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Accelerator Division
Alba Synchrotron Light Source
c/ de la Llum, 2-26
08290 Cerdanyola del Valles, Spain

Small Footprint Evaluation of Metal Coatings for Additive Manufacturing

Patrick Krkotić #*1, Albert Aguasca#, Juan M. O'Callaghan#3

#CommSensLab, Universitat Politècnica de Catalunya (UPC)

*ALBA-CELLS, E-08290 Cerdanyola del Vallés, Barcelona, Spain

1pkrkotic@cells.es, 3joano@tsc.upc.edu

***Abstract* — Rutile resonators have been used in the past to characterize high-temperature superconductors. In this work we describe a novel use of this type of resonator: the characterization of surface resistance in metal coatings deposited on plastic substrates at 9.1 GHz. A very small sample surface (about 1 cm^2) is required due to the high permittivity of rutile. We describe the use of the resonator to analyze pairs of small samples and to analyze the homogeneity of a sample set diced from a larger metallized surface. We also show a novel experimental method to determine the geometrical factors in dielectric resonators.**

***Keywords* — metal coating, additive manufacturing, rutile resonator.**

I. Introduction

Additive manufacturing has triggered the interest in methods of coating plastics with metals having good properties at RF and microwave frequencies. Several coating methods have been explored [1], including:

- Application (by brush or immersion) of colloidal solutions of silver particles in volatile solvents.
- Aerosol printing.
- Several forms of electrodeless deposition.
- Electrolytic deposition.

The surface resistance (R_s) of these coatings is normally tested using some form of the cavity end-wall replacement method [2], in which the R_s of a test surface is calculated from the changes in quality factor (Q) that it produces in a microwave cavity when that surface is used to replace another whose R_s is known.

Testing surface resistance of these coatings using standard cavities [1] may force the use of very high frequencies or very large surfaces. As described herein, the use of low-loss, high permittivity dielectrics to load measurement cavities allows the measurement of smaller samples at moderate frequencies. It also enables the study of the homogeneity of metal depositions by making round robin measurements of small sample pairs obtained from dicing a larger metallized sample.

II. Rutile Dielectric Resonator

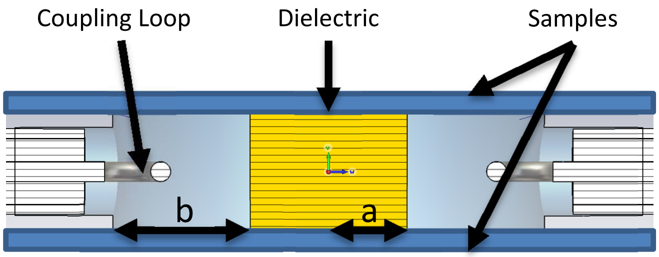

Fig. 1. Cross-sectional view of the dielectric resonator. The dielectric has 3 mm height and 4 mm diameter.

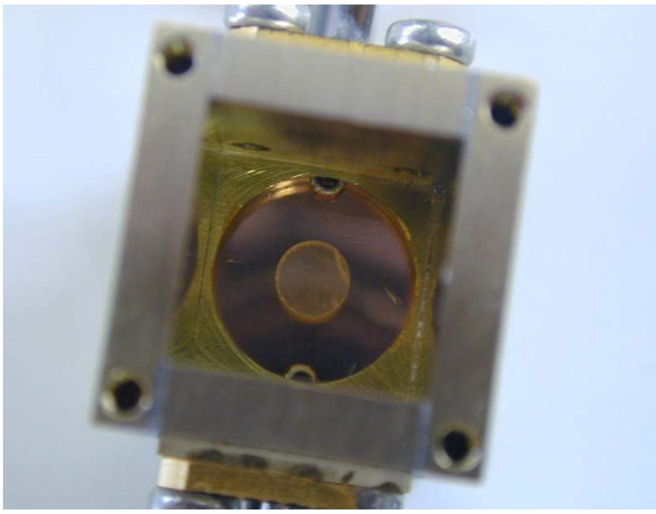

Fig. 2. Photograph of the measuring resonator with centered rutile. In this picture the rutile is placed onto one of the metal samples to be measured. An identical sample (not shown) is placed onto the dielectric. The top and bottom samples are held with CuBe springs.

