Investigation of surface strain by digital image correlation and charge trap characteristics of epoxy alumina nanocomposites

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Abstract

Epoxy alumina nanocomposites were fabricated through shear mixing of alumina nanoparticles in to epoxy resin under optimum process conditions for its potential application as insulator in transformer. The fundamental insight in to the influence of ageing of nanocomposite insulator due to its continuous exposure to UV irradiation and water in terms of their charge trap characteristics was explored. The surface potential decay and strain variation of nanocomposite samples were measured by using Electrostatic voltmeter and Digital Image Correlation technique (DIC), respectively. Diffusion of water into epoxy alumina nanocomposite is less compared with pure epoxy resin. It has been observed that surface potential decay rate and trap depth have shown increasing and decreasing trend, respectively, with the addition of alumina nanoparticles in to epoxy resin. The UV and water aged nanocomposites exhibited a decreasing trend of surface potential decay rate and the governing reasons could be due to surface damage causing increase in deep trap formation. The tensile strength and stiffness of the samples have increased with the addition of alumina nanoparticles in to the epoxy resin. It could be due to the better bonding of alumina particles with epoxy resin. The surface strain induced in to the sample during tensile loading and the surface potential decay rate of nanocomposite has shown a direct correlation, as observed in the present work.

1. Introduction

Epoxy-resin is widely used as an insulant in transformers, bushings, electrical machines windings, etc because of its excellent electrical, thermal and mechanical properties [1]. Nano particles, on addition to base resin can further enhance its electrical, thermal and mechanical properties and alumina is found to be the best nanofiller as reported in the literature [2, 3]. Exposure to the ultra violet (UV) radiation can have adverse impact, if the insulant is used for outdoor structures. UV radiation destroys the polymeric structure due to degradation of fundamental electrical insulating properties and tensile properties of polymeric materials [4, 5]. Hence, it is essential to study the impact of UV radiations on electrical properties and mechanical properties especially tensile strength and strain distribution of the epoxy alumina nanocomposites material.

In addition, outdoor insulators are exposed to rain and humid environments. In the process, the deposited water layer, over a period of time can diffuse into the bulk volume of insulant and can alter its fundamental properties. Hui et al reported the formation of water shell around the nanoparticles which lead to change in dielectric behaviour of cross linked polyethylene silica nanocomposites [6]. Qiang et al clearly indicated that water absorption is highly influenced by the surface morphology and structure of the particles. They have experimentally demonstrated the existence of water shell structure around the nanoparticles and studied the influence of moisture content on the dielectric properties of epoxy silica and boron nitride nanocomposites and concluded that boron nitride filler added epoxy nanocomposites is more suitable for its application in humid
In the present study, epoxy resins are used as a base material and alumina nanoparticles of 99.9% purity with size of 30 nm were used for the fabrication of epoxy-alumina nanocomposites. Alumina nanoparticles were kept at 150 °C in oven for 24 hours in order to remove the moisture. The dried alumina nanoparticles were mixed with ethanol by using high frequency sonicator. The alumina-ethanol solution was mixed with required amount of epoxy resin by using a high speed shear mixer operated at a speed of 4000 rpm for 6 hours followed by high frequency sonification (for one hour at 20 kHz). On completion of the process, required amount of curing agent (Tri-ethylene tetra-amine purchased from HUNTSMAN) was added to the epoxy-alumina mixture and left for degassing in order to remove the trapped air bubbles. The degassed mixture was moulded in required size and left for curing for 24 h, at room temperature. Epoxy nanocomposites with 1 wt%, 3 wt%, and 5 wt% of alumina nanoparticles were fabricated.

2.2. UV ageing and water ageing test
An UV chamber equipped with a xenon lamp was used to perform UV ageing of epoxy-alumina nanocomposites. The UV light covers the wavelength of 200 nm to 400 nm. Samples were kept at a distance of 20 cm from the xenon lamp. UV ageing of samples was performed for 150 and 300 hours. Water ageing test was performed using deionised water. Epoxy-alumina nanocomposites were immersed in the deionised water and were kept in oven at a temperature of 90 °C. The water ageing was performed for all the samples until the saturation in the weight is gained by the epoxy nanocomposites.

