Unexpected Effect of Field Angle in Magnetoresistance Measurements of High-Purity Nb

L. F. Goodrich, T. C. Stauffer, J. D. Splett, and D. F. Vecchia

***Abstract*—We report on unexpected field-angle dependence of magnetoresistance measurements of commercial, high-purity Nb discovered during our study of residual resistivity ratio (RRR) measurements. The RRR value indicates the purity and the low-temperature thermal conductivity of the Nb and is used as a specification for superconducting radio-frequency cavities. The RRR is typically defined as the ratio of the electrical resistivities measured at 273 K and 4.2 K. One way to obtain the normal-state resistivity at 4.2 K is to measure the resistivity versus magnetic field at 4.2 K and extrapolate to zero field. The field-angle dependence was measured when a specimen was rotated while the field was transverse to the specimen current. The resistance changed by about 10% as the angle varied at 8 T and the local maxima and minima of the resistance were separated by about 30°. This was observed on three polycrystalline Nb bar specimens with nominal dimensions of $64\ \mathrm{mm} \times 6.7\ \mathrm{mm} \times 3.3\ \mathrm{mm}$ and voltage taps separated by 30 mm. A similar field-angle dependence was obtained on a polycrystalline Cu wire specimen. This phenomenon has implications for Nb RRR measurements and interlaboratory comparisons.**

***Index Terms*—Angular dependence, niobium, residual resistivity ratio, resistance measurement, superconductor.**

I. Introduction

THE value of residual resistivity ratio (RRR) indicates the purity and the low-temperature thermal conductivity of Nb and is often used as a material specification for superconducting radio-frequency cavities [1]–[3]. A typical specification is that the RRR has to be larger than 260 with an accepted limit to random error of 5% [4]. Our goal is to answer some fundamental questions about the best measurement of RRR and the relative differences associated with different measurement methods, model equations, and magnetic field orientations. This paper is a continuation of this work. Further experimental details were reported [5]. We hope to achieve a better understanding of the relative differences, as large as 10%, that have been observed in interlaboratory comparisons [4].

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The RRR is typically defined as the ratio of the electrical resistivities or resistances measured at 273 K and at 4.2 K (the boiling point of helium at standard atmospheric pressure). However, pure Nb is superconducting at 4.2 K, so the low-temperature resistance is commonly defined as the normal-state (nonsuperconducting state) resistance extrapolated to 4.2 K and zero magnetic field. The two methods to obtain the extrapolated normal-state resistance are: (1) measure the normal-state resistance as a function of field at 4.2 K and extrapolate to zero field (field-extrapolation method) [5]; and (2) measure the normal-state resistance as a function of temperature in zero field and extrapolate to 4.2 K (temperature-extrapolation method).

In this paper we focus on data for the field-extrapolation method. For this method, resistance versus magnetic field is measured in liquid helium. Because liquid helium boils near 4.0 K at the atmospheric pressure of our test site, all data are presented at 4.0 K rather than 4.2 K. The systematic differences in resistances between these two temperatures should be 0.5% or less. We also limit the magnetoresistance data presented here to the case of the applied field transverse to the specimen current in a radial-access magnet.

An unexpected field-angle dependence of the measurements occurs as the specimen is rotated while the field is kept transverse to the specimen current. A worm and worm-wheel gear set, located at the top of the cryostat, was used to rotate and hold the field angle. The resistance changed by about 10% as the angle varied at 8 T, and the local maxima and minima of the resistance were separated by about 30°. Because of the magnitude and strong angle dependence, this phenomenon has implications for Nb RRR measurements, interlaboratory comparisons, and any future consideration for developing a reference material for these measurements.

The effect was seen in three (nominal RRR value of 343 (#1), 414 (#13), and 362 (#14)) polycrystalline Nb bar specimens with nominal dimensions of $64\ \mathrm{mm} \times 6.7\ \mathrm{mm} \times 3.3\ \mathrm{mm}$ and voltage taps separated by 30 mm. We have not measured a specimen that did not show this effect. The 0° field angle was defined as the direction of the applied magnetic field when it is parallel to the wider face of the rectangular bar specimen. A similar field-angle dependence was observed for a polycrystalline Cu wire specimen. The Cu wire had a diameter of 1.02 mm, a length of about 70 mm, and had voltage taps separated by 40 mm. The nominal RRR of the Cu wire was about 677. The voltage connections to the specimen were made with pressure contacts in the case of Nb and soldered contacts in the case of Cu.

