Temperature Sensitivity of Coaxial Probe Complex Permittivity Measurements: Experimental Approach

Bruce G. Colpitts

***Abstract*— An experimental investigation of the temperature sensitivity of the Teflon dielectric semi-rigid coaxial probe used in complex permittivity measurements is presented. Measurements are performed over the frequency range extending from 100 MHz to 26.5 GHz using 2.2 mm and 3.6 mm coaxial probes at a number of temperatures. An acute sensitivity of the probe-tip geometry to temperature is revealed along with its affect on measured complex permittivity. Measurements are further complicated by the nonlinear thermal phase response of the probe which results in hysteresis apparitions appearing in the measured complex permittivity during thermal cycling. The potential for removing these errors through temperature correction and the use of thermally stable probes is discussed.**

I. Introduction

THE COAXIAL probe is being used extensively in nondestructive measurement of complex permittivity, its small size makes it suitable for in vivo applications and TEM operation allows broadband coverage. There are now several techniques for relating probe impedance to the complex permittivity of the sample material [1]–[6] all requiring either a detailed knowledge of the probe geometry or a calibration procedure involving the measurement of standards. The technique has been demonstrated to have suitable accuracy for biological measurements [7] and has recently been improved by [2], [5], but the subject of temperature induced probe errors has received little attention.

This work demonstrates the temperature sensitivity of complex permittivity measurements using the coaxial probe. Small probe dimensions and measurement of the aperture impedance make this procedure very sensitive to geometrical deformation caused by changing temperature which induces errors in the measured results. The two error sources identified are those of probe deformation caused by differing thermal expansion coefficients between the dielectric and conductors, and the hysteresis that results from friction between the conductors and dielectric. Temperature induced errors are investigated experimentally for the case of operation both above and below the calibration temperature by measuring 0.1 M saline at a number of temperatures. The 0.1 M saline has been selected for this study as it provides a well defined reference with properties similar to those of biological materials.

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TABLE I
Thermal Expansion Coefficients for Copper, Teflon and Steel

Material	Thermal Expansion Coefficient /°C
Copper	17.7×10^{-6} [8]
Teflon	$100. \times 10^{-6}$ [9]
Steel	12.6×10^{-6} [10]

II. Temperature Sensitivity

It is common when making in vivo measurements that the samples are not at the same temperature as the probe and that in other cases one may wish to create a temperature profile of the complex permittivity. Both of the above procedures create a situation in the probe where its dimensions are altered through thermal expansion. The typical semi-rigid cable is formed from a copper outer conductor, a Teflon dielectric, and a silvered copper-covered steel inner conductor, each material having the thermal expansion coefficients of Table I. The greatest concern is generated by the high thermal expansion coefficients of the Teflon which causes the dielectric to protrude from the probe face when the probe is heated and the dielectric to recede when the probe is cooled as shown in Fig. 1. This phenomenon results in large errors in measured permittivity because of the sensitivity of the probe aperture admittance to changes in the probe-tip region (within one probe radius of the outer conductor [11]). There are differing rates of expansion for the center and outer conductors but they are very small compared to those of the dielectric and will not receive further consideration here. Probe hysteresis resulting from friction between the dielectric and conductors is an additional detrimental effect to the measurements along with the non-linear thermal expansion coefficient of Teflon. Also the overall length of the probe will vary with temperature causing additional errors in the measured results.

III. Theory

The method of Staebell [5] is used to transform measured reflection coefficient data into complex permittivity results. Other methods were considered such as [2] or [12], but these require an accurate description of the probe geometry which, in the cases evaluated here, produced less accurate results when published probe dimensions were used. The reasoning behind this is pointed out in [2] as a partial cancellation of errors that is particularly beneficial when the complex permittivity of the unknown is close to that of the calibration liquid as is the case in this work. The method used requires calibrating the system with four standards over the frequency band. The

