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# Frequency dependence of the microwave surface resistance of MgB<sub>2</sub> by coaxial cavity resonator



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#### ABSTRACT

We report on the microwave (mw) properties of a cylindrical MgB<sub>2</sub> rod prepared by the reactive liquid Mg infiltration technology. The MgB<sub>2</sub> rod, 94.3 mm long, is used as inner conductor of a coaxial cavity having a Cu tube as external conductor. By analyzing the resonance curves of the cavity in the different resonant modes and at different temperatures, we have determined the temperature dependence of the mw surface resistance,  $R_s$ , of the MgB<sub>2</sub> material, at fixed frequencies, and the frequency dependence of  $R_s$ , at fixed temperatures. Our results show that the  $R_s(f)$  curves follow a  $f^n$  law, where n decreases on increasing the temperature, starting from  $n \approx 2$ , at T = 4.2 K, down to  $n \approx 0.7$  at  $T \ge T_c$ . The double-gap nature of MgB<sub>2</sub> manifests itself in the presence of a wide low-T tail in the  $R_s(T)$  curves, which can be ascribed to the quasiparticles thermally excited through the  $\pi$  gap even at relatively low temperatures. © 2014 Elsevier B.V. All rights reserved.

### 1. Introduction

Investigation of the microwave (mw) surface resistance,  $R_s$ , of superconductors (SC) is of great interest for both fundamental and applicative aspects. Indeed, measurements of  $R_s$  allow one to investigate the mechanisms responsible for mw energy losses as well as to determine specific characteristics of the samples, fundamental for their applications in mw devices.

Although MgB<sub>2</sub> SC has been indicated by several authors as suitable material for mw application [1–3], the frequency dependence of  $R_s$  in this material has not been comprehensively investigated. Up to now, in the literature there are only few papers discussing results of frequency dependence of  $R_s$ , obtained mainly in MgB<sub>2</sub> films [4,5]. Nevertheless, several authors in order to compare  $R_s$  values obtained in different samples use the  $f^2$  scaling to compare results at different frequencies [6]. To our knowledge,  $R_s(f)$  of bulk MgB<sub>2</sub> samples has been investigated only by Dmitriev et al. in the range of frequencies 10–100 MHz [7].

The frequency dependence of  $R_s$  of SC can be conveniently measured by coaxial cavity resonators, with the SC sample as inner conductor, provided that the SC rod is long enough to feed the cavity in different resonant modes [8–11]. Recently, we have done a feasibility study [12] to build coaxial cavities using bulk MgB<sub>2</sub>

produced by reactive liquid Mg infiltration technique (Mg-RLI) [13]. The study has been carried out using a MgB<sub>2</sub> rod about 45 mm long; so, in the range of frequencies available with our experimental apparatus, it has been possible to investigate only three resonant modes. In this paper, we discuss the mw properties of a cylindrical rod of MgB<sub>2</sub> produced by the Mg-RLI technique, 94.3 mm long and 3.8 mm thick, which has been used as inner conductor of a coaxial cavity having a Cu tube as outer conductor. The cavity exhibits eight resonances in the frequency range 1-12 GHz. By measuring the quality factor of the different resonance curves, we have determined the frequency dependence of  $R_s$  of the MgB<sub>2</sub> rod, at fixed temperatures, and the temperature dependence of  $R_{\rm s}$ , at fixed frequencies. Our results show that, in the range f = 1-9 GHz, the  $R_s(f)$  curves follow a  $f^n$  law, where n decreases on increasing the temperature, starting from  $n \approx 2$  at T = 4.2 K down to n = 0.7 at  $T \ge T_c$ .

#### 2. Coaxial cavity: theoretical aspects and design

It is well known that coaxial cavity resonators support TEM modes, corresponding to standing waves in which an integer number of half-wavelength nearly matches with the length of the inner conductor [8]. The unloaded quality factor of an open-circuit-end cylindrical resonator, fed in a TEM mode, is given by

$$Q_{U} = \mu_{0}\omega \ln(b/a)(R_{sa}/a + R_{sb}/b)^{-1},$$
(1)



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where *a* is the radius of the inner conductor, *b* the inner radius of the outer conductor,  $R_{sa}$  and  $R_{sb}$  are the surface resistances of the inner and outer conductors, respectively, and  $\omega$  is the angular frequency of the considered mode.

