Measurement of Complex Permittivity of Polystyrene Composite at 11.64 GHz Using Cavity Perturbation Technique

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Abstract ─ Complex permittivity of polystyrene composites of different percentage of carbon black fillers are measured over the wide frequency range 8-12 GHz using X-band rectangular cavity resonator. The measurement technique uses the cavity perturbation technique. Validity of present measurement technique has been checked by measuring the dielectric properties of well-known dielectric materials (Teflon and Rexolite). Polystyrene composites are very useful for lightweight shielding and absorbing materials. It would be of great interest for the community to find its dielectric properties over wider frequency range. Due to lack of experimental data on dielectric parameters of carbon black composite in literature, the experimental study has been conducted to measure the dielectric properties of polystyrene composite over wide frequency range of X-band (8-12 GHz). In addition, the estimation of measurement error associated with this technique is also discussed.

Index Terms ─ Cavity perturbation, cavity resonator, complex permittivity, dielectric constant, dielectric loss, dielectric material

I. INTRODUCTION

Complex permittivity cannot be measured directly; it is usually calculated via other measurable parameters such as transmission/reflection coefficients (s-parameters), propagation constant, etc. Measurement of these parameters requires very precise and accurate experimental approach associated with specific formulas. Accurate information for the dielectric properties is required to study the possible hazard of EM field [1]. The dielectric properties of materials are determined by its molecular structure, if the molecular structure changes its dielectric properties changes. Any single technique for complex permittivity measurement is not suitable over a wide frequency range and complex permittivity. Numerous techniques for determination of dielectric properties of dielectric material have been developed [2-3]. Among various frequency domain methods for the measurements of complex permittivity, cavity perturbation technique is one of the most widely used because of its relative simplicity for precise microwave measurements of conductivity, dielectric, and magnetic properties of materials. In this technique, one measures the adiabatic change of the characteristics of a resonator upon the introduction of a foreign body (sample). This technique requires the accurate determination of quality factor and resonant frequency of microwave cavity resonator. Different cavity perturbation techniques have different limitations, i.e., the insertion of sample in the cavity [4]; size and shape of sample; type of cavity resonators etc.; hence further progress in this field is required.

Normally, the samples required for measurements in a rectangular cavity are equal to the height of the cavity. When the samples are smaller than the height of the cavity, accurate measurements are often difficult [5]. The objective of cavity perturbation technique is to measure accurately and precisely the quality factor Q and resonance frequency f_o of unloaded and loaded microwave cavity resonator using transmission or reflection coefficient (s-parameters) as a function of frequency [6-8].

A rectangular cavity resonator has been designed here with very small hole at the centre of broader side of the X-band waveguide resonator in order to insert a sample material under test for the measurement of complex permittivity [9]. Since cavity is operated in TE_{10n} mode, it has a number of resonant frequencies. The resonance frequency and quality factor of empty cavity and sample loaded cavity are measured at different resonances to perform experiment. Five samples of polystyrene composite with different percentage of carbon black are prepared by mixing polystyrene and carbon black together at room temperature. We use the similar process here to prepare the samples as given in

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the cited reference [10], the only difference is that we use a glass capillary of 4 mm inner diameter to cast the mixture for uniform cylindrical shaped samples of 4 mm diameter. Measurements are performed at room temperature. The effect of casting process, pressure and temperature profile of sample preparation is not studied here.

The measurement setup uses cavity resonator and VNA. The real and imaginary parts of the complex permittivity have been calculated from the shift in the resonance frequency and change in the Q-factor. In order to verify the validity of the measurement technique, real and imaginary parts of the complex permittivity of certain standard dielectric material (Teflon and Rexolite) are measured at X-band and the measured vales are compared with the values available in literature [11]. The complex permittivity of different compositions of carbon black has been measured at X-band. The experimental results obtained at 9.77 GHz with the present method have been compared with the existing values in literature [10]. However, the dielectric data of carbon black composite over the entire frequency range of 8-12 GHz are not available for comparison.

II. THEORETICAL BACKGROUND

It was Bethe and Schwinger [12] who proposed cavity perturbation technique for the first time. Thereafter, many researchers have reported theoretical and experimental analysis of cavity perturbation techniques [6-17]. The measurements of complex permittivity (**ε^***) and permeability (**μ^***) are performed by inserting a small, appropriately shaped sample into a cavity and determining the properties of the sample from resultant change in the resonant frequency and loaded quality factor.

The basic idea of the cavity perturbation is the change in the overall geometric configuration of the electromagnetic fields upon the insertion of a small sample must be small. Based on this assumption, a detailed derivation of the perturbation equation for the frequency shift upon the insertion of a sample into a cavity was given by Harrington [14].

