

Current-Voltage and Capacitive Behavior of Zinc Oxide:Poly(methyl methacrylate) composite films

Gaurav Yadav¹, Vishakha Roy¹ and Neeraj Kumar¹

¹Department of Physics, Amity School of Applied Sciences (ASAS), Amity University Rajasthan, Jaipur, (Rajasthan) 302006, India

Abstract

In the present investigations, simple solution casting technique has been employed for the preparation of novel composite films of Zinc Oxide (ZnO) and Poly(methyl methacrylate) (PMMA). Different filler concentrations of ZnO have been prepared to study the capacitive behavior of the composite film at room temperature. The dielectric permittivity (ϵ) was measured as a function of frequency at different voltages (0, 2 and 5 V) over the range of 100 Hz – 10 MHz at room temperature. The dielectric permittivity of the composite films increases as enhanced the filler concentrations of ZnO in PMMA. The capacitance vs. frequency characteristics of the composite films reveals that the value of capacitance increases ~ 2 – 5 times from higher frequency region to lower region with increase in filler concentration of ZnO at room temperature. The I-V characteristics of the composite films were also investigated at room temperature.

Keywords: Zinc Oxide (ZnO), Poly(methyl methacrylate), Solvent caste composite, Dielectric permittivity and I-V measurement.

1. Introduction

The progress in the field of nanotechnology for coming decades strongly depends upon the novel materials and their composites, to develop and fabricate electronic products, supercapacitors and non-conventional devices [1]. Zinc Oxide (ZnO) is a promising candidate having a lot of potential applications in the field of science and technology [1-5]. ZnO has wide direct band gap (3.37 eV) at room temperature. Therefore its most common applications are in laser diodes and light emitting diodes (LEDs) [1].

There are many other applications in optoelectronic devices based on ZnO thin films. Compared to GaN, ZnO has a similar band gap (~3.4 eV) at room temperature but ZnO has a larger exciton binding energy (~60 meV). Therefore ZnO is favorable for electronic applications because of its stability to high-energy radiation, to wet chemical etching [3] and laser devices [5]. One application which has begun to be commercially available is the use of ZnO as the front contact for solar cells or of liquid crystal displays [LCD's] [6]. On the other hand the Transparent Thin-Film Transistors (TTFT) can be produced with ZnO.

Nanorods of ZnO based sensors are main devices to detect changes in electric current passing through zinc

oxide nanowires due to adsorption of gas molecules [9, 10]. In spintronics applications ZnO doped ferromagnetic ions have also been considered for ferromagnetic properties at room temperature [11]. Such room temperature ferromagnetism in ZnO:Mn has been observed. Piezoelectric properties in ZnO have been shown capable of fabricating "self powered nanosystems" with everyday mechanical stress from wind or body movements [12,13].

ZnO based polymer composites have a great effect in the field of materials science community because of their high electron mobility which makes it good active material for batteries and its eco-friendly nature makes it promising electrode material for supercapacitor[3].

1.1 PMMA Based Composites

PMMA is a versatile, low cost, and exceptional polymeric material. PMMA based composites have had a great interest towards the direction of microelectronic applications [11]. From research point of view, it is always preferred because of its utility as a good binder and also it is easy to handle in the process of formation in thin composite films. PMMA is one of remarkable interest polymer in the scientific community due to its ability to form composites with metaloxides for charge storage devices[1, 11]. In general, the incorporation of ZnO nanoparticles into Poly(methylmethacrylate) matrix can to a great extent modify the electrical, mechanical and dielectric properties. The lack of a centre of symmetry in wurtzite, combined with large electromechanical coupling, results in strong piezoelectric and pyroelectric properties and the consequent use of ZnO in mechanical actuators and piezoelectric sensors. For the preparation of thin composite films there are various physical and chemical techniques like in situ method, spin coating, sputtering, solvent casting method and sol-gel method have been preferred [5].

All these types of unique properties attract researchers to work on this material, therefore PMMA has been taken as a binder for the formation of metal oxide polymer composite thin films with different ZnO wt. % ratio. A lot of work has been carried out by many researchers on ZnO and its composite films to obtain the different applications [14-20].

After studying the I-V electrical characteristics at room temperature present work can help to understand the effect of PMMA on different filler concentrations composition of ZnO.

