A zirconia cylindrical TM_{010} cavity for permittivity measurements at 1 GHz

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***Abstract*—The most accurate dielectric measurements are made by resonant cavity methods, the circular TM010 type being the simplest and most common. However, an airfilled such cavity at 1 GHz needs to be 250 mm in diameter. There is another problem as well: its limited applicability with very lossy samples, due to a too low *Q* value. This paper describes the development and properties of a metalized zirconia ceramic cavity for use at about 1 GHz. With its permittivity $\varepsilon' = 30$ its diameter becomes 40 mm instead of 150 mm for the airfilled version. Additionally and importantly, the dynamic range of the loss factor ε'' is greatly expanded. The calibration procedure using numerical retro-modelling is described and a measurement example of a ternary alcohol mixture is carried out. The accuracy is also estimated.**

Keywords— microwave resonant cavity, zirconia resonator, permittivity measurements.

I. Introduction

The first methods for microwave permittivity measurements had to depend on absolute and analytical methods, i.e. such where only geometric data and the frequency must be known, and solving of analytical equations provides the end result. A first overview is given in [1], but the first published measurements were by the Collie, Hasted and Ritson group in the UK, beginning in 1946. Developments were along two lines: the filled waveguide method and the cavity perturbation method. The latter as more convenient to use since the material under test (MUT) did not to be in very good contact with the metal of the measurement applicator but could be in a small vial or glass tube. A disadvantage was, however, the limitations of applicability of the perturbation theory [2] which was used to provide approximate absolute results (i. e. by using analytical functions with the system geometry). Another disadvantage was and is still the use of resonance, i.e. measurements at several frequencies require cavities with different dimensions.

The dynamic range in complex permittivity is another important aspect. As an example for the simplest circularly cylindrical TM_{010} cavity at 2,45 GHz and water with added salt, the MUT vial must not have a diameter exceeding 2 mm and the MUT loss factor ε'' must not exceed about 20 for the resonant condition to be at all measurable. However, the applicability of perturbation theory is then somewhat uncertain.

Based on the availability of data produced by the absolute methods, it became possible to employ calibration methods. The first significant use of such a method for food measurements was around 1970, at SIK in Sweden [3]. The resonant cavity was of the circular TM_{012} type, having null field nodes at ¼ and ¾ of the height and thus a sample length of half the overall height, resulting in a system insensitivity to the exact length of the MUT and also allowing e.g. metal caps of pressurised vials. Another advantage was in sample preparation: lossy samples could now be 5 mm in diameter at 2,45 GHz.

A scientific survey of the measurement techniques is given in [4], and an overview in [5].

Numerical software has since several years made it possible to simplify calibration methods by retromodelling techniques using only one reference substance and accepting nonlinearities of the measurement applicator. However, quick results require the use of interpolation among the modelling results.

The resonant closed applicator of the cylindrical TM_{010} type has a very simple geometry and is used in this work, and the properties when using a high-permittivity ceramic filling rather than air is investigated, in particular with regard to the possible range in complex permittivity. Of course, the practicality of a much reduced cavity diameter was also a reason for the work.

Both the air- and ceramic-filled cavity require a material under test (MUT) sample diameter of about 5 mm at 1 GHz, for providing a sufficiently high quality factor (*Q* value) with lossy MUTs. With the high permittivity filling/shaping in the center hole, it is possible to use MUT materials that can be easily cleaned off. In such use there must also be metal seals at the MUT ends. Else, a sample vial is needed. The MUT in this must then be significantly longer than the applicator height, for avoiding/controlling the leakage out through the applicator openings.

II. A comparative investigation of a ceramic and airfilled measurement applicator/resonator

The loop feeds of the zirconia (ZrO_2) ceramic with $\varepsilon' \approx 35$ was designed with a suitable loop coupling for achieving some few dB undercoupled coupling factor Γ_R at resonance. The same applied to the airfilled cavity. The electric field pattern is seen in Fig. 1 and the obtained resonant values in Table 1.

It is seen that the attenuation difference without/with MUT is much larger in the airfilled cavity case. This is related to the inherent properties of the TM_{010} mode in combination with the field impedance mismatching to high-ε' samples, and can also be explained by the relative amplitudes of the exact Bessel function

TABLE I. COMPARATIVE MODELING RESULTS

Item	Cer. cavity	Air cavity
Empty f_R (MHz)	1020,2	917,6
Empty Γ_R (dB)	–2,53	–6,98
Blood MUT f_R (MHz)	987,2	882,6
Blood MUT Γ_R (dB)	–19,80	–46,81
Δf_R empty/blood (MHz)	33,0	35,3
$\Delta \Gamma_R$ empty/blood (dB)	**17,27**	**39,83**

solution [6] having a different quotient inside/outside the MUT in the two cases. As a matter of fact, the airfilled cavity is more sensitive and different loops typically need to be used with the airfilled cavity for different sample ε′. As seen from the Table 1, the situation is greatly improved by the more similar ε′ values of the ceramic and MUT.

