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Understanding the impact of gamma irradiation of epoxy titania nanocomposites on surface and bulk charge characteristics

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Abstract

Epoxy titania nanocomposites were prepared under optimum process conditions through shear mixing of titania nanoparticles in to epoxy resin, for its potential application as insulant in nuclear power plants and space applications. The complex intrinsic nature of properties, its characteristic variation due to ageing of nanocomposite insulating material upon its continuous exposure to gamma irradiation, and their charge trap and space charge characteristics are explored. Surface potential variation studies were carried out under DC voltage. In the present study, the charge trap performance was assessed under switching impulse voltage. It is observed that surface potential decay and shallow trap formation are high with gamma irradiated specimen. In addition, the potential decay is high under switching impulse voltage compared to DC voltage. Also, the trap depth formed is less under switching impulse voltage compared to DC voltage and it is high under negative DC voltage. The space charge analysis through Pulsed electro acoustic (PEA) studies has shown increase in accumulation of space charge and enhancement of electric field with increase in dosage of gamma-irradiation. Polarity reversal tests have revealed that the electric field enhancement is high before reversal of polarity, irrespective of level of gamma irradiation dosage. The direct correlation between characteristic variation in trap depth values with the gamma irradiated specimen and its contact angle was observed.

1. Introduction

Epoxy resins are used as dielectric material in a harsh environment, especially in nuclear power plants and in space applications, because of their superior properties such as low dielectric loss, high resistivity and high breakdown strength [1]. In recent times, epoxy nanocomposite materials are found to exhibit enhanced mechanical, thermal, and electrical properties [2]. Smith et al have indicated that metal oxide nano fillers show better performance [3]. Among the several metal-oxides, TiO₂ has some unique electrical and chemical properties and it is widely studied to understand the performance of the epoxy titania nanocomposites [4]. Thus it has become essential to understand the electrical performance of gamma irradiated epoxy titania nanocomposites. Chang et al have fabricated Carboxyl-functionalized multi-walled carbon nanotube (c-MWCNT) with epoxy resin using gamma radiation and found out that the resulting epoxy nanocomposites are having enhanced radiation resistance properties [5]. Gao et al have observed that electron beam irradiated epoxy/Al₂O₃ nanocomposites materials show high partial discharge resistance [6]. Multiple changes occur to the surface of the polymer insulating material, on irradiation of polymer material, which can cause characteristic variation in its fundamental insulation properties [7–9].

Surface charge accumulation is one of the major problems with the insulating material used in the power apparatus, which can initiate the surface discharge process to occur causing early failure of insulating material. Also, the insulating material needs to be free from space charge. Wang et al have studied the impact of moisture on space charge variation in LDPE silica nanocomposites and have indicated that mono layer moisture present in the insulating material have a high impact on charge dynamics and breakdown voltage [10].
observed that any space charge present in the insulating material during polarity reversal can cause field enhancement in the insulating material leading to catastrophic failure of insulating material [11]. Tantipattarakul et al, have clearly indicated that chemical defects due to ageing can cause space charge variation in material [12]. The fundamental understanding of the impact of irradiation on electrical performance especially the surface charge accumulation characteristics and the space charge characteristics of epoxy nanocomposites is scanty.

Having known all these aspects, the following experimental studies are carried out with gamma irradiated epoxy titania nanocomposites, to understand the impact of gamma irradiation on its performance. (i) The surface potential variation and charge trap characteristics of epoxy titania nanocomposites under DC and switching impulse voltage (ii) The space charge accumulation characteristic studies in epoxy nanocomposite through pulsed electro acoustic (PEA) measurement studies.

