STUDY AND MEASUREMENT OF ELECTRICAL PROPERTIES OF SOME CONDUCTING POLYMERS AT MICROWAVE FREQUENCIES USING AUTOMATION TECHNIQUE

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ABSTRACT

This paper deals with the design and study of an automatic technique for acquiring the properties of conducting polymers Absorption Coefficient, dielectric constant, Conductivity at microwave frequencies using microcontroller controlled stepper motor. Electrical properties of conducting polymers are measured manually by using microwave bench at X- band. These measurements are not accurate when done manually. Hence, microcontroller based automatic measurement of the electric properties parameters is designed. The probe of slotted section of X-band microwave bench is moved by the stepper motor which is controlled by microcontroller. When the stepper motor is operated in full and half stepping mode respectively in forward or backward directions the positions of the probe are detected by an optical section through 0.00650 mm and 0.00325 per step. This system measures the width at the twice minima or maxima method obtained from the feedback signals of Standing Wave Ratio meter and is further used for the measurement of free space wavelength (λo) and waveguide wavelength (λg). This work also makes a comparative study of the above properties of chosen conducting polymers such as PEDOT, PPDA, PPY, PTH and PANI.

Keywords: Dielectric Constant, Absorption Coefficient, Conductivity, Optical Sensors, Stepper Motor, Conducting Polymers.

I. INTRODUCTION

The electrical properties of conducting polymers at microwave frequencies can be known mostly by the parameters dielectric constant or permittivity, absorption coefficient, electrical conductivity. These properties are inbuilt characteristics of conducting polymers which explains the behavior and the degree of wave matter relations at microwave frequencies. For measuring the microwave electric properties Robert and Von Hippel method is generally preferred. In this method, the microwave bench with a slotted section with a probe is operated manually. In the present work, microcontroller (μc) based automatic technique is used to measure the electrical properties of conducting polymers. In this technique the microcontroller based stepper motor is assembled with a gear to control the movement of the probe in the slotted section. The stepper motor makes the slotted region to move either in forward or reverse direction as per the digital signals given to it. The stepper motor is connected through a gear wheel to the probe in slotted section of the microwave bench. The microcontroller is used to operate the stepper motor and by software program written in Keil C language. The Stepper motor moves the probe in the slotted section of the microwave bench to find position of minima or maxima in proper direction. A data acquisition card in the crystal detector is used to convert the output into digital form and is send to microcontroller port. The number of steps of the motor in certain direction and the difference between the two maxima or minima are displayed.

Intrinsically conducting polymers are conjugated molecules. In their neutral state or faultless state they are either insulator of wide gap semiconductors. By doping they can be changed into metallic type conductors. These conducting polymers like polyacetylene, polyaniline, polytiophener (PTH), polyfuran (PFU), para phenylene vinylene have pi-electron conjugated structure are synthesized to use in many applications. Dielectric properties of conducting polymers are important because of its wide areas of applications such as coating in reflector antennas, electronic equipments, surfaces for selected frequencies, microwave absorbing materials, radar, microchip antennas. In other applications Non-biological materials screening dielectric properties are used in hyperthermia applicators, assess microwave imaging systems and to determine electromagnetic absorption patterns.

II. EXPERIMENTAL

2.1 Preparation of Conducting Polymers

Using anhydrous ferric chloride (FeCl3) as oxidant in nitrobenzene solvent by chemical oxidative polymerization of thiophene PTH (Polythiophene) is prepared. Then it is doped in I M FeCl3 solution in nitromethane for 24 hrs. PEDOT (Poly 3,4 – ethylenedioxytiophene) was prepared using Fecl3 as oxidant in aqueous dodecyl benzene sulfonic acid (DBSA) micellar solution. PANI (Polyaniline) was prepared by chemical oxidative polymerization of aniline in aqueous FeCl3 and was doped with 1 M solution of camphor sulphonic acid for 24 hrs. PPY (Polypyrrole) was prepared by the polymerization of pyrrole with Fecl3 in methanol solvent. The polymer as doped with 1 M FeCl3 solution in nitromethane for 24 hrs. PPDA (Polyparaphenelyne diazomethine) was prepared by solution condensation of glyoxal trimeric hydrate in DMF and paraphenylene at a molar ratio of 1:1 at 120'C for 5 hrs. PPDA as doped with 1 M of HCl for 24 hrs

2.2 Sample Preparation

Samples of different thickness and of rectangular shape 0.9 inches by 0.4 inches which is the size of the rectangular waveguide in X- band microwave bench were prepared. Rubber sample is also made cutting the firmer into 0.9 inches by 0.4 inches of desired thickness.

