# NIOBIUM SHIELDED SAPPHIRE RESONATOR FOR FIELD-DEPENDENT SURFACE RESISTANCE MEASUREMENTS OF SUPERCONDUCTING FILMS

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Abstract. For successful realization of planar superconducting microwave devices (e.g. high power filters in communication systems) large area films  $(1'' \le \emptyset \le 3'')$  on dielectric substrates with low surface resistance  $R_s$  up to high microwave field amplitudes  $B_s$  are required. Therefore, we have developed a very sensitive dielectric resonator technique to investigate the temperature and field dependence  $R_s(T, B_s)$  of both, low- $T_c$  (Nb, Nb<sub>3</sub>Sn) and high- $T_c$  unpatterned films (YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub>, Tl<sub>2</sub>Ba<sub>2</sub>Ca<sub>1(2)</sub>Cu<sub>2(3)</sub>O<sub>x</sub>). The measurement system is based on a low-loss sapphire rod ( $\emptyset = 7 \text{ mm}$ , h = 3.5 mm, tan  $\delta \le 3 \times 10^{-8}$ ) resonant at  $f_0 = 19$  GHz in the TE<sub>011</sub>-mode. This sapphire is shielded on one side by an open niobium cavity and on the other side by the film under test which is thermally isolated and can therefore separately be heated up to  $T_c$ . Adjustable coupling antennas allow an *in situ* variation of the coupling strength.  $Q_0$ -values above  $3 \times 10^7$  for a niobium film at T = 1.8 K reflect low parasitic losses equivalent to  $R_s = 20 \ \mu\Omega$ . Maximum  $B_s$ -values of about 50 mT have been obtained for both, low- and high- $T_c$  films in pulsed power measurements at 4.2 K.

# 1. Introduction

One of the most promising applications of epitaxial superconducting films on dielectric substrates is the realization of planar microwave devices [1-3]. For such applications (e.g. high power filters in communication systems) large area films  $(1'' \le \emptyset \le 3'')$  with low surface resistance  $R_s$  up to high microwave field amplitudes  $B_s$  are required. Nonlinear losses have to be avoided in field ranges (typically  $B_s \le 5$  mT) and at temperatures (40 K  $\le T \le 80$  K) which are envisaged for applications [4-7]. In order to investigate and optimize these properties, sensitive measurement techniques for the surface resistance and its field dependence are needed. Since data obtained with patterned films (e.g. microstrip resonator techniques) or devices may be influenced by the patterning process or current enhancement effects [8], suitable techniques for basic studies must enable non-destructive measuring  $R_s(T, B_s)$  of unpatterned, single films.

Different dielectric resonator setups have successfully been used for microwave characterization of superconducting films (see e.g. [9-11]). The dielectric has the main advantage of concentrating the microwave field energy which makes it easier to achieve high field levels  $B_s$  than with large-size cavities. The majority of these measurement systems use two superconducting samples to shield the

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two end faces of a sapphire cylinder [11]. Therefore, with such systems only average data for two samples can be obtained. In order two avoid this disadvantage, one sample can be substituted by a shielding cavity. For normalconducting shielding cavities, the sensitivity is limited by the background losses [10] and is often insufficient for high-quality superconducting films.

Therefore, we use a sapphire resonator attached to a superconducting niobium shielding cavity with very low background losses [12, 13]. The sample under test is thermally isolated in an open endplate configuration. This leads to very high Q values and, consequently, results in a very sensitive technique for temperature dependent  $R_s$  measurements up to high microwave field levels  $B_s$ .

After a detailed description of our niobium shielded sapphire resonator, some data for the temperature and field dependence  $R_s(T, B_s)$  of high- $T_c$  (YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub>) and low- $T_c$  films (Nb, Nb<sub>3</sub>Sn [14]) will be presented. These will allow for both, an estimation of the resonator performance (e.g. sensitivity limit and field range) and a comparison of the properties of high- and low- $T_c$  films. Possible loss mechanisms and field limitations will only be mentioned shortly here and have been discussed in more detail elsewhere [15-20].