2. Experimental studies

2.1. Sample preparation
The dielectric properties of the nanocomposites rely on the filler type, its functionalisation, size and shape, preparation process [13]. Thus in the present study, rutile TiO$_2$ (MT100S- TYCA corporation, Japan) with an average diameter of 15 nm is shear mixed with epoxy resin for 5 wt%. To the uniformly dispersed epoxy nano filler mixture, acid anhydride hardener is added. The resulting mixture is degassed, cast for 0.5 mm thick samples, and left for curing to get epoxy/titania nanocomposite. The schematic representation of the sample preparation is shown in figure 1. The obtained epoxy nanocomposite material was subjected to gamma irradiation in air ambiance through Caesium 137 at a dosage rate of 10.58 Gy min$^{-1}$ using BL2000 up to total doses of 4, 8 and 12 kGy.

2.2. Surface potential measurement setup
Figure 2 shows the schematic representation of the experimental setup for the surface potential measurements at room temperature. Needle-plane electrode configuration was used for depositing the unipolar charges on the surface of the epoxy nanocomposites, by generating corona discharges at 12 kV under DC voltage and switching impulse voltage of 100 $\mu$s/1000 $\mu$s using Trek high voltage amplifier (Model 20/20C) with input from a signal generator (Tektronix AFG 3051C). On the deposition of charges for 2 min (at position 1), the surface potential on the insulating material was measured using the Kelvin probe (at position 2) connected to the electrostatic
voltmeter (Trek Model 341B). The measured potential is recorded by using a digital storage oscilloscope (LeCroy WaveRunner 62Xi, 5 GSa s⁻¹, 600 MHz).

2.3. PEA Measurement setup

The space charge accumulation studies were carried out with a flat plate sample (with dimension 40 × 40 × 0.5 mm) by using PEA (Pulsed Electroacoustic Analysis) under the high DC electric field. The schematic diagram of the PEA measurement unit is shown in figure 3. The main components in the PEA system include a 30 kV high voltage source (TECHIMP), a 500 V pulse generator (TECHIMP 024FP052) with 10 ns pulse width operating at a frequency of 150 Hz, Oscilloscope (Tektronix DPO5034B, 350 MHz, 5 GSa s⁻¹) and a piezoelectric transducer. To obtain the impedance matching, a semiconductive layer is fixed on the surface of the high voltage electrode. A sequence of very short time pulses was applied to the test specimen placed between the high voltage electrode and the bottom plane aluminium electrode, which is at ground potential. Each pulse produces an electric force displacing internal charges and thus generating pulsed acoustic pressure waves, which are detected by a polyvinylidene fluoride (PVDF) piezoelectric electric transducer. The output of the transducer connected to an amplifier and the output is recorded in the oscilloscope, for the required time. For restoring the original signal, the obtained data is processed by using the calibration trace and deconvolution technique.

2.4. Selection of poling time

In the present study, two different tests were carried out which include (a) 1 h poling time (voltage-on) and 30 min depoling time (voltage-off). The poling time is fixed based on several trials, where it is observed that space charge accumulation is found to be negligible. Since the sample is thin, the space charge accumulates very fast and increases very slowly after one hour during poling and decays very fast within 30 min on depoling. (b) Polarity reversal test, where the required voltage is applied to achieve required $-V_{appl}$, for 1 h, and the applied potential is reversed to the opposite polarity to achieve $+V_{appl}$ and stressed for one hour. In the present study, the polarity reversal time is fixed as the 40 s. The schematic representation of the voltage application duration for the DC test and polarity reversal test is shown in figure 4.

3. Results and discussions

3.1. Surface potential variation with gamma irradiated epoxy nanocomposites

3.1.1. Under DC voltage

Figure 5 shows the variation in surface potential of epoxy nanocomposites on the removal of the charge deposition process by DC corona activity. There are several processes by which the surface charge deposited on the sample gets dissipated such as lateral charge spreading, neutralization with the ions in the air, transportation of charges through the bulk of the sample, etc. Liang et al have indicated that charge transport through the bulk is considered as the main decay process [14]. It is observed that the magnitude of negative charge is high compared to that of the positive charge in the surface charge characteristics. Similar characteristics were observed by Du et al and have indicated that negative corona can be induced more easily than a positive corona with an identical absolute value of applied voltage thereby, the number of negative charges generated be high compared to positive charges [7]. This generated charges gets deposited on the surface of the insulating material. As the charge is transferred through the bulk, charge trapping and detrapping play an important role in the decay characteristics. The charge decay rate is more on the removal of the charge deposition process by corona activity.
and becomes less with time as shown in figure 5. The fast decay rate is due to the detrapping of shallow traps and later on, the charge needs to be detrapped from the deep traps. As the gamma-ray irradiation dosage is increased the decay rate gets enhanced due to structural variation occurred due to irradiation forming chain scission, oxidation on the surface of the specimen enhancing the shallow trap site [15].

