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Measurement of dielectric properties of medium loss samples at X-band frequencies

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This work describes and evaluates a technique for determining dielectric properties, and presents results of dielectric measurement of some substances. Dielectric properties of some known dielectric materials are first measured and verified with our technique and then applied to conducting polymer materials. A non-destructive method based on the shift in resonance frequency and quality factor measurement of a resonant cavity placing a small-sized sample is used. Dielectric constant and loss factor measurement is performed with the aid of Network Analyzer in the frequency range from 8GHZ to 12GHz. 3-D EM simulation studies using HFSS 11 software of the cavity with loading is compared with the experimental measurements. The results indicated that using the resonant cavity technique is suitable for dielectric parameter measurement for small shaped medium loss samples having thickness in the range 4-6mm at X-Band frequencies.

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1. Introduction

There is a continual demand for accurate measurements of the dielectric properties of solids and liquids. Typical application of dielectric measurements are for cancer research, building material, negative index material, electromagnetic shielding, for the propagation of wireless signals etc. Various methods are available for the measurement of dielectric properties of materials at microwave frequencies among them techniques based on cavities are the most accurate [1]-[3]. In this method dielectric constant and loss factor of the material has been determined by inserting a small, appropriately shaped sample into the cavity and determining from the shift in resonance frequency and quality factor with respect to the unloaded cavity [4]-[9].

The earliest treatment of the cavity perturbation theory was given by Bethe and Schwinger [10]. Later Waldron derived a detailed theory of the cavity perturbation method based on following three assumption i.e. (1) original medium in the cavity should have no loss, (2) Sample is homogeneous and is much smaller than the cavity and (3) electromagnetic field outside the sample does not change. As the sample used in cavity are in the form of a thin rod, the length of which equals the height of the cavity, so that both the ends of the specimen are in contact with the cavity walls [11].

In the cavity perturbation method small holes can be drilled in the cavity walls and the sample can be inserted into the sample holder. By using this technique the cavity need not be taken apart for placing the sample and errors due to misalignment of the cavity may be reduced. This technique is to insert the full length sample through hole drilled in the wall of the cavity used by Kumar et al [12].

However it often becomes difficult to obtain large samples. Furthermore with the large samples the transmitted signal level becomes too low for accurate measurement of the change in frequency and Q factors as a result of perturbation. The need therefore arises to observe the changes for samples whose dimensions are much smaller than that of the cavity.

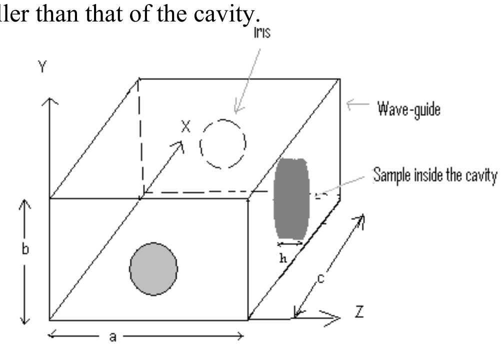

Fig 1. Placement of Sample inside the cavity

The technique described in this paper allows the sample to be placed against one of the narrow walls of the cavity where the perturbation of the electric field is least (Fig 1). It was applied to determine the conductivity and permittivity of standard samples viz Teflon, Perspex and Bakelite and then applied to determine these parameters for a test sample (EVA rubber composite). The technique adopted is quite simple, cheap and has the advantage of determining the permittivity and conductivity of samples which are difficult to obtain in large sizes.

2. Principle of cavity perturbation method

Consider two almost identical cavities denoted by subscripts 1 and 2. Cavity 1 is empty and cavity 2 contains a very small size dielectric material. The cavity walls are assumed to be lossless. Maxwell's equations for these cavities can be written as:

$$\vec{\nabla}\times\vec{E}_j=-j\omega_j\mu_j\vec{H}_j \tag{1}$$

$$\vec{\nabla}\times\vec{H}_j=j\omega_j\varepsilon_j\vec{E}_j \tag{2}$$

where j=1 and 2 for the respective cavities.