As can be seen in Figs. 1 and 2, the dielectric resonator (DR) [3] is composed of a closed metallic body housing a small dielectric cylinder shielded axially by two samples to be measured. The structure is held together with copper-beryllium springs to avoid a shifting of the dielectric. The magnetic coupling is achieved through the lateral walls by a pair of semi-rigid coaxial cables with a loop at the end. The coupling can be adjusted by changing the insertion depth of the cables. The resonant frequency of the DR is basically determined by the physical dimensions of the cavity and the dielectric constant of the material. The sample size is 12x12 mm. Several types of materials have been successfully measured with this device, including metal plates, metal coatings deposited on plastic as well as HTS coated conductors. A polished c-axis oriented high-purity rutile dielectric cylinder with a diameter of 4 mm and a height of 3 mm is used. The use of rutile in this type of resonator follows the work by Klein *et al.* [4] who used this material to characterize high-temperature superconductors because of its very low-loss factor and high permittivity. The usefulness of rutile resonators in additive manufacturing applications stems from the high permittivity of rutile and the small size of the resonator, since it enables determining the surface resistance of metal coatings on small samples. It also enables determining the surface resistance homogeneity of larger surfaces when diced into smaller (~1 cm^2) ones.

A. Electromagnetic Fields

The electromagnetic analysis of the DR [3] is done by assuming a perfect conducting cylindrical shielding wall as boundary condition. Inside the DR, two regions corresponding to the rutile dielectric and air are considered. Solving Maxwell's equations in this cylindrical structure leads to an infinite number of resonant modes. However, for microwave characterization of the samples the TE_{011} mode is typically used since it does not have radial currents and therefore, the cavity performance does not depend on the contact between the samples under test and the metal hosing of the cavity. The fields of this mode can be obtained from the fields of the TE_{01} mode in a partially-loaded circular waveguide [3]. With these considerations, the nonzero field components of the DR with cavity radius b and dielectric radius a operating in the TE_{011} mode can be described using a cylindrical coordinate system (r, φ, z) as done in [5]:

Inside the dielectric, for $r \leq a$

$$E_{\varphi 1}(r,z) = -iA\frac{2\pi f\mu_0}{\xi_1}J_1(\xi_1 r)\sin(\beta z) \quad (1)$$

$$H_{r1}(r,z) = -A\frac{\beta}{\xi_1}J_1(\xi_1 r)\cos(\beta z) \quad (2)$$

$$H_{z1}(r,z) = AJ_0(\xi_1 r)\sin(\beta z) \quad (3)$$

Outside the dielectric, for $b \geq r \geq a$

$$E_{\varphi 2}(r,z) = iB\frac{2\pi f\mu_0}{\xi_2}F_1(\xi_2 r)\sin(\beta z) \quad (4)$$

$$H_{r2}(r,z) = B\frac{\beta}{\xi_2}F_1(\xi_2 r)\cos(\beta z) \quad (5)$$

$$H_{z2}(r,z) = BF_0(\xi_2 r)\sin(\beta z) \quad (6)$$

with

$$F_0(\xi_2 r) = I_0(\xi_2 r) + K_0(\xi_2 r)\frac{I_1(\xi_2 b)}{K_1(\xi_2 b)} \quad (7)$$

$$F_1(\xi_2 r) = -I_1(\xi_2 r) + K_1(\xi_2 r)\frac{I_1(\xi_2 b)}{K_1(\xi_2 b)}, \quad (8)$$

where A, B are constants, μ_0 is the magnetic permeability of free space, f is the frequency, $J_0, J_1, I_1, I_2, K_0, K_1$ are the corresponding Bessel and Hankel functions. Furthermore, $\beta = \frac{\pi}{L}$ is the z-direction propagation constant and the r-direction wave numbers ξ_1 and ξ_2 are given by $\beta^2 = k_0^2\varepsilon_r - \xi_1^2 = k_0^2 + \xi_2^2$ with $k_0 = \frac{2\pi f}{c}$ describing the propagation constant and c is the speed of light. Note that for the TE_{011} mode, the electromagnetic fields decay almost exponentially along the radial direction away from the surface of the rutile cylinder. Therefore, if the radius of the cavity (b) is sufficiently large compared to the radius of the dielectric (a), then the fields in the lateral walls are negligible [6].