Charge trapping and detrapping play a major role in charge transportation through the sample. The demarcation energy, $E_m$ indicates the border between the emptied and occupied traps. The trap distribution characteristics can be derived from the surface potential decay characteristics, by adopting isothermal current decay theory. From Simmons and Tam’s theory [16], trap density and the energy gap in the charge trap sites are given as [17].

$$\Delta E = E_c - E_m = kT \ln (vt)$$

$$N(E) = \frac{2 \varepsilon_0 \varepsilon_r}{qL^2kT_0(E)} \frac{dV_s}{dt}$$

Where $\Delta E$ is trap depth, $E_c$ is the conduction band energy level, $T$ is the absolute temperature in kelvin, $k$ is Boltzmann constant, $t$ is time, $v$ is an attempt to escape frequency and its value is set to $10^{12}$, $N(E)$ is trap density at energy level $E$, $\varepsilon_0$ is the permittivity of free space, $\varepsilon_r$ is relative permittivity of the sample, L is thickness of the sample, $q$ is charge of an electron, $V_s$ is the surface potential on the sample and $f_0(E)$ is the occupancy rate of initial electrons.

Figure 6 shows the trap distribution characteristics of epoxy nanocomposites for charge deposited due to corona activity formed under positive and negative DC voltage of 12 kV. As the irradiation is increased, the initial surface potential on the sample is decreased and there is a left shift in the peak of the energy density, $N(E)$ which shows that the energy gap is decreasing. This indicates the formation of shallow trap sites with gamma-ray irradiated samples when compared with virgin samples, as can be observed from table 1. Xing et al have studied the charge trap characteristics with gamma irradiated epoxy AlN nanocomposites and have indicated the left
shift in the trap depth [18]. Haque et al have indicated that charge trap sites occur in solid insulation due to physical changes, chemical impurities or by the radiation caused damage [19]. In general, high energy irradiation can form chain scission and oxidation to the insulating material enhancing surface trap site and deep trap sites. Huang et al have indicated that the deep trap formed can enhance the surface flashover voltage [8]. However, the trapped charges can gain energy by several mechanisms including electron tunneling, impact ionisation, thermal de-trapping, and photon assisted detrapping [20]. Thus depending on the trap depth and local condition, the trapped charges cause local electric field enhancement causing localised discharges leading to catastrophic failure of insulating material [21].

3.1.2. Switching impulse voltage

The surface charge is deposited on the sample by corona charging using a series of switching impulse voltage, 100 μs/1000 μs at 100 Hz for both positive and negative polarities with a peak voltage of 12 kV. Figure 7 shows the surface potential decay of epoxy nanocomposites after the sample surface is charged for 2 min. With an

![Figure 6. Trap distribution characteristics of epoxy nanocomposites under (a) +12 kV DC and (b) −12 kV DC.]

| Table 1. Surface potential decay and trap characteristics for DC and switching impulse voltages. |
|---------------------------------------------------------------|
| Voltage sample     | DC voltage                      | Switching impulse voltage                  |
|                   | Positive polarity | Negative polarity | Positive polarity | Negative polarity |
| A                 | B               | A             | B               |
| Virgin            | 0.0009 0.90      | 0.0007 0.90   | 0.0023 0.87      | 0.0028 0.87       |
| 4 kGy             | 0.0013 0.89      | 0.0009 0.90   | 0.0036 0.86      | 0.0050 0.85       |
| 8 kGy             | 0.0018 0.88      | 0.0013 0.89   | 0.0049 0.85      | 0.0071 0.84       |
| 12 kGy            | 0.0026 0.87      | 0.0023 0.87   | 0.0085 0.84      | 0.0104 0.83       |