2.3. Surface potential decay measurement
Figure 1 shows the experimental setup to measure the surface potential decay. It consists of needle electrode of tip radius 0.3 mm and sliding ground electrode mechanism. Function generator was used to generate positive and negative dc voltages which was further amplified by trek amplifier (Trek model 20/20C) to get high dc voltage. High voltage was connected to needle electrode. The gap between the sample surface and the tip of needle electrode was maintained 5 mm throughout the experiment. In the present study, sample was kept under needle electrode (position 1) and charged for 5 min by applying dc voltage of ±10 kV and, then brought under sensor (position 2) which was connected to electrostatic voltmeter (Trek Model 341B) for measuring the surface potential. Digital storage oscilloscope (DSO) was used to record the surface potential decay characteristics.

Surface charge accumulation over a period of time on the surface of insulating materials can lead to surface flashover at normal operating voltage itself [8]. Therefore, it is essential to understand the influence of nanofiller on the surface potential variation due to surface charge accumulation under normal, UV ageing, and with water aged specimens.

In addition, insulating materials should have good mechanical properties. Earlier research indicated that tensile properties of materials are highly dependent on the proper dispersion of nanofiller in the base matrix, and interfacial adhesion between nanofiller and matrix [9, 10]. The conventional method of measuring the stress and strain falls under destructive test, by using an universal testing machine in order to get cumulative strain. An alternate method ought to be used to get localised point strain using surface of the sample. Digital image correlation (DIC) is one of the non-contact methods to measure the localised strain [11, 12]. Thus, an attempt has been made to analyse the strain distribution at room temperature under different ageing conditions and at elevated temperatures, using the conventional tensile test method and the DIC technique, and to correlate the strain distribution using these two methods. Since the strain distribution is a surface phenomenon, an attempt has been made to correlate the surface potential decay characteristics and strain distribution to understand the electromechanical behaviour of the epoxy-alumina nanocomposites.

With the view of above literature, methodical experimental studies are carried out to understand the following aspects with epoxy alumina nanocomposites with and without exposure to UV ageing and water ageing treatment. (i) To investigate the surface potential variation, and (ii) To investigate the tensile behaviour and strain distribution of the nanocomposites at different temperatures using the conventional tensile test and the DIC technique.

2. Experimental studies

2.1. Sample preparation
In the present study, epoxy resins (Araldite CY 205 in liquid, solvent-free, unmodified Bisphenol A epoxy purchased from HUNTSMAN) as a base material and alumina nanoparticles of 99.9% purity with size of 20–30 nm (purchased from Richem international, USA) were used for the fabrication of epoxy/alumina nanocomposites. Alumina nanoparticles were kept at 150 °C in oven for 24 hours in order to remove the moisture. The dried alumina nanoparticles were mixed with ethanol by using high frequency sonicator. The alumina-ethanol solution was mixed with required amount of epoxy resin by using a high speed shear mixer operated at a speed of 4000 rpm for 6 hours followed by high frequency sonification (for one hour at 20 kHz). On completion of the process, required amount of curing agent (Tri-ethylene tetra-amine purchased from HUNTSMAN) was added to the epoxy-alumina mixture and left for degassing in order to remove the trapped air bubbles. The degassed mixture was moulded in required size and left for curing for 24 h, at room temperature. Epoxy nanocomposites with 1 wt%, 3 wt%, and 5 wt% of alumina nanoparticles were fabricated.

2.2. UV ageing and water ageing test
An UV chamber equipped with a xenon lamp was used to perform UV ageing of epoxy-alumina nanocomposites. The UV light covers the wavelength of 200 nm to 400 nm. Samples were kept at a distance of 20 cm from the xenon lamp. UV ageing of samples was performed for 150 and 300 hours. Water ageing test was performed using deionised water. Epoxy-alumina nanocomposites were immersed in the deionised water and were kept in oven at a temperature of 90 °C. The water ageing was performed for all the samples until the saturation in the weight is gained by the epoxy nanocomposites.
2.4. Mechanical test

2.4.1. Tensile test

The tensile test was conducted on the samples with different wt% of alumina filled epoxy nanocomposites, using an universal testing machine (KIC-2-0200-C) with a loading capability of 20 kN. The loading rate of the upper cross head was fixed to be 2 mm min\(^{-1}\) and was kept constant for all the samples under the test. In the present study, tensile samples with different wt% of alumina nano filler were prepared as per ASTM D-638 (Type IV) standard with dumbbell shape having dimensions of 115 X 6 X 3 mm and a gauge length of 25 mm. The tensile test was conducted at room temperature and also at elevated temperatures of 40 °C and 50 °C.