#### 2. Niobium shielded sapphire resonator

### 2.1. Experimental setup

Fig. 1a shows the cryogenic setup of our measurement system. The sapphire resonator (see also close-up in Fig. 1b) is integrated into a vacuum chamber which can also be filled with helium exchange gas for cooling purposes. The whole setup is immersed into a liquid helium cryostat and cooled down to 4.2 K. As dielectric we use a low-loss sapphire rod (HEMEX Ultra High-Purity Quality, Crystal Systems Inc., USA) with a height of 3.5 mm and a diameter of 7 mm which is glued by an adhesive polymer (opti-clean polymer<sup>TM</sup>) to the niobium shielding cavity. These sapphire dimensions lead to a resonant frequency of about 19 GHz for the  $TE_{011}$ -Mode (see Fig. 2a). The film under test  $(1'' \le \emptyset \le 3'')$  is thermally isolated by a gap from the shielding cavity with the sapphire and is mounted onto a copper sample holder which can be adjusted by means of three 120°-arranged micrometer screws. The parallelism of sample and shielding cavity and the width of the gap (typically 100 -200 µm) are controlled by thin Kapton foils which are removed before closing the vacuum chamber. The resulting tilt angle between sample and niobium cavity is less than  $0.1^{\circ}$ . Due to the thermal isolation, which is also supported by teflon parts in the suspension of the sample holder, the sample can be separately heated up while the niobium cavity and the sapphire rod stay at 4.2 K. Beside low background losses (see sec. 3.1) at all temperatures, this has also the advantage that no temperature dependent losses have to be subtracted from the total losses to evaluate the surface resistance of the film under test.

The input and output coupling to the resonator is performed by open loop antennas formed by the inner conductors of two semi-rigid coaxial cables. The lengths of these antennas are different, i.e. the longer one is used for the input coupling. Both antennas can be simultaneously moved in vertical direction (see arrow in Fig. 1b) *in situ* to adjust the coupling strength. Thereby the external Q values can be variied between  $10^5$  and  $10^9$  (between  $10^7$  and  $10^{11}$ ) for the input (output) coupling. This allows for both, negligible coupling coefficients for accurate determination of  $R_s$  and critical input coupling for high field measurements.

Frequency domain measurements of the temperature dependence  $R_s(T)$  and of the field dependence  $R_s(B_s)$  for low input powers ( $P_{in} \le 10$  dBm) are performed with a HP Vector Network Analyzer (HP 8720C). For negligible coupling coefficients, the error for the low-field Q determination is about 5%. In order to avoid heating phenomena, high field measurements are performed in the time domain regime by applying short RF pulses (typically 200 µs pulse length and



# Fig. 1a:

Cryogenic setup for the niobium shielded sapphire resonator. The sample holder (see Fig. 1b) can be adjusted by three 120°-arranged micrometer screws.

Fig. 1b:

Close-up of the niobium shielded sapphire resonator. The gap allows for a controlled heating of the sample  $(\emptyset \le 3'')$  while the shielding cavity with the sapphire stay at 4.2 K. The coupling antennas can be adjusted *in situ* (see arrows).

1 Hz pulse period). For time domain measurements we use a HP Sweep Generator (HP 8340B) which is externally pulsed by a HP Pulse Generator (HP 8013B). The sweep generator drives an AEG microwave amplifier (VTL 20021) with a maximum output power of 43 dBm (20 W). The input power  $P_{in}$  and the power which is transmitted through the resonator  $P_{trans}$  are measured with detector diodes (Millitech DXP-42-00). The unloaded Q of the cavity can be calculated from the ratio  $P_{trans}/P_{in}$  and from the external Q values which are determined with the network analyzer. At elevated field levels the measurement error of  $R_s$  increases to 10%. The accuracy for the  $B_s$  determination is about 20% for the highest field levels due to the simultaneous change of both couplings.





Comparison of calculated and measured resonant frequencies as function of the gap width (spacing) between sapphire and sample





MAFIA results for the field calibration factors of the cavity  $\eta_{cavity}$  and the sample  $\eta_{sample}$ .

#### 2.2. Determination of the measurement parameters

For a controlled operation of the cavity and for the determination of the surface resistance of and the field level on the film, a precise knowledge of the measurement parameters, such as resonant frequency, geometry factor and field calibration factor, as well as their dependence on the gap width is necessary. Therefore, we performed numerical calculations of these numbers for the  $TE_{011}$  mode with the computer code MAFIA [21]. Fig. 2a shows a comparison of calculated and measured values for the resonant frequency  $f_{\theta}$  for different gaps between sample and cavity. The deviation between those values is less than 1‰. Obviously, the decrease of  $f_0$  with the gap width ( $\cong$  3 MHz/µm) allows for a sensitive control of the spacing.