A: Decay rate (s⁻¹); B: Trap depth at the peak value of trap density (eV)
increase in irradiation dosage, the initial surface potential is decreased and the decay rate is increased, irrespective of the polarity of charging through switching impulse voltage (table 1). Due to gamma irradiation of polymer composites, matrix structure gets altered due to chain scission, oxidation, and crosslinking. As a consequence, carbonyl (C=O) and hydroxyl (–OH) groups are observed in epoxy composites. The adjacent radicals can cross link thereby reducing the surface charge trap sites leading to a reduction in surface potential [22]. On removal of charging the surface, the recombination and lateral motion of charges occur and the increase in conductivity due to shallow traps formed enhances the decay rate. Thus, with an increase in irradiation dosage, the initial surface potential is decreased and the decay rate is increased, irrespective of the polarity of charging through switching impulse voltage. The initial surface potential is less and the decay rate is increased with charge deposited under switching impulse voltage compared to the charges deposited under DC voltage, irrespective of polarity, and level of irradiation.

Du et al have investigated the trap distribution characteristics of epoxy resin for a repetitive pulse voltage and have observed reduction in trap depth, irrespective of the polarity of the applied voltage pulse [23]. With an increase in irradiation dosage, there is a reduction in the trap depth as given in table 1, which indicates that shallow traps are increased. The characteristics are much similar to the trend observed under DC voltage (table 1). Comparing trap depths under DC and switching impulse voltages, the charge gets trapped deeper in insulating material under negative DC voltages. When the epoxy nanocomposite is gamma ray irradiated, structural changes occur to the surface of the material. Pulikkalparambil et al carried out accelerated weathering studies with the bio epoxy ionic liquid blends and have indicated an increased contact angle [24]. In the present study, to check the surface condition of the insulating material for hydrophobicity, contact angle measurements are performed. A typical photograph of the water droplet for contact angle measurement with epoxy nanocomposites is shown in figure 8. The results of the study indicate a marginal reduction in contact angle (table 2). The characteristics of the trap depth and contact angle shows a direct correlation.

3.2. Space charge variation in gamma irradiated epoxy nanocomposites

Figure 9 shows the space charge profiles of epoxy nanocomposites obtained by the PEA technique, during poling period, under different electric fields. It is observed that the space charge accumulation increases with an increase in gamma-ray irradiation dose. The homo charges are formed by the balance between the electron
injection or extraction from electrodes to the recombination or diffusion through sample bulk [25]. With an increase in the applied electric field, there is an increase in homo charge accumulation near to the electrodes. It is essential to understand the electric field limit to avoid any catastrophic failure of insulating material.

Dissado et al. have calculated the mean magnitude of stored charge, \( Q_m \) to determine the electric field threshold limit of the sample and is derived as the total charge stored in the sample just at the beginning of the depoling stage [26]. The mean magnitude of stored charge, \( Q_m \) (C m\(^{-3}\)) is calculated as

\[
Q_m(E, t) = \frac{1}{(x_1 - x_0)} \int_{x_0}^{x_1} |\rho(x, t)| \, dx
\]

Where \( \rho(x) \) is the charge density, \( x_1 \) and \( x_0 \) are the electrode positions and the induced charge at the electrode is not taken into consideration.

Figure 10 shows a variation in charge density with the different applied electric fields for gamma-ray irradiated samples. The space charge accumulation enhances with increasing electric field and above certain electric field, space charge accumulation in the nanocomposite enhances rapidly, is the point of threshold electric field limit. The threshold electric field for the epoxy nanocomposites decreases with an increase in irradiation dose, as shown in Table 2.

| Sample  | Threshold electric field (kV mm\(^{-1}\)) | Contact angle (degree) |
|---------|-----------------------------------------|------------------------|
| Virgin  | 8.872                                   | 64.87                  |
| 4kGy    | 8.753                                   | 62.51                  |
| 8kGy    | 8.667                                   | 60.06                  |
| 12kGy   | 8.540                                   | 58.93                  |