2.3 Measurement of Dielectric Constant at Microwave Frequencies

For precise measurement of dielectric constant, width at twice minima or maxima method was used. The schematic diagram of experimental setup is as shown in Fig. 1.

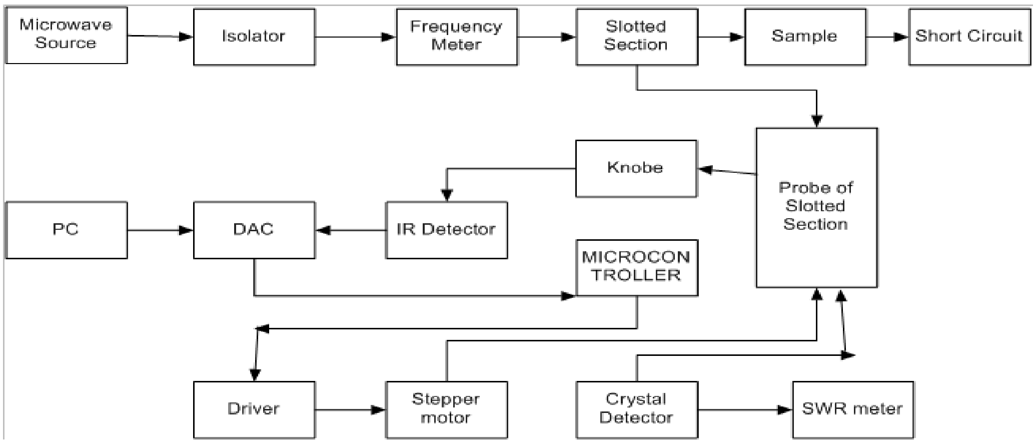

Fig. 1 The Schematic Diagram of Experimental Setup

Initially the frequency of the dielectric constant was determined by taking micrometer reading of cavity resonator when dip in output is produced. Then the cavity resonator was detuned. The standing wave pattern with and without sample is show in the Fig. 2.

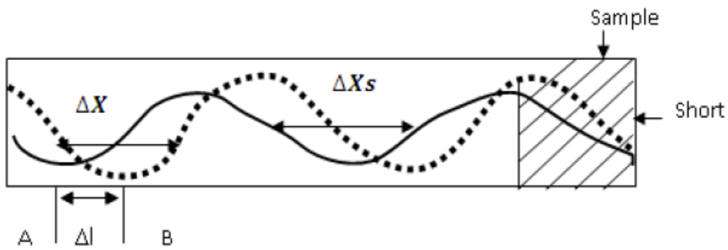

Fig. 2: Standing Waves in the Waveguide with and Without Sample.

2.4 Stepper Motor

The stepper motor is operated in two types of stepping modes viz full and half. Only one coil winding is energized at a given instant of time in full stepping mode. The stator is energized according to the sequence.

A → B → C → D or AB → BC → CD → DA

Whereas in half stepping mode there is a combination of both full wave and full step (1 and 2 Phases on) drive modes. For every second step only one phase is energized and during the other steps one in each stator is energized. The stator is energized according to the following sequence.

AB → B → BC → C → CD → D → DA → A

2.5 IR Detector

To detect the position of the starting and ending position of probe in microwave bench an IR detector MOC 7811 is used. This will be generally used in printer to detect page.

2.6 Stepper Motor Driver

To drive the stepper motor NPN transistor SL 100 is used and the circuit is as shown Fig. 3. A switching circuit consisting of power transistors drives stepper motor unit. The current to the field coils of the motor is provided by these power transistors. Here four medium power transistors are used to drive the coils of motor. The 4-bit

(D0- D3) output of the microcontroller port is fed to the base of transistors which in turn are used to control the movement or rotation of the motors which is controlled by software.

