Available online at www.sciencedirect.com
ScienceDirect
Journal of the European Ceramic Society 27 (2007) 2823–2826
www.elsevier.com/locate/jeurceramsoc

Measurements of the surface resistance and conductivity of thin conductive films at frequency about 1 GHz employing dielectric resonator technique

Jerzy Krupka[a,*], Krzysztof Derzakowski[b], Tomasz Zychowicz[a], Bradley L. Givot[c], William C. Egbert[c], M.M. David[c]

[a] Instytut Mikroelektroniki i Optoelektroniki Politechniki Warszawskiej, Koszykowa 75, 00-662 Warszawa, Poland
[b] Instytut Radioelekroniki Politechniki Warszawskiej, Nowowiejska 15/19, 00-665 Warszawa, Poland
[c] Corporate Analytical Technology Center, 3M Corporate Research Laboratory, 3M Center, St. Paul, MN 55144-1000, United States

Available online 8 December 2006

Abstract

A dielectric resonator technique has been developed for measurements of conductivity and surface resistance of thin metal films deposited on a dielectric substrate. This technique allows for measurements of films having surface resistances that are smaller than 5 Ω without requiring the need to perform measurements of the substrate thickness. The uncertainty of the surface resistance measurements is about 2–3% for both thin films and bulk materials. The accuracy of the conductivity measurements of the thin films is similar to the accuracy of the measurements of their thickness. Several samples have been measured having thicknesses that range from 66 nm to 50 μm.

Keywords: Nanocomposites; Electrical conductivity; Non-destructive evaluation; Thin metal films

1. Introduction

Measurements of surface resistance and conductivity at microwave frequencies have been known for many decades. The most accurate measurements can be performed on bulk materials using resonant cavity fully made of (or plated with) material under test. Using this technique surface resistances as low as 1 nΩ on niobium made cavities at temperatures in the range 1–2 K have been measured.[1] For such structures uncertainty of the surface resistance measurements is essentially the same as uncertainty of the unloaded Q-factor measurements, assuming that geometric factor for specific mode of the cavity under test is accurately known. With discovery of high temperature superconductors that were grown on flat dielectric substrates several new techniques have been proposed that are applicable for measurements of such samples. These techniques included TE_{011} mode cavities with one endplate terminated by sample under test,[2,3] parallel plate resonators[4,5] and dielectric resonators.[6–8] Most of these techniques are directly applicable for measurements of bulk materials having thickness few times larger that the skin (or penetration) depth of such materials at a specific frequency.

Measurements of thin conductive or superconductive films, having thickness comparable or smaller than the skin (or penetration) depth are more difficult since electromagnetic fields penetrate through them which makes electromagnetic analysis of test structures difficult. One of the structures (sapphire resonator technique) which is applicable for measurements of thin superconducting films has been described in.[9] In this paper application of dielectric resonator technique for measurements of thin conductive films deposited on dielectric substrates at is presented.

2. Theory and experiments

Resonant structure that has been proposed for measurements of conductivity and the surface resistance of thin conductive films at frequency about 1 GHz is shown in Figs. 1 and 2.

Evaluation of the quasi TE_{011} mode resonant frequencies Q-factors of the structure shown in Fig. 2 requires advanced numerical modeling. This have been done employing rigorous mode-matching technique.[10,11] Alternatively other numerical

* Corresponding author. Tel.: +48 22 722 7287; fax: +48 22 825 3055.
E-mail address: krupka@imio.pw.edu.pl (J. Krupka).