3.2.1. Space charge profile during poling
Figure 11 shows the variation in space charge profiles of epoxy nanocomposites stressed with 12 kV mm\(^{-1}\) at different instants of time during the poling period. It is observed only the homo charges are present at the electrodes. Also, not much of a peak shift is noticed with an increase in poling time, especially with the virgin epoxy nanocomposites. This indicates that no charge transport into the bulk of the insulating material has occurred. With gamma irradiated specimen, a marginal spread in the homo charges is observed, near to the surface of the specimen. On comparing with figure 6, increase in the surface charge trap predominated than the deep trap formation in the bulk of the insulating material. Canadas et al. studied space charge behaviour with UV
irradiated PET material and have observed shallow trap sites in irradiated material than deep traps [27]. As time progresses, the homocharge formed near to the electrode opposes the applied electric field thereby reducing the further charge injection from the contact electrode [28].

Figure 10. Variation in charge density with gamma irradiated epoxy nanocomposites for different electric fields.

Figure 11. Variation in space charge profiles when stressed with 12 kV mm\(^{-1}\) at different instants of time during poling for (a) Virgin and (b) 12 kGy epoxy nanocomposites.
3.2.2. Space charge profile during depoling

Figure 12 shows the space charge profile in epoxy nanocomposites, at different time instants, during depoling. As time progresses, the homo charge near the electrodes and the trapped charge in the bulk of the specimen is detrapped or recombined. Chen et al. have said that space charge decay is determined generally by the concentration of carriers, their mobility and by the local electric stress \[29\]. The rate of dissipation depends on the detrapping time constants of the shallow and deep traps \[30\]. As irradiation causes the structural variations in the sample, the mobility of the carriers in the virgin sample is different from the gamma-ray irradiated samples.

Figure 13 shows the space charge, \(Q_m\) decay after being poled for 1 h. In general, the depoling process involves three different processes which include charge de-trapping, charge transport, and charge injection, and each mechanism has a different time period of operation \[31\]. They also have indicated that the space charge decay and the initial charge density at the start of the depoling stage increase with an increase in irradiation dosage. From figure 13, we can observe two different trends in the decay process of space charge.

\[Q_m(t) = q_s e^{-\tau_s t} + q_d e^{-\tau_d t}\] (4)

By fitting the space charge decay curve to the double exponential as given in equation (4) we can find the characteristic parameters during depoling as given in table 3.

In table 3, the parameters \(q_s\) and \(q_d\) (C m\(^{-3}\)) represents the amount of trapped space charge. The parameters \(\tau_s\) and \(\tau_d\) (s\(^{-1}\)) represent the space charge decay rates. As the absolute values of \(q_s\) and \(\tau_s\) are large when compared with \(q_d\) and \(\tau_d\), it indicates the shallow traps are more. Zhou et al. have studied the space charge decay process in composite insulators using the double exponential function and have indicated similar characteristics as observed with epoxy titania nanocomposites \[30\].

3.3. Space charge Variation in gamma irradiated epoxy nanocomposites on polarity reversal

Figure 14 shows the variation in space charge profile in epoxy nanocomposites on polarity reversal with time. The polarity reversal time is fixed for 40 s. During the polarity reversal process, we can see the transition of positive to negative charges near the Al electrode and negative to positive charge near the SC electrode which clearly indicates the accumulation of homo charges near to the electrodes.
Figure 13. Space charge decay in epoxy nanocomposites during depoling.

