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Dielectric Parameters of Doped Poly (Methyl Methacrylate) Dielectric Films At Microwave Frequencies

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***Abstract*— The paper entitled "Dielectric Parameters of Pure and Doped Poly Methyl Methacrylate (PMMA) Films at Microwave Frequencies" presents different dielectric parameters of some pure and doped dielectric films corresponding to the X-band frequencies (8 – 12 GHz). The interested dielectric parameters are real and imaginary parts of the dielectric constants $\in_r$& $\in_i$, loss tangent (tan δ), conductivity σ, relaxation timeτ, absorption coefficient k and refractive index n. A novel waveguide method recently introduced for the study of dielectric parameters of samples in the form of films is used in this study. An X-band microwave test bench with a Gunn diode as a source, consisting of a slotted sample holder with a thin film sample is used for the study. Pure and doped PMMA films of 100µm to 1000µm thickness are to be prepared and various parameters are analyzed for different thickness t, temperature T, and frequency f. The dopents used are $AgNo_3$ and CdS. Since the method is proposed for thin films, it is very efficient for costly materials. A very thin film, even with very small area may be selected for the study.**

1. INTRODUCTION

In contrast to metals that are generally good conductors, dielectrics are a class of materials that are poor conductors of electricity. But they can be polarized easily by an applied electric field. The electrical natures of these materials are described by their dielectric properties. The development and application of electromagnetic waves (microwaves) to communications and electrical measurements, have led to the need for determining electrical constants at these wavelengths. In recent times polymers are finding an important place in different research laboratories for the study of their various properties. In view of the applications of insulation, isolation and passivation properties of polymers in microelectronics and optical waveguide systems, studies of the dielectric properties of polymers are of considerable interest. In polymeric materials, the polarization and depolarization behaviour are related to the dielectric relaxation process.

The present work describes the results of our investigations on dielectric properties of pure PMMA and doped by Cadmium sulphide (CdS) and silver nitrate (AgNO3) the form of thin films at 9.72 GHz microwave frequency, at room temperature and increasing temperatures.
Conventional transmission and cavity resonance methods are difficult to use in the dielectric study of thin samples, since they have no strong interaction of the RF signal with the specimen. Also sample thickness should be made a sizable fraction of the wavelength (~λ/4 and its multiples in most of the methods) to obtain accurate measurements using these methods. Waveguide measurements are highly sensitive, sample holder preparation is relatively easy and mechanical connections of waveguide sections are simple and robust, waveguide sample holders have effectively been applied for precise and accurate dielectric characterization.

In this paper we present a simple method, in which thin film of the sample is inserted longitudinally at the centre of the waveguide excited in the normal dominant TE10 mode and the corresponding VSWR (voltage standing wave ratio) and phase shift is measured. Using these various dielectric parameters could be obtained.

2. EFFECT OF TEMPERATURE ON DIELECTRIC CONSTANT

There will be significant variation of dielectric constant with temperature for a dielectric material. The dielectric constants of all samples did not vary with the temperature between 80 and 250 K. The dielectric constant exhibits a much faster increment with further increasing temperature above 300K. This is the typical behaviour in the transition region for the polymers having dipolar structure. Varying of the dielectric constant with temperature for a polymer sample was attributed to the temperature dependence of the orientation activity of the dipole groups.

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3. EFFECT OF FREQUENCY ON DIELECTRIC CONSTANT

As frequency increases, polarization decreases and hence its dielectric constant drops.The decreasing of the dielectric constant with increasing frequency can be explained by frequency dependency of orienting dipoles. The orientation moment is fully established at low frequencies and therefore, dielectric constant reaches its maximum values. On the other hand, at higher frequencies, the dipoles will not have enough time to orientate and thus, the dielectric constant decreases.

4. NEED FOR PERMITTIVITY MEASUREMENT

Dielectric materials are essential for modern electronics, for circuit board, antenna manufacturing materials etc. So in order to choose appropriate materials for each purpose measurement of each material is essential. In industrial fields hundreds of processes are to be monitored and maintained throughout the manufacturing cycle in order to take raw materials from one form and combine them to create a finished product. Many times, the combination of materials must be monitored to maintain the quality of the product. By relating the permittivity of the material mixture to the composition of the individual components; it is possible to determine the individual concentrations of each material quickly and efficiently.

