NEW RESULTS ON RF PROPERTIES OF SUPERCONDUCTING NIOBIUM FILMS USING A THERMOMETRIC SYSTEM

M. Fouaidy, IPN Orsay (CNRS/IN2P3-Univ. Paris XI), France

P. Bosland, M. Ribeaudeau, S. Chel, M. Juillard, SEA/DAPNIA/DSM CEA Saclay, France

Abstract

A cylindrical bulk Niobium cavity is used for measuring, with a removable end-plate method, the surface resistance R_S of superconducting thin films (Nb or NbTiN) sputtered onto copper disks (Nb/Cu). An alternative device, for characterization of the RF properties at two frequencies (4 GHz: TE011 mode, 5.6 GHz: TE012 mode) of Nb/Cu samples was developed. The main feature of this technique, which is based on vacuum insulated surface thermometry, is an improved accuracy and sensitivity as compared to the usual RF method. Precise calibration of samples RF losses is performed with a DC heater and 24 thermometers mounted on the back-side of the test-disk and placed inside a vacuum can. This thermometric method along with a thermal model was used for studying the absolute R_S distribution of several test-samples (bulk Nb and thin Nb films). This new facility allows also the determination of all the thermal parameters involved in the model (substrate thermal conductivity and heat transfer coefficient at the solid-LHe interface). After validation runs, the RF properties of several Nb/Cu samples were successfully studied with this device. Interesting data were obtained and analyzed. We investigated in particular, the effect of the Nb film and Cu roughness resulting from different substrate surface preparations, on the RF properties of the samples.

1 INTRODUCTION

SRF cavities based on superconducting thin films (Nb or NbTiN) sputtered onto OFE copper substrate are still considered as an alternative solution to bulk niobium cavities for future high energy accelerators and colliders. Such cavities offer several economical and technical advantages as compared to bulk niobium structures : lower BCS losses, no quench or thermal breakdown, no magnetic shielding needed, cost effective, reduced sensitivity to Lorenz force detuning and microphonics… However the RF performance of Nb films cavities especially at high accelerating fields (i.e $E_{acc} > 15$ MV/m) are still much lower than those obtained with bulk niobium cavities. More precisely, the RF losses or residual surface resistance of Nb films increases strongly with E_{acc} : the unloaded quality factor Q_0 decreases typically by one order of magnitude when E_{acc} is increased from ~0 to 25 MV/m at $T \cong 1.7$ K. In order to understand this limitation and to get more insight into the

physics of this phenomena, we investigated the RF properties of sputtered superconducting niobium films.

A dedicated device was developed to perform the measurement of these properties on samples.

2 EXPERIMENTAL ARRANGEMENT AND VALIDATION TESTS

2.1 Purpose

Due to the lack of accuracy and sensitivity of the usual end plate replacement method for measuring RF surface resistance R_S at 4.2 K of sputtered superconducting niobium films it was necessary to develop a new technique. Moreover the main advantages of the thermometric method briefly described here after are : a) it allows local and overall measurement of R_S, b) absolute measurement (no reference disk needed), c) measure solely the test-sample RF losses (i.e extra RF losses in the cavity, indium gasket, RF couplings ports are excluded).

2.2 Experimental set-up and procedure

The experimental set-up and measurement procedure were already described in a previous paper [1]. A picture of the experimental arrangement is shown in Fig. 1 and a close view photograph of the thermometric system is presented in Fig. 2.
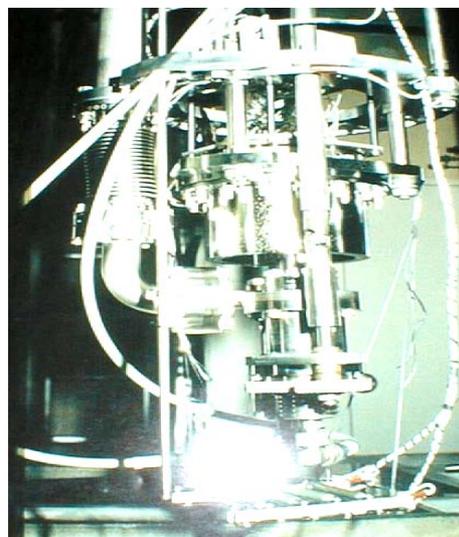

Figure 1: The experimental set-up showing the cylindrical TE011 cavity with the thermometric system.