Several parameters were deduced and tensile stress, tensile strain, and Young’s modulus were estimated. Equations (1) and (2) respectively represents tensile stress (\(\sigma\)) and tensile strain (\(\varepsilon\)),

\[ \sigma = \frac{P}{A_o}, \]

where \(P\) shows the axial tensile loading, \(A_o\) is the original cross-section area of the sample.

\[ \varepsilon = \frac{L_f - L_i}{L_i}, \]

where \(L_i\) is the original length of the specimen, \(L_f\) is the final length of the sample. In the elastic region, stress versus strain curve follows the Hook’s law, and slope of the curve represents Young’s modulus. Young’s modulus is calculated using equation (3) to measure the stiffness of unaged and aged samples,

\[ Y = \frac{\sigma}{\varepsilon} \text{(MPa)}, \]

2.4.2. Digital image correlation (DIC) method

DIC is a non-contact optical based method which uses images to acquire different strain distribution at different points on the surface of materials under test. In this method, images are captured during loading condition and later these images are processed in VIC-2D software, to obtain strain distribution at different points. Prior to the test, samples are coated with white spray and after drying, random black speckle pattern are created. While processing the images, reference image has been set, and the correlation function in DIC trace the position of speckle pattern of the reference image in the subsequent images. A region with different pixels called subset has been defined, and the reference subset is correlated with deformed one to obtain surface displacement of speckles and further strain distribution is obtained. The accuracy of surface displacement depends on the maximum correlation between the subset of the image in the reference and deformed state. Figure 2 shows the schematic diagram of DIC setup. In the present study, displacement in speckle pattern was recorded by a Grasshopper CCD camera (with Sony ICX625 2/3’ sensor) of 5.0 megapixel with a maximum resolution of 2448 × 2048 pixels placed at 50 cm in front of the sample gripped between the cross heads of universal testing machine. The focal length of the lens (Canon) used with the camera was 50 mm. A light source was focussed on the sample under the test in order to brighten the speckle pattern. The sample images captured by the camera during loading condition was continuously recorded on the computer using Grasshopper GRAS-50S5 IEEE-1394b graphical user interface. The optimized subset of 29 and step size of 5 was used for performing the DIC analysis.
3. Results and discussion

3.1. Water diffusion studies

Figure 3 shows the variation in % weight gain of epoxy nanocomposites due to water ageing at 90 °C. It was observed that water diffusion was high with epoxy resin material and was minimum with 5 wt% alumina added epoxy nanocomposites. Addition of alumina nanoparticles suppressed the water uptake in nanocomposites and water uptake decreased with the increase in wt% of alumina nanofiller. Subjecting the polymeric specimens to water ageing at high temperatures is one of the established methods to simulate the accelerated ageing or long-term consequences of water uptake phenomenon [13]. The similar characteristics were observed by Sarathi et al with epoxy clay nanocomposites and the reason for the water uptake suppression was the increment in hydrophobicity of samples on addition of nanofiller [14]. Water diffusion coefficient (D) was calculated as [15].

\[ D = \frac{\pi L_{0.5}^2}{64t_{0.5}} \]  

(4)

where \( L_{0.5} \) is the thickness of the specimen and \( t_{0.5} \) is the time of absorption. Table 1 shows the diffusion coefficient for all the samples and reduction in diffusion coefficient value was observed on incorporation of alumina nanoparticles.

