may therefore show higher values of  $H_{c1}$  than the bulk. From our measured values of  $H_{c1}$  and the known correlation of  $H_c$ ,  $H_{c1}$ , and  $\kappa$ , we may deduce a value for  $\kappa$  in the near-surface layer. From either the  $H_{c2}$  or resistivity measurements, another value of  $\kappa$  may be determined for the bulk. From the two independently determined values of  $\kappa$ , our apparatus can, in principle, contribute to the knowledge of the radial impurity distribution.

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TABLE I

Measured and Calculated Properties of Nb Samples at 4.2° K

Sample	r	$\kappa$	$H_{c2}$ (R-A-G) Oe	$H_{c2}$ meas. Oe	$H_{c2} = \sqrt{2} \kappa H_c$ Oe	$H_{c1}$ Oe	$l_e$ Å
Nb1A	26	1.62	3770	3800	3660	990	600
Nb1B	36	1.51	3460	3600	3410	1040	830
Nb2	46	1.44	3280	3200	3250	1080	1060
Nb3	83	1.35	3000	-	3050	1100	1900
Nb cavity A	143	1.29	2850	-	2920	1150	3300
Nb cavity B	500	1.24	2700	2700	2800	1170	11,600

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