The measured (loaded) quality factor,  $Q_L$ , differs from the unloaded quality factor of Eq. (1) because of the additional energy losses out of the ports for coupling the cavity to the external circuit. However,  $Q_U$  can be calculated from the measured  $Q_L$  considering the coupling coefficients  $\beta_1$  and  $\beta_2$  for the excitation and detection lines (see Chap. 4 of Ref. [8])

$$\mathbf{Q}_U = \mathbf{Q}_L (1 + \beta_1 + \beta_2). \tag{2}$$

From Eqs. (1) and (2), one can obtain  $R_s$  of the inner conductor provided that  $R_s$  of the outer conductor is known.

We have manufactured a coaxial cavity with a Cu tube as outer conductor and a MgB<sub>2</sub> rod as inner conductor. The external Cu tube is 105.4 mm long and has an inner diameter of 10.2 mm. The MgB<sub>2</sub> rod has been prepared by the reactive liquid Mg infiltration technique, which consists in the reaction of pure liquid Mg and a preform of B powder, in a sealed stainless steel container. This technique allows one to prepare large MgB<sub>2</sub> objects, as it has been demonstrated elsewhere [13]. In particular, a steel tube with an internal diameter of 4 mm has been filled by crystalline B powder of average sizes less than 38 µm (99.5% purity) and the powder has been pressed reaching a packing density of almost 1.4 g/cm<sup>3</sup>. At the ends of the container, two cylinders of Mg have been put in contact with the B powder and the whole system has been subjected to a thermal treatment at 850 °C for 3 h. The resulting MgB<sub>2</sub> rod has been machined out of the reaction container to have a diameter of 3.8 mm and a length of 94.3 mm. The surface of the MgB<sub>2</sub> rod has been polished by conventional diamond-based tools.

The Cu tube and the MgB<sub>2</sub> rod are put together coaxially inserting the inner rod into two PTFE stoppers, having a blind hole 2 mm deep, which match with the external tube. The ends of the tube are closed by two modified SMA connectors, which are connected to the excitation end detection lines. Each stopper forms a gap of fixed width between the antenna and the ends of the inner rod. Fig. 1 shows a photo of the coaxial cavity along with one of the modified SMA connectors (inset).

The coaxial cavity has been characterized by measuring its frequency response in the range 1–13 GHz by an hp-8719D Network Analyzer, in the temperature range 4.2–77 K. From the analysis of the resonance curve in the different modes, we have determined the mw surface resistance of the inner MgB<sub>2</sub> rod. In order to determine the temperature and the frequency dependencies of  $R_s$ , we have detected, at fixed temperatures and/or at fixed frequencies, the resonance curves and measured the coupling coefficients. This operation is done by a data-acquisition program that performs a Lorentzian fit of the resonance curve, determines  $Q_L$  and measures the S<sub>11</sub> and S<sub>22</sub> parameters from which  $\beta_1$  and  $\beta_2$  are calculated [8].

#### 3. Experimental results and discussion

In the range of frequencies investigated, the cavity supports eight TEM modes resonating approximatively at 1.35, 2.74, 4.19, 5.69, 7.20, 8.76, 10.22 and 11.67 GHz. The spectrum, obtained at T = 4.2 K, is shown in Fig. 2.

From  $Q_L$  and Eq. (2), we have determined the unloaded quality factor and, using Eq. (1), the mw surface resistance of the MgB<sub>2</sub> material by which the inner rod is made,  $R_s$ . In order to know the mw surface resistance of the outer Cu cylinder,  $R_s^{Cu}$ , we have assembled a coaxial cavity in which the MgB<sub>2</sub> rod has been replaced by a Cu rod of the same dimensions; we have investigated the mw response of the Cu/Cu cavity and determined  $R_s^{Cu}$  as a function of the temperature.