3.2. Surface potential measurement and trap density analysis

Figure 4 shows the surface potential decay characteristics of the unaged and UV irradiated epoxy nanocomposites. The surface potential variation with time for alumina filled epoxy nanocomposites can be approximated as,
where $V_0$ is the initial surface voltage for the samples, $\lambda$ is a decay rate. The decay rate for the different compositions of alumina filled epoxy nanocomposites with UV aged specimen is shown in table 2. Decay rate has increased with the increase in wt% of alumina nanofiller in epoxy nanocomposites. The decay rate was high under positive DC voltage. Similar characteristics were observed by Du et al with epoxy titania nanocomposites.

$$V(t) = V_0e^{-\lambda t}, \quad (5)$$
It was observed that UV ageing (table 2) has reduced the decay rate. This reduction could be due to chain scission, radical formation or due to occurrence of cross-linking upon exposure to UV irradiation [17].

Figure 5 shows the surface potential variation with the water aged epoxy alumina nanocomposites and the decay rate is shown in table 3. It was observed that the decay rate decreased with the water aged samples compared to unaged samples. The cause could be due to diffusion of water into the bulk volume of insulation and can occupy the intermolecular spaces causing charge trap site thereby leading a reduction in decay rate [18].

According to Simmons and Tam’s theory, trap energy density, $N(E)$ can be estimated from a surface potential decay process as [19]

$$N(E) = \frac{2eV}{qE_l(E)t} \frac{dV}{dt},$$

where $q$ is the electron charge, $L$ is thickness of sample, $k$ is Boltzmann constant, $T$ is absolute temperature, $t$ is time, and $l_0(E)$ is occupancy rate of initial electrons. The trap energy level $\Delta(E)$ can be expressed as

$$\Delta E = E_C - E_M = kT \ln(\nu t),$$

where $E_C$ is conduction band energy and $E_M$ trap level energy, $k$ is the Boltzmann constant, $T$ is the thermodynamic temperature, and $\nu$ is the attempt to escape frequency in order of $10^{12} \text{ s}^{-1}$.

Figure 6 shows the variation in trap energy level of the unaged, and UV/ water aged epoxy alumina nanocomposites. It was observed that, with the increase in alumina content, trap energy level decreased and with the increase in number of UV ageing hours, trap energy level increased (table 2). Trap energy level corresponds to number deep traps. Higher trap energy level represents high number of deep traps which obstruct the mobility of charge carrier leading to decrease in surface potential decay. With UV aged sample right shift in the peak of trap characteristics was observed. This right shift indicates the increase in the threshold energy for charge de-trapping due to which surface potential decay rate has decreased.

In case of water aged samples, increment in the trap energy level (table 3) was observed as compared to unaged samples. This increment in trap energy leads to slow decay of surface potential. Even though a marginal variation in trap depth was observed with UV aged and water aged samples, which indicated a marginal increase

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**Table 3.** Variation of decay rate and trap depth of water aged alumina filled epoxy nanocomposite under DC voltage.

| Sample wt\% | + DC Decay rate ($\lambda$) | Trap depth (eV) | - DC Decay rate ($\lambda$) | Trap depth (eV) |
|-------------|-----------------------------|-----------------|-----------------------------|-----------------|
| 0           | 0.003198                    | 0.8574          | 0.00298                    | 0.8603          |
| 1           | 0.003507                    | 0.8545          | 0.003207                   | 0.8588          |
| 3           | 0.003775                    | 0.8541          | 0.003475                   | 0.8569          |
| 5           | 0.004186                    | 0.8488          | 0.003986                   | 0.8524          |
in deep trap formation. Thus, in general, it was observed that the surface potential decay rate and the trap depth show inverse correlation.

3.3. Mechanical analysis

3.3.1. Tensile testing

Figure 7 shows the stress-strain curve at different loadings (figure 7(a)) and the variation in strain distribution with respect to time in epoxy alumina nanocomposites. Pure epoxy sample showed the lowest tensile strength at the lowest strain. The addition of alumina nanofiller enhances the tensile stress-strain behaviour of epoxy polymer matrix. Addition of nanoparticles increases the stiffness of nanocomposites thereby Ultimate tensile
strength ($S_u$) has increased with the increase in wt% of alumina nanofiller. Epoxy nanocomposite with 5 wt% of alumina nanofiller shows highest tensile strength, as can be observed in figure 7(a).