The apparatus consists of cylindrical bulk niobium TE011 cavity (height : 66 mm, I.D : 110 mm) with a removable end plate (sample to be tested: disk of 126 mm diameter) and the vacuum insulated surface thermometric system including a calibration heater (DC Joule heating). The 24 removable thermometers, which are distributed as shown in Fig. 2 are pressed against the sample back-side of the test-sample by means of individual copper beryllium springs and placed inside a vacuum can.

Figure 2 : The thermometric system including the thermometers array and the calibration heater mounted of the backside of a Nb/Cu sample.

Briefly, the experiment is performed in three steps [1-2] : 1) measurement of the RF surface resistance R_S of the sample versus the applied surface magnetic field H_S at a fixed temperature T by the usual end-plate replacement method (RF measurement), 2) calibration Resistance R vs. Temperature T of the thermometers with no heater power (Q=0) in the temperature range 1.5 K-4.2 K, 3) thermometers thermal response ΔT vs. Q, 4) surface temperature distribution ΔT on the sample as function of H_S.

2.3 Validation tests

The first results [1] have shown that the thermometric method lead to surface resistance values in good agreement with those obtained by the usual RF method. An example is illustrated in (Fig. 3-Fig. 4).
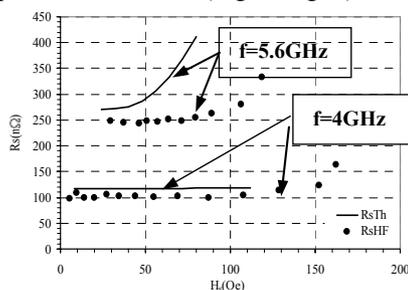

Fig. 3 Bulk Niobium R_S data measured by the two methods for two frequencies at 1.7 K *(solid dots : usual RF method, solid line: thermometry)*
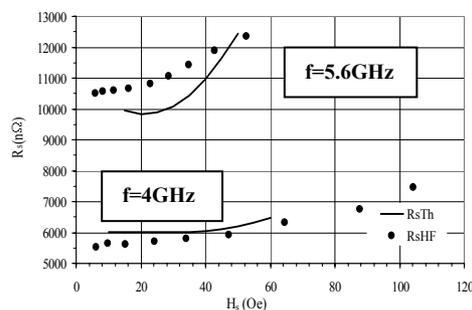

Fig. 4: Bulk Niobium R_S data measured by the two methods at 4.2 K *(solid dots : usual RF method, line: thermometry)*

Finally the experimental data on sputtered Nb films [2] shows also that the thermometric method is more accurate (factor 3) than the usual RF method.

3 TEST RESULTS ON SPUTTERED NIOBIUM FILMS

Among various parameters (sputtering conditions for example [2]) we investigated in particular, the effect of the Cu substrate roughness at the μm scale (~niobium film thickness) resulting from different substrate surface preparations, on the RF properties of the samples.

3.1 Preparation of the substrate surface

The growth phenomena, nucleation process and crystalline structure of the sputtered niobium film depend on the morphology of the copper substrate surface. More precisely the Cu surface roughness induces geometric defects in the Nb film during sputtering process [3]. Moreover a previous study have shown [4] a clear correlation between the density of geometric defects (surface peaks) on the surface of the Cu substrate and the slope of the RF characteristics R_S vs. H_S. However these features [4] were observed on Nb/Cu samples which was not produced in the same conditions in terms of sputtering parameters. As our purpose is to study solely the effect of Cu roughness, all the Nb/Cu samples concerned here after were prepared using the same optimal values [2] for the sputtering parameters (i.e argon pressure, sputtering power,…). The copper disk surface is prepared [2] prior to sputtering Nb films according the following process : 1) ultrasonic cleaning in a hot alkaline bath, 2) pure (resistivity : 18 MΩ) water rinsing, 3) surface polishing (chemical etching or mechanical polishing or electro-polishing), 4) pure water rinsing, 5) deoxidizing (sulfamic acid with water) 6) ultrasonic cleaning in pure water bath, 7) fast drying in filter air. Notice that all the copper disks were machined

from the same Cu sheet. Four different chemical etching baths methods were studied. The main characteristics of three of them are summarized in Table 1 (C_j is the molar concentration of the 'j' chemical specie).