When a small sample is inserted in a cavity having electric field E_0 and magnetic field H_0 in the unperturbed state, the fields in the interior of the sample are E and H. For loss less sample, the variation of resonance frequency is given by [12,13] as:

$$\frac{f_s - f_0}{f_s} = -\frac{\int(\Delta\varepsilon E.E_0^* + \Delta\mu H.H_0^*)d\tau}{\int(\varepsilon E.E_0^* + \mu H.H_0^*)d\tau}, \qquad (1)$$

where ε and μ are the permittivity and permeability of the medium in the unperturbed cavity respectively. $d\tau$ is the elementary volume, $\Delta\varepsilon$ and $\Delta\mu$ are the changes in the above equations due to the introduction of the sample in the cavity. Without affecting the generality of Maxwell's equations, the complex frequency shift due to lossy sample in the cavity is given by Waldron [15] as:

$$\frac{-df^*}{f^*} = \frac{(\varepsilon_r - 1)\varepsilon_0 \int_{v_s} E.E_0^* dv + (\mu_r - 1)\mu_0 \int_{v_s} H.H_0^* dv}{\int_{v_c} (D_0.E_0^* + B_0.H_0^*)dv}, \qquad (2)$$

where df^* is the complex frequency shift as the permittivity of materials is a complex quantity, so the resonance frequency is also complex. B_0, H_0, D_0 and E_0 are the fields in the unperturbed cavity and E and H is the field in the interior of the sample [16].

In terms of energy, the numerator of Equation (2) represents the energy stored in the sample and the denominator represents the total energy stored in the cavity. The total energy $\boldsymbol{W = W_e + W_m = 2\,W_e = 2W_m}$, where $\boldsymbol{W_e}$ and $\boldsymbol{W_m}$ are the electrical and magnetic energies, respectively. Two assumptions are applied in equation (2). The fields in the empty part of the cavity are negligibly changed with the insertion of the sample. The fields in the sample are uniform over its volume. Both of these assumptions can be considered valid if the sample is sufficiently small relative to the resonant wavelength. The negative sign in Equation (2) indicates that by introducing the sample the resonance frequency is lowered. When a dielectric sample is inserted into the cavity resonator at the position of maximum electric field, only the first term in the numerator is significant, since a small change in ε' at a point of zero electric field or a small change in μ at a point of zero magnetic field does not change the resonance frequency. Therefore, Equation (2) can be reduced to:

$$\frac{-df^*}{f} = \frac{(\varepsilon_r - 1)\int_{v_s} E.E_{0\max}^* dv}{2\int_{vc} |E|^2 dv}. \qquad (3)$$

III. MEASUREMENT OF COMPLEX PERMITTIVITY

The cylindrical sample is taken with uniform cross sectional area '***s***' and length is greater than narrow dimension '*b*' of the cavity, so that it will occupy the entire narrow dimension of the cavity. Due to the change in the overall capacitance and conductance of the cavity on the introduction of the sample, the resonance frequency decreases from f_0 to f_s and the quality factor from Q_0 to Q_s.

The procedure for the determination of complex permittivity is described as follows.

1. The resonance frequency (f_0) and unloaded quality factor (Q_0) of the cavity resonator are measured with the empty cavity at the position of maximum electric field.
2. The sample is inserted in the cavity at the position maximum electric field without dismantling the

cavity resonator. The shifted resonance frequency f_s and loaded Q-factor Q_s are measured.

3. Knowing the volume of the cavity and the sample, the shift in resonance frequency and Q-factor, the dielectric constant and dielectric loss of the sample can be computed.

The complex resonant frequency shift is related to measurable quantities by [4], [5]:

$$\frac{df^*}{f} = \frac{f_s^2 - f_0^2}{f_s^2} + \frac{j}{2}\left(\frac{1}{Q_s} - \frac{1}{Q_0}\right). \tag{4}$$

On equating real and imaginary parts of Equation (3) and (4), we have:

For real part,

$$\frac{-(f_s - f_0)}{f_s} = \frac{(\varepsilon_r^{/} - 1)\int_{V_S} E.E_{0\max}^* dv}{2\int_{V_C} |E_0|^2 dv}. \tag{5}$$