0955-2219/$ – see front matter
doi:10.1016/j.jeurceramsoc.2006.11.008

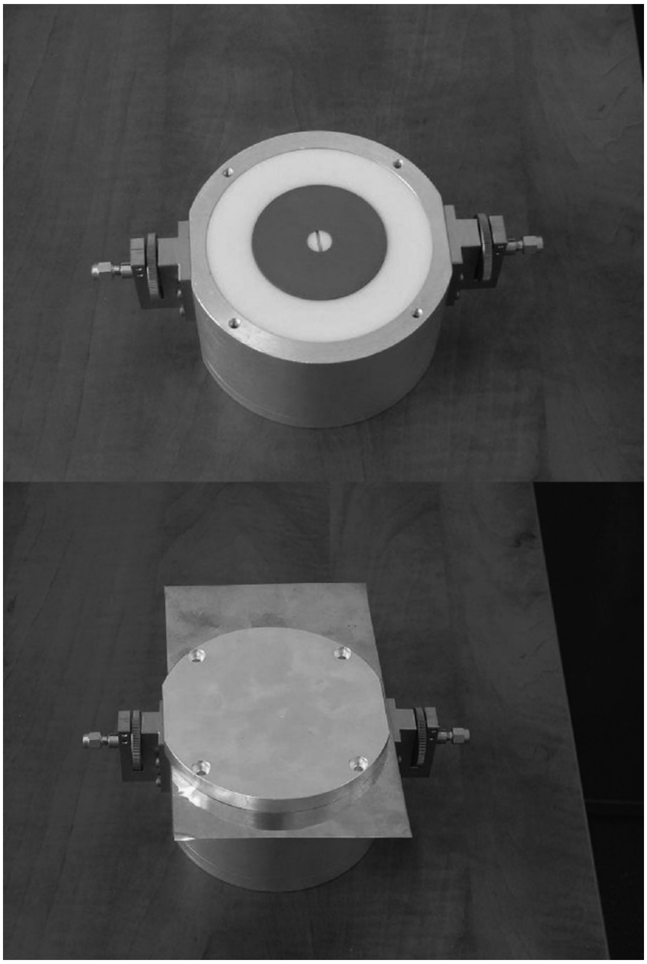

Fig. 1. Photographs of 1 GHz dielectric resonator structure used for measurements of conductivity of thin conductive films.

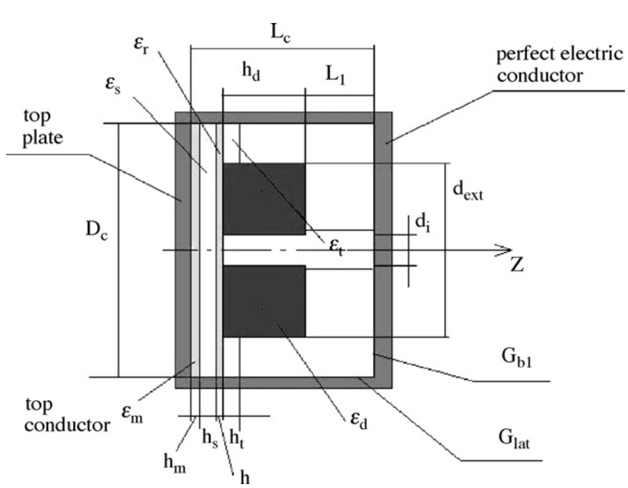

Fig. 2. Simplified structure used in electromagnetic modeling. Parameters: $D_c = 95.0$ mm, $d_{ext} = 56.9$ mm, $d_i = 4.75$ mm, $h_d = 28.2$ mm, $h_t = 5.0$ mm, $h_m = 0.01$ mm, $L_1 = 30.0$ mm, $\varepsilon_d = 36 - 8.86 \times 10^{-4}$j, $\varepsilon_t = 2.06 - 3 \times 10^{-4}$j, $\varepsilon_m = 1 - 8.43 \times 10^{8}$j, $\varepsilon_s = 3 - 3 \times 10^{-3}$j.

techniques such as finite element or finite difference can be used for this purpose.

In order to perform rigorous electromagnetic modeling all dimensions and material properties (complex permittivities of dielectrics and surface resistances of metals) of the test structure have to be known. Material properties of major parts of the structure have been determined based on careful measurements that are described in details below.

The most important parameter of the resonant structure is Q-factor which in general depends on conductor losses in conductive film under test and on conductor and dielectric losses in all other parts of the structure. This can be written as follows:

$$Q^{-1} = Q_c^{-1} + Q_p^{-1} \tag{1}$$

where Q is the measured unloaded Q-factor of the resonant structure; Q_c the Q-factor associated with conductor losses in measured conductive film; Q_p is the Q-factor due to parasitic losses in all other parts of the resonant structure.

