A NEW INDUCTIVE METHOD FOR MEASURING THE RRR-VALUE OF NIOBIUM

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Abstract
A new method for measuring the RRR-value of niobium is presented. The principle of the measurement uses low frequency induction in a niobium sheet placed close to a pair of coils. In contrast with the usual resistive method, the present one gives information on the local value of the RRR, with a spatial resolution of the order of 1 cm. In addition, it is non destructive, thus opening the way to mapping RRR measurements on cavities. This tool will permit the measure of RRR inhomogeneities in cavities due to sheet forming or heat treatments, and the systematic check of the quality of weld seams.

1. INTRODUCTION

Niobium purity is a major issue for the technology of superconducting cavities. A clear correlation between cavity performance and material purity has been established (ref.1). In this context, tools enabling the characterization of this purity are especially useful. So far, the most convenient criterion for the evaluation of niobium purity is the residual resistance ratio (RRR) defined as $RRR = \rho_{300K} / \rho_{4K}$. The room temperature resistivity of niobium is a constant (ρ_{300K}=1.45 10^{-7} Ω.m) and the residual resistivity ρ_{4K} depends primarily on the material purity and, to a lesser extent, on its crystalline state (ref. 2). In practice, for niobium, the low temperature resistivity is measured either at 4 K in the normal conducting state, or at 10K in order to avoid the onset of superconductivity. ρ_{10K} is not very different from ρ_{4K}, and appropriate corrections can be applied to deduce the true RRR from ρ_{10K} .

Usually, the Niobium RRR is measured by "4-wires" resistivity measurements from elongated metal samples at cryogenic temperature. This very widely used technique has two severe drawbacks : it is non local, since the measured quantity is the resistance averaged over the sample length (typically 10 cm). Moreover, it is destructive, since the measured strip has to be cut out from the bulk. This practically precludes the application of the method to measure the RRR on real accelerating cavities, even though RRR measurements on finished accelerator structures would be most valuable.

In order to overcome these shortcomings, we developed a new method for measuring non-destructively the local RRR-value of Niobium, using low frequency induction.

2. PRINCIPLE OF MEASUREMENT

The mutual inductance of a pair of coils can be modified by the presence of a neighbouring piece of metal. The magnitude of the modification depends on the details of the geometry, and on the metal conductivity.

In the present case, the pair of coils is placed close to the niobium surface (fig. 1).

A known AC current is injected in the primary coil. The AC voltage V induced in the secondary coil is measured. In order to minimize systematic errors, V is measured both with the niobium sheet in the normal conducting state at 10K and in the superconducting state. The measurement in the superconducting state being used as reference, the ratio V_N/V_S is plotted as a function of frequency (fig. 2).

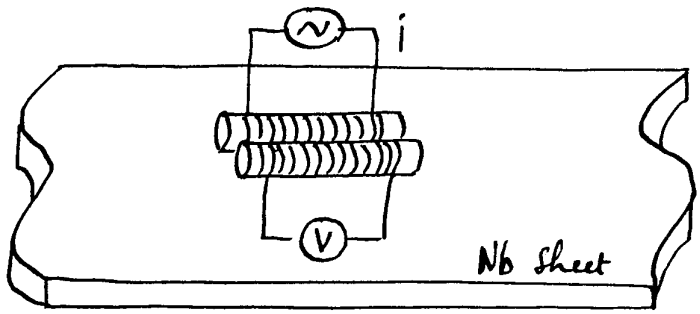


Fig. 1 Experimental setup

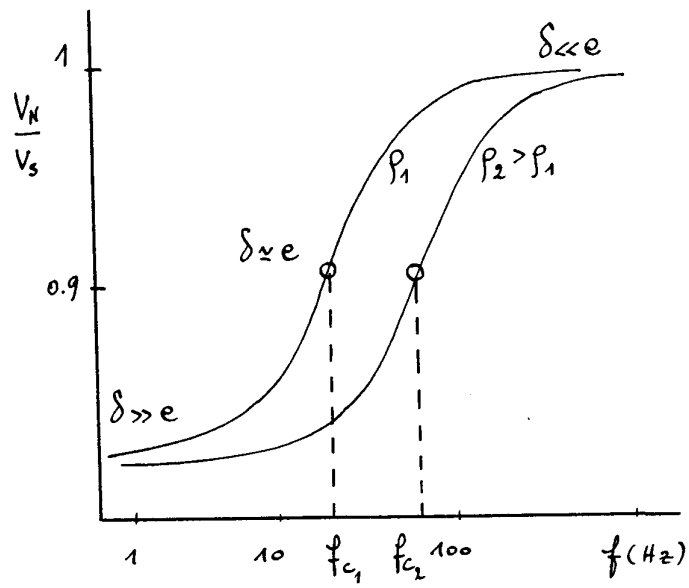


Fig. 2 The ratio V_N/V_S as a function of frequency.