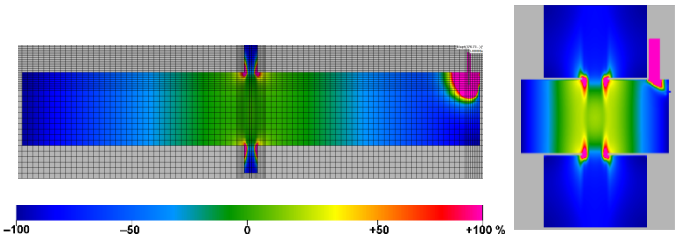

Fig. 1. The total electric momentaneous maximal field at resonance in the vertical center cross section of the airfilled 250 mm in diameter cavity with 5 mm diameter blood sample ($\varepsilon' = 58$; $\sigma = 1{,}3$), left. The same in a zirconia cavity with a diameter of 40 mm (i.e six times smaller), right. Also note that the height of the airfilled cavity is 40 mm and of the zirconia cavity 20 mm.

III. THE EXPERIMENTAL CERAMIC APPLICATOR

This was manufactured from gold-(Au)-plated zirconia (ZrO_2) ceramics, with dimensions given in Fig. 2. It was originally assumed that the real permittivity ε′ of the material was about 35, which was used for obtaining an empty resonant frequency about 1,00 GHz. However, it turned out to be about 29. The Au plating was for corrosion resistance, with an underlying base layer of molybdenum (Mo) by a chemical process for very good adhesion. The conductivity of Mo is about $2{,}5\times10^8$ S/m, but there is a lower conductivity of the compound layer. In addition, there are also surface roughness issues.

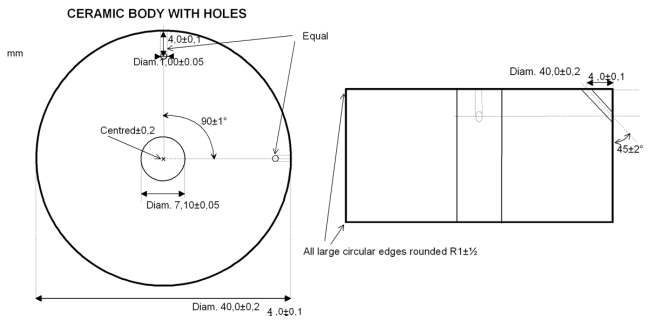

Fig. 2. Dimensions and feeds of the ceramic cavity. Its height is 20,0 mm.

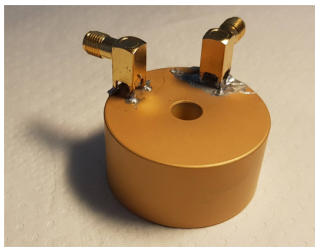

Fig. 3. The experimental cavity.

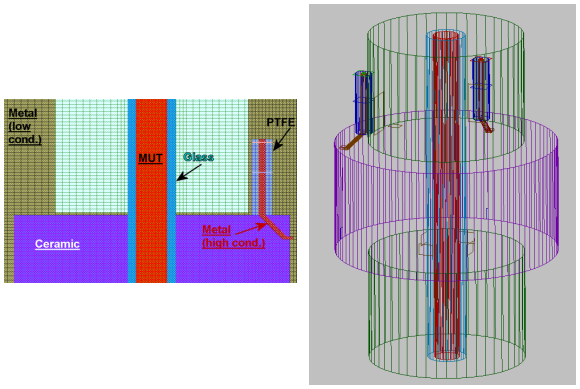

Fig. 4. Software editor images of the applicator.

In all, the "effective" conductivity becomes lower by at least a factor 10. Silver (Ag) wire was mounted and soldered in the holes for coupling loops, with SMA chassis contacts; see Fig. 3.

IV. THE CALIBRATION BY NUMERICAL MODELING

This was carried out in multiple steps, similarly to [7], see Fig. 5. The FDTD commercial Quickwave™ software [8] was used, throughout. Figure 4 shows images of the cavity, from the software editor.

A. Empty Applicator

The procedure is started with empty cavity, based on the measured values. Firstly, ε′ of the ceramic must be set; this is quite simple, by the resonant frequency. The result was $\varepsilon' \approx 29$ for later finetuning after all other scenario data have been iteratively adjusted.