II. Procedure

All of the measurements presented were made with a transport current of about 4 A in a constant transverse magnetic field and fixed field angle. The minimum measurement sequence consisted of the specimen current starting at zero; then the current was ramped up and held for about 11 s at about 4 A; and finally, the current was ramped back to zero. Readings of specimen voltage and current were made before, during, and after each current cycle. Ninety-three readings, over about 1.8 s, were made during the initial and final zero-current regions. The settling time after the current returned to zero was at least 1.4 s. Five sets of 93 readings were made during the 11 s that the current was held constant. The settling time before the first reading with the current on was about 2 s. The thermoelectric and meter offset voltages were approximately canceled using the initial and final readings, assuming a linear change with time. The total time for this minimum measurement sequence was about 17 s. Five resistance (R) values were calculated using the five different sets of 93 readings during the finite current portion of the cycle. No significant time dependence appears within these five R values.

Measurements were also made with both positive and negative currents at 8 T for various angles to detect any possible Hall-effect voltages. No significant dependence of R with current direction was measured; therefore, no significant Hall-effect voltages were observed.

The reproducibility of the R measurements was limited by the reproducibility of the magnetic-field angle (θ). We quantify the reproducibility of the magnetic-field-angle by its standard deviation, which we estimate to be 0.5°. For measurements at 8 T and at the steepest portion of the curve, an angle change of 0.5° would result in a 0.25% change in R for a Nb specimen, and a 1% change in R for the Cu specimen. The measurements of R at a given field and angle had an average coefficient of variation (standard deviation divided by average) of less than 0.033%.

III. Results

Fig. 1 is a plot of R versus θ at 8 T and 4 K for Nb specimen #13. The local maxima and minima of R are separated by about 30°. The range of R was about 9.9% at 8 T relative to R (0°). Two sets of measurements are shown with different symbols. The first set of measurements was made with increasing angle setpoints started at near 0° and finishing at over 100°. The second set was taken with the angle decreasing between each set point to check reproducibility and to fill in data at different angles. The reproducibility was within the measurement uncertainty. There are at least five R values at each set angle, although the five symbols often appear as one because the readings were very repeatable. This is true for all figures. Table I summarizes the results. The R values listed in Table I were those measured near 0°.

Similar R versus θ measurements at 8 T and 4 K for Nb specimen #14 are shown in Fig. 2. Again the local maxima and minima of R are separated by about 30°; however, the minimum near 60° is significantly lower than the minimum near 0°. The local maxima near 30° and 90° are also quite different. The range of R was about 13.9% at 8 T relative to $R(0°)$.

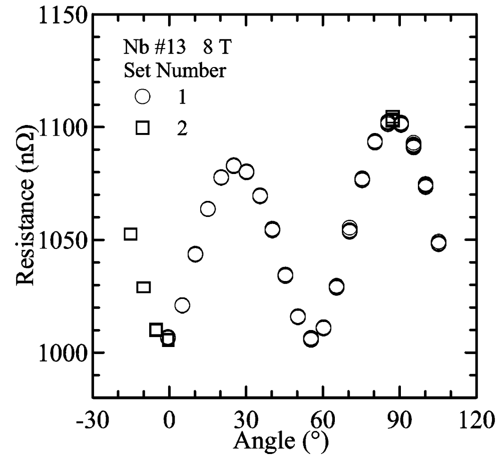

Fig. 1. Pure Nb specimen #13's resistance versus magnetic-field angle at 8 T. Two sets of measurements were made to check reproducibility and fill in data at different angles.

TABLE I
Summary of Properties

Specimen	RRR	R(0 T) range	R(1.6 T) range	R(4 T) range	R(8 T) range
Nb #1	343	581 nΩ[a]	671 nΩ	852 nΩ	1115 nΩ
		–	1.1 %	2.1 %	7.8 %
Nb #13	414	474 nΩ	565 nΩ	746 nΩ	1007 nΩ
		0.1 %	0.3 %	2.6 %	9.9 %
Nb #14	362	553 nΩ	645 nΩ	833 nΩ	1101 nΩ
		0.3 %	1.7 %	4.7 %	13.9 %
Cu	677	1126 nΩ	4389 nΩ	8339 nΩ	13922 nΩ
		0.3 %	6.3 %	13.5 %	21.4 %

[a]For the Nb specimens, R values at 0 T were extrapolated.

