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Understanding the dielectric properties of EPOXY molybdenum disulfide nanocomposites

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An attempt has been made to understand the surface discharge resistance of epoxy molybdenum disulphide nanocomposite material under AC and DC voltages. To characterize the material, surface charge accumulation studies and basic dielectric properties of the material viz., permittivity and tan(□) of the material were carried out. Also the level of degradation of insulating material characterized through contact angle measurement and by surface roughness measurements. The results of the study conclude that 0.5 wt% of epoxy nanocomposites showed high discharge resistance.

Keyword *: Nanocomposite, Surface Discharge, MoS_2, surface roughness*

1.0 INTRODUCTION

EPOXY resin is basically a high performance material and is used as an insulant in all high voltage power apparatus because of its high electrical, thermal and mechanical properties. The epoxy resin is one of the best structural materials and more than that, it is one of the best insulant. In addition, epoxy resin is effectively used because of its cost-effectiveness. One of the major requirement with electrical insulating material is improvement of its withstand capability. In addition, the bulk volume of insulting material should not have any localized electric field enhancement. To achieve this, in recent times inorganic fillers like silicon carbide is used. It is well established now the Molybdenum disulphide material, which is inorganic filler material, its performance on electrical, thermal and mechanical properties is studied.

During operation of electrical machine, under normal operating voltage, local electric field concentration can occur and initiate corona activity, surface discharge activity or discharges in voids etc. These discharges can cause local degradation causing carbonization and lose its insulating property. Hence it is essential to develop an insulating material, which has high discharge resistance with required dielectric properties. In recent times, polymer nanodielectric materials have been studied for different insulating properties. It is reported that there is great improvement in dielectric properties of the material with electrical discharge resistance properties [1]. Kozako *et al* [2, 3] reported that electrical discharge resistance of polyamide layered silicate nanocomposites improved greatly compared with the unfilled polyamide and the micro composites.

Epoxy resin with silicon carbide reinforcement exhibits good partial discharge resistance characteristics [4]. In power apparatus, it is used as stress grading material [5, 6]. A number of studies have reported the use of non-linear dielectrics in power apparatus operating at normal ambient temperatures [7]. Sarathi *et al.* studied surface discharge activity with epoxy clay nanocomposites and observed that injected current pulses have nanosecond rise time exciting signals in the UHF signal range [8].

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Having known all this, In the present work, the following investigations are made to understand the following important aspects such as (i) Variation in Surface Discharge Inception Voltage (SDIV) in epoxy MoS_2 nanocomposites material under AC and DC voltages, (ii) Analysis of the damage caused due to surface discharge through measurement of surface roughness, and contact angle (iii) surface charge accumulation level with epoxy nanocomposites (iv) Variation in permittivity and tan(δ) of the epoxy molybdenum disulphide nanocomposites.

2.0 EXPERIMENTAL STUDIES

2.1 Sample Preparation

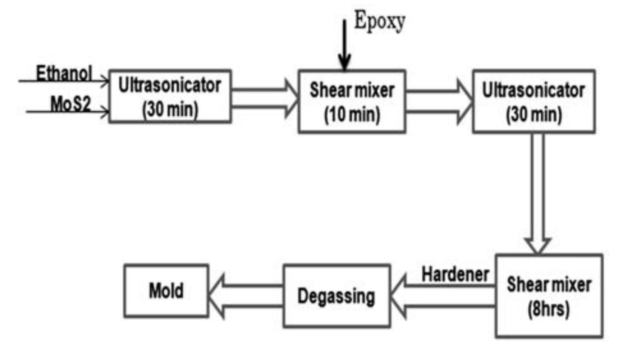

FIG 1. SCHEMATIC REPRESENTATION OF SAMPLE PREPARATION PROCEDURE

In this study, bisphenol-A epoxy resin (DGEBA, CY205, Ciba Geigy Inc.) and tri-ethylene tetra-amine (TETA) hardener were used. Molybdenum disulfide nano filler was purchased from loba chemie. To get the improved properties homogenous dispersion of filler in the matrix is necessary. To achieve uniform dispersion of the nano particles, as a first step, required quantity of MoS_2 was mixed with ethanol and sonicated for 30min. Then epoxy resin and the nanofiller were mixed thoroughly at first stage by using high shear mixer at 700 rpm for 10 minutes and then by using ultrasonic mixer for 30 minutes. The ultrasonic mixer operates at 20 kHz.After thatultra-sonicationshear mixing was carried out for 8 Hrs. On completion, required quantity of hardener was added and the mixture was degassed and cast into mould ofrequireddimensions. The schematicrepresentation is shown in Figure1.