Fig. 3 shows the temperature dependence of the unloaded quality factor of the MgB<sub>2</sub>/Cu cavity and the mw surface resistance of the MgB<sub>2</sub> rod, obtained in the fundamental mode. In this mode, we obtained the highest quality factor, which results  $Q_U \approx 17,000$  at T = 4.2 K; it remains of the order of  $10^4$  up to about 30 K and reduces by a factor of about 20 when the SC rod goes into the normal state. The correspondent values of  $R_s$  go from  $R_s \approx 0.1$  mΩ, at T = 4.2 K, up to  $R_s \approx 20$  mΩ at  $T = T_c \approx 38.5$  K. The inset shows the  $R_s^{Cu}$  vs. *T* curve that has been used to extract  $R_s$  from  $Q_U$ .

The  $R_s(T)$  curve of Fig. 3 exhibits a low-T tail much wider than that generally observed in other SC. This behavior is a peculiarity of MgB<sub>2</sub> [14] and can be ascribed to an excess of quasiparticles coming from the  $\pi$  band, which are thermally excited through the small gap even at relatively low temperatures. Several authors have observed a decrease of  $R_s$  from about 15 K down to 4.2 K [6,15,16] in the  $R_s(T)$  curves of MgB<sub>2</sub> films. Some of them, fitting the data at  $T \leq 15$  K with an exponential law at which a constant value is added, determine the energy gap related to the  $\pi$  band and the residual surface resistance [15,16]. Actually, also our results show a linear dependence in a semi-log scale indicating an exponential law, but the slope is too small to give a reasonable value of the  $\pi$  gap. Although some authors have obtained similar results [6], we think that it is not reasonable to perform such a fitting; it is possible that this result is due to a high value of the residual surface resistance. So, we can only affirm that the residual mw resistance is of the order of  $0.1 \text{ m}\Omega$ .

Fig. 1. A photo of the assembled coaxial cavity; the inset shows one of the modified SMA connectors.

The same procedure used to obtained  $R_s(T)$  of Fig. 3, has been carried out in the higher-frequency modes. Fig. 4 shows the



Fig. 2. Spectrum of the cavity at the liquid-He temperature.



**Fig. 3.** (left axis)  $Q_U$  of the coaxial cavity as a function of the temperature at the frequency of the fundamental mode. (right axis) Microwave surface resistance of the inner MgB<sub>2</sub> rod, extracted from  $Q_U$  by taking into account the  $R_s^{Cu}(T)$  curve shown in the inset.



Fig. 4. Temperature dependence of the mw surface resistance of the inner  $MgB_2$  rod for the first six resonant modes.

temperature dependence of  $R_s$  of the MgB<sub>2</sub> rod extracted from the experimental data obtained for the first six resonant modes. The results relative to higher-frequency modes are not reported here since the resonance curves falling at  $f \gtrsim 9$  GHz are noisy and a not good Lorentzian fit has been obtained for these curves. As one can see from the figure, on increasing the frequency the  $R_s(T)$ curve broadens in proximity of  $T_c$ , as expected, and decreases slowly going toward low temperature; this is reasonably due to the higher values of the residual surface resistance at higher frequencies.

In order to determine the frequency dependence of  $R_s$ , the temperature was set at desired values, then the resonance curves for the different modes were acquired and analyzed to obtain the quality factor and the coupling coefficients. For each temperature, the deduced values of  $R_s$  plotted as a function of the frequency in a log–log scale have highlighted a linear behavior indicating a  $f^n$  law; two examples of  $R_s(f)$  curves, one at low T and one near  $T_c$ , are shown in the inset of Fig. 5. By fitting the data obtained at different T, we have found that n decreases on increasing the temperature, starting from  $n \approx 2$  at T = 4.2 K down to  $n \approx 0.7$  at  $T \ge T_c$ . The main panel of Fig. 5 shows the temperature dependence of n.