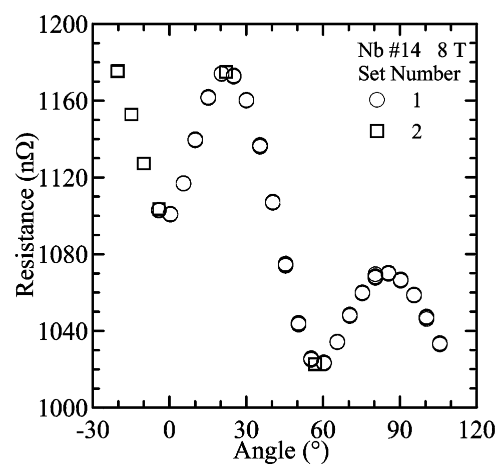

Fig. 2. Pure Nb specimen #14's resistance versus magnetic-field angle at 8 T. Two sets of measurements were made to check reproducibility and fill in data at different angles.

A plot of R versus θ at 8 T and 4 K for the Cu specimen is shown in Fig. 3. The adjacent local maxima and minima of R appear to be separated by about 5° in some cases; however, on a broader angle scale, there appear to be maxima and minima separated by 30°. Four sets of data were taken to try to fill in the finer structure of the angle dependence. Where these points were filled in, the dependence is shown to be reproducible with continuity to the level expected due to the uncertainty of the

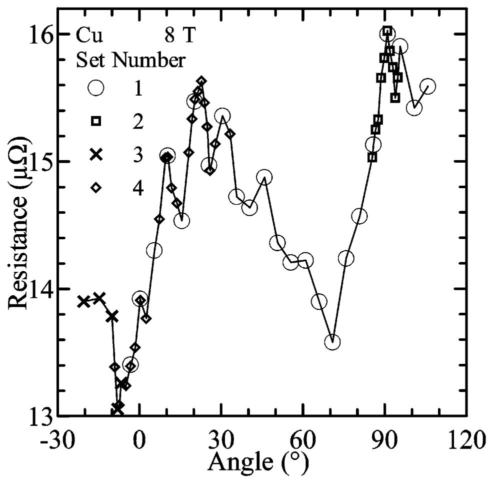

Fig. 3. Cu specimen's resistance versus magnetic-field angle at 8 T. Four sets of measurements were made to check reproducibility and fill in data at different angles. A line was drawn through all points to show the trend.

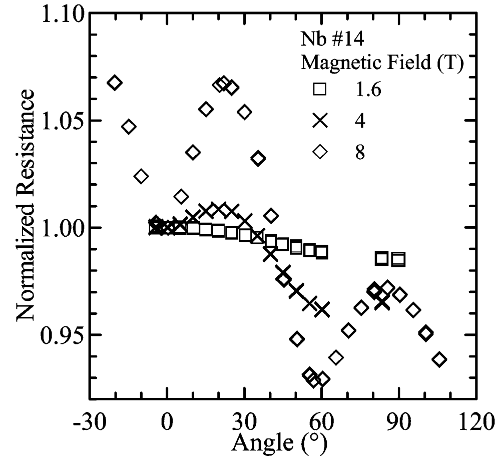

Fig. 5. Pure Nb specimen #14's normalized resistance versus magnetic-field angle at various fields. The resistance was normalized at each field by the measured value near 0°.

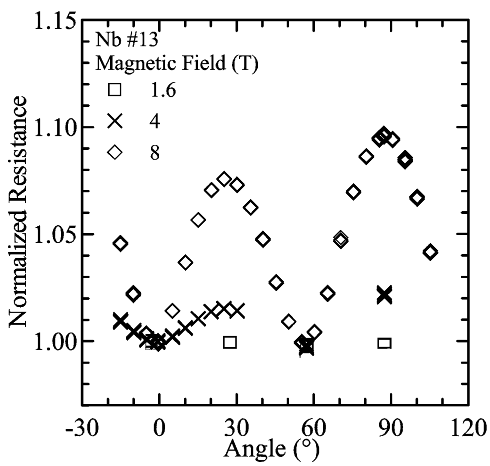

Fig. 4. Pure Nb specimen #13's normalized resistance versus magnetic-field angle at various fields. The resistance was normalized at each field by the measured value near 0°.