The second procedure is the most complicated, since the inherent losses in the ceramic, the equivalent conductivity of the metallization and also that of the coupling loops, and the coupling factor at resonance must all be varied. This is since obtaining an agreement with the resonant coupling factor in the numerical scenario and the resonant bandwidth (at e.g. 20 dB down from the value at resonance) is needed. The metal conductivities must also be set to approximately correspond to the known data for the metals.

The measured data for the empty cavity were resonant frequency fR =1118,0 MHz; reflection factor at resonance ΓR = –5,53 dB. The frequency bandwidth 20 dB down, i.e. ΓR-20 (in this case Γ at –25,53 dB) was 27,4 MHz, see top Fig. 6. ΓR-20 is of course directly related to the loaded quality factor (Q-value).

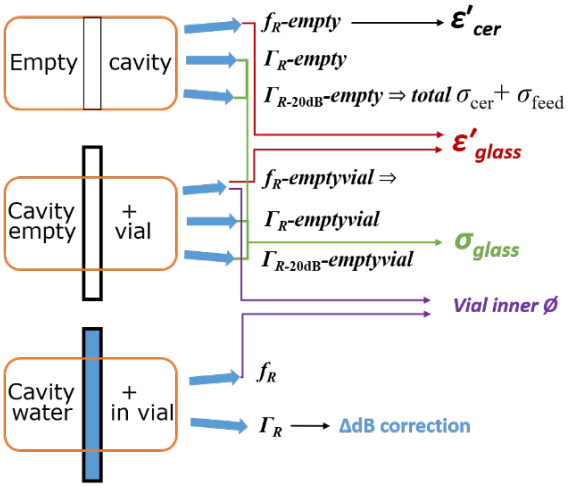

Fig. 5. Calibration flowchart.

Also $\Gamma_{R\text{-}30}$ was used; according the theory for the Cauchy/Lorentzian shape of the resonant curve, the $\Gamma_{R\text{-}30}/\Gamma_{R\text{-}20}$ quotient between these is $\sqrt{1000-1}/\sqrt{100-1} \approx 3{,}177$. This gave 27,32 MHz and indicates a very pure TM_{010} mode which is not disturbed by a nearest higher mode TM_{110}. A contributing reason for this is that the transmitting and receiving loops are at 90º. The S_{11} image (Fig. 7) shows that the empty cavity is undercoupled, as it has to be.

The modelling S21 result with the empty cavity is shown in bottom Fig. 6. ΓR-20 was 26,85 dB.

The final data of the empty cavity were: ceramic permittivity ε′ = 29,15 ; ceramic conductivity σ = 0,0015 S/m ; metallization

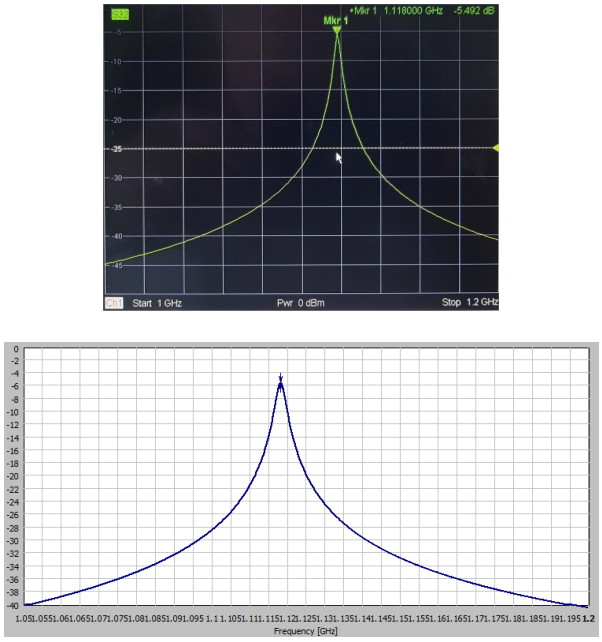

Fig. 6. S_{21} for empty cavity. Top = measured; bottom = modeled. The range on the x-axis is from 1,051 to 1,2 GHz, and on y-axis from 0 to -40 dB.

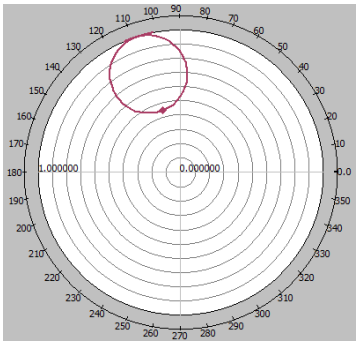

Fig. 7. S11 for empty cavity

conductivity $\sigma = 5\times10^7$ S/m. The loop conductivity was set to $\sigma \approx 5\times10^8$ S/m. It is to be noted that the conductivity of pure silver (Ag) is $1{,}6\times10^8$, pure Au is $2{,}5\times10^8$ and Mo $5{,}2\times10^7$ S/m. There is also a manganese (Mn) alloy layer, having 6.2×10^5 S/m There is a chemical bonding between the metallization layers and the ceramic