The relation between the unloaded  $Q_{\theta}$  of the sapphire resonator and the surface resistance of the cavity  $R_{s,cavity}$  and sample  $R_{s,sample}$  is given by geometry factors  $G_{sample}$  and  $G_{cavity}$  according to  $1/Q_0 = 1/Q_{cavity} + 1/Q_{sample} = R_{s,cavity}/G_{cavity} + R_{s,sample}/G_{sample}$ . The calculated geometry factors are plotted in Fig. 2b as function of the gap. Measurements on samples with known  $R_s$  as well as comparison to data from other groups and measurement systems confirm the calculated geometry factors.

For a determination of the RF field level  $B_s$ , the field calibration factor  $\eta$  must be known according to  $B_s = \eta (P_0 Q_0)^{\frac{1}{2}}$  where  $P_0$  is the dissipated power in the resonator. The dependence of  $\eta_{sample}$  and  $\eta_{cavity}$  on the gap width has also been calculated with MAFIA and is shown in Fig. 2c. According to these numbers, maximum field levels of about  $B_s = 50$  mT can be achieved for  $Q_0 = 10^{\circ}$ and  $P_0 = 10$  W. The field calibration factor  $\eta_{cavity}$  of the shielding cavity is slightly larger and its geometry factor  $G_{cavity}$  smaller than the numbers for the sample because of the field concentration in the sapphire, which is nearer to the niobium cavity. As a consequence of these results, we usually work with a gap width between 100 and 200 µm.

#### 3. Experimental results

#### 3.1. Temperature dependence of R<sub>s</sub>: measurement limit

We have measured the temperature dependence of  $R_s$  for several high- and low- $T_c$  films. Typical results are shown in Fig. 3a on a reduced temperature scale  $T_c/T$ . The open symbols show directly measured data while full symbols represent the same data after subtraction of a residual resistance  $R_{res}$ . The data for high-quality low- $T_c$  films at  $T \le 4.2$  K yield important information concerning the measurement limit of our sapphire resonator. For a niobium film, sputtered onto a copper substrate, we obtained a quality factor of  $Q_0 \ge 3 \times 10^7$  ( $Q_0 = 3 \times 10^6$ ) at T = 1.8 K (4.2 K). This corresponds to a surface resistance of  $R_s = 80 \ \mu\Omega$  at 4.2 K, which has also been calculated numerically for f = 19 GHz from the isotropic BCS theory with material parameters of  $T_c = 9.25$  K,  $\Delta(0)/kT_c = 1.90$ ,  $\lambda_{L}(0) = 38 \text{ nm}, \xi = 64 \text{ nm}, l = 520 \text{ nm}$  (for RRR = 200) [22]. These  $Q_0$  values also allow an estimation of the loss tangent of the sapphire rod: tan  $\delta \leq 3 \times 10^{-8}$  at 4.2 K (the upper limit reflects possible radiation losses through the gap). The maximum Q value of  $Q_0 \ge 3 \times 10^7$  is equivalent to a measurement limit of  $R_s = 20 \ \mu\Omega$  (by scaling with  $f^2$  this means 50 n  $\Omega$  at 1 GHz). This limit becomes relevant for Nb films only below 4.2 K but for Nb<sub>3</sub>Sn films already at 4.2 K [19, 20]. Up to now, we measured the lowest  $R_s$  value for high- $T_c$  films to be (110 ± 10)  $\mu\Omega$  for both, YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> and  $Tl_2Ba_2Ca_{1(2)}Cu_{2(3)}O_x$  films at 19 GHz and 4.2 K [17].

Therefore, our measurement technique yields an excellent sensitivity, especially for the investigation of high- $T_c$  films. Consequences from the  $R_s(T)$  dependence for envisaged applications of double-sided high- $T_c$  films on various substrates [5] or a quantitative analysis of the  $R_s(T)$  curves for  $YBa_2Cu_3O_{7-\delta}$  [23] and Nb<sub>3</sub>Sn films [20] can be found elsewhere. SRF97D15 929



Fig. 3a:

Comparison of  $R_s(T)$  dependences for a Nb (squares), Nb<sub>3</sub>Sn (circles) and for a YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> film (diamonds), respectively. While open symbols show directly measured data, full symbols show the data after subtraction of the residual value  $R_{res} = 20 \ \mu\Omega$ . This measurement limit (dashed line) becomes relevant only for low- $T_c$  films at  $T \le 4.2$  K.