We may assume that $E = E_0$ and the value of E_0 in the TE_{10p} mode is $E_0 = E_{0max} \sin(p\pi z/l) \sin(p\pi z/l)$, where a is the broader dimension of the cavity and l is the length of the cavity. Integrating and rearranging the Equation (5), we obtain:

$$\frac{f_0^2 - f_s^2}{f_s^2} = \frac{4V_s\varepsilon^{/}}{V_c},$$
$$\varepsilon^{/} = \frac{V_c(f_0^2 - f_s^2)}{4V_s f_s^2}, \tag{6}$$

where V_c is volume of the cavity = $a \times b \times l$ (dimensions of the cavity) and V_s is the volume of the sample = $\pi r^2 h$ (r is the radius and h is the length of the sample):

For imaginary part,

$$1/2\left(\frac{1}{Q_s} - \frac{1}{Q_0}\right) = \frac{\varepsilon_r^{//}\int_{v_S} E.E_{0\max}^* dv}{2\int_{v_S} |E|^2 dv}. \tag{7}$$

Integrating and rearranging the Equation (7), we obtain:

$$\left(\frac{1}{Q_s} - \frac{1}{Q_0}\right)\frac{f_0^2}{f_s^2} = \frac{4V_S\varepsilon^{//}}{V_C},$$
$$\varepsilon^{//} = \left(\frac{1}{Q_s} - \frac{1}{Q_0}\right)\frac{V_C f_0^2}{4V_S f_s^2}, \tag{8}$$

where Q_s is the quality factor of cavity loaded with sample and Q_0 is the quality factor without sample. The measured values of Q (Q_0 & Q_s) of the cavity are calculated by the equation given below:

$$Q = \frac{f_{resonant}}{\Delta f_{3dB}} = \frac{f}{f_R(3dB) - f_L(3dB)}. \tag{9}$$

Equation (5) and (7) are the expression for complex permittivity using cavity perturbation technique [9].

IV. EXPERIMENTAL SET UP

A transmission type rectangular cavity resonator is designed and fabricated using standard WR-90 waveguide at X-band with inner dimension of 23 x 10 mm. Depending upon the modes to be propagate, the length has been chosen. The fundamental mode is TE type with *10n* where *n* is the number of half wavelength along the propagation direction. Two thin conducting sheets are used to close the two ends of the waveguide to form a cavity resonator. The inductive coupling is provided with two symmetric holes of diameter 4 mm on these end sheets. The design specifications are given below:

- Frequency Range: 8 GHz - 12 GHz (X-band);
- Cut of wavelength: 46 mm;
- Modes Propagates: 5 (TE_{105}, TE_{106}, TE_{107}, TE_{108}, TE_{109});
- Inner dimension of cavity: 22.9 x 10 x 140 mm^3;
- Outer dimension of cavity: 25 x 12 x 140 mm^3;
- Material used: Brass;
- Coupling hole: 4 mm.

In order to insert a sample material in the resonator cavity without disassembling it, a narrow hole is constructed at the centre of the broader side of the cavity. The narrow opened hole has negligible effect on changing the geometrical configuration of electromagnetic field inside the cavity. The width of the sample hole is equal to the diameter of the cylindrical sample. This rectangular waveguide cavity resonator is connected to two ports of the Network Analyzer for S-parameters measurement. A conventional thru-reflect-line (TRL) calibration technique is applied for calibration. The cavity resonator has multiple resonant frequencies in particular frequency band, TRL calibration was reused in each resonant frequency range and the measurement was separately performed in each resonant frequency range.

V. ERROR ANALYSIS

No measurement of a physical quantity can be entirely accurate. There is an inherent error margin associated with the results. Error in measurement is the difference between the measured value and the unknown, true, value of the measured quantity [18]:

$$\sigma^2_{error} = \sigma^2_{Measurement} - \sigma^2_{True}.$$

The precision of measurement can best be improved through the correction of the causes of variation in the measurement process. However, it is frequently desirable to estimate the confidence interval for the mean of measurements which includes the measurement error variation. The confidence interval for the mean of these measurements is reduced by obtaining multiple readings. To estimate the total measurement error, uncertainty analysis is conducted on the present measurements. The calculation of uncertainty requires a detailed budget which breaks down the variance of measurement error into consistent components, each of which can be separately estimated. The detailed model becomes like: $\sigma^2_M = \sigma^2_{instrument} + \sigma^2_{fixture} + \sigma^2_{enivironment} + \sigma^2_{calibration} + \sigma^2_{sample} + \sigma^2_{analysis}$.

Uncertainty due to repeatability, i.e., series of observations/readings at different interval of time, variations in cables, connectors, relevant accessories, AC mains, change of operators, variations in environment conditions, i.e., temp, humidity etc. have been calculated by taking standard deviation of the mean of the readings of five observations of each parameters. Uncertainty due to standard deviation (Usd) is calculated as follows:

$$U_{sd} = \sqrt{\begin{matrix}(1/5(5-1)) \times \{(X_1 - X_M)^2 + (X_2 - X_M)^2 + \\ (X_3 - X_M)^2 + (X_4 - X_M)^2 + (X_5 - X_M)^2\}\end{matrix}}, \quad (10)$$

where X_1, X_2, X_3, X_4, X_5 are readings at different interval of time and X_M is mean value. The degree of freedom = (No. of observations) – 1, i.e., 5-1=4.

