Using the Coaxial Probe Method for Permittivity Measurements of Liquids at High Temperatures

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Abstract—The use of the Coaxial Probe Method for dielectric characterization of liquid materials and the cares that should be taken to make reliable permittivity measurements at high temperatures are described in this work. Due to the lack of a standard procedure in the literature for these measurements, the validation of the method involved the characterization of different liquids of known properties (water and alcohols) at different frequencies and temperatures. An experimental procedure was developed according to manufacturer’s recommendations and our own experience in the lab. The reliability of the technique was evaluated by comparing the deviation of the measured results from material data in the literature with the accuracy limits supplied by the manufacturer of the probe.

Index Terms—thermal variables measurement; microwave measurements; permittivity measurement.

I. INTRODUCTION

Though microwaves are usually associated to telecommunications systems, those high frequency electromagnetic waves are also widely employed as an alternative form of material heating. The best known example in this context is their use in domestic microwave ovens. Microwave heating has also been adopted in large scale industrial applications, such as in food preparation and drying, for instance. Other applications involve the processing of miscellaneous materials, such as rubber and ceramics, and a growing interest in the field of Chemistry is also worth mentioning [1], [2]. In all these applications, the knowledge of the intrinsic electrical properties of the materials to be heated is of fundamental importance, both for the designer of the heating devices and for the final users of such equipment.

There are many techniques for measuring the permittivity of materials. The choice of the most appropriate technique depends on several characteristics associated to the material, especially: its state (solid, liquid or gaseous); the range of temperatures the material will be submitted to; and the frequency range of interest.

For the electric characterization of liquid and semi-solid materials in the microwave frequency range, one of the most used techniques is the Coaxial Probe Method, which is quite flexible and allows permittivity measurement at high temperatures [3]. Such flexibility, however, brings some
difficulties of practical order, with relevant implications to the reliability of the results. In a recent research work led by the authors, where it was required to measure the permittivity of some materials at high temperatures, several questions have arisen when implementing the method. When looking for the pertinent literature, a lack of references that could explain useful considerations of practical order or that could clearly describe a standard procedure for the measurements was noticed.

The above mentioned difficulties motivated the accomplishment of this work, which describes the use of the Coaxial Probe Method for dielectric characterization of liquid materials at high temperatures, with focus on the cares that should be taken to produce reliable measurements. The validation of the experimental procedure involved the characterization of some liquids with known properties (water and alcohols) at different frequencies and temperatures. The reliability of the method was verified by evaluating if the deviations of the measured data from the expected results were within the accuracy limits supplied by the probe’s manufacturer. Although we have made measurements with several materials for temperatures up to 180°C, we report here the results for selected materials up to about 100°C.

This article is organized as follows. Section II presents a brief overview of the main techniques for permittivity measurements. The following section describes the adopted configuration and measurement procedure, with emphasis on guidelines and special cares to obtain accurate and reliable results. The validation of the technique is presented in section IV. Section V concludes the paper.

II. TECHNIQUES FOR MEASURING THE DIELECTRIC PROPERTIES OF MATERIALS

In general, a system for dielectric measurements consists of three parts: measurement equipment; a test fixture to hold the material under test (MUT); and software capable of accomplishing the necessary calculations from basic measurements, and of presenting the results in forms of tables, graphs or listings. The type of equipment to be used depends, among other factors, on the material under test, the parameter to be measured, the frequency range, the measurement technique and the required precision.

Several techniques for measuring the dielectric properties of materials exist in the literature [3]-[6], covering a wide range of the electromagnetic spectrum. Among them, some groups can be identified. There is a group of measurements based on the use of resistive cells, for characterizing materials of high resistance (insulating type), in which a high testing voltage is applied to the MUT for determining its surface or volumetric resistivity. Measurements with parallel plate systems, configured to form a capacitor with the MUT, lie in another group. They are used for permittivity measurements only.

There are techniques with one and two-port systems for measuring either permittivity, permeability, or both. Measurements with coaxial probes and reflected waves lie in the first case, e.g. [7], [8]. Measurements with transmission lines and waveguides lie in the second, e.g. [9], [10]. There are still
other groups, as those with resonant cavities, with resonant circuits, with open resonators, and the free space technique, e.g. [6], [7], [11], [12].