Using the four equations obtained from (1) and (2), and integrating over the cavity volume one obtains: [13]

$$\frac{\omega_2-\omega_1}{\omega_2}\approx-\frac{\iiint_{v_s}\left(\Delta\varepsilon E_1^*\cdot E_2+\Delta\mu H_1^*\cdot H_2\right)dv}{\iiint_{v_c}\left(\varepsilon_1 E_1^*\cdot E_2+\mu_1 H_1^*\cdot H_2\right)dv} \tag{3}$$

where electric and magnetic fields of the empty cavities are E_1 and H_1, E_2 and H_2 are the fields inside the perturbing sample. V_c and v_s are the volumes of the cavity and of the sample respectively. Ω_2 and ω_1 are respectively the resonant angular frequencies of the cavity with and without the sample.

For non-magnetic samples $\mu_1=\mu_2=\mu_0$ and $\varepsilon_1=\varepsilon_0$ for the empty cavity the (3) can be expressed as

$$\frac{\omega_2-\omega_1}{\omega_2}=-\frac{\varepsilon_r^{'}-1}{2}\frac{\iiint_{v_s}E_1^*\cdot E_2\,dv}{\iiint_{v_c}\left|E_1\right|^2dv} \tag{4}$$

Now considering complex angular frequencies we can write $\omega=\omega_r+j\omega_i$ and assuming small perturbation

$$\frac{\omega_2-\omega_1}{\omega_2}=\frac{\left(\omega_{r_2}-\omega_{r_1}\right)+j\left(\omega_{i_2}-\omega_{i_1}\right)}{\omega_{r_2}\left(1+j\frac{\omega_{i_2}}{\omega_{r_2}}\right)} \tag{5}$$

If Q_1 and Q_2 are the Q factors respectively for the empty cavity and that with the sample and considering that Q_2 generally has values between 100 to 1000 (5) reduces to

$$\frac{\omega_2-\omega_1}{\omega_2}=\left(\frac{f_2-f_1}{f_2}\right)+j\left(\frac{1}{2Q_2}-\frac{1}{2Q_1}\right) \tag{6}$$

From (4) and (6) we have

$$\left(\frac{f_2-f_1}{f_2}\right)+j\left(\frac{1}{2Q_2}-\frac{1}{2Q_1}\right)=-\frac{\varepsilon_r^{'}-1}{2}\frac{\iiint_{v_s}E_1^*\cdot E_2\,dv}{\iiint_{v_c}\left|E_1\right|^2dv} \tag{7}$$

Hence,

$$2\frac{f_1-f_2}{f_2}=\varepsilon_r^{'}-1\frac{\iiint_{v_s}E_1^*\cdot E_2\,dv}{\iiint_{v_c}\left|E_1\right|^2dv} \tag{8}$$

$$\frac{1}{Q_2}-\frac{1}{Q_1}=\varepsilon_r^{''}\frac{\iiint_{v_s}E_1^*\cdot E_2\,dv}{\iiint_{v_c}\left|E_1\right|^2dv} \tag{9}$$

Now if ε_r' and ε_r'' are the real and imaginary parts of the permittivity and σ the conductivity of the material, and ω_0 and ω are the resonant frequencies of the empty and loaded cavity respectively, then

$$\sigma=\varepsilon_r^{''}\cdot\omega\varepsilon_0 \tag{10}$$

3. Measurement system

Three rectangular X-Band cavities having lengths 27.5mm, 21.8mm and 18.3mm with widths 22.9mm and height 10.2mm were used to resonate respectively at 8.51GHz, 9.46GHz and 10.47GHz for the measurement of the dielectric properties. The narrow wall of the cavity had a thin slot nearly 4 cm in length and 1.2mm in width. The sample is placed firmly by means of a spring in the sample holder. A micrometer screw gauge is attached to the sample holder which gives the distance moved by the sample inside the cavity. The sample was gradually inserted inside the cavity with the increment of 1 mm at which measurements are taken. Small irises having diameter 2.5mm at either end of the cavity coupled the power into and out of it.