Uncertainty arises from flaw in the measurement which repeats each time a measurement is made. It is mainly due to errors in the calibration of the measuring instruments. It depends upon calibrator's accuracy, its resolution, calibration certificate, and its temperature drift specifications etc. Uncertainty due to calibrator's accuracy (U_{a1}), calibrator's resolution (U_{a2}), calibrator's calibration certificate (U_{a3}), and due to temp drift in calibrator's specifications (U_{a4}) at 95%, confidence is calculated. Combined uncertainty (Uc) will be:

$$Uc = \sqrt{(U_{sd})^2 + (U_{a1})^2 + (U_{a2})^2 + (U_{a3})^2 + (U_{a4})^2}. \quad (11)$$

Overall measurement uncertainty ±U = kUc; where k = 1.96 for 95% confidence level. At 95% confidence interval, the error is ± 1.5 ppm.

VI. RESULTS AND DISCUSSION

The dielectric constant and dielectric loss of Teflon and Rexolite are measured at X-band. Table 1 shows a comparison of complex permittivity of Rexolite obtained with the present method and other method in literature [11]. The experimental results for Rexolite are in good agreement with those obtained using other method in literature.

The values for Teflon have been presented earlier in reference [9]. The cavity was connected to a network analyzer and could be excited to operate in five modes. Accordingly, five resonant peaks corresponding to frequencies around 8.46, 9.15, 9.96, 10.7 and 11.64 GHz appeared on the screen of the analyzer. The S-parameter (transmission co-efficient) measurement for Rexolite in X-band is shown in Fig. 1. Figure 1 shows the expected shifts in resonance frequency and quality factor. 3 dB down point on the resonance curves are noted down to calculate quality factor (Q_0) of empty cavity and sample loaded cavity (Q_s). Using Equations (6) & (8), dielectric constant and dielectric loss of sample materials have been calculated.

Complex permittivity of different samples of polystyrene composite are measured at X-band (8-12 GHz). Comparison of the dielectric constant and dielectric loss for different composition of carbon black at resonance frequency of 9.96 GHz obtained with the present experiment and available in literature [10] is presented in Fig. 2.

Table 1: Comparison of dielectric constant & dielectric loss of Rexolite

	Measured			Literature		
f_0 (GHz)	f_S (GHz)	ε′	ε″	f_S (GHz)	ε′	ε″
8.46	8.245	2.34	0.0021	8.78	2.35	0.0012
9.15	8.93	2.209	0.0013	-	2.36	0.00128
9.96	9.705	2.35	0.0012	9.927	2.33	0.0002
11.64	11.34	2.37	0.0002	11.08	2.33	0.0002

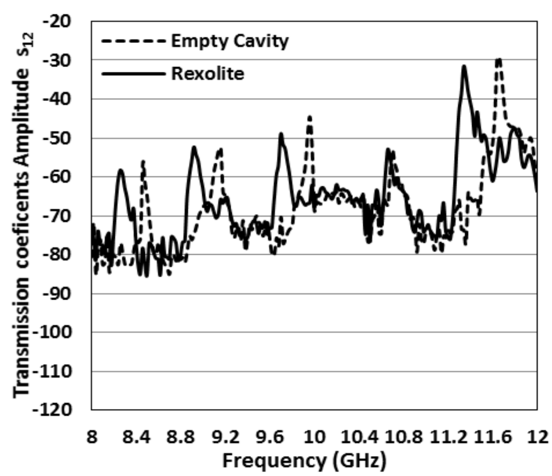


Fig. 1. Transmission coefficient measurement for Rexolite in X-band.

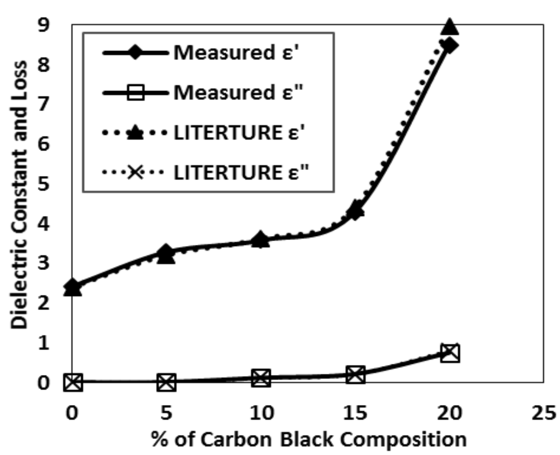


Fig. 2. Comparison of ε' & ε" at different % of carbon black composition.