In the normal conducting state, the eddy current induced in the niobium sheet circulates on a skin depth $\delta = \sqrt{\frac{2\rho}{\omega\mu_0}}$ (eq. 1). δ vanishes for high frequencies. The electromagnetic wave is then totally reflected, like in the superconducting state, where the current circulates on the negligibly small superconducting penetration depth λ. The ratio V_N/V_S thus approaches 1 for high frequencies. At very low frequency, the skin depth becomes larger than the Niobium sheet thickness, and the sheet becomes transparent to the AC electromagnetic field. V_N/V_S then saturates at a constant value, smaller than one.

Between these two extreme limits, the curve V_N/V_S (f) has an inflexion for a frequency f_c such that the skin depth δ is of the order of the sheet thickness e. For a given coil geometry and sheet thickness, the curve V_N/V_S (f) depends only on the material RRR. Considering two

metal sheets of equal thickness with resistivity ρ_1 and ρ_2 respectively, it can be seen from eq. 1 that the penetration of the magnetic field is the same in both sheets for two frequencies f_1 and f_2 such that $f_1 / f_2 = \rho_1 / \rho_2$. More generally, these two sheets have homothetic V_N/V_S (f) curves : V_N/V_S (f) (metal 1) = V_N/V_S (f.ρ_2 / ρ_1) (metal 2). The measurement of the experimental V_N/V_S (f) curves for both metals can thus give the ratio ρ_1 / ρ_2. Instead of two different sheets, one single metal sheet can also be measured at 300 K and at 10K. The frequency ratio between the curves V_N(300K) /V_S (f) and V_N(10K)/V_S (f) then yields ρ_{300K} / ρ_{10K}, i.e. the RRR-value of the metal sheet. The above considerations are completely independent of the geometrical arrangement of the coils. This method of measurement of the RRR is thus (to some extent) free from systematic errors.

3. EXPERIMENTAL DETAILS

Choice of the coils and geometrical arrangement

Two key components of the device are the primary and secondary windings. Special care was taken in the choice of these coils. The following criteria were used :

-Small coils, for a good spatial resolution.

-The useful part of the field created by the coils is the one located close to the Niobium surface. The stray field must thus be as large as possible, for both primary and secondary windings. Flat coils were chosen to this end.

-The voltage induced in the secondary coil is proportional to its inductance L. For a large signal to noise ratio, L should be as large as possible.

-L may also vary with temperature, causing undesirable variations in the voltage induced in the secondary coil. The choice of a coil having a low temperature coefficient is important.

The chosen coils (reference SIGMA 289.85880 , L = 1 mH, R(300K) = 37 Ω, R(10K) = 1 Ω, length 10 mm, diameter 4 mm) meet these requirements, and have the additional advantage of being commercially available at low cost. For simplicity, the same component was used for the primary and the secondary coils.

The coils have to be on the same side of the sheet. This arrangement is desirable if one wishes to use the device on cavities. The geometrical disposition of the coils with respect to the sheet was chosen so as to maximize the influence of the metal sheet. Several arrangements were tried, with coil axis perpendicular or parallel to the sheet plane, and coaxial or parallel coils. A satisfactory arrangement was found with parallel coils, with their axis parallel to the niobium surface, the coils lying as close to each other as possible (fig. 1). In this geometry, a crucial parameter is the distance between the coil doublet and the niobium surface. In order to maximize the influence of the underlying metal, this distance should be maintained as small as possible. The influence of this distance has been investigated. As can be seen from fig. 3, the signal decreases when the the coil-sheet distance increases. The shape of the V_N/V_S(f) curve thus varies when d varies (fig. 4), but the critical frequency f_c stays the same, so systematic errors induced by changes of the coil-sheet distance between the warm and cold measurements will probably be small.

Excitation of the primary coil was made with an AC generator (ref.SEFRAM 4434) with an output level of the order of 10 volts. The detected voltage across the secondary coil was of the order of 0.1 mV at low frequency and 300 mV at high frequency.

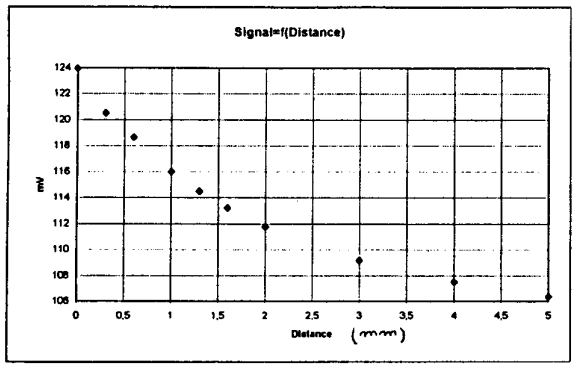
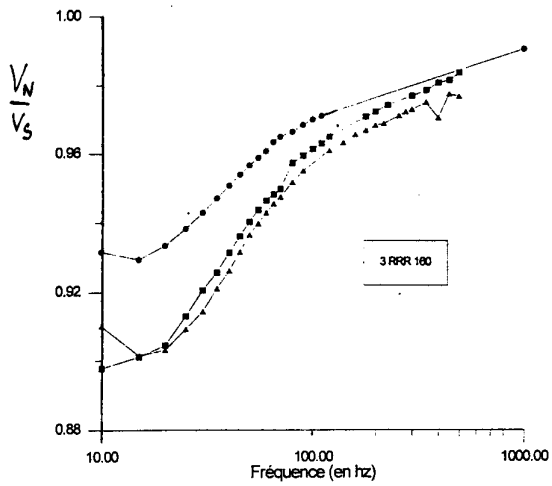