2. Experimental Techniques

For the fabrication of thin films there are a lot of techniques that can be used. In this paper we have used the sol-gel technique to prepare the samples. The sol-gel process is a synthesis route consisting in the preparation of a sol and successive gelation and solvent removal.

2.1 Selection Of Materials

To fabricate the ZnO:PMMA composite films, we have used Zinc oxide in powder form (Fischer scientific, India) and the granules of PMMA ($C_5O_2H_8$)_n manufactured by Aldrich USA) and Chloroform ($CHCl_3$) purchased by Sigma Aldrich, has been used for the preparation of the homogeneous solution for thin composite films. Both chemicals used in the present work were of analytical grade and were used without further purifications.

2.2 Synthesis And Fabrication Process Of The Composite Films

The investigated composite films were prepared on the basis of ZnO particles embedded into the PMMA polymer matrix. The granules of PMMA (20 mg by wt.) were dissolved in 5 ml of Chloroform in the ultraclean borosil beaker and stirred well at room temperature until the granules of PMMA was completely and homogeneously dissolved in chloroform (for this process minimum 2 hrs is required).

Further, the different (30%,50%,70%) filler composition of Zinc Oxide was then added to the solution of PMMA and the composite solution was then placed on magnetic stirrer (2NLH magnetic stirrer manufactured by Remi Equipment Pvt. Ltd. INDIA) for 60 seconds @ of rpm 200/minutes. A cleaned glass slide of dimensions (75 × 25 × 1.25 mm) was vertically and carefully dipped in to the composite solution of ZnO and PMMA. After few seconds, the slide of composite solution was carefully removed from the beaker and placed horizontally (making an angle of 180°) in to enclosed environment covered with a glass bell jar to prevent with the excessive moisture and unwanted impurities.

After 24 hrs the prepared thin composite films were carefully removed from the glass slide. The vacuum

deposited Indium tin oxide (ITO) was used as electrodes on both the surfaces for electrically contact to examine the electrical properties of the composite film to make a capacitor as shown in figure 1. The area of the deposited electrode on the glass slide was to be measured $25 \times 10^{-2} \text{ cm}^2$. Thickness of the samples was found to be 25-30 μm .

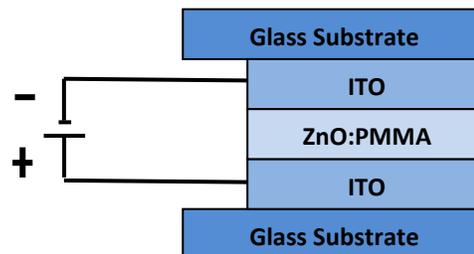


Fig. 1 Cross-Sectional Schematic of measurement of Electrical Properties.

3. Measurements And Characterization

The I-V measurements were carried out using 4284A LCR Meter (Agilent Technology) and the dielectric measurements were carried out at room temperature using Impedance/Gain phase analyzer (Hewlett Packard).

3.1 Current-Voltage (I-V) Characteristics

The I-V characteristics for 30%, 50% and 70% filler composition ZnO: PMMA composite films have been carried out at room temperature as shown in figure 2(a) to 2(b). In figure 2 (a) the graph was plotted in linear scale and in figure 2 (b) the graph has been plotted in semi log scale to know the behavior of currents as the current and voltage have an exponential relation so the semi log plot represents the data more accurately. The current voltage characteristics were measured by applying a pulse voltage swept from (0 → 40 → 0 → -40 → 0) at room temperature. The current was observed with a resistance connected in series with the composite films. The I-V curve for 30% filler composition composite films indicated in black color with square circles while the I-V curve for 50% filler composition composite film reflects in red color with circles.

The composite films showed highly non-linear conduction when voltage was applied as seen in the I-V hysteresis loop formed. This behavior of the composite film is due to the ZnO particles bound by PMMA. At the time of I-V measurements, initially the voltage was swept in complete cycle from 0 → +V → 0 → -V → 0. In positive bias region (0 → +V) as voltages were swept from 0 to 40 V the current flowing in positive direction

in the composite film from the upper part of the electrode and negative bias to create the dipoles for electrical polarization and same trends of flow of currents found in the opposite direction to polarized the dipoles created by the interactions of tiny particles as embedded in the composite films as shown in figure 2 (a).