Q-factor due to parasitic losses can be further evaluated as follows:

$$Q_p^{-1} = p_{e1} \tan\ \delta_{DR} + p_{e2} \tan\ \delta_{Teflon} + p_{e3} \tan\ \delta_{substrate} + \frac{R_{s1}}{G_{b1}} + \frac{R_{s1}}{G_{lat}} \tag{2}$$

where $p_{e(n)}$ is the electric energy filling factors in appropriate dielectric parts of resonant structure; G_{b1} the geometric factor for lower bottom of resonant structure; G_{lat} the geometric factor for lateral surface of resonant structure; R_{s1} the surface resistance of the lower bottom and lateral surface of resonant structure.

All electric energy filling factors and geometric factors of the structure can be rigorously evaluated employing mode-matching technique. The major problem in Q-factor determination is the knowledge of the surface resistance R_{s1} and loss tangent of dielectric resonator $\tan\delta_{DR}$. Contribution of dielectric losses in Teflon parts of the structure and in dielectric substrate is below the level of experimental errors and can be neglected (this is because electric energy filling factors in these pars are very small).

Measurements of permittivity and dielectric losses in the dielectric resonator puck have been performed in large copper cavity having diameter of $D_c = 150$ mm and length of $L_c = 75$ mm puck. Dielectric puck was situated at the center of the cavity and its quasi TE_{011} mode resonant frequency was 931.3 MHz with the unloaded Q-factor of $Q = 36070$. Because geometric factor of the cavity with dielectric puck was $G = 1608\ \Omega$ so Q-factor due to metal wall losses $Q_c = 190900$. As the result Q-factor due to dielectric losses was determined: $Q_d = 44480$ with $p_{e1} = 0.977$ at 931.3 MHz. This losses were then linearly scaled with frequency to 1.069 GHz (frequency of test fixture). Surface resistance of metal plates was determined from measurements of Q-factor of empty test fixture. Measured Q-factor of the TE_{011} mode of the empty cavity was $Q = 38665$ at 4.6309 GHz. Evaluated surface resistance of 18.79 mΩ was then scaled to the frequency of interest 1.069 GHz (proportionally to the square root of frequency law) and at this frequency it was 8.42 mΩ. Parasitic losses of the test structure can be then evaluated in all of its parts except the top bottom plate. The top bottom plate losses will be included in further analysis of the structure. Evaluated Q-factor due to parasitic losses was determined to be $Q_p = 34430 \pm 2\%$. Validity of these measurements and analysis have been checked by measurements of bulk metals. If resonant structure is terminated at the top by bulk metal plate (infinitely thick) then the surface resistance (or conductivity) of the metal plate can be easily evaluated form formulae:

$$Q_c^{-1} = Q^{-1} - Q_p^{-1} = \frac{R_{s\infty}}{G_{b2}} \quad (3)$$

and

$$\sigma_\infty = \frac{\omega\mu_0}{2R_{s\infty}^2} \quad (4)$$

For our test structure geometric factor for top metal plate was $G_{b2} = 312.1\ \Omega$ and measured Q-factor value of the structure terminated by bulk copper plate was $Q = 17{,}450$ so conductivity evaluated at 1.069 GHz for bulk copper is $\sigma_\infty = 5.41 \times 10^7$ S/m which is in good agreement with all other experiments. Assumption that sample is bulk is valid if sample thickness $h > 3\delta$ where δ denotes the skin depth defined as:

$$\delta = \sqrt{\frac{2}{\omega\mu_0\sigma}} \quad (5)$$

For bulk copper at 1.069 GHz, $\delta = 2.1\ \mu$m.

For films having thickness comparable or thinner than the skin depth electromagnetic filed penetrates through them and Q-factor due to conductor losses in films under test (Q_c) depend not only on their conductivity but also on film thickness and thickness of the dielectric substrate. If film thickness becomes smaller than penetration depth then Q_c factor depends rather on the product of film thickness times its conductivity than on conductivity itself. It can be seen in Figs. 3 and 4. One can see in Fig. 3 that if the thickness of the substrate is sufficiently large and if $\sigma h > 1$ (Ω^{-1}) then Q_c does not depend on h_s. In such a case Q_c depends generally on two variables film thickness and its conductivity. For extreme cases if $\sigma h < 10$ (Ω^{-1}) then Q_c depends only on σh and if $h > 3\delta$ Q_c depends only on σ. In the first extreme case (semi-transparent sample) only σh can be determined and in the second one σ can be directly evaluated. In the first case determination of conductivity requires measurements of film thickness. Numerical computations of Q_c factor have been performed for number of conductivities and film thickness and their results are shown in Fig. 5. Results of these computations were used in MATLAB program that evaluates conductivity of thin and bulk metal films from measurements of the unloaded Q-factor terminated by metal film deposited on a dielectric substrate. If the thickness of substrate is small than then measurements of Q-factors should be performed with additional dielectric sheet having thickness in the range 0.8–1.0 mm inserted between the top metal plate and original substrate.