Regarding test equipment, it is worth mentioning high impedance meters, LCR meters, impedance analyzers and network analyzers, each one with its proper characteristics, frequency range of application and accuracy. In specialized setups, other equipments can be used, such as power meters and spectrum analyzers, for instance.

Broadly speaking, high impedance meters are used for characterizing materials in DC conditions. LCR meters and impedance analyzers cover the electromagnetic spectrum for frequencies up to about 1 GHz. Network analyzers, on the other hand, cover a wide range of the spectrum, with upper frequency as high as 110 GHz.

The choice of the test fixture, usually related to the measuring technique, is as important as the choice of the technique itself and measurement equipment. It should be flexible, cover the desired frequency range, hold or fit the MUT properly, operate in a wide range of temperatures, be resistant to humidity and corrosion, and reduce the introduction of errors or uncertainties in the measurements, among possibly other conditioning factors.

Most test fixtures for the above mentioned measuring techniques can be adapted to fit liquid materials. However, the most suitable and the most employed technique for this purpose is the one that uses coaxial probes.

III. CONFIGURATION AND ADOPTED PROCEDURES

In this work we used a network analyzer Agilent E5070B and a coaxial probe Agilent 85070D, shown in Fig. 1 [13]. The used software was 85070E, also from Agilent [7].

In the coaxial probe specifications, it is required that the MUT has minimum diameter and depth of 20 mm and 20/$\varepsilon_r^{1/2}$ mm, respectively, where $\varepsilon_r$ is the expected value of the relative permittivity of the material. It is also assumed that the MUT has “infinite” dimensions, it is non-magnetic ($\mu_r = 1$), isotropic and homogeneous.

![Coaxial probe Agilent 85070D](image)

Fig. 1. High temperature dielectric probe Agilent 85070D [13].
Fig. 2 shows the typical accuracy of the coaxial probe Agilent 85070D. Typical system accuracy is better than ±5% for permittivity and ±0.05 for loss tangent, according to the manufacturer [13]. For low frequencies, however, the accuracy is reduced, which is intrinsic to the measuring technique. For materials with \( \varepsilon_r = 2 \), the accuracy is about 20% at 2.5 GHz.

![Fig. 2. Typical relative permittivity and loss tangent accuracies vs. frequency for materials with \( \varepsilon_r = 2, 5, 20, 50 \) and 80 [13].](image)

A. **Theoretical Background**

The coaxial probe of Fig. 1 is usually modeled as a coaxial aperture opening on an infinite ground plane, assuming radial symmetry. The use of the coaxial probe requires an accurate model relating the reflection coefficient at the coaxial probe aperture to the permittivity of the material contacting the probe. Useful models range from equivalent circuits to variational techniques, imposing a compromise between computational speed and accuracy over the desired frequency range. Comments about these models can be found in [14], where a model based on fitting a full-wave moment method to a rational function was used to address the issue of range and accuracy at a reasonable speed.

Concerning the vector network analyzer calibration to correct for systematic measurement errors, a one-port model is usually used for the coaxial probe. In this model, the reflection coefficient measured by the network analyzer \( \Gamma_M \) differs from the actual reflection coefficient \( \Gamma_A \) according to

\[
\Gamma_M = E_D + \frac{\Gamma_A E_R}{1 - E_S \Gamma_A}
\]

where \( E_D, E_R \) and \( E_S \) are the systematic errors of directivity, frequency response (reflection tracking), and source match, respectively [15]. Calibration involves measuring three known reflection coefficients, computing the systematic errors at each measurement frequency from (1), and mathematically removing them from subsequent measurements.

Three calibration choices are available for Agilent 85070E software. The first requires a measurement of a 50-ohm load connected to the end of the cable, measurement of the probe in air and measurement of the probe with a shorting block attached. The second requires the measurement of the probe in air, with the shorting block attached to the probe and with the probe immersed in water. The third allows the user to define the calibration standards, requiring the measurement of the probe.
immersed in three liquids.

The use of well known reference liquids as calibration standards for the coaxial probe allows relative easy calibration at the probe aperture [16], with the advantage that the calibration is done straightly at the measurement interface. Reference liquids for calibration can be found in the literature, e.g. [17].

Air, a short circuit and deionized water are readily available as well-defined calibration standards. Water is a useful standard for the coaxial probe because the dipole losses of water are sufficiently high at frequencies where radiation from the probe aperture is appreciable, which minimizes reflections from finite boundaries at the measuring setup. These standards provide very good accuracy for a wide range of materials [14].