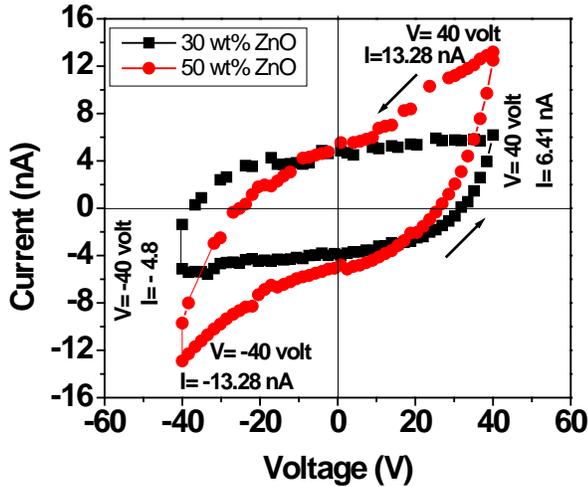


Fig. 2 (a) I-V characteristics for 30, 50 wt% ZnO:PMMA composite films.

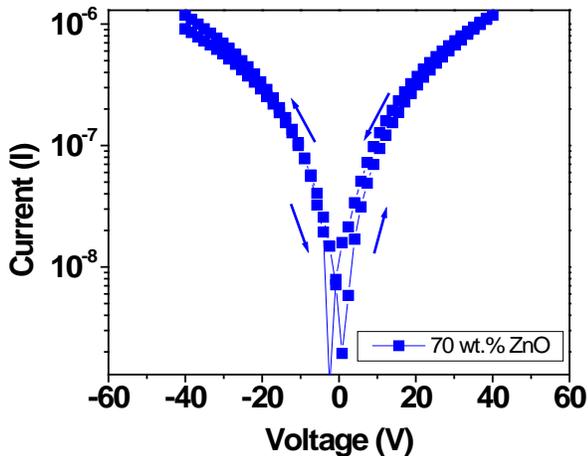


Fig. 2 (b) I-V characteristics for 70 wt% ZnO:PMMA composite films plotted on semi-log scale.

I-V characteristics strongly revealed the shape of hysteresis loop for both (30% and 50% filler composition) samples. By referring to the shape of the I-V curves in figure 2(a), it showed that both samples demonstrate rectifying behaviors as they are in well symmetrical form of hysteresis loops and perfectly crossed at origin as shown in figure 2 (c).

The 70% filler composition composite film also showed a symmetric non-linear I-V behavior, which is due to the effect of work function of the electrodes during applied voltages on the composite films. Such type of I-V characteristics reveals a good p-n junction for rectifying properties of diodes [3-9].

As figure 2 (a), showed the consecutive sweeps of 30% to 50% filler composition sample to prove the hysteresis behaviour of the composite films, we applied the positive bias (0 V → 40 V → 0 V) to the sample, the sample will transit from high resistivity state to low resistivity state, then for negative bias (0 V → - 40 V → 0 V) the resistivity was reduced from high resistivity state to low resistivity state. Hence the hysteresis behavior existence in the samples of 30 to 50wt.% and rectifying properties exhibited in 70% filler composition.

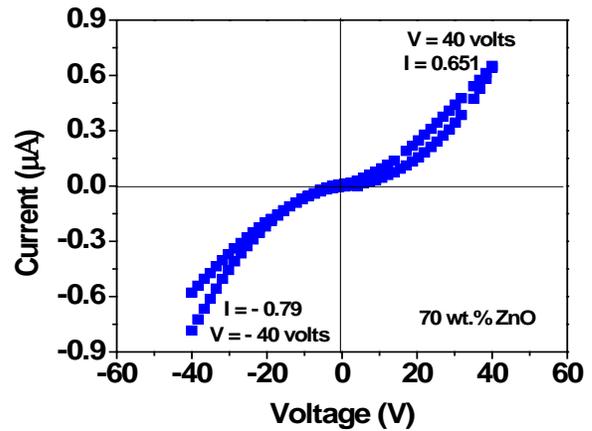


Fig. 2 (c) I-V characteristics of 70 wt.% ZnO:PMMA composite films

According to the theory applied by various researchers on such types of composites [31-33]; the resistivity of the device or films will reduce when positive voltages are applied (SET) because of the oxygen vacancies from the upper metal oxide active layer are repelled into the lower active layer thus increasing the conductivity of the device (ON STATE). When negative voltages are applied (RESET), it will attract oxygen vacancies from the lower active layer to the upper active layer which will decrease the conductivity and increase the resistivity thus making the whole device become resistive (OFF STATE) [33-35].