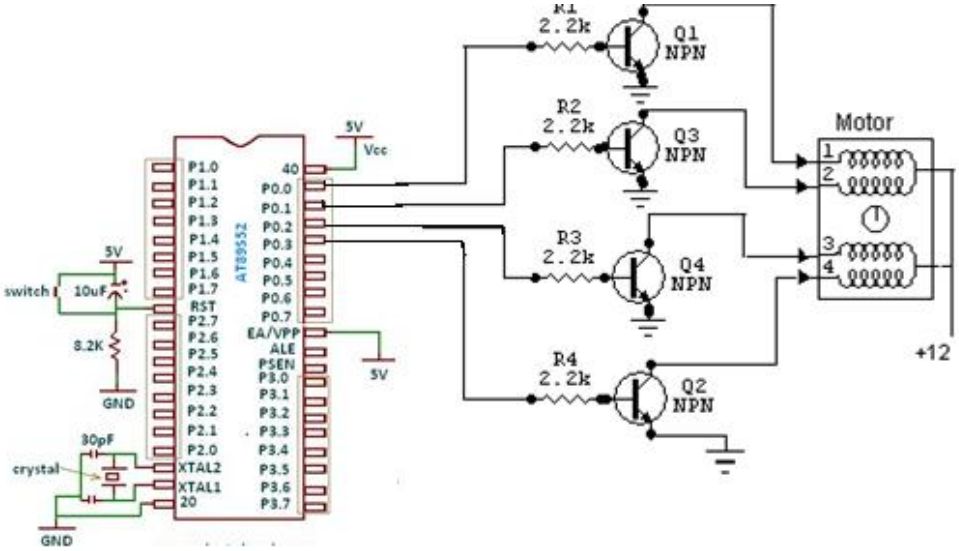

Fig. 3: Stepper Motor Driver Circuit

III. EXPERIMENTAL SETUP

To determine the dielectric constant the experimental setup is shown in Fig. 4. The hardware which is specially designed consists of three parts viz. control board, Data Acquisition Card and Low cost Stepper motor controller.

The microcontroller 8051 is compatible for these parts. The analog current from the probe is converted into digital form by DAC. The full step or half step rotation in clock wise or anti clock wise rotation of stepper motor is done by the suitable software written in embedded ‘C’ language. This technique moves the probe of the slotted section 0.00650 mm per step in full stepping mode and 0.00325 mm per step in half stepping mode of stepper motor. The probe of the slotted section is welded with a nut and is connected to the shaft of stepper motor through a gear. The minima or maxima of standing wave are measured for a sample dielectric in front of circuit and initial guide wavelength is measured. The thickness of the conducting polymer is measured by a micrometer accurately. The exact position of the first and second minima or maxima from the short circuit end is noted using Standing Wave Ratio Meter (SWR). The short circuit now is removed and the conducting polymer is inserted into the aperture of the wave guide touching the short circuit. Now the position of the first minima or maxima from the short circuit end is measured and the shift is determined.

To detect the starting and ending position of the probe an IR detector is used. The four switches are used for controlling the movement of probe of slotted section. The details of pin allocation to switches and IR detector are shown.

978-81-931039-6-8

IV. SOFTWARE

The stepper motor can be operated in various stepping modes. The program to control stepper motor is written in embedded 'C' language. This language is user friendly and is easy to understand when compared to any other programming languages. The various modes driven by the software are

1. Reset System 2. Full Stepping mode 3. Half stepping mode
4. Initial position 5. End position 6. Start 7. Stop

The flow chart is given in Fig. 4.

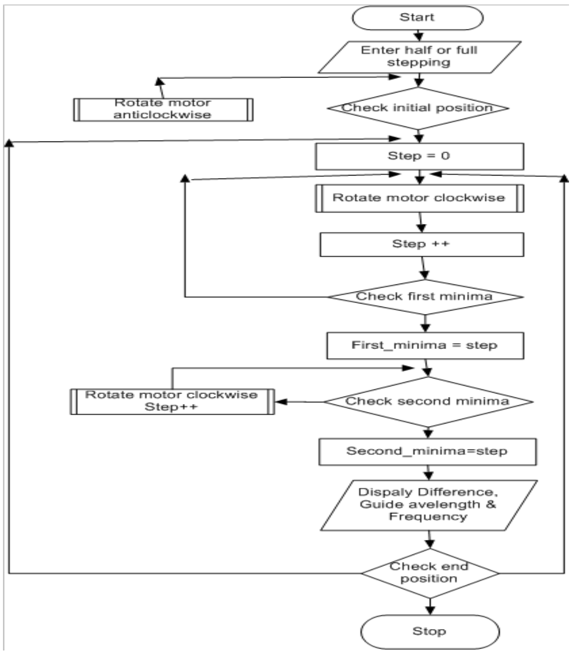

Fig. 4: Flow Chart to Control Stepper motor

V. THEORETICAL CALCULATIONS

A solid of length le is loaded in rectangular wave guide against short circuit that touches it well. If D & Dr are the positions of first voltage minima of the standing wave position when the wave guided is unloaded and loaded with conducting polymer then the respective distances from the short circuit will be (l + lϵ) & (l_R + lϵ). Let β and βϵ are the respective propagation constant.