2.2 Surface Discharge Inception Voltage (SDIV)

The experimental setup used for investigating the surface discharge studies of epoxy molybdenum disulphide nanocomposites is shown in Figure 2. The IEC (b)electrode system was used for generating surface discharges and the experiment was conducted in a medium of air under AC and

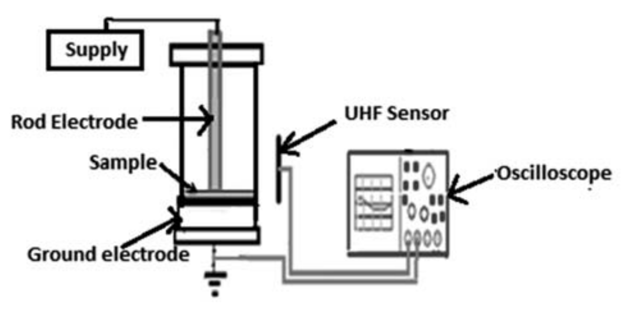

FIG 2. EXPERIMENTAL SETUP FOR SDIV

DC voltages. The epoxy nanocomposite sheet of size 30 mm x 30 mm x 1 mm was used as specimen. The broadband UHF sensors used for the present study is placed at a distance of 20 cm away from the test specimen to acquire UHF signals radiated during surface discharge process.

2.3 Surface Charge Measurement

Figure 3 shows typical experimental setup used for measurement of surface charges. Electro Static Voltmeter (ESV) is used for measuring the surface charge accumulation on epoxy/MoS_2 composite. The charges were deposited on surface by corona discharge maintaining the DC voltage at 6 kV for 5 minutes. The sensor was placed 2 mm away from the material, which can measure the charge deposited on material. The charge deposited on the surface was measured using electrostatic voltmeter as,

$$Q = V \frac{\varepsilon_0 \varepsilon_r A}{d} \quad ...(1)$$

Where, ε_o and ε_r are the permittivity of the vacuum and the medium respectively, A is the Area of cross section of sensor,d is the distance between sensor and the sample surface and V is the voltage measured by ESV.

In general, the charge decay process can be quantified as,

$$Q(t) = Q_0 \, e^{\lambda t} \quad ...(2)$$

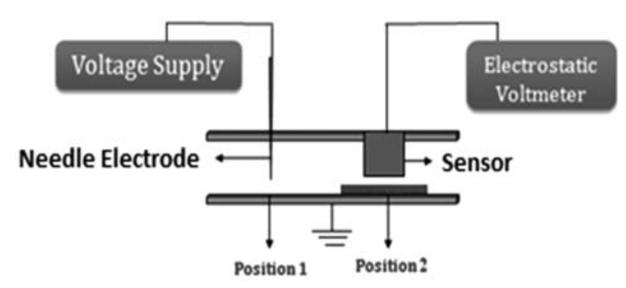

FIG. 3. EXPERIMENTAL SETUP FOR CHARGE MEASUREMENT

2.4 Contact Angle And Surface Roughness Measurement

Hydrophobicity nature of material can be quantified by measuring the contact angle. The static contact angle was measured by liquid droplet method [9]. The liquid volume of water used for the study is about 20 µl. The contact angle was measured as

$$\theta = 2\tan^{-1}\frac{2h}{d}$$

Where, d is the diameter of liquid drop and h is the vertical height of liquid drop. For each specimen, the contact angle was measured at six different locations and averaged.Surface roughness was measured through laser surface profilometer. For each sample, surface roughness was measured at five positions nearelectrically damaged area and averaged.