B. Applicator with Glass Vial

Glass vials with 7,0 mm outer diameter and about 1 mm material thickness were used. The vial inner diameter had now first to be determined. The glass trial ε′ = 4,0 and conductivity σ = 0,005 S/m obtained from earlier work was used as start data, with water data from [7] at the actual temperature (+23± 0,5) °C of the deionised water: ε = 78,90–j4,325 at 1066 MHz, corresponding to σ = 0,2565 S/m. A good agreement on the difference between the measured f_R with empty vial and water-filled vial (1115,0 – 1066,0 = 49,0 MHz) with the modelled (1115,1 – 1065,9 = 49,2 MHz) was obtained with an inner vial diameter of 4,65 mm.

With these data, the resonant data with empty vial had to be matched with regard to the Γ_R difference with/without empty vial. The modelling result for obtaining the best data was glass ε′ = 4,0; σ = 0,003 S/m This gave the modelling data with empty vial: f_R =1115,1 MHz; Γ_R = –5,56 dB.

C. Cavity with Deionised Water at 22 °C in the Vial

Since the water true ε′ had been used as reference for determination of the vial inner diameter, its conductivity was next, for determination of the deviations between measurements and modelling with regard to conductivity losses.

The cavity surface current distribution changes with complex permittivity of the sample, modifying the overall surface squared current losses and by that changing the equivalent no load transmission loss that is used here for determination of the sample loss factor. The quotient between these true empty and current distribution modified empty cavity transmission losses typically becomes about 0,5 dB. It can be shown that this value becomes quite constant when the overall transmission quotient with a lossy sample exceeds about 6 dB. It is, however, of principal importance that one factor of knowledge is now 'lost', since the metal surface current distribution is in principle unknown, due to the complex permittivity of the MUT substance also being in principle unknown [7].

The modelled water Γ_R reduction was –5,56 – (–15,82) = 10,26 dB and the measured was –5,55 – (–14,96) = 9,41 dB. The difference of 0,85 dB is to be applied for all further measurements as a correction for the modelling, provided the additional Γ_R decrease to that with empty vial is down about 6 dB, i.e. Γ_R is totally less than about –12 dB.

V. Measurements and Calculations on a Ternary Alcohol Mixture at +23°C

The mixture was 199 g (250 mL) 1-butanol, 194 g (250 mL) isopropanol, and 500 g (500 mL) water. This liquid mixture is intended to represent certain kinds of human tissue for phantom development. The temperature of the sample was +23 °C.

The measured data were f_R = 1085,0 MHz; Γ_R =–23,0 dB. With the Γ_R difference above, the target values for the iterative numerical modelling become f_R = 1085,0 MHz; Γ_R =–23,0 – 0,85 = –23,85 dB.

For ε' = 45,8; σ = 0,75 S/m the modelling result was f_R = 1085,1 MHz; Γ_R = –22,71 dB. With instead σ = 0,85 S/m, the result was f_R = 1085,0 MHz; Γ_R = –23,64 dB. Interpolation gives σ = 0,78 S/m. This corresponds to ε'' ≈12,9.

VI. Accuracy and Discussion

The repeatability and resolution of the f_R and Γ_R values is within 0,1 MHz and 0,1 dB, respectively, both for the measurements and the numerical modelling. As a consequence, the accuracy depends on the differences between the real and modelled cavity, and the temperature and purity of the water used as reference.

The ε' accuracy is within about ±0,2 MHz totally, which is translated to 0,2/49·79 = ±0,3 units in ε'.

The σ accuracy is more complicated, due to the 0,85 dB real/modelling difference, the network analyzer attenuation non-linearity, and the interpolation of data for obtaining the target value. In the most favorable cases with ε' higher than about 20 and Γ_R being lower than about –15 dB, the inaccuracy corresponds to about ±0,3 dB. Translated into σ inaccuracy at these corresponding (complex) ε values, one gets ±0,03 S/m. At the actual f_R values 1,0…1,2 GHz, this corresponds to about ±0,5 in ε''.

These inaccuracies are significantly less than with the common coaxial probe method.

VII. Conclusion

The high-permittivity ceramic measurement cavity can be used with a wider dynamic range of complex permittivity data of the MUT than corresponding airfilled cavities. It is also very practical to use due to its 150 times smaller volume than that of an airfilled cavity.

The method of successive and iterative retromodelling for calibration of the measurement system is initially time-consuming, but provides high resolution and accuracy. Calibration with known and quite accurate water data also provides a quite high accuracy of the obtained results for other liquids.

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