Expanding tangent sum angle

$$\frac{\tan\beta(Dr - D + l\epsilon)}{\beta l\varepsilon} = \frac{tan\beta\varepsilon l\epsilon}{\beta\varepsilon l\varepsilon}$$

Where $\beta = {}^{2\pi}/_{\lambda g}$

The value of λg can be calculated using the formula

$$\left(\frac{l}{\lambda_o}\right)^2 = \left(\frac{l}{\lambda g}\right)^2 + \left(\frac{l}{\lambda c}\right)^2$$

i. Dielectric constant(ϵ'_r) : Dielectric Constant is given by following formula

$$\epsilon'_r = \frac{\left(\frac{a}{\pi}\right)^2 \left(\frac{\beta\epsilon l\epsilon}{l\epsilon}\right)^2 + 1}{\left(\frac{2a}{\lambda g}\right)^2 + 1}$$

Where 'a' is the wave guide dimension, λc is twice the waveguide dimension (= 4.582) and λo is the ratio of velocity and frequency of microwave (=3.03 variable).

ii. Loss Tangent (Tan δ): The loss tangent was calculated using the formula

$$\text{Tan } \delta = \left(\frac{(\Delta Xs - \Delta X)}{\varepsilon' d}\right)\left(\frac{\lambda_o}{\lambda g}\right)^2$$

Where

ΔXs = Width at twice minimum or maximum with sample

ΔX = Width at twice minimum or maximum without sample

iii. Loss Factor (ε''): The loss factor is determined from the following formula

$$\varepsilon'' = \varepsilon' \text{ Tan } \delta$$

iv. Absorption Coefficient (α_f): The absorption coefficient is calculated by the formula

$$\alpha_f = \frac{\varepsilon_r' f}{nc}$$

v. Electrical Conductivity (σ_f): The Electrical Conductivity is calculated by the formula

$$\sigma_f = 2\pi f \varepsilon_0 \varepsilon_r''$$

VI. RESULTS AND DISCUSSION

6.1 Dielectric Constant (ε')

The dielectric constant of special conducting polymers at X band frequency was compared and found that the dielectric constant decreases with increase in frequency. This is basically due to orientation polarization in the microwave field. As the frequency of the field increases the polarization has no time to reach its steady field and the orientation polarization falls firstly. It is also clear that dielectric constant is highest for PEDOT (3, 4-ethylenedioxythiophene) and gradually decreases in the order of PANI (Polyaniline), PTH, PPY and is lowest for PPDA (Polyparaphenelyne diazomethane). The deviation dielectric constant with frequency for various conducting polymers is as shown in the Fig. 5.

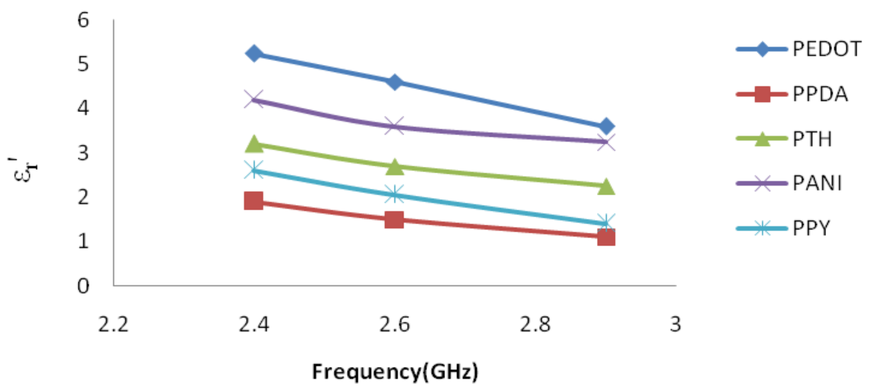

Figure 5: Deviation of dielelctric constant with Microwave frequency for various conducting polymers PEDOT, PPDA, PTH, PANI and PPY

6.2 Absorption Coefficient (α_f)

Based on the transparency of wave passing through the medium, which specifies the absorption of electromagnetic waves, the materials can be classified. This transparency is defined by absorption coefficient. This parameter is related to the dielectric loss and shows the same behavior of dielectric loss. In a conducting polymer with a low absorption coefficient, light is only poorly absorbed, and if the material is thin enough, it will appear transparent to that wavelength. The absorption coefficient depends on the material and also on the wavelength of light which is being absorbed. Fig. 6 shows that PEDOT has high absorption coefficient and PPDA has lowest absorption coefficient with PANI, PTH and PPY in between them.