B. Quality Factor

The influence of absorption on the resonator modes can be characterized by the quality factor Q_0, since $Q_0/2\pi$ equals the ratio between stored energy and energy dissipated in a cycle. From this loss equation one can easily derive the relationship between the unloaded quality factor and the losses in the different materials, which are established by the filling factors p_i and geometrical factors R_{G_i} given by [3]

$$\frac{1}{Q_0} = \sum_i p_i \tan(\delta_i) + \sum_i \frac{R_{s_i}}{R_{G_i}}, \quad (9)$$

where $\tan(\delta)$ is the loss tangent describing dielectric losses and R_{s_i} is the surface resistance. The first sum considers the losses of the dielectric bodies in the resonator and the second sum takes into account its lossy metal surfaces. The dimensionless filling factors (p_i) indicate the ratio of the energy stored in the dielectric to that stored in the entire resonator. Note that we have changed the notation of the geometrical factors of the metal walls with respect to that used in previous works [3,4] where the sign G is used. Since these geometrical factors have units of resistance - and G refers to admittance in engineering contexts -, we have used the sign R_G to be consistent with the units.

C. Determining the Geometrical Factors

The geometrical factors can be defined over the ratio of the magnetic field H in the resonator volume V to the tangential magnetic field H_t on the surface A_i given by [6,7]

$$R_{G_i} = \frac{\iiint_V \mu\omega|H|^2 dV}{\iint_{A_i}|H_t|^2 dA_i} \quad (10)$$

$$p_i = \frac{\iiint_{V_{dielectric}} \frac{\varepsilon_0\varepsilon_r}{2}|E_{\varphi 1}|^2 dV_{dielectric}}{\iiint_{V_{dielectric}} \frac{\varepsilon_0\varepsilon_r}{2}|E_{\varphi 1}|^2 dV_{dielectric} + \iiint_{V_{air}} \frac{\varepsilon_0}{2}|E_{\varphi 2}|^2 dV_{air}}. \quad (11)$$

Note that the geometrical factors and the filling factors depend entirely on the geometry of the resonator and the excited field mode, and not on the materials used. Both quantities can be estimated by substituting the nonzero electromagnetic fields from (1)-(8) into (10), (11) and solving the integrals. This leads to a geometrical factor at room temperature for the upper and lower surfaces of the resonator R_{G_S}=251.75 Ω and for the metallic enclosure R_{G_m}=138697 Ω, with a filling factor (p) of approximately unity. This supports the assumption that most of the electromagnetic energy is stored in the dielectric and that the field decays outside the dielectric rapidly. Furthermore, the high geometrical factor for the lateral walls indicates that the losses of the housing wall can be neglected for the calculations of the sample surface resistance. With this, we can rewrite (9) to determine the surface resistance for the DR used in this work as

$$R_s(T) = \frac{R_{G_S}}{2}\left(\frac{1}{Q_0(T)} - p\tan(\delta)\right). \quad (12)$$

To experimentally determine the geometrical factor of the top and bottom cover of the DR and the loss tangent of the dielectric, one can measure the unloaded quality factors of different materials of known R_s in different configurations. In this work we used sheets of copper (Cu) and copper-beryllium (CuBe) whose R_s was calculated from DC conductivity obtained with four-point-probe measurements. Quality factors where then measured for the three possible combinations: Cu-Cu, Cu-CuBe and CuBe-CuBe. Taking the equation of the surface resistance (12) we can rewrite it into a matrix equation where we consider the different material combinations used:

$$\underbrace{\begin{pmatrix} 2R_{S_{Cu}} & 1 \\ 2R_{S_{CuBe}} & 1 \\ R_{S_{Cu}} + R_{S_{CuBe}} & 1 \end{pmatrix}}_{K} \underbrace{\begin{pmatrix} 1/R_{G_S} \\ p\tan(\delta) \end{pmatrix}}_{x} = \underbrace{\begin{pmatrix} 1/Q_{0_{Cu}} \\ 1/Q_{0_{CuBe}} \\ 1/Q_{0_{Combined}} \end{pmatrix}}_{b}. \quad (13)$$

Recall that top and bottom geometrical factors must be the same as well as their surface resistances. Moreover, we assume that the losses of the lateral walls are negligible as proved in the previous section.

Solving the matrix equation leads to the solution

$$x = (K^T K)^{-1} K^T b. \quad (14)$$

The measured geometrical factor is $G_s = 254.23\ \Omega$ which is very close to the analytically calculated value. Note that this experimental procedure rules out the possible impact that small imperfections in the dielectric (scratches and cracks) may have on the geometrical factor.

The solution of the overdetermined system of equations above (13) also yields a value for $p\tan(\delta)$ from which the loss tangent of the dielectric can be determined (in our case $p\tan(\delta) \approx 1.24 \cdot 10^{-4}$).

In the finite-element method tool CST [8] we can compute the geometrical factor by simulating the same materials as those in the measurements and use the resulting quality factors in (13). Here, we get a value for the geometrical factor of $R_{G_S} = 249.91\ \Omega$. Knowing the geometrical factor, we can use the DR to measure the unloaded quality factor to determine the unknown surface resistances of further samples.