In highly homogeneous SC, it is expected that  $R_s$  varies quadratically with the frequency except at temperatures near  $T_c$ , where



**Fig. 5.** Temperature dependence of the exponent *n* obtained by fitting the  $R_s(f)$  curves with the  $f^n$  law at fixed temperatures. The inset shows the frequency dependence of  $R_s$ , with the best-fit curves, obtained at T = 4.2 K and T = 34.6 K; the best-fit lines have been obtained with n = 1.9 at T = 4.2 K and n = 1.34 at T = 34.6 K.

the frequency dependence of the normal skin depth may play a role. In granular SC, deviations from this law have been highlighted [10] and have been ascribed to normal-material inclusions at the grain boundaries. The frequency dependence of the mw surface resistance of MgB<sub>2</sub> has not been comprehensively investigated; in the literature, there are only few papers concerning results obtained mainly in films [4,5,15] and these have been obtained measuring  $R_s$  at two or three different frequencies.  $R_s(f)$  of bulk samples has been investigated in the range of frequencies 10-100 MHz by Dmitriev et al. [7]. The investigation up to now carried out has highlighted deviations from  $R_s \propto f^2$  in both bulk and films even at relatively low temperatures; Zhukov et al. [4] have found a more than quadratic dependence, Klein et al. [15] by comparing results obtained at two different frequencies found that the  $f^2$  scaling works only at  $T \leq 10$  K and that for  $T \geq 25$  K a good agrement is obtained with a linear *f* dependence. Jin et al. [5], comparing results obtained at f = 7 GHz and at f = 18 GHz, found a good agreement using n = 2, but n = 1.5 was necessary at THz frequencies.

In a previous work, we have investigated bulk MgB<sub>2</sub> produced by Mg-RLI technique [12]. Because of the shorter rod used in that work, the fits of the  $R_s(f)$  curves were done on only three points, so the results could be inaccurate; for this reason, we have investigated a longer MgB<sub>2</sub> rod. In the longer rod we have obtained values of  $R_s$  in the SC state lower than those obtained in the shorter one, indicating a better quality of the long rod; however, the results of Fig. 5 confirm the  $R_s(f)$  dependence already obtained in the shorter rod. Moreover, the  $R_s(f)$  dependence we obtained in the SC state is consistent with that reported by Dmitriev et al. at lower frequencies [7]; on the contrary, in the normal state we obtained a frequency dependence closer to the expected one with respect to the linear dependence obtained by Dmitriev et al. We would like to remark that for normal metals in the normal-skineffect regime  $R_s \propto \sqrt{f}$  is expected, whereas we obtained  $R_s \propto f^{0.68}$ up to T = 77 K at least. Although this value of the exponent *n* is expected in the anomalous skin effect, we do not think that our MgB<sub>2</sub> material can be in the anomalous-skin-effect regime; indeed, calculating the skin depth from the values of the surface resistance in the normal state, we obtain that  $\delta$  ranges from 2 to 4  $\mu$ m, which is much larger than the mean-free path expected for MgB<sub>2</sub> materials (not greater than 10 nm). So, the reason for which the normalstate surface resistance of MgB<sub>2</sub> varies more rapidly than  $\sqrt{f}$  is not vet clarified.

#### 4. Conclusion

We have investigated the mw response of a coaxial cavity constituted by an outer Cu tube and an inner MgB<sub>2</sub> rod. 94.3 mm long, produced by the reactive liquid Mg infiltration technique. Taking advantage of the large length of the MgB<sub>2</sub> rod, which allowed us to feed the cavity in different TEM modes, we have performed an accurate investigation of the frequency dependence of the mw surface resistance of bulk MgB<sub>2</sub> in the range 1-12 GHz, which has not been discussed in the literature up to now. From the analysis of the different resonance curves at different temperatures, we have determined both the temperature dependence of  $R_s$  of the MgB<sub>2</sub> rod, at fixed frequencies, and the frequency dependence of  $R_s$ , at fixed temperatures from T = 4.2 K to T = 77 K. The estimated values of the residual surface resistance go from  $\approx 0.1 \text{ m}\Omega$ , at  $f \approx 1 \text{ GHz}$ , to  $\approx 3 \text{ m}\Omega$  at  $f \approx 9$  GHz. Although a high homogeneity is hardly obtained in samples of large dimensions, the values of the residual surface resistance of our MgB<sub>2</sub> material are smaller than those reported in the literature for bulk MgB<sub>2</sub>; they are higher than those reported for the best  $MgB_2$  films, but, since  $R_s$  of our sample remains of the same order from 4.2 K up to about 20 K, the material is competitive with MgB<sub>2</sub> films at temperatures achievable with modern crvo-coolers. From the investigation at different frequencies we have found that the  $R_{\rm s}(f)$  curves follow the  $f^n$ law with n decreasing on increasing the temperature, starting from  $n \approx 2$  at T = 4.2 K down to  $n \approx 0.7$  in the normal state, up to T = 77 K at least. Our results confirm that, in MgB<sub>2</sub>,  $R_s(f)$ deviates from the  $f^2$  dependence at temperatures of the order of 10 K, as already obtained at lower frequencies.