5. PROPOSED METHOD

For this paper we have used the technique used by Dube and Natarajan. Here the sample was mounted along the axis of the waveguide. Standing waves are produced by short-circuiting the system. These are detected in the slotted line by means of a travelling wave guide detector. From the measured VSWR (voltage standing wave ratio) and phase shift, dielectric constants can be obtained.

This method is comparatively simple than other methods of measurements. It is used for microwave measurements and the results obtained are consistent i.e. they have a high degree of reproducibility.

6. METHODOLOGY

6.1 Sample Preparation

For the experimental study, films of poly methyl methacrylate(PMMA) and films of PMMA doped with CdS (Cadmium sulphide) and $AgNO_3$ (silver nitrate) are used.For the sample preparation of pure PMMA sample, 50 ml of methyl methacrylate was taken in a beaker and about 1gm of benzoyl peroxide is added. By inserting a magnet in to it, the solution was placed on the hot plate of the magnetic stirrer apparatus and then switched on. The magnet inside the solution starts rotation due to magnetic induction. The speed of rotation can be controlled by the speed controlling knob. The temperature of the hot plate can be adjusted by the temperature varying knob. The solution is made to stir until it became viscous. After that the solution is filtered using a filter paper. The blend is poured in to the moulds of different thickness and covered well to avoid evaporation. After 5-6 hours the film is detached carefully from the mould. For the preparation of doped PMMA films, desired amount of dopant material was added in to methyl methacrylate solution and repeated the above process. PMMA doped with CdS and $AgNO_3$ are prepared in this way. These PMMA film and doped PMMA films are then used for the experiment.

6.2 Mould Preparation

A glass slide was placed above the surface of a table and a paper mould which is made by removing a square portion from the center of the paper strip of thickness 100µm was placed above it. The prepared solution was poured on. Then another glass slide was placed above the paper and the four sides of the mould was sealed in order to avoid evaporation. Then the process was repeated by using paper strips of different thickness to get thin films of different thicknesses.

6.3 Preparation of PMMA doped with $AgNo_3$ (0.1%)

About 0.1% of $AgNo_3$ is dissolved in 1ml of ethanol. This solution is added to the methyl methacrylate solution while stirring. This mixture is made to stir about one hour. And when the solution becomes viscous, it is poured in to the suitable mould. After 5 to 6 hours It is carefully detached from the mould.

Density of PMMA = 0.942gm

6.4 Preparation of PMMA doped with CdS (0.2%)

Weight of 5 ml of PMMA = 5 x 0.942 =4.72g

0.2 weight percentage CdS = $\frac{0.2}{99.8}$ x 4.72 = 9. $^{-3}$g4589x 10

1 mol of Cd in CdS = $\frac{Weight\ of\ Cadmium}{Weight\ of\ Cadmium\ sulhide}$ = $\frac{112.41}{(112.41+32.066)}$ = 0.7781

Cadmium in 0.2 weight percentage CdS = 0.7781g x 9.4589 x 10^{-3} = 7.359 x 10^{-3} g

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Weight of cadmium acetate = $\frac{weight\ of\ cadmium\ Acetate}{1\ mol\ of\ Cd\ weight}$ x amount of cd in 9.4589 x 10^{-3}g of CdS = $\frac{226.52}{112.41}$ x 7.359 x 10^{-3} = 0.01483g

In 9.4589 x 10^{-3}g of CdS, the amount of sulphar= 9.4589 x 10^{-3}g - 7.359 x 10^{-3} g = 2.099 x10 $^{-3}$g

Therefore the weight of Thiourea required = $\frac{76.12}{32}$ x 2.099 x10 $^{-3}$g = 4.9929 x10 $^{-3}$g

CdS is not available in powdered form. So it is difficult to dissolve it in any solution. The recommended solution is ethanol. For the preparation of CdS, we are mixing a properly weighed amount of cadmium acetate and thiourea and finally dissolve it in to ethanol.