The frequency was kept stable by using Agilent E8257D signal generator. The cavity was connected to a sensitive spectrum analyzer for the detection of shift in resonance frequency due to insertion of sample in the cavity. The quality factor was determined by 3dB method using a Precision Attenuator HP X382A [Fig 2]. The cavity was excited at TE_{011} mode.

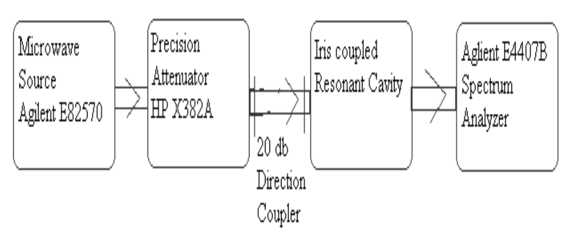

Fig 2. Experimental Set-up.

4. Measurement samples

Bakelite sample, grade 'P 1001' from Raj Udyog, India (Having Tensile strength 1050kg/cm^2, cross breaking strength 1550kg/cm^2 and specific gravity 1.42) was used. EVA rubber, containing vinyl acetate (28%), grade 'Pilene 2806' from polyolefin industries limited, India (Having density of 0.95g/c.c. and melt flow index, MFI of 6.0) was used for the entire study.

Carbon Black: - Extra conducting furnace (XCF) carbon black, grade 'Vulcan XC-72' ISO classification N472, from Cabot Corporation, USA (having ensity of 1.67g/c.c. , average particle diameter of 30nm, nitrogen surface area of 220m^2/g, Dibutyl phthalate (DBP) absorption of 220 c.c./100g and iodine absorption value of 270mg/g) was used.

Here we have used carbon (25phr) as the conducting and EVA (100phr) as the insulating component. In the text V indicates Vulcan XC-72 carbon black.

The sample preparation procedure is the same as reported in [6].

5. Theory

5.1 Rectangular cross section

If a rectangular sample of length, breadth and thickness W, B and d respectively is placed inside a rectangular cavity having corresponding dimensions c, b and a then for TE_{011} mode

$$E_1 = 2A\left[\frac{a}{\pi}\right]\omega\mu_0 \cdot \sin\left[\frac{\pi z}{a}\right]\sin\left[\frac{\pi x}{c}\right]e^{j\omega t}) \qquad (11)$$

$$\iiint_{v_c} |E_1|^2\, dv = \left[4A^2\frac{a^2}{\pi^2}\mu_0^2\omega_0^2\right]\frac{bac}{4} \quad . \qquad (12)$$

For thin samples it can be assumed that the electromagnetic fields in the cavity do not change due to the introduction of the sample as shown in hence E_1=E_2 and

$$\iiint_{v_s} E_2{}^2\, dv = \left[4A^2\frac{a^2}{\pi^2}\mu_0^2\omega_0^2\right]\int_0^{\mathbf{d}}\sin^2\frac{\pi z}{a}dz\int_{\frac{\mathbf{C}}{2}-\frac{\mathbf{W}}{2}}^{\frac{\mathbf{C}}{2}+\frac{\mathbf{W}}{2}}\sin^2\frac{\pi x}{c}dx\int_0^{\mathbf{B}}dy \qquad (13)$$

$$= \left[4A^2\frac{a^2}{\pi^2}\mu_0^2\omega_0^2\right]\cdot\frac{WB\pi^2 d^3}{3a^2} \qquad (14)$$

