A New Approach for RRR Determination of Niobium Single Crystal Based on AC Magnetic Susceptibility

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INTRODUCTION

Residual resistivity ratio (RRR) value is an important characteristic of material purity.

AC magnetic susceptibility of a number of single crystal niobium samples for different orientations of type $\langle 100 \rangle$, $\langle 011 \rangle$, $\langle 111 \rangle$ and treatments (BCP 70, 150 μm , annealing 800°C/2h) were measured.

The RRR value was determined on base of these results using a relation between the imaginary part χ'' of AC magnetic susceptibility at low frequency f of AC magnetic field and resistivity ρ of the sample: $\chi'' = k \cdot f / \rho$.

Main principles of RRR determination

The AC susceptibility caused by eddy current can be expressed for spherical sample in terms of its radius a , and the skin penetration depth δ :

$$\chi_{AC} = \chi' + i\chi''$$
$$\chi' = \frac{9}{4} \left(\frac{\delta}{a} \right) \frac{[\sinh(2a/\delta) - \sin(2a/\delta)]}{[\cosh(2a/\delta) - \cos(2a/\delta)]} - \frac{3}{2}$$
$$\chi'' = \frac{9}{4} \left(\frac{\delta}{a} \right) \frac{[\sinh(2a/\delta) + \sin(2a/\delta)]}{[\cosh(2a/\delta) - \cos(2a/\delta)]} - \frac{9}{4} \left(\frac{\delta}{a} \right)^2$$

$$\delta = 1/(\pi\mu_0\mu\sigma f)^{0.5} = (\rho/(\pi\mu_0\mu f))^{0.5}$$

$\mu_0 = 4\pi \times 10^{-7}$ H/m; μ - the relative permeability; ρ - resistivity; f - frequency.

AC method: at low f - χ'' can be expressed as $\chi'' = A1 + A2 \cdot f$. In homogeneous sample $A1=0$, $A2=k \cdot \sigma$ ($k=\text{const}$); $\sigma = 1/\rho$; $1/A2 = \rho/k$; σ - electrical conductivity

$$\chi'' = k \cdot f / \rho$$

$$RRR = \rho_{300K} / \rho_{4.2K}$$

• Magnetic susceptibility of superconductors and other spin systems, Ed. By Robert A. Hein et. al., Plenum Press New York, 1991, page. 213 [A. F. Khoder, M. Gouach, Early theories of χ' and χ'' of superconductors for controversial aspects]

Single crystal samples

- Sample N1 (as delivered)
- Sample N2, BCP, 70 μm
- Sample N3, BCP, 150 μm

$800^\circ\text{C} / 2h$

The single crystal samples of company Heraeus have been used. The samples were cut out using EDM method.

Magnetic field applied along directions of type $\langle 100 \rangle$, $\langle 011 \rangle$, $\langle 111 \rangle$

Equipment

The Quantum Design **MPMS 5XL** SQUID Magnetometer uses a (SQUID) detector is extremely sensitive for all kinds of AC and DC magnetic measurements.

Magnetic moments down to 10^{-8} emu ($G \cdot cm^3$) (10^{-11} Am²) can be measured. The **MPMS** has a temperature range between 1.9 K and 400 K, the superconducting magnet can reach magnetic fields up to 5 T.

Multiple functions make possible in particular following:

- A supplement for measuring anisotropic effects of magnetic moments
- An addition for measuring electrical conductivity (magneto-resistance) and Hall constant
- AC susceptibility measurements which yield information about magnetization dynamics of magnetic materials

AC-method: $h = h_a \sin(2\pi f)$, h – intensity of AC magnetic field, h_a – amplitude value of h , f – frequency
 $h_a = 0.1 - 4$ Oe; $f = 3 - 1000$ Hz

Measuring contour

Squid response

Superconducting solenoid for DC fields + copper coils for AC fields

Sample N1 (as delivered)
magnetic field along $\langle 111 \rangle$

frequency extrapolation

Frequency dependencies of imaginary part of AC-susceptibility for different values of applied magnetic field. At low frequency at $B < 3$ T observed the scattering of the points (left figure). At $B \geq 3$ T change of the curve slope (right figure).

Sample N1 (as delivered)
magnetic field along $\langle 111 \rangle$, $\langle 110 \rangle$, $[100]$

At $B \geq 3$ T – Kapitza linear law - $R=K*f(B)$ (normal conducting state): $1/A2$ [RRR] ($T=2K$, $B=0$) = 3532, at $T = 300$ K: RRR($T=300K$, $B=0$) = 1095290

$\langle 111 \rangle$ $B=0$ RRR = **310**;

$\langle 110 \rangle$ $B = 3$ T: RRR = **270**; $B = 0$ RRR $\cong$ **280 – 300**;

$[100]$ $B = 3$ T: RRR = **260**; $B = 0$ RRR $\cong$ **280 – 300**;

frequency extrapolation

RRR (4-point DC method, $I \parallel [110]$, as delivered) = 269
RRR (4-point DC method, $I \parallel [111]$, as delivered) = 280
- good correlation with current results

$$RRR = \frac{1/A2_{300K}}{1/A2_{2K}}$$

Sample N2, 70 μ m BCP 800°C/2h annealing,
magnetic field along [100], <011>, <111>

frequency extrapolation

Frequency dependencies of imaginary part of
susceptibility at B=0; 3T (T=2K; 300K). Angle
between the curves at B=0; 3T (T=300K) shows
the small magnetoresistivity.

B=3 T; B	RRR
[100]	169 (207 B=0T)
<011>	198
<111>	226

Sample N2, 70 μ m BCP 800°C/2h
annealing, magnetic field along [100]

temperature extrapolation

RRR=166 (B || [100]) by temperature extrapolation method

RRR=169 (B || [100]) by frequency extrapolation method

1/A2 dependence of T^3

correlation of RRR values obtained by frequency and temperature extrapolation

Sample N3, 150 μ m BCP 800°C/2h
annealing magnetic field along [100]

At $B \geq 1.5$ T curve $1/A2$ vs B follows
the Kapitza law: $R=K*f(B)$

Similar bend at definite magnetic
fields was observed on DC
magnetic resistance.

This bend is probably caused by
transition from SC to normal
conducting state of niobium

Summary

- One more approach for determination the RRR values by means of AC-susceptibility examined
- RRR values for main crystallographic orientations of Nb single crystals are obtained
- Good correlation with results for RRR obtained by 4 point DC method
- The magnetic field dependence of value R follows to the Kapitza law $R=K f(B)$
- The advantage of this method is possibility to measure simultaneously the different magnetic and transport properties such as a very small values of resistivity. Determination of resistivity can be done by taking into account the size and the shape of the sample.