3.2 Measurement Of Dielectric Permittivity

The measurement of dielectric permittivity (ϵ) as carried out using Impedance/Gain phase analyzer (Hewlett Packard) as shown in figure 1. The capacitance of the material was observed over the frequency range of

100Hz-10MHz at applying voltages 0V, 2V and 5V (why these three voltages why not just one). This process was used to find the permittivity of the different % filler composition of the samples.

Effect of filler concentration

Variation of real part of permittivity (ϵ) as a function of frequency for various % filler composition of ZnO:PMMA composites at room temperature for different voltages are shown in figure 3(a). It has been found that the permittivity increased with increases in wt% of ZnO. The higher effective permittivity is observed for higher % filler compositions.

Effect of frequency

The frequency dependent characteristics of dielectric permittivity reveal that the value of permittivity increases from higher frequency to lower frequency range. It is due to the higher value of permittivity of pure ZnO than PMMA. Figure 3 (a)-(c) show that the value of ϵ is reduced drastically with increases in the value of frequency from 100 Hz onwards. The decrease in the value of ϵ is due to the structure of both the materials in the composite films and their concentrations. The low value of dielectric permittivity is due to the porous structure of both the constituent in the composites, with porosity lies between 50% to 80% [39-41].

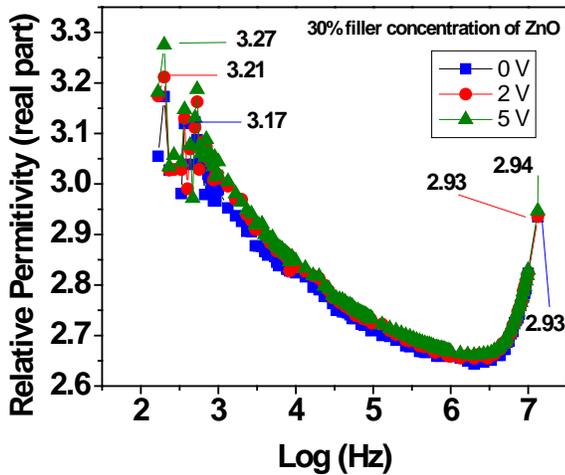


Fig. 3 (a) Dielectric permittivity vs. Log(f) graph of 30% filler concentration of ZnO

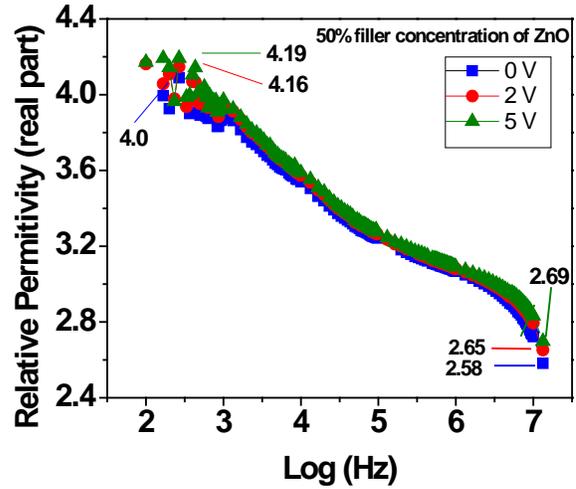


Fig. 3 (b) Behaviour of Dielectric permittivity vs. Log(f) for 50% filler concentration of ZnO