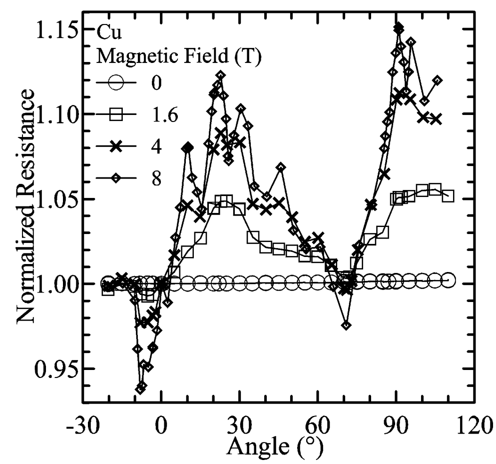

Fig. 6. Cu specimen's normalized resistance versus magnetic-field angle at various fields. The resistance was normalized at each field by the measured value near 0°. A line connects the points of each field to show the trend.

angle variable. The range of R was about 21.4% at 8 T relative to $R\ (0^\circ)$.

For all specimens, R versus θ measurements were also made in magnetic fields of 1.6 and 4 T. A transverse field of 1.6 T was about the lowest field in which the Nb specimen was completely in the normal state. Normalized R versus θ plots for two Nb and one Cu specimen are shown in Figs. 4–6. The Cu specimen was measured in 0 T to verify the absence of any significant field-angle dependence with no applied field. In all cases, R at each value of field was normalized by the measured resistance near 0°. The plots indicate that there is less dependence on angle for lower magnetic fields than for higher magnetic fields. The normalized R versus θ plot for a third Nb specimen (#1) is not shown to save space, but the curves were similar to those of specimen #13 and these data are summarized in Table I. For each of the three Nb specimens, the range of R values at 1.6 T was less by at least a factor of 7 than the range at 8 T.

We did compare R values extrapolated to 0 T for two different sets of magnetoresistance data. For Nb specimen #13, we acquired data with the angle set near 0° and another set near 90°. The extrapolated $R(0\ \mathrm{T})$ [5] for these two angles was within 0.1%. For Nb specimen #14, we acquired data with one angle set near 0° and another set near 60°. The extrapolated $R(0\ \mathrm{T})$ for these two angles was within 0.3%. These results indicate that the RRR can still be measured precisely at different orientations.

IV. Discussion

The measured dependence on field angle was not expected for polycrystalline specimens. In trying to develop a theoretical understanding of the magnetoresistance of polycrystalline copper, it was concluded in [6] that the grains of a drawn and annealed polycrystalline copper wire were not randomly oriented. Two different crystal orientations were likely parallel to the wire axis. This argument can plausibly be extended to nonrandom orientations of grains transverse to the wire axis due to deformation texturing and the preference of lower-energy grain alignments. The Nb bar specimens will have a different texture because of the different crystal structure and the rolling deformation; however, they are still likely to be textured. Thus, the measured field-angle dependence may be due to a nonrandom grain alignment for the section between the voltage taps in both drawn and annealed Cu and in rolled and annealed Nb. We have concluded that the observed angle dependence was not likely an experimental artifact, such as the Hall-effect discussed earlier.

The angle dependence effect has implications for magnetoresistance measurements. The first is that reproducibility in interlaboratory comparisons could be seriously degraded by angular variations. A second is that there is no unique shape for the curve of R versus magnetic field. Since for the same specimen, R at 1.6 T varies by less than 1.7% and at 8 T can vary by more than 13%, the detailed shape cannot be unique. Thus, the fitting routine has to be robust.

V. Conclusion

We measured magnetoresistance of high-purity Nb as a function of magnetic-field angle while keeping the magnetic field perpendicular to the specimen current. We measured unexpected dependence on field angle in polycrystalline Nb and Cu specimens. The Nb resistance changed by about 10% with angle at 8 T and the local maxima and minima of the resistance were separated by about 30°. A plausible source of this effect is nonrandom grain alignment, which may be typical of all deformed and annealed specimens. Angle dependence makes it more difficult to reproduce measurements and results in nonunique magnetoresistance curves. However, it is possible to extrapolate measurements at different angles and obtain nearly the same residual resistivity ratio for a given specimen.

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