The values for the dielectric constant and conductivity σ of the test sample can be obtained from (10), noting the sample and cavity resonator dimensions. If a rectangular sample of length, W, breadth, B and thickness, d is loaded in a rectangular cavity resonator having corresponding dimensions C, b and a respectively, then the values for ε_r' and σ can be expressed as

$$\varepsilon_r^{'} = \left[\frac{f_1^3 - f_1^2 f_2}{f_2^3}\cdot\frac{3a^3bc}{2WB\pi^2 d^3}\right] + 1 \qquad (15)$$

$$\sigma = \frac{Q_1 - Q_2}{Q_1 Q_2}\cdot\frac{f_1^{\,2}}{f_2}\varepsilon_0 \cdot \frac{3a^3bc}{2WB\ \pi\ \ d^3} \qquad (16)$$

where the resonant frequencies of the cavity without and with the sample are f_1 and f_2 respectively, and Q_1 and Q_2 are the corresponding loaded Q's of the empty cavity and that with the sample.

5.2 Circular cross section

When a cylindrically shaped sample of thickness d and radius r is inserted inside the cavity the stored energy inside the sample can be expressed as

$$\iiint_{v_s} E_2^2\, dv = \left[4A^2\frac{a^2}{\pi^2}\mu_0^2\omega_0^2\right]\int_0^{\mathbf{d}}\sin^2\frac{\pi z}{a}dz\int_0^{2\pi}\sin^2\frac{\pi\rho\phi}{c}\rho d\phi\int_0^r d\rho \qquad (17)$$

$$= \left[4A^2\frac{a^2}{\pi^2}\mu_0^2\omega^2\right]\cdot\frac{2\pi^7 d^3 r^4}{9a^2c^2} \qquad (18)$$

Substituting the value of the integral we can obtain the values of the and then loss tangent, . Hence,

$$\varepsilon_r^{'} = \left[\frac{f_1^3 - f_1^2 f_2}{f_2^3}\cdot\frac{9a^3bc^3}{4\pi^7 d^3 r^4}\right] + 1 \qquad (19)$$

$$\varepsilon_r^{''} = \frac{Q_1 - Q_2}{Q_1 Q_2}\cdot\frac{f_1^2}{f_2^2}\cdot\frac{9a^3bc^3}{8\pi^7 d^3 r^4} \qquad (20)$$

$$and\ \tan\delta = \frac{\varepsilon_r^{''}}{\varepsilon_r^{'}} \qquad (21)$$

Further conductivity (σ) can also be obtained from

$$\sigma = \varepsilon_r^{''}\cdot 2\pi f_1.\varepsilon_0 \qquad (22)$$

Therefore,

$$\sigma = \frac{Q_1 - Q_2}{Q_1 Q_2}\cdot\frac{f_1^2}{f_2}\varepsilon_0\cdot\frac{9a^3bc^3}{4\pi^6 d^3 r^4} \qquad (23)$$

5.3 Modeling and simulation

High Frequency Simulation Software based on finite element method is used to calculate the shift in resonance frequency and quality factor of the sample under consideration. The system that we modeled consists of the cavity coupled with two irises at the two end of the cavity. This model will be also used later for the evaluation of measurement accuracy.

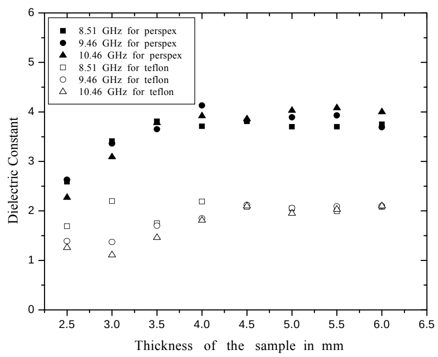

Fig. 3. Variation of measured dielectric constant of Teflon and Perspex as a function of sample thickness at frequency 8.51 GHz, 9.46 GHz, 10.46 GHz for Rectangular Shape Sample

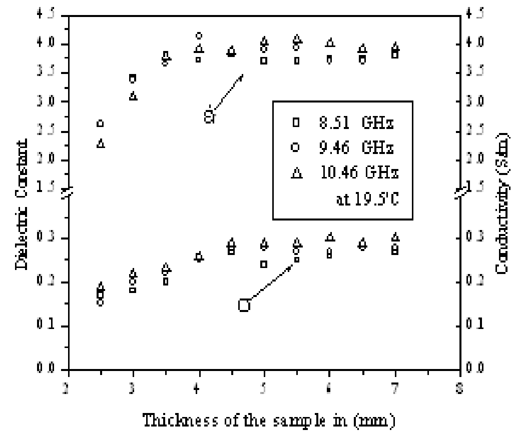

Fig. 5. Variation of measured dielectric constant and conductivity of Bakelite as a function sample thickness at frequencies 8.51 GHz, 9.46GHz and 10.47 GHz.