B. Adopted Procedure

Measurements of some liquids of interest at high temperatures in the preliminary phase of our project produced unsatisfactory results. In the very first tests, the results looked fine. However, with repetition of the procedures, sometimes the results were repeated (considering average values only), sometimes the results diverged considerably from the previous measurements. So, it was difficult to clearly identify in which cases the measurement errors were due to the method itself (systematic errors), and in which cases other factors were affecting the measurements. In fact, it was observed later that test conditions had enormous influence on the results, which was an indicative that special cares would be required for the measurements. To clarify some questions about the tests, the manufacturer was consulted. Fig. 3 shows a photo of the measuring system.

![Basic measurement setup](image)

Fig. 3. Basic measurement setup.

Regarding the practical size of the samples, it was suggested that the MUT should have minimum diameter and depth of 30 and 20 mm, respectively, for the coaxial probe in use, whose contact interface with the MUT has a 19 mm diameter (Fig. 1). Calibration was done with air, a shorting
The correct form of inserting the coaxial probe in the MUT (if normal to its surface or with a slope, and at which depth) also had strong influence in the results. It was verified later that the penetration depth of the coaxial probe in the MUT was not so important, once minimum MUT dimensions next to and below the probe were preserved. Naturally, a problem of practical order exists, because the probe cannot be inserted too deep in the material, otherwise the MUT will make contact with the access connector which links the coaxial cable to the test equipment. In practice, it was suggested that the probe should be slightly inserted in the MUT (about 2 to 3 mm, for insertion normal to the MUT’s surface).

An inclination of the probe in relation to the surface of the liquid is also recommended, to avoid the formation of air bubbles between the surface of the probe and the material. The coaxial probe is capacitive and, as such, it is based on the distribution and intensity of the electric field around it. The change of the dielectric medium around the probe with air bubbles of unpredictable form and size produces unexpected results. The inclination of the probe helps to eliminate these bubbles. However, the inclination should not commit the requirements of MUT dimensions or violate the mentioned limitations regarding penetration depth of the probe in the material. The use of a plastic or wood toothpick was useful for removing bubbles.

In spite of the range of operating temperatures for the coaxial probe Agilent 85070D be specified from −40°C to 200°C, measurement in function of temperature is not a straightforward procedure. The manufacturer indicates that the use of the probe is guaranteed only for temperatures from 0 to 55°C [7]. Our first thought was that the calibration should be made in the same temperature of the measurements. The manufacturer, however, did not give support to this solution. It was suggested the use of the software 85070E to compensate for measurement errors for temperatures up to 75°C, by entering theoretical values of the calibration standards and measuring the dielectric constant of the MUT at test temperature. Nevertheless, as we intended to make measurements at temperatures above 100°C, this solution would not be feasible. It would not be possible to insert the dielectric constant of water or to calibrate the system with water at temperatures above 100°C at room pressure for instance. After several rounds of discussions with the manufacturer, we decided to perform system calibration at 25°C (room temperature).

Thus, we decided that the following cares should be taken during system calibration and measurements:

- Perform system calibration at room temperature;
- Keep the coaxial probe out of the sample while the MUT was heated up or cooled down;
- Insert the coaxial probe in the sample and carry out the measurements in a short time, after the temperature of the MUT is reached;
- Should the variations of the permittivity values be abrupt, use the Refresh Cal option of the
software for updating the calibration; and

- Repeat the procedure three times and average the results (to minimize instability.

It is worth mentioning that the procedures above did not eliminate measurement errors completely. However, they reduced the degree of uncertainty of the measurements considerably. The results became repeatable and reliable, as shown in the following section.

IV. TECHNIQUE VALIDATION

A. Permittivity Measurements of Water

Fig. 4 presents the measurements with Agilent 85070E of the relative permittivity of pure water at 25, 50 and 90°C with the recommended procedure (only three curves are shown due to a limitation of the software). In Fig. 4 and in the following ones, e’ and e” are, respectively, the real and imaginary parts of the measured relative permittivity of the sample.