Table 4 represents the tensile parameters extracted from the stress-strain curve of virgin epoxy nanocomposites, UV aged specimen and for water aged specimen. It can be observed that epoxy-alumina nanocomposites shows higher Youngs modulus value, peak load that it can withstand and ultimate tensile strength. Enhancement in Youngs modulus of nanocomposites on increasing the wt% of nanofiller can be attributed to restriction in mobility of polymer chain under loading condition [20], and also strong interfacial adhesion of nanoparticles allows equal and efficient load distribution in the samples. Similarly, failure strain increased for the higher wt% of alumina filled UV aged epoxy nanocomposites. This increment was due to the strong interfacial adhesion between the nanoparticles, base epoxy matrix, and the newly formed radicals and crosslinked bonds due to UV irradiation. For water aged specimen, the stiffness was decreased, which could be due to the loosening of polymer bonds by water ageing. Failure strain also have decreased for the water aged epoxy-alumina nanocomposites.

Figure 7(b) shows the strain distribution of pure epoxy and alumina-epoxy nanocomposites as a function of time. It is observed that maximum % strain corresponds to $S_u$ (failure strain) increased, and also breakage time under same loading condition increases on addition of alumina nanoparticles to base epoxy resin. The possible cause for enhancement could be due to strong adhesive bond between the alumina nanoparticles and base epoxy resins and effective load distribution between base matrix and nanofiller.

Figure 8 shows the stress strain variation at 50 °C and corresponding tensile parameters at different temperature were given in table 5. At higher temperature, gradual reduction in Youngs modulus and increment in failure strain for all samples are observed. This reduction in the stiffness of the samples can be attributed to the significant reduction in interfacial bonding between nanofiller and epoxy matrix, and due to the distortion in the cross-linked structure because of the vibrational motion of the polymer molecules at higher temperatures [21]. At 50 °C, epoxy-alumina nanocomposites exhibit ductile behaviour with yielding, entering into the plastic
region of the stress-strain curve, and drastic reduction in tensile strength is observed. At higher temperature of 40 °C and 50 °C, drastic enhancement in the failure strain was observed.

3.3.2. Digital image correlation (DIC) analysis

Figure 9 shows the typical strain distribution contours at different instances for the alumina filled epoxy nanocomposites. Images were taken at different time during the tensile test. Deformation in speckles pattern of the images were continuously monitored and were correlated with the first reference image to obtain strain distribution of the sample. It was observed that over the time, maximum longitudinal strain field was concentrated at the breakage point of sample (circled in the figure 9).

Influence of alumina nanofiller on the strain distribution was carried out using DIC technique. Figure 10 shows the variation in % strain distribution at breakage point for pure epoxy and alumina-epoxy nanocomposites. Corresponding failure time and maximum strain at breakage point is shown in table 6. A clear deviation in strain distribution was observed, measured from DIC technique as compared to conventional tensile testing. It was observed that time taken for sample failure was more for the alumina-epoxy nanocomposites as compared to pure epoxy, and 5 wt% sample has shown the highest failure time. Addition of nanoparticles increased the interfacial adhesion and restricted the polymer bond for any deformation, therefore
increment in failure time was observed for higher wt% of nanofiller sample. Similar variation was observed with the conventional tensile testing as shown in figure 7.

Strain distribution was calculated using DIC technique for 300 hours UV aged samples as well as water aged samples. The strain contour plots of UV aged and water aged samples were analysed and it was observed that sample broke at the point with maximum concentrated stress. Corresponding failure time and maximum strain at breakage point was given in table 6. Increment in failure time was observed for 3 wt% and 5 wt% UV aged sample as compared to unaged one. In case of water aged samples, water diffusion into the bulk of samples weakens the adhesive bond between the matrix and nanoparticles, as a result decrement in the failure time was observed for water aged sample. 1 wt% shows the opposite nature and showed higher failure time compared to unaged sample. Similar behaviour for both UV aged and water aged samples was observed during the conventional tensile testing method.

Strain distribution analysis was carried out at different temperatures, using DIC technique (table 6). Strain distribution for all the samples has increased till breakage of sample with the increase in application of load. Strain distribution is higher for the higher wt% of alumina filled epoxy nanocomposites at 50 °C. Addition of nanofiller increased the failure time when subjected to uniform loading at higher temperature. In general, irrespective of temperature, the range at which the strain measurements were carried out adopting DIC, it clearly indicates that increase in wt% of nano filler enhances the strain of the material.