Fig. 3 Signal variation with distance between the coils and the metal sheet (T=300 K, F=1000 Hz).



Fc (en hz)	Température (en K)	RRR
58.3	10	165
62.5	10	158
60.7	10	153

Fig. 4 Result of three independent measurements on the same sample, with various distances between the coils and the metal sheet

4. ERROR BARS

Sensitivity of RRR measurement to temperature errors

During the normal state resistivity measurement, the sheet is maintained above the critical temperature, i.e. at about 10 K in the case of Niobium. This resistivity $\rho_n(T)$ varies with temperature as follows :

$$\frac{1}{RRR} \approx \frac{\rho_n(T)}{\rho_n(300K)} - 4.10^{-7} T^3$$

This gives the following error evaluation :

$$\frac{dRRR}{RRR} \approx -\frac{d\rho_n}{\rho_n} + 1.210^{-6}.RRR.T^2.dT$$

The RRR measurement could thus be affected by errors on the temperature evaluation. The temperature of the sheet is controlled with a typical uncertainty of less than 1 degree. This corresponds to a relative uncertainty of 2 or 3 % on the RRR-value.

Systematic errors of the RRR measurement

Several Niobium samples of known RRR-value have been measured by the inductive method described above (fig. 5). The response curves $V_N/V_S(f)$ are fairly different for the different samples, so the discriminating power of the measurement will be good. The main limitation comes from the dispersion of the V_N/V_S values at various frequencies. From the dispersion apparent in the curves of fig. 5, we estimate that the RRR-value of the sample can be evaluated with an error of the order of 5% for RRR-values of 200.

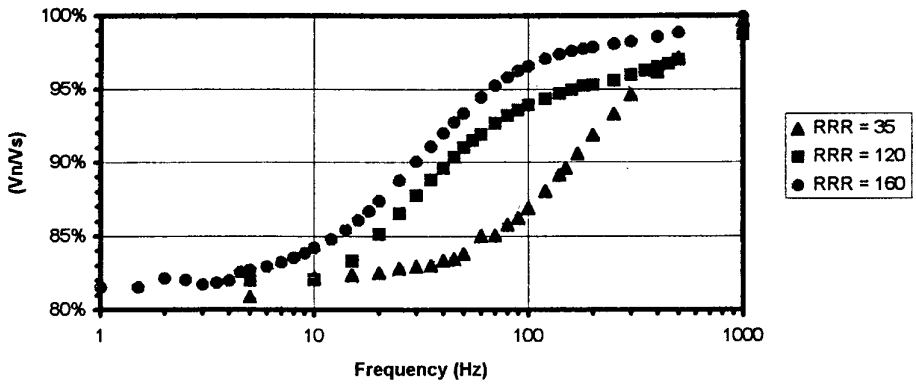


Fig. 5 VN/VS(f) for three Niobium samples of different RRR-values

Reproducibility

The same sample was also measured three times in three independent experiments, with a purposedly different distance between coil and sheet (fig. 4). The three corresponding $V_N/V_S(f)$ curves appear to be rather different, but the RRR-values which can be extracted from the determination of the critical frequency f_c agree with each other within 5%.

From the analysis of the three sources of error (temperature uncertainty, systematic errors, reproducibility) mentioned above, we conclude that the overall relative precision of the RRR measurement is about 5% for RRR-values of 200. This is confirmed by the comparison of inductive and resistive measurements of the RRR of several samples : both methods agree within 5%.

5. CONCLUSION

We believe that the inductive method of RRR measurement described above is a valuable new tool for characterizing superconducting material. Its main domain of application will probably be the control of the RRR homogeneity of the Niobium sheets supplied by industry, and the local RRR-value of fabricated cavities. For this last application, we are presently preparing a rotating arm to make a complete RRR mapping of a cavity. This arm will use the same mechanical arrangement as the temperature mapping arm in use in our laboratory. Given the size of the coils and the geometry of the system, the expected spatial resolution will be of the order of 1 cm . This tool should permit to check the RRR modifications due to sheet forming, heat treatments, and to measure the local RRR of weld seams.

6. REFERENCES

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