Table 1 : Main parameters of the chemical etching baths
*(*EDTA : Ethylene Diamin TetrAcetic Disodic)*

Bath	SN6	SN2	SN10
C_{H2S04} (M)	5.8	7.7	4.8
C_{HNO3}(M)	0.87	1.15	0.72
C_{HCl}(M)	$1.6\ 10^{-2}$	$1.0\ 10^{-2}$	$1.3\ 10^{-2}$
C_{CuS04}(M)/EDTA*(M)	0.3	0.24	0.1/0.2 *
θ (° C)	25	35	23
Etching duration (min)	105	40	60
Removed layer (µm)	450	150	50
Roughness Ra (µm)	0.10	0.025	0.015

The fourth chemical etching bath (SSCR2), which include two salts, has the following composition : C_{H2S04} = 1.8M, $C_{HS03NH2}$ =1.15M, $C_{K2CR207}$= 0.05M. It must be stressed that Ra was measured over an area of 10 µm.

3.2 Substrate surface roughness

The measured roughnesses Ra are 0.015 µm and 0.01 µm for (SSCr2 + SN10) etching and electro-polishing respectively. For the other etching baths see Table 1. Note that SSCR2 bath alone leads to Ra= 0.15 µm. These results show that the substrate roughness depends strongly on the chemical treatment. The observed morphologies of the substrate surface after the above treatments as well as the sputtered niobium films are presented in Fig. 5.
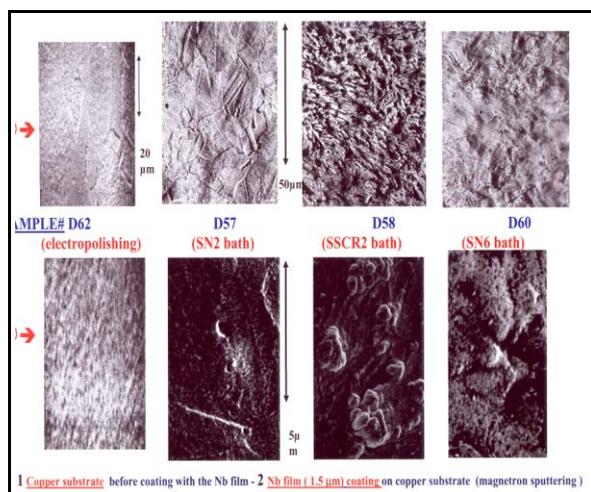

Fig. 5 Effect of different treatment on the substrate surface (upper micrographs) and niobium films (lower micrographs)

3.2 Surface roughness and RF properties

The variation of the residual surface resistance with the magnetic field, measured at T=1.7 K and 4 GHz by the thermometric method for the 4 samples (#d57, #d58, #d60 and #d62) are presented in Fig. 6. These results clearly
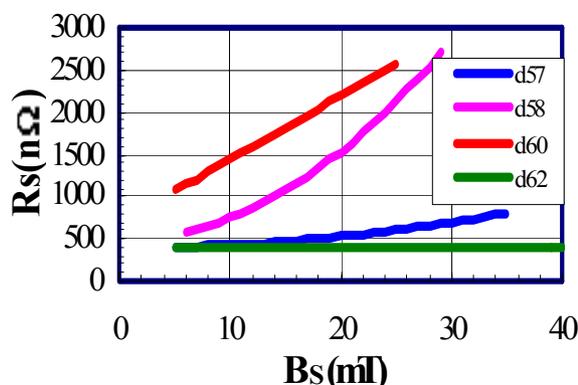

Fig. 6 Effect of substrate roughness on Nb films surface resistance at T=1.7 K (f=4 GHz)

show the correlation between the substrate roughness and the Nb film RF properties : the surface resistance and the slope of the R_S vs. H_S or (B_S) curve decrease strongly with the roughness. At low field (i.e B_S<6mT) a factor of ~3 is observed between the samples #d62 and #d60 while the slopes are respectively ~0 and 100 nΩ/mT respectively for the samples #d62 and #d58. Notice that similar behavior were observed on these four samples for f=5.6 GHz. The data show a linear frequency dependence of sputtered niobium films residual surface resistance which is weaker as compared to bulk niobium (quadratic). Finally the increase of Nb films surface resistance with the substrate roughness might be attributed to an enhancement of crystal defects densities during the film growth in some regions with substrate geometric defects.

4 ACKNOWLEDGEMENT

5 REFERENCES

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