2.5 Dielectric Response Spectroscopy

The dielectric constant and tan(δ) of the epoxy molybdenum disulphide composites were measured in the frequency range 0.1Hz to 106Hz by using NOVA CONTROL Impedance Analyzer.

3.0 RESULTS AND DISCUSSIONS

3.1 Surface Discharge Inception Voltage (SDIV)

Figure 4 shows characteristic variation in surface discharge inception voltage with different percentages of MoS_2 in epoxy nanocomposites. The surface discharge inception voltages are defined as the voltage at which the first UHF signal is generated on application of voltage to the test cell. It is observed that, when the wt% of MoS_2 is low in epoxy resin, the surface discharge inception voltage is high but above certain wt% of MoS_2 in epoxy resin, a marginal reduction in inception voltage is observed. Raised inception voltage at lower nanofiller loadings can be attributed to the barrier effect of the nanofillerpresent inthe matrix. Kozako [2] has indicated that the nanoparticles act as barrier and hinder the flow of electrical charge. The formation of conductive path will be redirected along the surface of the nanoparticles as the charge carriers reach the nanoparticles. The possible reason for decrease in surface discharge inception voltage after 0.5% could be the agglomeration of nanoparticles, due to which the interfacial interactions between polymer and nanoparticles will be less, there by initiation of discharge happens at lower

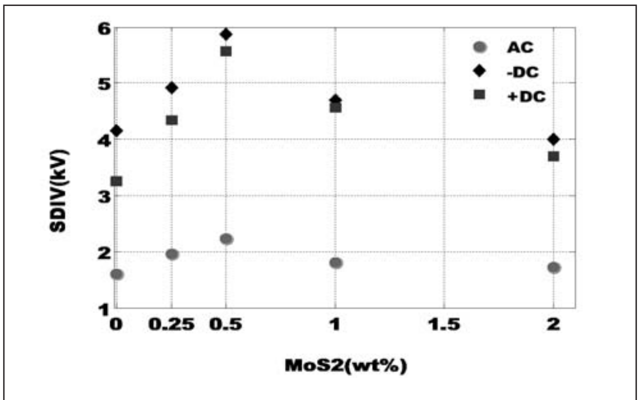

FIG 4. SURFACE DISCHARGE INCEPTION VOLTAGE VARIATIONS WITH RESPECT TO CONCENTRATION OF MOS_2.

voltages. It is observed that SDIV under AC voltage is much lower than the DC inception voltage, irrespective of percentage of MOS_2 added to epoxy resin. The reason could be due tothe charges injected in one half cycles be taken out in the next half cycle under AC voltages, which can aid the discharge process causing reduced discharge voltage.Under DC voltages, charge accumulates on surface of the insulating material. The electric field formed on the surface of the insulting material opposes the applied electric field thereby enhancing the surface discharge inception voltage.

3.2 Surface Charge Measurement

Figure 5 shows charge decay of epoxy molybdenum disulphide nanocomposites. Charge accumulated on the surface of epoxy nanocomposite is measured using electrostatic voltmeter and is observed that charge retention and magnitude of charge on surface is decreasing as the concentration of MoS_2 is increasing. It is mainly because that the MoS_2 has large electro negativity, due to which it captures charges and forms a shield. The charges captured in this shielding layer generate an internal field opposite to the applied electric field,which decreases effective electric field strength and further charge are kept from injecting into the bulk.