6.5 Measurement Techniques

In this technique the thin specimen which is pure PMMA film or doped PMMA film is mounted along the axis of the waveguide. The advantage of this method is that the thin specimen is placed longitudinally at the center of the broad side of a hollow rectangular waveguide excited in the TE_{10} mode so that the whole specimen remains in the maximum electric field. Standing waves are produced in the rectangular waveguide by short circuiting the system. These are detected in the slotted line by means of a travelling waveguide detector.

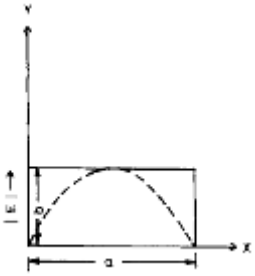

Fig 6.5.1 Electric field distribution in a rectangular waveguide in the TE_{10} mode

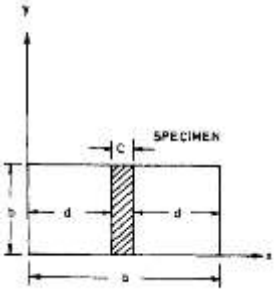

Fig 6.5.2 Cross section of a rectangular waveguide in the specimen

In this experimental technique, travelling wave detector is used to measure the shift in the minimum position when specimen is introduced. Let the specimen length be L cm and the phase shift produced by x cm, then phase introduced by the specimen per unit length is,

$$\beta_s = \beta_0 \frac{x}{L}$$

Thus the total phase constant β_r is given by

$$\beta_r = \beta_0 + \beta_s$$

$$\beta_r = \beta_0 \left(1 + \frac{x}{L}\right)$$

For measuring the dielectric loss, the VSWR is taken with and without specimen, let these be p_1 and p_2 , then

$$r = \frac{p_1 - 1}{p_2 + 1} = e^{-\alpha}$$

$$r' = \frac{p_2 - 1}{p_2 + 1} = e^{-(\alpha + 2\beta_i L)}$$

Where r and r' are the reflection coefficients of the system with and without the sample and α is attenuation constant. The VSWR thus obtained is used to obtain the dielectric parameters with the help of numbered equations.

$\tan\delta = \frac{2K}{1-K2}$ ……..*(3)*

$\tau = \epsilon_i / \omega \epsilon_r$..... (4)

$\sigma = \omega\ \epsilon_0\ \epsilon_i$……(5)

$\epsilon_i = 2n^2K$ .....(6)

6.6 Experiment Setup

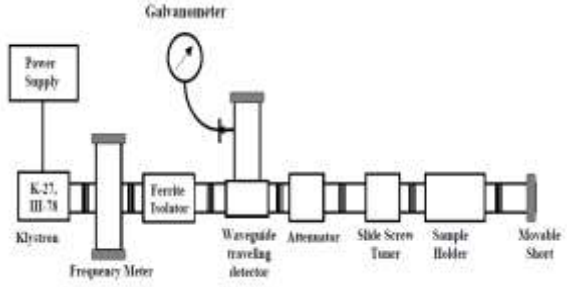

Fig 6.6.1 Schematic diagram of the experimental set up

Gunn diode is used to produce frequencies in the range of x-band to be used for the experiment. The frequency produced is measured using the frequency meter. The isolator is used to isolate input and output wave. The standing wave is produced in the travelling waveguide detector. Using a voltmeter, the maximum and minimum is noted and VSWR is calculated. Variable attenuator is used to attenuate the incoming signal. Slide screw tuner is used for the impedance matching. Then the in sample holder, the specimen is introduced, longitudinally. Then a movable short at the end.