3.3.3. Electromechanical behaviour

The strain distribution analysis using DIC technique indicates that addition of alumina nanofiller increased the tensile strength and reduced the time of failure of epoxy-alumina nanocomposites. Likewise, in case of surface potential decay, decay rate increased with the increase in wt% of alumina nanofiller and this increment can be attributed to decrement in trap energy level leading to detrapping of charge carriers easily. Thus, correlating strain distribution and decay rate of surface potential, it shows direct correlation. Also the strain distribution and trap depth shows inverse correlation. Thus surface potential decay measurement can be used as non-destructive diagnostic tool to understand the strain behaviour of the material due to ageing of material under mechanical stress.

4. Conclusions

The important conclusions accrued based on the present study are the following:

- Diffusion of water into epoxy alumina nanocomposite is less compared with pure epoxy resin.

| Sample | Failure time (s) | % Strain corresponds to Su |
|--------|------------------|---------------------------|
| wt%    | Unaged sample    | 300 hours UV aged | Water aged | 40 °C | 50 °C | 40 °C | 50 °C | 40 °C | 50 °C |
| 0      | 67.03            | 65.59                  | 59.5       | 80.5  | 147.4 | 1.521 | 1.852 | 1.706 | 1.206 | 1.879 |
| 1      | 78.2             | 50.78                  | 84.9       | 105.9 | 169.5 | 2.162 | 2.3   | 1.29  | 1.754 | 1.776 |
| 3      | 70.38            | 73.24                  | 60.1       | 93.84 | 147.4 | 1.691 | 2.388 | 1.74  | 1.558 | 4.06  |
| 5      | 79.11            | 101.2                  | 66.25      | 95.81 | 194   | 1.6371| 2.932 | 1.762 | 1.457 | 7.123 |

Table 5. Variation in tensile parameters for tensile test conducted at 40 °C and 50 °C.

| Sample | Young's modulus (GPa) | Peak load (N) | Ultimate tensile strength, Su (MPa) | % Strain corresponds to Su |
|--------|------------------------|--------------|------------------------------------|---------------------------|
| wt%    | 40 °C                  | 50 °C        | 40 °C                              | 50 °C                     | 40 °C | 50 °C |
| 0      | 1.099                  | 0.847        | 382.6                              | 471.61                    | 20.91 | 16.13 | 5.11  | 9.42 |
| 1      | 1.309                  | 1.056        | 649.59                             | 633.94                    | 35.5  | 26.4  | 6.85  | 10.79|
| 3      | 1.715                  | 1.315        | 743.39                             | 634.95                    | 40.62 | 27.26 | 6.08  | 9.38 |
| 5      | 2.091                  | 1.874        | 906.35                             | 869.31                    | 49.53 | 37.52 | 6.11  | 12.21|

Table 6. Variation in failure time and maximum strain distribution at breakage point.
• Surface potential measurement showed the decrement in decay rate for the UV and water aged nanocomposites compared to unaged samples. Addition of alumina nanofiller increased the decay rate and trap distribution characteristics showed left shift in the peak indicating formation of shallower traps.

• Incorporation of alumina nanoparticles to epoxy resin increased the tensile strength and stiffness of the samples. Tensile parameters of the nanocomposites showed marginal reduction, at higher temperatures. UV aged samples showed higher tensile strength for 3 wt% and 5 wt% samples. Variation in tensile strength was not significant due to water ageing.

• Surface strain distribution was measured for unaged and UV/water aged nanocomposites using DIC technique and correlated with the conventional method. It is concluded that, sample failure time increased on addition of alumina nanoparticles and, it was higher for 3 wt% and 5 wt% of UV aged sample, and 1 wt% of water aged sample. At higher temperature, incremental increase in failure time was observed.

• Surface strain formed during tensile test and the surface potential decay rate, shows direct correlation. Thus surface potential measurement can be used as a diagnostic tool to understand the strain behaviour of the material due to ageing of material under mechanical stress.

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