The shift in resonance frequency & Q-factor depend on the dielectric properties of the material; the dielectric constant and dielectric loss of each composites of carbon black are measured on that specific frequency. In present measurement, the shift in resonance frequency for various carbon compositions varies from 9.70 GHz to 9.57 GHz. The experimental values of dielectric constant and dielectric loss for different polystyrene composite at 9.77 GHz are in good agreement with those obtained in literature. Finally, the dielectric properties of polystyrene composites are measured at X-band using this two port transmission type cavity resonator. Tables 2 (a) & 2 (b) shows experimental values of dielectric constant and dielectric loss for different composites of carbon black.

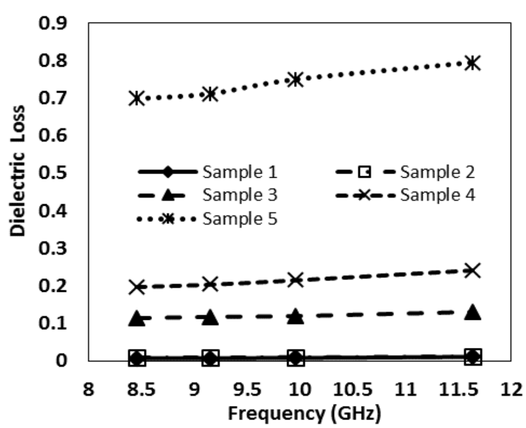
The dielectric parameters of five different samples; sample 1 (100% PS + 0% CB), sample 2 (95% PS + 5% CB), sample 3 (90% PS + 10% CB), sample 4, (85% PS + 15% CB) and sample 5 (80% PS + 20% CB) of polystyrene composite at wider frequency range of 8-12 GHz are also represented in Figs. 3 (a) & (b).

Table 2 (a): Dielectric constant **(ε′)** of polystyrene composite at various resonance frequencies (f_0) in X-band (8-12 GHz)

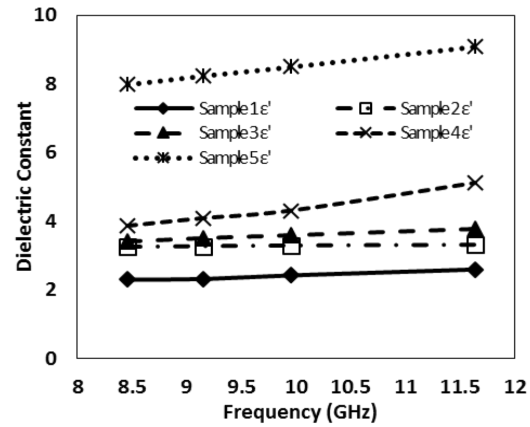
f_0 (GHz)	0% CB	5% CB	10% CB	15% CB	20% CB
8.46	2.29	3.249	3.856	7.95	7.95
9.15	2.313	3.265	4.076	8.234	8.234
9.96	2.415	3.28	4.3	8.5	8.5
11.64	2.59	3.3	5.1	9.08	9.08

Table 2 (b): Dielectric loss **(ε")** of polystyrene composite at various resonance frequencies (f_0) in X-band (8-12 GHz)

f_0 GHz)	0% CB	5% CB	10% CB	15% CB	20% CB
8.46	0.007	0.0084	0.115	0.198	0.699
9.15	0.0076	0.0087	0.118	0.204	0.712
9.96	0.0080	0.0098	0.1200	0.215	0.750
11.64	0.011	0.0127	0.1295	0.24	0.795



(a)



(b)

Fig. 3. (a) Variation of measured dielectric constant as a function of frequency, and (b) variation of measured dielectric loss as a function of frequency.

VII. CONCLUSION

In this paper, cavity perturbation technique for measuring the complex permittivity of polystyrene composite at X-band is described. We have presented measured results compared with those existing in literature to show good agreement. It has been observed from Tables 2 (a) & 2 (b) that, the values of dielectric constant and dielectric loss are increasing with the increase of carbon black concentration in polystyrene. For polystyrene composites, the dielectric data over entire frequency range are not available, so the dielectric behavior of polystyrene composites presented here is of great interest for the community. Measurement error has been estimated and found that at 95% confidence interval, the error is ± 1.5 ppm. The dielectric properties of the material can be measured with existing cavity resonator with an accuracy of 1.5 ppm.

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