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6.7 Procedure

1. The particular frequency of x-band is set using the Gunn diode.
2. The frequency of the signal is measured using the frequency meter. Frequency meter is rotated slowly and the voltage is noted at the voltmeter. Voltmeter shows a dip at a particular frequency only, the frequency for which sudden dip is shown is the frequency of the signal. Thus the frequency of the signal used is obtained.
3. By using the travelling wave guide detector and the voltmeter connected to it, the maximum and the minimum voltages and the corresponding positions are noted. Thus VSWR without inserting the sample ρ_2 is obtained by calculating V_{MAX}/V_{MIN}.
4. Now the prepared sample of PMMA or the doped PMMA is inserted to the sample holder longitudinally. Then as before, using the travelling wave guide detector and the voltmeter, the maximum and minimum position and the voltages are noted. Thus the VSWR after inserting the sample ρ_1 is obtained.
5. It should be noted that ρ_1 should be greater than the ρ_2. By adjusting the slide screw tuner this condition is obtained.
6. The shift x in the minimum position, before inserting and after inserting the sample is noted.
7. The length, the thickness of the sample, the shift, ρ_1 ρ_2, the frequency used etc. are used in the suitable equations and thus the dielectric parameters and the conductivity are obtained. A program designed in the MATHLAB is used for this calculation.
8. Thus the procedure is repeated for various samples similarly and the dielectric parameters and the conductivity is noted.

6.7.1 Temperature Variation:

1. The sample holder is heated with the hot plate apparatus. The temperature of the sample holder is noted with help of a thermometer.
2. The procedure is repeated as before. The VSWR with and without the sample is noted. The shift corresponding to the minimum position is noted.
3. The dielectric parameters and the conductivity is calculated.
4. The temperature is changed and set of readings are taken for a new temperature.
5. Thus the process is repeated for different temperatures.

6.7.2 Frequency Variation:

1. The frequency of the Gunn diode is changed.
2. The respective frequency set is measured using the frequency meter.
3. Procedure is repeated as above. The shift, ρ_1, ρ_2, .etc. are noted
4. The dielectric parameters and the conductivity is obtained.
5. Then another frequency in the range of x-band is set and the experiment repeated.

The observations and the calculations are tabulated. The graphs are plotted corresponding to temperature and the frequency variation.

7. CONCLUSION

The method used for the analysis of different parameters of dielectrics, both lossless and lossy is found to be very effective. The major advantage of this method is related to the sample size. The dielectric parameters measured are real and imaginary parts of refrectiveindex, losstangent, conductivity, relaxation time, refractive index and absorption index. These parameters are analyzed for varies values of frequency corresponding to X-band. The parameters are also studied for different temperature. The materials selected for the study are PMMA films, both pure and doped with some lossy compounds. Different thin film samples of pure PMMA are prepared and the paramerters are studied using an X-band microwave test bench. Some films doped with $AgNo_3$ and CdS are also prepared and used for the study. The method of investigation uses the measurement of wavelength change inside the sample due to its dielectric properties. A standing wave is generated inside mached test bench and the film sample is inserted into the waveguide through the linear slot of the sample holder. This changes the wavelength inside the guide and hence a shift is observed for any minimum point of the standing wave distribution. This shift is directly related to the real part of the dielectric constant of the material. The change in the value of voltage standing wave ratio, VSWR is a measure of its imaginary part of the dielectric constant. The other parameters such as loss tangent, conductivity etc are all related to these values and hence the evaluations of them are easy.

Pure PMMA samples:

Thickness variation

For pure PMMA, the imaginary part of the dielectric constant is less. It shows that the conductivity also is less.As the thickness of the film increases, the shift in minima and VSWR slightly changes.The method is devised for thin films. So the thickness of the samples should be

small. If its value increases to 1mm or above, there is possibility for increase of error.

Frequency variation

As the frequency increases the real part of the dielectric constant of the material decreases. This is in agreement with standard results.The imaginary part of the dielectric constant increases with frequency. The loss tangent and conductivity also shows an increase. But the relaxation time decreases with frequency.

Temperature variation

As the temperature of the sample increases, almost all parameters increases. But therelaxation time shows some irregularities.

Doped PMMA samples:

For doped samples the real part of the dielectric constant shows an increase.Doping shows a tendency to reduce the imaginary part of the dielectric constant. Correspondingly all the other related parameters also shows a decrease.As the frequency and temperature changes, the doped samples shows changes somewhat similar to pure samples.The conductivity of PMMA can be increased by using a suitable dopant material. All the doped films show better conductivity as compared to pure PMMA films.The increase in conductivity is accounted due to creation of additional hopping sites for the charge carriers in doped samples.Hopping process is the probable mechanism of conduction phenomenon.

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