Table 3. Space charge decay parameters.

| Sample | $q_s$ (C m$^{-3}$) | $\tau_s$ (s$^{-1}$) | $q_d$ (C m$^{-3}$) | $\tau_d$ (s$^{-1}$) |
|--------|-------------------|-------------------|-------------------|-------------------|
| Virgin | 1.341             | 0.035             | 0.235             | 0.0002            |
| 4 kGy  | 1.592             | 0.049             | 0.169             | 0.0002            |
| 8 kGy  | 1.725             | 0.057             | 0.251             | 0.0002            |
| 12 kGy | 1.992             | 0.066             | 0.285             | 0.0002            |

Figure 14. Space charge profile during polarity reversal from $-12$ kV mm$^{-1}$ to $+12$ kV mm$^{-1}$ for (a) Virgin and (b) 12 kGy epoxy nanocomposites.
The space charge pattern with epoxy titania nanocomposites during the polarity reversal test from $-12 \text{ kV mm}^{-1}$ to $+12 \text{ kV mm}^{-1}$ is shown in figure 15. On polarity reversal, the reversed charge peaks occur with an increase in voltage, near to the electrodes. During this process, the homo charges present in the system before reversal will not respond quickly for the reverse voltage variation thereby an image hetero charge on the electrodes in addition to the capacitive charge be formed\[32\]. And also, it is observed that on polarity reversal, the rate of charge migration to the opposite electrode is high with gamma irradiated specimen compared with virgin epoxy nanocomposites.

Figure 16 shows the variation in electric field distribution in epoxy nanocomposite on polarity reversal with time. With an increase in irradiation dosage, the electric field across the sample is increasing. As discussed earlier, the trapped charge after polarity reversal distorts the local electric field as shown in figure 16. Wang et al have indicated that the trapped charges during polarity reversal distort the local electric field distribution\[33\].

The electric field distortion in the sample is defined by the electric field enhancement factor (EF) is calculated as
Where $E_{\text{m}}$ is the electric field in the sample bulk (kV mm$^{-1}$), $E_{\text{appl}}$ is the applied electric field (kV mm$^{-1}$) and $EFEF$ is the electric field enhancement factor. During polarity reversal, the applied electric field, $E_{\text{appl}}$ is changed from $-12$ kV mm$^{-1}$ to $+12$ kV mm$^{-1}$ and the electric field inside the sample bulk, $E_{\text{m}}$ before and after the polarity reversal i.e., at 3620 s and 7260 s resp., is used in the calculation of electric field enhancement factor. As the polarity reverse period is only 40 s, the distortion in the electric field is determined by the trapping or recombination of the fast electron and slow hole during polarity reversal \textsuperscript{[11]}. With an increase in irradiation dosage, space charge accumulation in the sample increases. So the electric field distortion also increases which in turn increases the electric field enhancement factor. It is realised that the level of change in field enhancement after polarity reversal is less with epoxy nanocomposites compared to gamma irradiated specimen. The cause for it could be due to the trapped charges in the bulk volume of nanocomposites present during poling period would have got recombined during polarity reversal thereby reducing enhancement in local electric field and is more with gamma irradiated specimen as shown in figure 17.

Figure 18 shows the space charge decay variation with time during depoling in DC and polarity reversal test respectively. With gamma-ray irradiated specimen, the decay rate is fast compared to the virgin specimen. But space charge decay during depoling in DC test is fast compared with the polarity reversal test. On polarity reversal, the electric field applied is reversed in a very short time so, some charges trapped in the bulk after polarity reversal take more time for detrapping or for recombining with the trapped charge.
4. Conclusions

The important conclusions accrued based on the studies are the following:

- Surface potential variation with gamma irradiated epoxy nanocomposites indicates a reduction in initial surface potential, an increase in decay rate with the left shift in the energy trap distribution characteristics, confirming an increase in shallow traps. The characteristic variation in trap depth values and the contact angle shows a direct correlation.

- Comparing trap depths under DC and switching impulse voltages, the charge gets trapped deeper in insulating material under negative DC voltages.

- Increase of gamma irradiation dosage enhances the accumulation of space charge during poling and with high decay of charge during depoling.

- The electric field enhancement in epoxy nanocomposites is increased when the dosage of gamma irradiation is high. The threshold electric field limit is decreased with an increase in gamma-ray irradiation dose. Also, on polarity reversal, irrespective of level of irradiation, the electric field enhancement is reduced, which is the indicative of deep trap formation in the material.

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