#### Fig. 3b:

Field dependence of  $R_s$  in pulsed power measurements on high- and low- $T_c$  films. Maximum field levels were always limited by the available power. Data for Nb films depend on the substrate material (see text).

# Fig. 3c:

 $R_s(B_s)$  dependence for a sputtered YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-8</sub> film at different temperatures. The arrows indicate field breakdowns.

## **3.2.** Field dependence of $R_s$ : maximum achievable field levels

We performed pulsed high power RF measurements (see sec. 2.1.) on high- and low- $T_c$  samples. Fig. 3b shows a comparison between the high field performance of a Nb bulk sample, Nb and Nb<sub>3</sub>Sn [14] films and one YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> film [24] at 4.2 K. We obtained maximum field levels of  $B_s = 50$  mT for both, low- and high- $T_c$  samples, which were limited by the available RF power of 20 W. Up to now, Tl<sub>2</sub>Ba<sub>2</sub>Ca<sub>1(2)</sub>Cu<sub>2(3)</sub>O<sub>x</sub> films [25, 26] yielded a significant lower power handling capability [17]. In the case of Nb films, we observed an effect of the substrate material on the data. First, the low-field  $R_s$  values depend on the substrate material: for a Nb film on copper we measured  $R_s = 80 \ \mu\Omega$ , while for a Nb bulk sample and for Nb films on sapphire only minimum values of  $R_s = 90 \ \mu\Omega$  could be obtained. These data can be distinguished within their errors of 5% (see sec. 2.1.). Secondly, several Nb films on sapphire substrate interface as is discussed in more detail in [19, 20]. For all other films in Fig. 3b, we measured comparable  $R_s(B_s)$  slopes, which might be caused either by the samples or by the niobium shielding cavity.

Fig. 3c shows the  $R_s(B_s)$  dependence of a YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> film measured at different temperatures. We deposited this film by a DC sputtering technique [27] onto a LaAlO<sub>3</sub> substrate ( $\emptyset = 2''$ ). For  $T \le 50$  K we observed only weak  $R_s(B_s)$  slopes and the maximum field levels were limited by the available power. For  $T \ge 70$  K the slopes increased and field breakdowns were observed (indicated by arrows in the plot) at field levels which are comparable to the expected  $B_{c1}$  values for RF currents in the *a*,*b*-planes of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> ( $B_{c1} = 15 - 20$  mT at 77 K, see e.g. [15]). The possible loss mechanisms and field limitations ( $B_{c1}$ , weak links, global and defect induced thermal phenomena) will not be discussed here. We reported about analysis on these mechanisms e.g. in [13, 15, 17, 18].

The achieved high power performance has to be compared with requirements of envisaged applications. Typically (e.g. for output multiplexers in mobile communication systems), nonlinear losses must be avoided for  $B_s \leq 5$  mT up to T = 77 K [3, 5-7]. Obviously, these specifications are already fulfilled by the best films (Fig. 3c).

# 4. Summary

Our niobium shielded sapphire resonator allows for very sensitive  $R_s$  determination of planar superconducting samples ( $1'' \le \emptyset \le 3''$ ) as a function of temperature and RF field level. We obtain  $Q_0$ -values above  $3 \times 10^7$  at 1.8 K corresponding to a measurement limit of 20 µ $\Omega$  at 19 GHz which is equivalent to 50 n $\Omega$  at 1 GHz by scaling with  $f^2$ . Therefore, our technique is particularly suited for the investigation of high- $T_c$  films ( $R_s \ge 110 \mu \Omega$ ) but can also be used for low- $T_c$  samples (e.g. for the investigation of substrate induced effects). By applying pulsed RF power, maximum field levels of  $B_s = 50$  mT have been achieved for both, high- and low- $T_c$  superconductors limited by the available power of 20 W.

We plan a further improvement of our measurement technique by substituting the Nb shielding cavity by Nb<sub>3</sub>Sn. This should lead to a further improvement of the sensitivity for measurements at  $T \ge 4.2$  K. Since probably in many cases field limitations are defect-induced, a scanning sapphire resonator technique is also under construction which will allow for spatially resolved  $R_s(T, B_s)$  measurements on samples up to 4 inch in diameter.

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