To observe steady values coming from the formulas simulated measurement of shift in resonance frequencies and Q-factor were made for each 1mm insertion of sample inside the cavity using the three different resonance frequencies. The dielectric constant obtained for each 1mm thickness of sample using equation (15) and (19) for two types of sample sizes were observed. The value increases with increase in sample thickness up to 4mm after which it attained a steady value up to 6mm of sample thickness [Fig 3 & 4]. For Perspex the profile shows the value of ε' obtained for 4-7mm thickness are almost constant .Similar trends were also obtained for the imaginary part or loss ε" as a function of sample thickness for Bakelite sample [Fig 5]. It is clear that ε' and ε" values obtained for the region 4-6mm sample thickness are quite accurate.

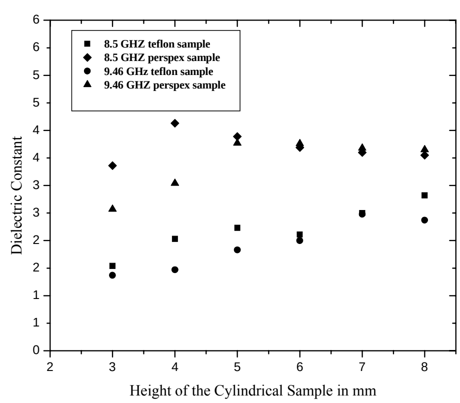

Fig 4 Variation of measured dielectric constant of Teflon and Perspex as a function of sample thickness at frequencies 8.51 GHz and 9.46 GHz for cylindrical shape sample

6. Results and discussion

The dielectric constant reported for Teflon is 2.08 [13] the value is close to the measurement results we obtained for the sample thickness between the 4-6mm for 8.51 GHz frequency and are close to the value of 3.81 reported for Perspex [14]. The loss value obtained for Bakelite sample for 4-6mm range is 0.45 which is in well agreement with the reported value. So similar measurement was done for EVA rubber composites with 25 volumes of carbon and observed with a value of 6.20 and 2.18 S/m respectively for Dielectric constant and conductivity at a frequency 9.46 GHz.

Measurement results obtained for the conductivity of the sample using the two-probe method, for Bakelite was found to be 1.67E-09 S/m at 19.50C and for EVA.25V it was 8.00E-03 S/m.

The non consistent values of ε' and ε" were obtained for sample thickness less than 4mm was due to the fact they are not causing sufficient perturbation of the cavity for accurate determination. Sample thickness greater than 6mm will absorb electric field and going to give non-consistent results hence are not within our region of interest.

7. Evaluation of accuracy

Knowing the standard value of the dielectric constant of the materials the relative measurement (and simulation) error δ_m (and δ_s) can be defined as

$$\delta_m = \frac{|\varepsilon_{rm} - \varepsilon_{rDm}|}{\varepsilon_{rDm}} \tag{24}$$

where ε_{rm} is the measured (simulated) value of the dielectric constant of the materials and ε_{rDm} is the standard value of the dielectric constant of the materials.

The data from the measured (and simulated) dielectric constant for Perspex are evaluated as follows

f in GHz	8.51	9.46	10.47
δ_m [%]	0.26	1.05	2.36
δ_s [%]	2.62	2.89	8.40

8. Conclusion

The determination of the dielectric parameters for small shaped samples having thickness 4-6 mm gives an accurate value with the resonant cavity technique. This way the requirement of full length sample can be avoided.

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