Results of measurements of various conductive films are shown in Table 1. Measurement uncertainties of conductivity

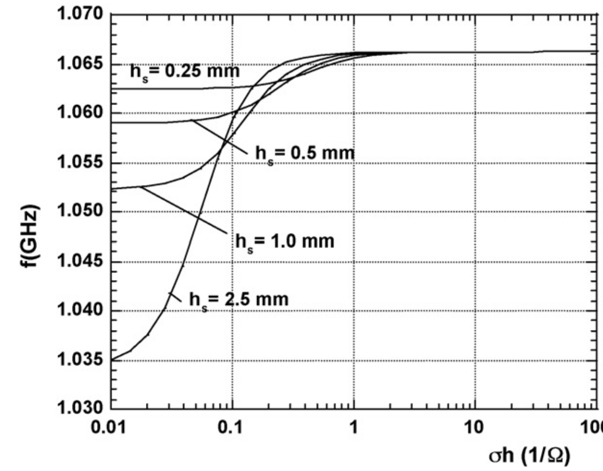

Fig. 4. Quasi TE_{011} mode resonant frequency of the resonant structure vs. σh product for various thickness of substrates.

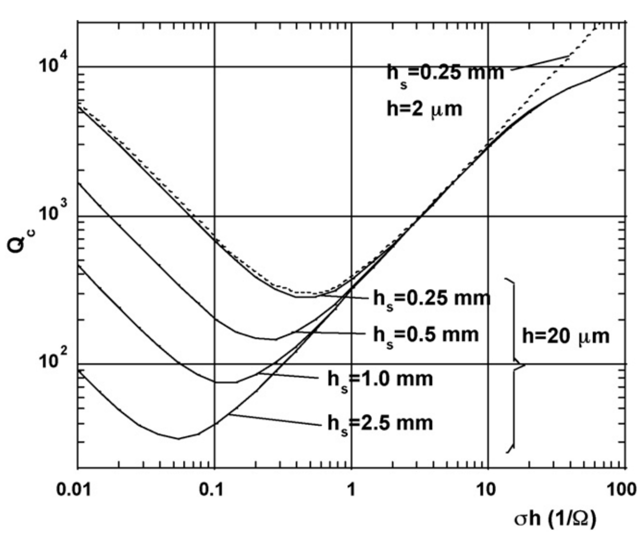

Fig. 3. Q-factor depending on metal film losses of the resonant structure vs. σh product for various thickness of substrates and films. Top plate metal losses included.

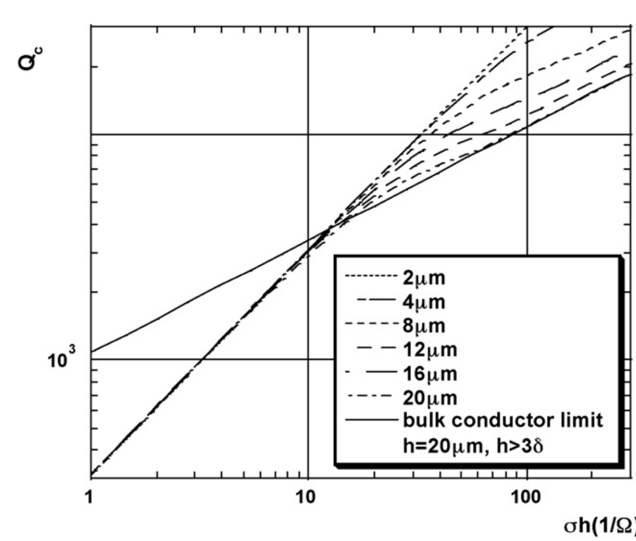

Fig. 5. Q-factor depending on metal film losses (Q_c) vs. σh product for various film thickness. Thickness of substrates $hs > 0.6$ mm.