D. Characterization of samples

In order to investigate the surface resistance of different materials we used sample pairs of the same material as test surfaces of the cavity. The materials tested are OFHC Cu with resulting surface resistance of $R_S = 25.65\ \mathrm{m\Omega}$, CuBe ($R_S = 59.26\ \mathrm{m\Omega}$), copper on polyactic acid – CuPLA ($R_S = 35.93\ \mathrm{m\Omega}$) and copper on polyvinylchloride – CuPVC ($R_S = 29.80\ \mathrm{m\Omega}$). The two latter materials where obtained by electrolytic deposition of copper onto a plastic surface which has previously been coated with a silver conductive primer (TIFOO product reference 01-18-00150). For comparison, we can calculate theoretical values of the surface resistance with $R_{S_{theo}} = \sqrt{\pi f_0 \rho \mu}$, where $\rho_{Cu} = 1.772\ \mu\Omega\mathrm{cm}$ and $\rho_{CuBe} = 9.729\ \mu\Omega\mathrm{cm}$, and $f_0 = 9.1$ GHz. The frequency results because of the dielectric number of the rutile in the TE_{011} mode. This gives us an estimate of a theoretical value for copper of $R_{S_{theo}} = 25.29\ \mathrm{m\Omega}$ and for copper-beryllium of $R_{S_{theo}} = 59.11\ \mathrm{m\Omega}$. We can see that the copper sheets as well as CuPVC are in good agreement with the theoretical value. The same is valid for BeCu. Nevertheless, CuPLA shows a surface resistance which is higher than expected.

Table 1. Summary of surface resistance measurements.

Material	Measured R_S	Theoretical R_S
OFCH Cu	25.65	25.29
CuPLA	35.93	25.29
CuPVC	29.80	25.29
CuBe	59.26	59.11

E. Characterization of metallization homogeneity

To test the homogeneity of the coating process, we electrodeposited Cu onto a 165x180 mm polyvinylchloride (PVC) piece and diced it into 12x12 mm pieces. We then took four pieces from different places of the original plate and set up a round robin in which all the combination pairs where measured. The resulting surface resistance of the four samples can be calculated by solving the over-determined system of equations below:

$$R_{G_s} \cdot \begin{pmatrix} 1/Q_{01} - p\tan(\delta) \\ 1/Q_{02} - p\tan(\delta) \\ 1/Q_{03} - p\tan(\delta) \\ 1/Q_{04} - p\tan(\delta) \\ 1/Q_{05} - p\tan(\delta) \\ 1/Q_{06} - p\tan(\delta) \end{pmatrix} = \begin{pmatrix} 1 & 1 & 0 & 0 \\ 1 & 0 & 1 & 0 \\ 1 & 0 & 0 & 1 \\ 0 & 1 & 1 & 0 \\ 0 & 1 & 0 & 1 \\ 0 & 0 & 1 & 1 \end{pmatrix} \begin{pmatrix} R_{S1} \\ R_{S2} \\ R_{S3} \\ R_{S4} \end{pmatrix}, \quad (15)$$

where we use the estimated values of the geometrical factor and loss tangent of the previous section. After measuring the six possible configurations the solution of the matrix equation (15) yields $R_{S1} = 30.12\ \mathrm{m\Omega}$, $R_{S2} = 26.96\ \mathrm{m\Omega}$, $R_{S3} = 32.24\ \mathrm{m\Omega}$, and $R_{S4} = 29.89\ \mathrm{m\Omega}$, which gives an average surface resistance of $\overline{R_S} = 29.80\ \mathrm{m\Omega}$. In order to discard that the spread in R_s values does not come from lack of repeatability in the measurements, we performed a series of 10 consecutive measurements on the same pair of samples. The resonator was re-assembled before each measurement. The resulting spread in Q-factor is less than 5%, which is significantly smaller than the spread in R_s values listed above (17% with respect to average). We can therefore conclude that the electrodeposition process is not fully uniform over the PVC surface.

III. Conclusion

We describe the use of a rutile loaded dielectric resonator for the characterization of surface resistance in metal coatings deposited on plastic substrates. We show that the device is useful to determine the average surface resistance of a pair of samples made of the same material. We also show the procedure to study the homogeneity of the metallization in a large surface. The materials and metallization processes described are only used to illustrate the characterization method, and no claim is made regarding the metallization properties or its possibilities for improvement.

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