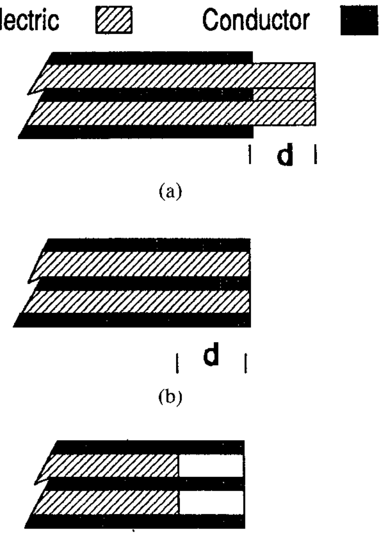


Fig. 1. Semi-rigid coaxial probe-tip shown for the case of (a) probe heated above the fabrication temperature, (b) probe at the fabrication temperature, and (c) probe cooled below the fabrication temperature.

calibration yields values for the unknown coefficient in a quadratic expression of the aperture admittance in terms of the complex permittivity of the unknown as in (1)

$$Y_L \simeq \epsilon_r^* + \zeta \epsilon_r^{*2} \tag{1}$$

where Y_L is the aperture admittance, ϵ_r^* is the relative complex permittivity of the sample, and ζ is the unknown coefficient which is sought. Since standard Automatic Network Analyzer (ANA) calibration techniques can not be applied at the probe aperture an equivalent two-port network is assumed between the aperture and the ANA to account for the probe and its connector. Now Y_L can be obtained if the system is calibrated using three standards, but if a fourth standard is measured the probe aperture dimensions need not be known. In this case once the calibration is performed the reflection coefficient for any unknown can be measured and translated into the unknown's complex permittivity.

IV. The Experiment

Two aspects of probe errors are experimentally investigated to demonstrate their significance in complex permittivity measurement where temperature is not constant. First, the effects of the dielectric moving with respect to the conductors creating either a void or protrusion at the probe-tip is investigated. Second, the effects of temperature cycling and the resulting hysteresis in the measurements is examined. This is followed by an evaluation of the receded dielectric probe.

A. Probe-Tip Deformation

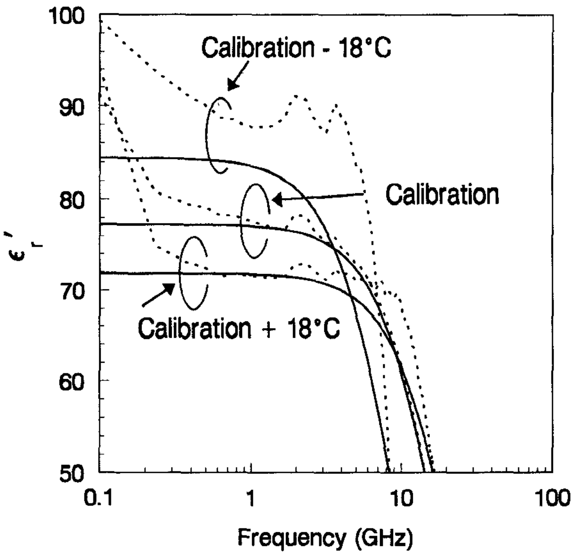
To illustrate the significance of temperature variations on permittivity measurements a Teflon dielectric semi-rigid coaxial probe of 3.6 mm was fabricated. This 24 cm probe has the thermal characteristics presented in Table I. The ANA system used in this work is an HP8510B Network Analyzer, an HP8340B Synthesized Sweeper, and an HP8515A S-Parameter Test Set with the probe connected directly to the test port. The probe is calibrated with the dielectric, center and outer conductors terminated in a single plane perpendicular to the cable axis using the following standards: open, short, water [13] and methanol [14]. The 0.1 M saline solution used as the test material is represented by complex permittivity data from [13]. In the case of measuring liquid samples care must be taken in order that the probe-tip not trap air as it is submerged, any trapped air is removed by tapping the probe until there is no change in the measured reflection coefficient.

The test procedure involves measuring 0.1 M saline at the calibration temperature then at temperatures 18°C above and below the calibration temperature. To achieve this the probe was immersed 5 cm into the saline solution and remained there until the measured reflection coefficient stabilized. The measured results now include the effects of the probe-tip deformation and the changing length of the probe. Fig. 2 presents the measured and reference [13] complex permittivity curves for the 0.1 M saline at the three temperatures.