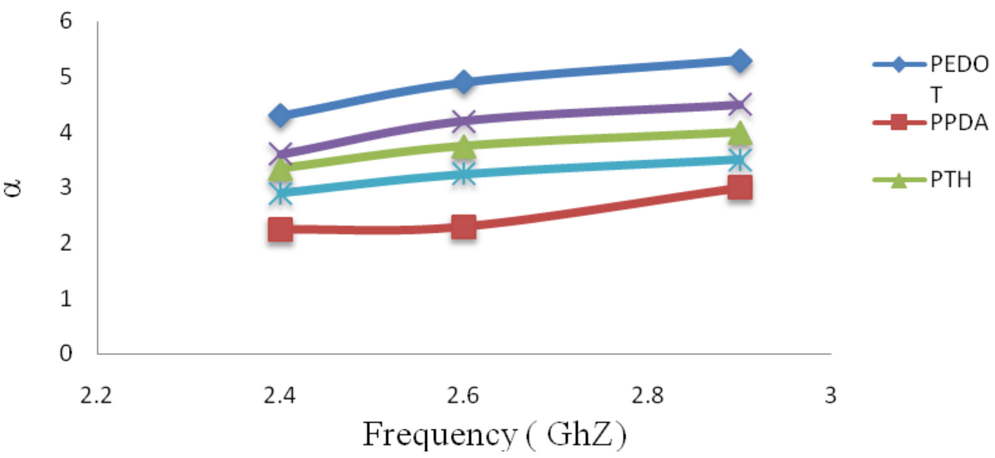

Figure 6 Variation of absorption coefficient with Microwave frequency for various conducting olymers PEDOT, PPDA, PTH, PANIand PPY

6.3 Conductivity (σ_f)

The conductivity at microwave frequencies is a directly related to dielectric loss and follows the same variation as the dielectric loss factor with frequency. Fig. 7 show that conductivity increases as frequency increases. In this also PEDOT shows best conductivity followed by PANI, PTH, PPY and PPDA.

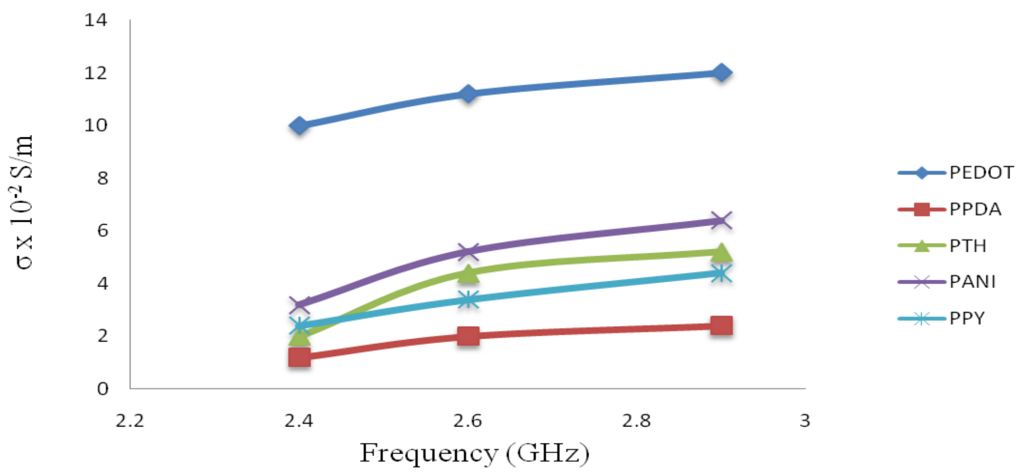

Figure 7 Variation of Conductivity with Microwave frequency for various Conducting Polymers PEDOT, PPDA, PTH, PANI and PPY

VII. CONCLUSIONS

The accuracy of the measurement of electrical properties like dielectric constant, Absorption coefficient and electrical conductivity are measured with high precision using X-band microwave bench with automation technique. This measurement depends only on measurement of guide wave length. Using this technique the above electrical properties for conducting polymers PPDA, PANI, PTH, PPY and PEDOT are studied and compared at X-band frequency. The observed values are in good agreement with expected values at 5 degree of freedom under χ^2 test. It was found that the dielectric constant, absorption coefficient and electrical conductivity show an inclination in the order PPDA<PPY<PTH<PANI<PEDOT. Among the above conducting polymers PANI and PEDOT show better electrical properties at microwave frequencies. The electrical properties of various important conducting polymers were studied with high precision at microwave region by automating the microwave bench with microcontroller.

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