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- [1] E.W. Collings, M.D. Sumption, T. Tajima, Supercond. Sci. Technol. 17 (2004) S595.
- [2] Y. Bugoslavsky, G.K. Perkins, X. Qi, L.F. Cohen, A.D. Caplin, Nature 410 (2001) 563.
- [3] T. Tajima, A. Canabal, Y. Zhao, A. Romanenko, B.H. Moeckly, C.D. Nantista, S. Tantawi, L. Phillips, Y. Iwashita, I.E. Campisi, IEEE Trans. Appl. Supercond. 17 (2007) 1330.
- [4] A.A. Zhukov, A. Purnell, Y. Miyoshi, Y. Bugoslavsky, Z. Lockman, A. Berenov, H.Y. Zhai, H.M. Christen, M.P. Paranthaman, D.H. Lowndes, M.H. Jo, M.G. Blamire, L. Hao, J. Gallop, J.L. MacManus-Driscoll, L.F. Cohen, Appl. Phys. Lett. 80 (2002) 2347.
- [5] B.B. Jin, T. Dahm, F. Kadlec, P. Kuzel, A.I. Gubin, E.-M. Choi, H.J. Kim, S.-I. Lee, W.N. Kang, S.F. Wang, Y.L. Zhou, A.V. Pogrebnyakov, J.M. Redwing, X.X. Xi, N. Klein, J. Supercond. 19 (2006) 617.
- [6] B.P. Xiao, X. Zhao, J. Spradlin, C.E. Reece, M.J. Kelley, T. Tan, X.X. Xi, Supercond. Sci. Technol. 25 (2012) 095006.
- [7] V.M. Dmitriev, N.N. Prentslau, V.N. Baumer, N.N. Galtsov, L.A. Ishchenko, A.L. Prokhvatilov, M.A. Strzhemechny, A.V. Terekhov, A.I. Bykov, V.I. Liashenko, Yu. B. Paderno, V.N. Paderno, Low Temp. Phys. 30 (2004) 284.
- [8] M.J. Lancaster, Passive Microwave Device Applications of High-Temperature Superconductors, Cambridge University Press, Cambridge, 1997.
- [9] J.R. Delayen, C.L. Bohn, Phys. Rev. B 40 (1989) 5151.
- [10] P. Woodall, M.J. Lancaster, T.S.M. Maclean, C.E. Gough, N. McN. Alford, IEEE Trans. Magn. 27 (1991) 1264.
- [11] A. Agliolo Gallitto, G. Bonsignore, M. Li Vigni, A. Maccarone, Supercond. Sci. Technol. 24 (2011) 095008.
- [12] A. Agliolo Gallitto, P. Camarda, M. Li Vigni, A. Figini Albisetti, L. Saglietti, G. Giunchi, IEEE Trans. Appl. Supercond. 24 (2014) 1500109.
- [13] G. Giunchi, Int. J. Mod. Phys. B 17 (2003) 453.
- [14] M. Bonura, A. Agliolo Gallitto, M. Li Vigni, C. Ferdeghini, C. Tarantini, Eur. Phys. J. B 63 (2008) 165.
- [15] N. Klein, B.B. Jin, R. Wördenweber, P. Lahl, W.N. Kang, Hyeong-Jin Kim, Eun-Mi Choi, Sung-IK Lee, T. Dahm, K. Maki, IEEE Trans. Appl. Supercond. 13 (2003) 3253.
- [16] G. Ghigo, D. Botta, A. Chiodoni, L. Gozzelino, R. Gerbaldo, F. Laviano, E. Mezzetti, E. Monticone, C. Portesi, Phys. Rev. B 71 (2005) 214522.