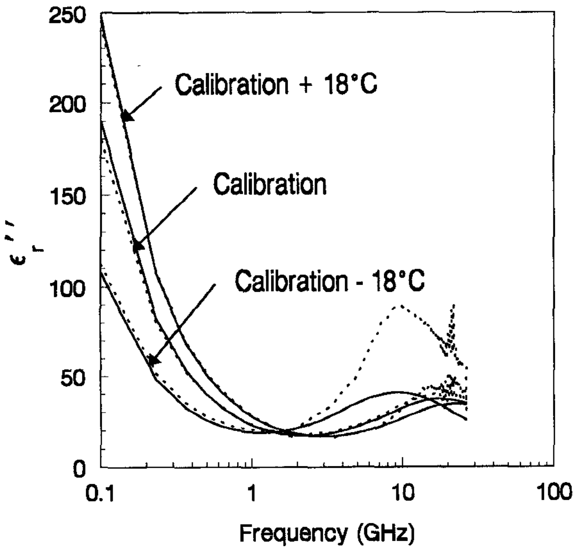
A comparison of the three cases reveals a distinct difference between the measurement of hot and cold saline solution where the cold solution yields larger errors. Table II presents a comparison of the mean percent error magnitudes for each of the measurements over a particular frequency range. Since the permittivity of the saline solution is a strong function of temperature it is necessary to examine the relative errors in each of the cases rather than absolute values. Examination of this table reveals that errors increase more rapidly when the probe is cooled compared to when it is heated. Thus, when measuring over a range of temperatures the probe should be faced and calibrated in the cool part of the range so as to minimize these errors.

B. Hysteresis

An additional complication to this temperature sensitivity is that the position of the dielectric relative to the conductors is not a linear function of temperature which is a result of friction between the conductors and the dielectric. When the probe is initially prepared for calibration the previous temperature extreme to which it has been exposed will have a great influence on any measurement that involves changing temperature. The last temperature extreme will determine whether at the calibration temperature the dielectric is under stress attempting to withdraw (was previously hot) or is attempting to advance (was previously cold). To demonstrate probe hysteresis a 12.5 cm probe was thermally cycled while monitoring the open circuit reflected phase. The experimental set up consisted of a water bath whose temperature could be varied between 0°C and 100°C and a heavy copper wire thermally connecting the water bath to the probe midpoint. The probe temperature was monitored on the outer conductor at a central location with a thermocouple. The phase versus temperature results are presented in Fig. 3 where hysteresis is clearly seen for two cycles of temperature variation from room temperature to 14°C then increasing to 35°C and back to room temperature. An important observation here is that as the



(a)



(b)

Fig. 2. Measured and reference values of the complex permittivity of 0.1 M saline obtained using a 3.6 mm probe at the temperatures indicated. The solid lines indicate the reference values [13] while the dashed lines represent the measured values for the (a) real permittivity and (b) imaginary permittivity.

TABLE II
MEAN PERCENT ERROR MAGNITUDE FOR 0.1 SALINE COMPLEX PERMITTIVITY MEASUREMENTS

Description/ Temperature	Frequency (GHz)	Error Real	Error Imaginary
Calibration/ 23° C	0.1 - 1.0	4.1	1.9
	1.0 - 10.0	1.1	4.3
	10.0 - 26.5	2.7	8.1
	0.1 - 26.5	2.3	6.6
18° C above calibration	0.1 - 1.0	4.9	2.0
	1.0 - 10.0	4.9	5.0
	10.0 - 26.5	8.8	30.7
	0.1 - 26.5	7.3	21.0
18° C below calibration	0.1 - 1.0	8.2	7.9
	1.0 - 10.0	16.6	61.7
	10.0 - 26.5	35.0	112.7
	0.1 - 26.5	27.8	91.5
Calibration with receded dielectric at 19° C	0.1 - 1.0	3.7	2.0
	1.0 - 10.0	6.2	16.9
	10.0 - 26.5	36.2	28.4
	0.1 - 26.5	24.9	23.4