![Complex Permittivity Measurements of Water](image)

(a)

(b)

Fig. 4. Measured (a) e' and (b) e" of pure water at 25, 50 and 90°C.
The measured data were compared with Meissner and Wentz model for pure water [18], whose results are presented in Fig. 5. The * marks are on the measured curves at 2.45 GHz. Each color is associated to a fixed temperature. The curves with soft variations correspond to the model. Fig. 6 presents a comparison of the measurements versus temperature with Meissner and Wentz model [18] at 2.45 GHz.

In Fig. 5(a) as well as in Fig. 6(a) it can be observed that the values of $e'$ are close to the model at low temperatures. It is also verified that the differences increase with the temperature raise, and that in all cases the measured values are higher than the obtained from the mathematical model. To confirm these results, new measurements were carried out, which led to similar results.

![Comparison between measured (a) $e'$ and (b) $e''$ of pure water and Meissner and Wentz model [18].](image-url)
The larger errors at higher temperatures roused suspicion about the adopted procedures. By keeping the coaxial probe at room temperature and immersing it in the sample just for the measurements could cause a local cooling of the sample (at the surface of the coaxial probe), which could make significant changes in the sample temperature, affecting the measured values of permittivity.

To verify if the statement about the probe temperature proceeded, new tests were performed. This time, the probe was left immersed in the sample all the time, so that it was always at the same temperature of the sample during measurements. In these measurements, it was noticed that the errors in the real part of the relative permittivity decreased in relation to the previous measurements, except 

Fig. 6. Comparison of measured (a) $\varepsilon'$ and (b) $\varepsilon''$ of pure water with Meissner and Wentz model [18] at 2.45 GHz.
for the highest temperatures. The errors of the imaginary part also decreased, but not as pronounced as those for the real part. Fig. 8 shows these new measurements.

![Graph](image)

Fig. 7. Comparison of the measured (a) $\varepsilon'$ and (b) $\varepsilon''$ of pure water with Meissner and Wentz model [18] at 2.45 GHz with the probe at the same temperature of the sample during measurements.

Considering the results for the real part of the permittivity at temperatures up to 70°C, there would be an indication that the probe should be immersed all the time in the sample. However, since the heating of the probe (and cables) would have more influence at higher temperatures (above 100°C), and due to calibration aspects and the good results of the previous measurements, we decided to keep the original procedure. Nevertheless, the measurements were always taken at a small time after the probe was inserted in the liquid, in order to heat the surface of the probe without heating up cables and connectors. Practice established the proper time for it.
B. Permittivity Measurements of Alcohols

During the laboratorial investigations, in order to evaluate the accuracy of the method, other materials were tested. Methanol and ethanol (ethyl alcohol) were among these materials. The results of the measurements were compared with data from the literature. Due to the lack of information at the time the measurements were performed, most comparisons were made at room temperature. For example, Fig. 8 presents a comparison between permittivity measurements of methanol and Debye model for this material [6]. The black curve represents the model. There was very good agreement between theoretical and practical data.

![Graph of permittivity measurements of methanol and Debye model at room temperature.](image)

Fig. 8. Comparison between measurements of (a) $\varepsilon'$ and (b) $\varepsilon''$ of methanol and Debye model at room temperature.
C. Global Analysis of Uncertainty

Fig. 9 presents the measured errors of the real part of the permittivity and the loss tangent of water, ethanol and methanol at 2.45 GHz. The solid curve in this figure represents the typical accuracy of the probe extracted from Fig. 2. The points described in the illustration as Water 0, Water 1, Water 2 and Water 3 are relative to the measurements of pure water. The data of Water 0 was obtained during preliminary tests, without the recommended procedure of the previous section.

As it can be observed, the measurement errors are smaller than expected values for the probe, except for Water 0. Measurements in function of temperature have shown less accuracy at high temperatures. Even so, errors were within acceptable values.

Fig. 9. Accuracy in the measurement of (a) permittivity and (b) loss tangent of water, ethanol and methanol at 2.45 GHz.
V. CONCLUSION

This work presented the use of the Coaxial Probe Method for characterizing the dielectric constant of liquid materials at high temperatures. Due to the lack of a standard procedure in the literature for these measurements, the validation of the method involved the characterization of different liquids of known properties at different frequencies and temperatures, observing the manufacturer's recommendations and our own experience in the lab. A measurement procedure was established, which was reliable regarding repeatability and measurement accuracy. The reliability of the technique was demonstrated by comparing the deviation of the measured results from the data in the literature with the uncertainty limits supplied by the manufacturer of the adopted equipment.

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REFERENCES


