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## **RF** and Surface Properties of Superconducting Samples

Tobias Junginger, CERN, Geneva, Switzerland and MPIK Heidelberg, Germany † Rebecca Seviour, Cockcroft Institute, Warrington and Lancaster University, United Kingdom Wolfgang Weingarten, CERN, Geneva, Switzerland Carsten Welsch, Cockcroft Institute, Warrington and University of Liverpool, United Kingdom

## Abstract

At CERN a compact Quadrupole Resonator has been developed for the RF characterization of superconducting samples at different frequencies. In this paper, results from measurements on bulk niobium and niobium filmon copper substrate samples are presented. We show how different contributions to the surface resistance depend on temperature, applied RF magnetic field and frequency. Furthermore, measurements of the maximum RF magnetic field as a function of temperature and frequency in pulsed and CW operation are presented. The study is accompanied by measurements of the surface properties of the samples by various techniques.

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#### Abstract

At CERN a compact Quadrupole Resonator has been developed for the RF characterization of superconducting samples at different frequencies. In this paper, results from measurements on bulk niobium and niobium film on copper substrate samples are presented. We show how different contributions to the surface resistance depend on temperature, applied RF magnetic field and frequency. Furthermore, measurements of the maximum RF magnetic field as a function of temperature and frequency in pulsed and CW operation are presented. The study is accompanied by measurements of the surface properties of the samples by various techniques.

## **INTRODUCTION**

The surface resistance  $R_{\rm S}$  of superconducting cavities can be obtained by measuring the unloaded quality factor  $Q_0$  [1],

$$R_{\rm S} = \frac{G}{Q_0},\tag{1}$$

where G is the geometry factor of the cavity, dependent only on the cavity shape and not on its size or material. G can therefore be accurately obtained by a numerical simulation. The surface resistance  $R_S$  may vary strongly over the cavity surface and the value obtained is the average  $R_S$ over the whole surface [1]. A convenient way to investigate the surface resistance of superconducting materials is to examine small samples, because they can be manufactured cheaply, duplicated easily and used for further surface analyses. An overview of different systems for the RF characterization can be found in [2]. Measurements with a high precision can be performed using an RF-DC compensation technique [3]. One of the devices exploiting this technique is a four-wire transmission line half-wave resonator using a TE<sub>21</sub>-like mode, called the Quadrupole Resonator.

The resonator was originally designed to measure the surface resistance of niobium film samples at 400 MHz, the technology and RF frequency chosen for the LHC at CERN. The device was used to measure the surface resistance at 400 MHz of bulk niobium and niobium on copper samples [4, 5, 6], refurbished in 2008 to extend the measurement range to 800 and 1200 MHz and to probe the critical field of the samples.



Figure 1: Surface resistance at 400 MHz (2.5 K (blue), 4 K (magenta)) and 800 MHz (2.5 K (black), 4 K (red)) of a micrometer thin niobium film sputtered on top of a copper substrate. The lines show predictions from a least squares multiparameter fit to a data set comprising 209 values  $R_{\rm S}(B, f, T)$ .

#### SURFACE IMPEDANCE

In this paper results on bulk niobium and on a micrometer thin niobium film sputtered on top of a copper substrate are presented. The reactor grade (RRR 50) bulk niobium sample was prepared by buffered chemical polishing (BCP). Precautions were taken that the acid temperature did not exceed 15 °C to avoid a larger surface resistance caused by hydrides [7]. The second sample is a niobium film DC magnetron sputtered with a normal incident angle on to a copper substrate. This sample was first tested in 1998, then kept under normal air and retested in 2011. Both samples were rinsed with ultrapure water at  $\approx 5$  bar before being mounted in the Quadrupole Resonator.

The residual resistance of the bulk niobium sample scales quadratically and therefore identically as the BCS resistance with frequency. The field dependent surface resistance can best be fitted by a quadratic increase of the BCS resistance with applied magnetic field [3].

For the niobium on copper sample the scaling of the residual resistance with frequency is linear, indicating other loss mechanisms. The field dependency of the surface resistance is also different. An exponential increase going into saturation for higher fields is found, see Figure 1. Localized states in Nb<sub>2</sub>O<sub>5</sub> allow tunneling of normal electrons in the superconductor, if the energy gained is larger than the energy gap  $\Delta$ . This tunneling model can explain the field

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<sup>&</sup>lt;sup>†</sup> tobias.junginger@cern.ch

dependent surface resistance  $R^E$  observed:

$$R^{E} = R_{400}^{E} \frac{f}{400 \,\mathrm{MHz}} \left[ e^{c/E_{\mathrm{rf}}} - e^{c/E_{\mathrm{rf}}^{0}} \right], E_{\mathrm{rf}} \ge E_{\mathrm{rf}}^{0},$$
(2)

with  $c = 2\kappa\Delta\epsilon_r/e\beta$ . Where  $\kappa \approx (1.6-5.3)$  nm is the decay constant of the wave function into Nb<sub>2</sub>O<sub>5</sub>,  $\epsilon_r \approx 10$ -15 the relative dielectric constant of Nb<sub>2</sub>O<sub>5</sub>,  $R_{400}^E$  the electrical surface resistance at 400 MHz, *e* the elementary charge and  $\beta$  the static field enhancement factor [8, 9, 10].