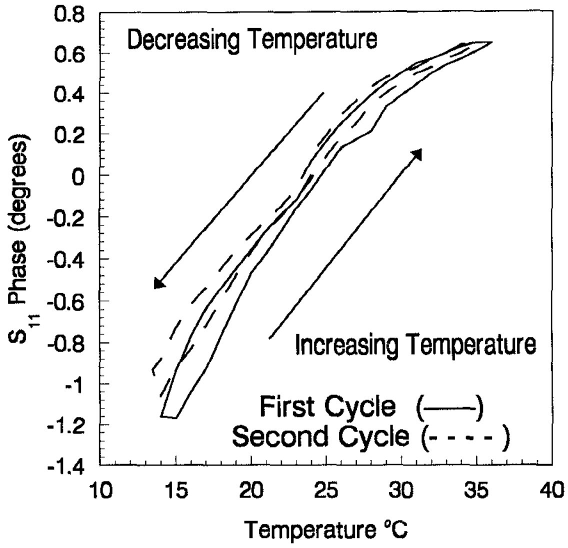


Fig. 3. Measured S_{11} phase versus temperature results for an open circuited 2.2 mm probe of length 12.5 cm at a frequency of 13.3 GHz.

temperature decreases, causing the probe dielectric to recede, the measured S_{11} phase is indicating a greater electrical length to the open circuit. This is opposite to the effect one would expect from a probe of decreasing length. In fact, the previously planar faced probe now has a receded dielectric producing a significant change in the reflection coefficient which appears to lengthen the cable. This phenomenon is a result of two parts, the first is the change in the probe-tip geometry while the second is the incremental change in the overall probe length. The slope of the curve in Fig. 3 is opposite to what is expected from a thermal length change in the cable, thus it appears that the change in probe-tip geometry is the dominant effect.

Further effects can be seen in the measurement of 0.1 M saline when the probe has undergone various preconditioning. Fig. 4 presents a family of curves showing the measured permittivity at room temperature for a number of preconditioning cases. Case 1 is for a probe exposed to −16°C for one hour then returned to room temperature for 12 hours before the measurement. In case 2 the probe-tip was immersed in 100°C water for 2 minutes then returned to room temperature before measurement. In case 3 the probe was cooled by immersion in 0°C water for 2 minutes and returned to room temperature. Cases 4 and 5 are the same as 2 and 3 respectively but represent a second temperature cycle. Note that the first change is the greatest while subsequent changes take on a pattern with the hot cycle showing a lower real permittivity while the cold cycle shows a higher permittivity. The large initial change resulted from stresses built up from the last cycle of preconditioning where the cable was exposed to −16°C for one hour.

C. Receded Dielectric Probe

The receded dielectric probe was calibrated in the withdrawn state (Fig. 1(c)) as if it were a normal probe as a means of determining if the calibration of this distorted probe

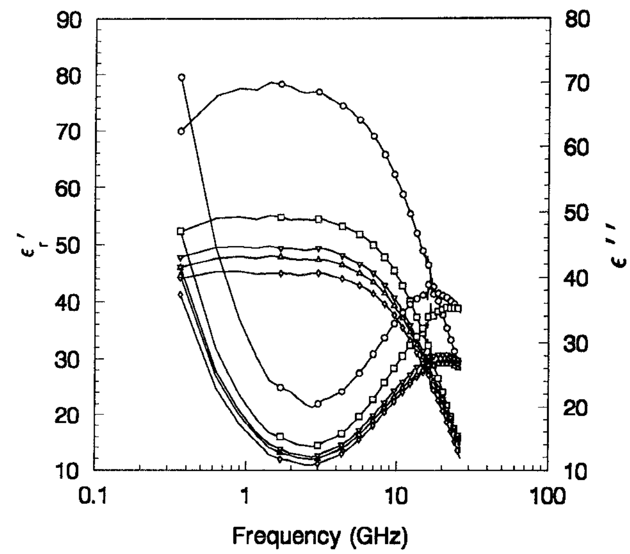


Fig. 4. Measured complex permittivity of 0.1 M saline using a 2.2 mm probe at room temperature that has been exposed to a sequence of temperature changes before the measurement as follows: 1. Previously exposed to −16°C (o—o—o), 2. exposed to 100°C water (◇—◇—◇), 3. exposed to 0°C water (□—□—□), 4. exposed to 100°C water (△—△—△), and 5. exposed to 0°C water (▽—▽—▽).