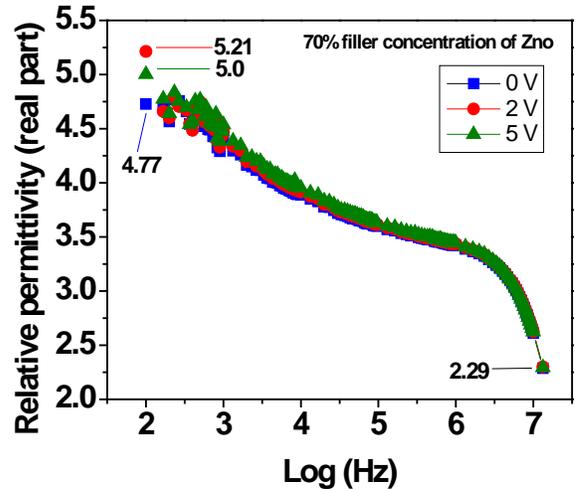


Fig. 3 (c) Dielectric permittivity vs. Log(f) graph of 70% filler concentration of ZnO

In figure 3 (a)-(c) clearly shown that the value of ϵ decreases with increase in frequency at low frequency region (1Hz- 3Hz). This is due to the space charge accumulation due to polarization because of irregularities of atoms in the composites. Therefore it consists of defects such as drooping bonds, vacancies, and pores at the grain that due to the high surface area per unit volume. Therefore, the defects on surface of the composite films cause dipole moment and induced polarization. Hence at low frequency, the hopping electrons are trapped by the imperfection at the interface.

As the frequency increases in the graphs of figure 3 (a)-(c) of dielectric permittivity vs. frequency there is no space charge polarization where dipoles fail to follow the change in electric field and there is no dispersion and finally leads to the decrease in dielectric permittivity.

3.3 Capacitance Vs Frequency Measurements

Capacitance vs. Frequency measurements have been extensively used in investigating the electrical properties of composite films. We have investigated the reveals that the trend of increasing capacitance is due to the increase of ZnO concentration in polymer.

For low value of the frequency, the charge built due to electric field originating from ionized donors in a ZnO layers of the composite films terminates on negative charges on the ITO electrodes. Here, the increase of ionized donors reduces the capacitance due to enhancement of the depletion layer. Figure 4 (a)-(c) shows the typical C vs. Log(f) characteristics of different filler composition of ZnO:PMMA composite films at room temperature between the frequency ranging from 100Hz-10MHz. All the three graphs shows that the resistance, interface states and interface layers between the ZnO:PMMA composites and the effect of electrode contact [3,9, 40-43]. The higher value of capacitance at

Capacitance curves at different frequencies at different ZnO filler composition at room temperature as shown in Figure 4(a)-(c). These curves show that the measured capacitance sharply decreases 1.7 times with increasing in frequency however the value of capacitance decrease downward 1.2 times. Moreover, the shapes of the Capacitance curves are strongly dependent on frequency and applied voltages.

In Figure 4 the capacitance for all the composites is higher than that of pure PMMA (4.15 pF), ranging from 18.5 pF to 21.5 pF as shown in figure 4 (a)-(c). The result

low frequency are due to the interface states in equilibrium with n-type of ZnO:PMMA with an application of ac signal. Further at low frequency the measured capacitance of the composite films at room temperature is dominated by the depletion capacitance of the rectifying contact.

value of capacitance decreases with increasing frequency. Such type of voltage and frequency dependent characteristics is due to the particular and prominent features of Schottky barrier, impurity level, high series

Fig.5 clearly shows that the value of capacitance and dielectric permittivity enhance almost linearly as increase in filler composition of ZnO. This is due to the contribution of ZnO particles embedded in polymer matrix.

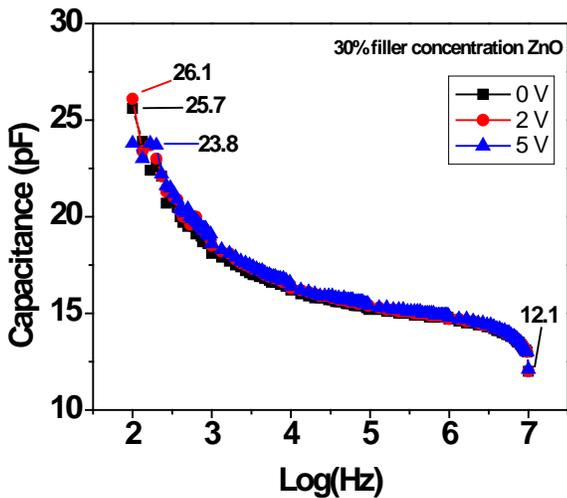


Fig. 4(a) Capacitance vs. Log(f) behavior graph of 30% filler concentration of ZnO