The electrical field E on the Quadrupole Resonator sample surface scales linearly with frequency for a given magnetic field B, as required by the law of induction as applied to the geometry in between the crooked ending of the rods and the sample. For a peak magnetic field  $B_{\rm p}$ =10 mT, the peak electric field is  $E_{\rm p}$  =0.35(0.70) MV/m for 400(800) MHz, respectively. These values are small compared to  $E_{\rm p}$ -field levels on elliptical cavities, but the area of high electric field is larger. In elliptical cavities the surface E field is mainly concentrated around the iris of the cavity, in the Quadrupole Resonator it is approximately spread over the same area on the sample surface as the magnetic field. The fact that the mean values of  $E_{\rm mean}/B_{\rm mean}$ for elliptical cavities and the Quadrupole Resonator are comparable is a valuable asset for the latter if real accelerator cavity surfaces are to be studied.

Figure 1 shows a small part of the data analyzed consisting of 209 values  $R_{\rm S}(B, f, T)$ . The lines show the result from a least squares multiparameter fit to the whole data set. Three parameters describe R(B) via the tunneling model:  $\beta$ =40-200,  $E_{rf}^0$ =0.9 MV/m and  $R_{400}^E$ =601 nΩ. The parameter  $\Delta$ =16.4 K mainly accounts for the temperature dependence via the BCS resistance. The residual resistance at 400 MHz,  $R_{\rm res,400}$ =268 n $\Omega$  was derived assuming a linear relation between residual resistance and frequency as found in the data and in previous publications on sputter coated niobium films [11]. An uncertainty  $\Delta R/R=10\%$ for each value of R was assumed, mainly accounting for errors from calibration and non-linearity of the field measurement giving a  $\chi^2$ =190 for the 209 values. Values for 1200 MHz were not included in the analysis due to limited available amplifier power at this frequency.

#### **CRITICAL FIELD**

The critical field under RF exposure has been investigated using pulses just long enough that the stored energy in the cavity reaches steady state (pulse length  $\approx 2$  ms), and in continuous wave (CW) operation.

When short pulses are used the critical field is found to be independent of frequency. For the bulk niobium sample the critical field under RF exceeds the thermodynamic critical field  $B_c$  of the material. The value found can be understood by a superheating field which can be calculated from Ginzburg-Landau theory [12, 13]. For the niobium on copper sample the critical RF field is lower than  $B_c$ . The sample has a low mean free path and therefore a high Ginzburg-Landau parameter. Therefore flux entry becomes



Figure 2: Quench field B of a bulk niobium sample at 400 (red) and 1200 MHz (black). The field was measured using short pulses (dots) and in CW operation (squares). After an additional chemical etching the values obtained in CW increased significantly (triangles).

energetically favorable at a field level below the Ginsburg Landau prediction. The field levels found can be explained by the vortex line nucleation model (VLNM) based on a thermodynamic energy balance [14, 15]. A detailed analysis is presented in [3].

When measured in CW the quench field becomes dependent on frequency and surface properties. In early tests the bulk niobium sample quenched at low field levels due to a local defect. A second etching (BCP 100  $\mu$ m) yielded higher field levels, still below the values found for short pulses, see Figure 2. The fact that  $B_p$  vs.  $1-(T_c/T)^2$  gives a straight line is an indication that an intrinsic superconducting field limitation is found for all curves. For the CW measurements this can be explained by a local defect heating its surrounding area. When the temperature in the vicinity of the defect exceeds the field dependent critical temperature the quench occurs.

#### SURFACE ANALYSES

The samples have first been analyzed using a light microscope. The niobium on copper sample is found to comprise substructures and holes of several tens of µm on the film, see Figure 3. On the right side of Figure 3 one can clearly see grain boundaries on the bulk niobium sample. The diameter of the Quadrupole Resonator samples is 75 mm. This size is too large for most surface analysis systems. A technique allowing contactless profile and surface roughness measurements of the whole samples is white light interferometry. The bulk niobium sample surface was found to be rougher than the Nb on Cu surface. Typical values for the average and rms roughness  $R_{\rm a}/R_{\rm q}$  are 200(300) nm for the niobium on copper and 900(1100) nm for the bulk niobium sample over an area of  $312 \times 234 \,\mu\text{m}$ . In the vicinity of grain boundaries the roughness of the bulk niobium sample may be even higher. At an interface between three grain boundaries  $R_a/R_q$ =3130(4100) nm was measured. Figure 4 shows a 3D image of this surface area.



Figure 3: Micrographs of the sample surface of a micrometer thin niobium film sputtered on top of a copper substrate (left) and a bulk niobium (right) sample.



Figure 4: Surface profile of a bulk niobium sample in the vicinity of a boundary between three grains obtained from white light interferometry. The surface area displayed is  $312 \times 234 \,\mu\text{m}$ .

### SUMMARY AND OUTLOOK

Results from RF measurements and contactless surface analysis on a bulk niobium and a niobium film on a copper substrate sample have been presented. The 75 mm diameter samples have now been cut into smaller pieces for further surface analysis (AFM, UFM and XPS). The Quadrupole Resonator will be used for other materials than niobium such as Nb<sub>3</sub>Sn, Mg<sub>2</sub>B [16] or multilayers [17] of superconducting and insulating layers in the future.

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