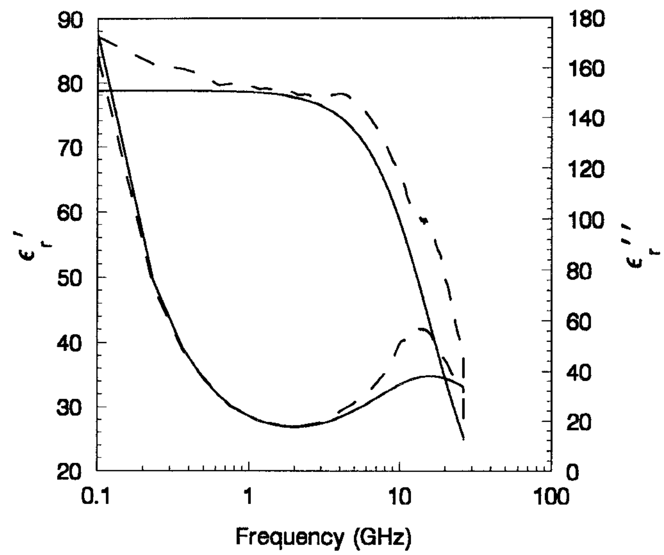


Fig. 5. Reference (—) and measured (- - -) values of the complex permittivity of 0.1 M saline for the case of a 3.6 mm probe calibrated with the dielectric receded by 0.25 mm.

was feasible. An important consideration in this test is to ensure that the liquid samples fill the void at the probe-tip. Processing of the measured data proceeded using the technique of [5] producing the results shown in Fig. 5. Table II presents the mean percent error magnitude of this measurement over several portions of the frequency spectrum for comparison to the properly faced probe. The 3.6 mm probe had a 0.25 mm section of the dielectric removed which is equivalent to a 24 cm probe cooling by approximately 12°C. With reference to Table II the below 1 GHz response is superior to the planar faced probe at calibration temperature for the real part of the permittivity and comparable for the imaginary part. This advantage is lost at the higher frequencies where the planar faced probe has much lower errors but it appears there is a significant improvement over the probe that is cooled without further calibration. Use of the receded dielectric probe appears to have two applications. One is that the low frequency accuracy of the measurements can be enhanced apparently by increasing the volume of the sample interrogated. This is advantageous for measuring small samples where the use of a larger probe size is not possible. The second application is to use the receded dielectric calibration along with the properly faced calibration and interpolate to produce calibration data for temperatures between. In this case care must be taken to monotonically move from one calibration temperature to the next and to use the same procedure during the measurement in order to minimize hysteresis effects.

V. Potential For Temperature Compensation

In order to compensate for temperature dependence in the probe-tip geometry it would be necessary to predict the dielectric position beyond the simple linear thermal expansion coefficients, particularly when large thermal excursions are experienced. The correction process would require a model of the probe-tip beyond simple transmission line theory in order to account for the higher order modes that will exist near the probe-tip, this will most likely require numerical techniques. The accuracy can be improved by calibrating the probe at two temperatures and interpolating for points between. With these difficulties arising in a temperature correction scheme it appears that the design of a thermally stable probe is the essential step in making use of this technique for complex permittivity measurements over broad temperature ranges. An air line with a thermally stable aperture window may be the solution provided the window can be properly accounted for in calibration or a coaxial line that has equal thermal expansion coefficients for both the conductors and dielectric. Artifacts of these temperature induced errors have appeared in the literature particularly of note is the hysteresis found in [11] when measuring leaves during freeze thaw temperature cycling. It appears, based on this work, that at least part of the hysteresis in these measured results could be explained by hysteresis in the probe since the temperature was cycled between +22°C and −32°C. Permittivity measurements have also been performed over a broad temperature range in [15] where a semi-rigid coaxial probe is used, these results would also be in suspicion at temperatures other than calibration.

VI. Conclusion

The dielectric filled semi-rigid coaxial probe is shown to be very sensitive to temperature when used in measuring the complex permittivity of 0.1 M saline. Although the accuracy is good at the calibration temperature it decreases when the probe is heated and the deterioration is even more severe when the probe is cooled. The extreme sensitivity of these complex permittivity measurements to small changes in the probe-tip geometry make temperature control or compensation essential for accuracy. If complex permittivity is to be measured over a temperature range as in the creation of thermal complex permittivity profiles then an analysis method that will account for the deformed geometry needs development. The procedure to overcome this is to use a probe constructed with conductors and dielectric having the same thermal expansion coefficients or to use an air dielectric cable with a thermally stable window at the aperture. One advantage of using a withdrawn dielectric probe is that the low frequency accuracy is slightly improved over the planar faced probe.

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