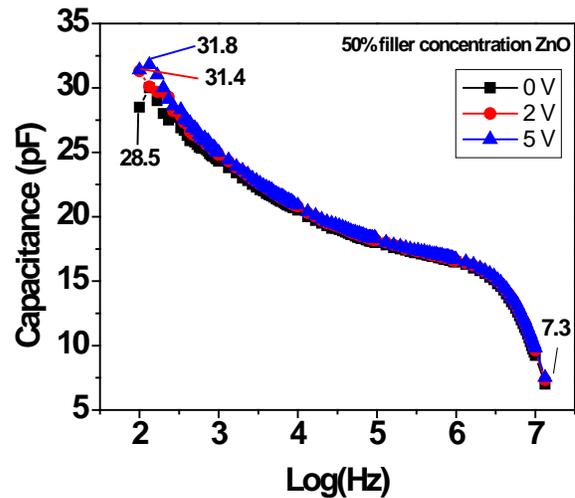


Fig. 4(b) Capacitance vs. Log(f) behavior graph of 50% filler concentration

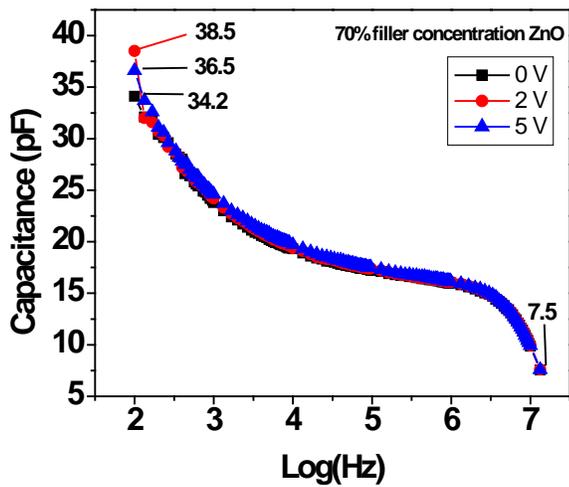


Fig. 4 (c) Capacitance vs. Log(f) behavior graph of 70% filler concentration of ZnO

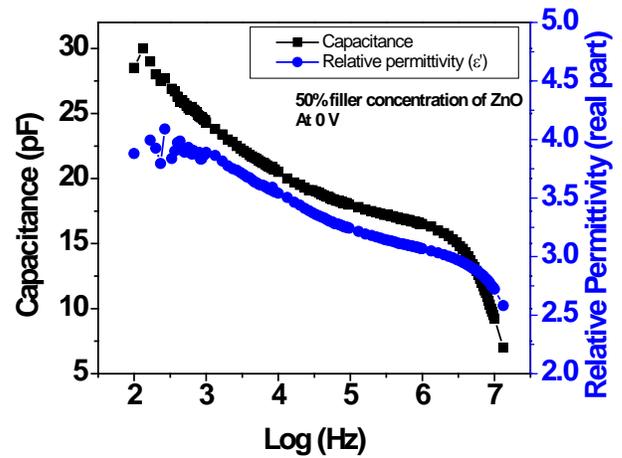


Fig. 4(d) Capacitance vs. Dielectric permittivity (real) at 0 V for 50% filler concentration.

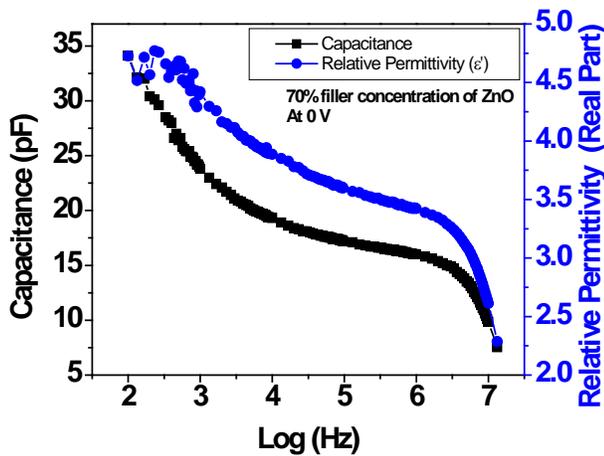


Fig. 4(e) Capacitance vs. Dielectric permittivity (real) at 0 V for 70% filler concentration.