Table 1
Results of measurements of various thin conductive films

Material	h (μm)	Q	σh (Ω^{-1})	Rs (Ω), 1.069 GHz	Rs (Ω), 1 MHz	σ (Ωm^{-1})
Cu (bulk)	50	17450	2685	0.000372		5.37×10^7
Cu (#2)	22	16900	1047	0.000955		4.76×10^7
Cu (#3)	26	16900	1238	0.000808		4.76×10^7
Cu (on Cr)	0.2	1896	6.44	0.155300		3.22×10^7
Cu	0.066	900	2.96	0.337500		4.49×10^7
Ag (plated on brass)	10	16600	447	0.002237		4.47×10^7
Al foil	10	14850	287	0.003484		2.87×10^7
Conductive ink	14	6600	34.6	0.028920		2.47×10^6
Conductive ink	6	3530	32.2	0.031040		5.37×10^6
ITO/metal layers	0.2	152	0.488	2.05	2.16	2.44×10^6
ITO/metal layers	0.35	318	1.028	0.973	1.02	2.94×10^6
Silver (155)	0.402	92	0.295	3.39	4.03	7.34×10^5
Silver (710)	0.201	70	0.226	4.42	8.06	1.13×10^6

are of the order of 3–4% for thick samples and of the order of 2% + $\Delta h/h$ for thin samples.

For samples with $\sigma h < 0.2$ (Ω^{-1}), equivalent to $R_s > 5\ \Omega$ measurement technique described above is not applicable. For such samples measurements are possible but thickness of substrates must be precisely know. As one see in Fig. 3 if thickness of substrate is smaller that 250 μm and $\sigma h < 0.2$ (Ω^{-1}), than the slope of Q_c-factor versus σh is steep and negative and σh can be determined for given thickness of substrate.

References

1. Bauer, P. Review of models of RF surface resistance in high gradient niobium cavities for particle accelerators–Revision 1, TD-04-014, 2004.
2. Beck, A. C. and Dawson, R., Conductivity measurements at microwave frequencies. *Proc IRE*, 1950, **10**, 1181.
3. Kuhleman, T. and Hinken, J. H., Measurements of the thickness dependence of the surface resistance of laser ablated high-T_c superconducting thin films. *IEEE Trans Magn*, March 1991, **27**, 872–875.
4. Taber, R. C., A parallel plate resonator technique for microwave loss measurements of superconductors. *Rev Sci Instrum*, 1990 August, **61**, 2200–2206.
5. Talanov, V. V., Mercaldo, L. V. and Anlage, S. M., Measurement of the absolute penetration depth and surface resistance of superconductors using the variable spacing parallel plate resonator. *IEEE Trans Appl Superconduct*, 1999, **9**(June (2)), 2179–2182.
6. Llopis, O., Parra, T., Ousset, J. C., Chrisey, D. B., Horwitz, J. S. and Graffeuill, J., Comparative study of microwave surface impedance of high-T_c superconductor samples. *Solid State Commun*, 1991, **78**(7), 631–633.
7. Wilker, C., Shen, Z.-Y., Nguyen, V. X. and Brenner, M. S., A sapphire resonator for microwave characterization of superconducting thin films. In *Applied Superconductivity Conference*, 1992.
8. Krupka, J., Klinger, M., Kuhn, M., Baranyak, A., Stiller, M., Hinken, J. and Modelski, J., Surface resistance measurements of HTS films by means of sapphire dielectric resonators. *IEEE Trans Appl Superconduct*, 1993, **3**(September (3)), 3043–3048.
9. Ceremuga, J., Krupka, J., Geyer, R. G. and Modelski, J., Influence of film thickness and air gaps in surface impedance measurements of high temperature superconductors using the dielectric resonator technique. *EICE Trans Electron*, 1995, **8**, 1106–1110 (Japan).
10. Maj, Sz. and Pospieszalski, M., A composite multilayered cylindrical dielectric resonator. In *IEEE MTT-S Digest*, 1984, pp. 190–192.
11. Krupka, J., Derzakowski, K., Abramowicz, A., Ceremuga, J. and Geyer, R. G., Application of mode matching technique for modelling of cylindrical dielectric resonators containing multilateral superconducting, metal and dielectric media. In *NUMELEC'97 Conference*, 1997.