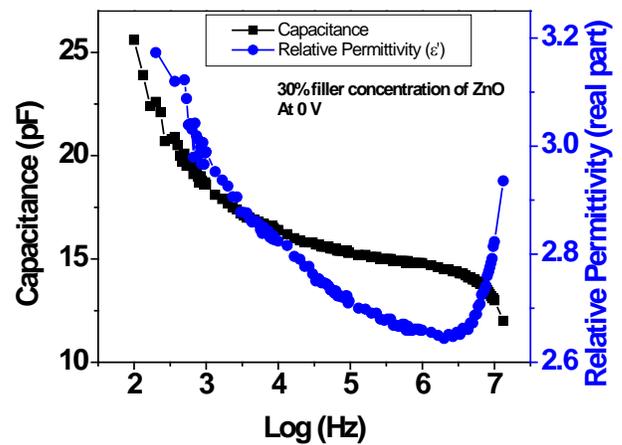


Fig. 4(f) Capacitance vs. Dielectric permittivity (real) at 0 V for 30% filler concentration.

The above fabricated films were cut into 2x1.5 cm pieces to fit a homemade silver electrode for characterization by measuring the dielectric properties. Precision LCR meter HP 4274 A connected with HP 4275 A and with Test Fixture HP 16047 A at frequency range 10^2 Hz to 10^5 Hz was used. The dielectric parameter as a function of frequency is described by the complex permittivity.

$$\epsilon^*(\omega) = \epsilon'(\omega) - j\epsilon''(\omega) \tag{Eq(1)}$$

where the real part ϵ' and imaginary part ϵ'' are the components for the energy storage and energy loss, respectively, in each cycle of the electric field. The measured capacitance C was used to calculate the dielectric constant, ϵ' using the following expression.

$$\epsilon' = Cd / \epsilon_0 A \tag{Eq(2)}$$

where d is the thickness between the two electrodes, A is the area of the electrodes, ϵ_0 is the permittivity of the free space, $= 8.85 \times 10^{-12} / N.m^2$ and ω is the angular frequency ($\omega = 2\pi f$), f is applied frequency. The dielectric loss ($\epsilon''(\omega)$) is described with eq. :

$$\epsilon''(\omega) = \epsilon'(\omega) \cdot \tan\theta(\omega) \tag{Eq(3)}$$

$\tan\theta(\omega)$ is tangent delta. The electric modulus is the reciprocal of the permittivity in complex form was found using eq.

$$M^* = 1 / \epsilon^* = M' + jM'' \tag{Eq(4)}$$

Where M' and M'' are the real and imaginary part of dielectric modulus and it was calculated by Eq.

$$M' = \epsilon' / \epsilon'^2 + \epsilon''^2 \tag{Eq(5)}$$

$$M'' = \epsilon'' / \epsilon'^2 + \epsilon''^2 \tag{Eq(6)}$$

3.4 Real and imaginary electrical modulus

In Figure 5(b) it can be seen that M' values increased with frequency. Nevertheless, the figure 5(a) peaks in M'' values were developed at the same frequency range, indicating the appearance of a relaxation process. The maximum of M'' increased when ZnO nanoparticles concentration amount increased, the frequency at the American Journal of Polymer Science 2012, 2(6): 135-140 139 maximum of the peak of M'' show the (ω') the relaxation frequency.

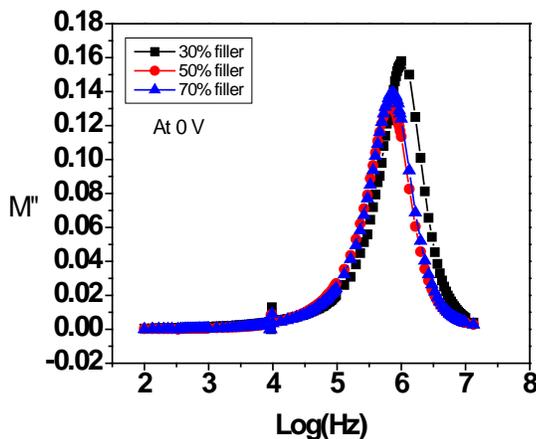


Fig. 5(a) Variation of imaginary electrical modulus of polymer at different filler concentration of ZnO nanoparticles composite.

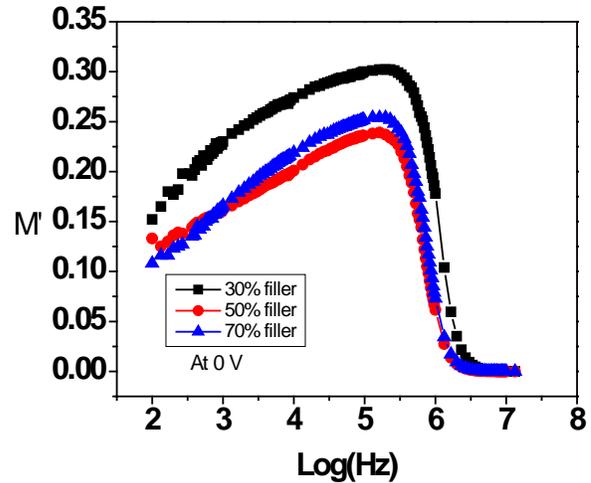


Fig. 5 (b) Variation of real electric modulus of polymer at different filler concentration of ZnO nanoparticle composites.

Relaxation peaks were displayed at higher frequencies since relaxation processes were influenced by the interfacial polarization effect which generated electric charge accumulation around the ZnO nanoparticles and the displacement of the peak as the particle content increased and this is identified with work of Tsangaris,G,et.al.

4. Conclusions

Novel composites film with varying different filler composition of ZnO in PMMA polymer matrix were successfully fabricated by the process of simple solution casting techniques. The capacitance and dielectric permittivity increased with increase in ZnO concentrations. Regions of low frequency relaxation are attributed to the space charge polarization. The composite films could be appropriate for electrical properties particularly for multilayer dielectric capacitors and related applications.

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Gaurav Yadav was born at village Pali of District Rewari (Haryana), India on 09 September 1994. He has received his Bachelor's degree (B.Sc.) in science from Maharishi Dayanand University, Rohtak Haryana in 2014 and now pursuing master's degree (M.Sc.) in Applied Physics under the Department of Physics at Amity University Rajasthan, Jaipur. During his M.Sc. degree, he has been deeply involved to carry out the advanced research work based on smart materials and their composites in thin film form.

Vishakha Roy was born at Lucknow (Uttar Pradesh), India on 02 June 1991. She has received her Bachelor's degree (B.Sc.) in science from Isabella Thoburn College affiliated to University of Lucknow in 2013 and now pursuing master's degree (M.Sc.) in Applied Physics under the Department of Physics at Amity University Rajasthan, Jaipur. In addition with this work she has showed enough interest in experimental research work based on polymer composite and their characterization for further processing.

Dr. Neeraj Kumar was born in Heempur Manak of District Bijnor U.P. India. He has received his Bachelor's and Master's degrees; both in first divisions from Vardhman (PG) College, Bijnor affiliated to Rohilkhand University Bareilly in 1997 and 1999 respectively. He has awarded his Degree of Doctor of Philosophy (Ph.D.) from Indian Institute of Technology (IIT) Roorkee in Experimental Solid State Physics with "A" Grade in 2005. He has done some advanced research work at Pennsylvania State University USA during his Post Doctoral Fellowship in 2006. Now he has been serving his duties as Head in the Department of Physics and the Coordinator of Directorate of Research and Publications at the Amity University Rajasthan, Jaipur since 2007. His area of research interest includes "Development and Characterization of Polymer – Ferroelectric Nanocomposite Films for Low Voltage Operated Devices, Piezoelectric Devices and Ferroelectric Devices. He has published his quality work in peer reviewed Journals of International repute like *Journal of Applied Physics*, *Ferroelectrics*, *IEEE Transaction on Dielectric and Electrical Insulation*, *Journal of Phys. D: Applied Physics*, *Polymer Engineering and Science* and many more. He has visited USA, China, and South Korea for his presentations as a speaker.