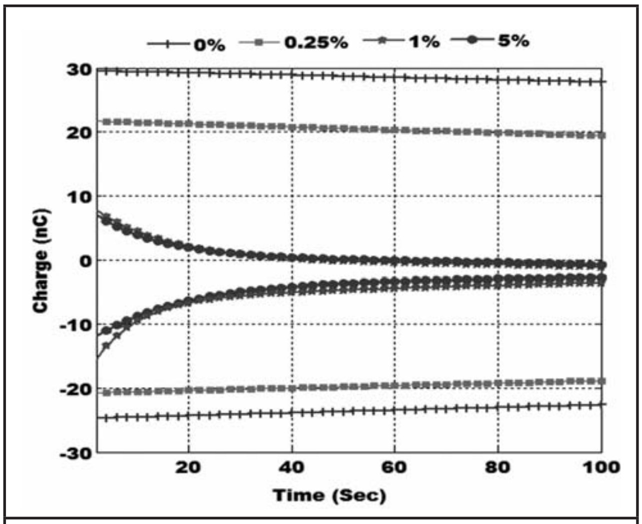

FIG 5. RELATIONSHIP BETWEEN SURFACE CHARGE AND DECAY TIME WITH INCREASE IN CONCENTRATION OF FILLER.

3.3 Surface Roughness

The surface roughness measured near the damage caused due to surface discharge activity is the value of average surface roughness (Ra). Before subjecting to discharge, surface roughness will remain same almost for all specimens. The discharge exposed composites shows decrement in surface roughness with increasing in wt% of MoS_2. This clearly indicates when the weight percentage of nano particle is increased, it improves the discharge resistance of the material This could be due to improvement in boding strength of the nano particle with the base resin. The variation in surface roughness with MoS_2 is shown in Figure 6.

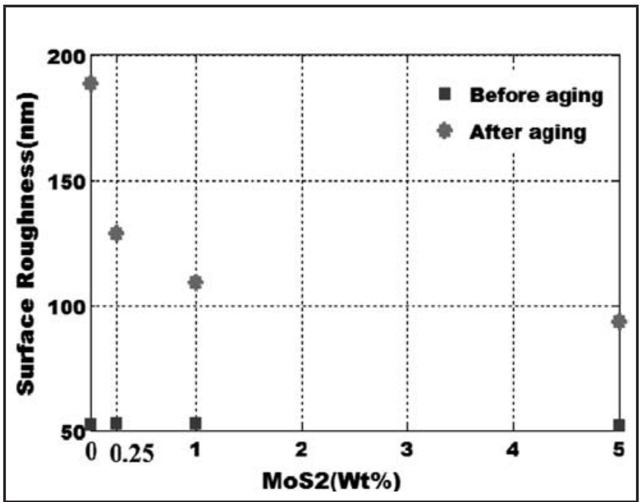

FIG 6. VARIATION OF SURFACE ROUGHNESS WITH RESPECT TO CONCENTRATION OF MOS_2

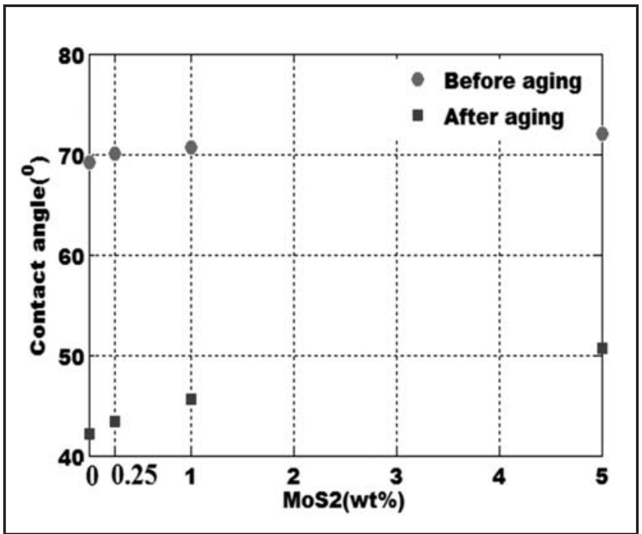

FIG 7. CONTACT ANGLE VARIATION WITH RESPECT TO CONCENTRATION OF MOS_2

3.4 Contact Angle Variation Of Epoxy Nanocomposites

The measure of contact angle of a water droplet is an indirectmeasure of hydrophobicity of the material. Figure 7 shows the variations in contact angle before and after subjecting sample to electrical aging. It is observed that a contact angle drastically reduces for the neat epoxy material and the reduction level is low with nanocomposites.

3.5 Dielectric Properties of Epoxy Nanocomposites

3.5.1 Permittivity

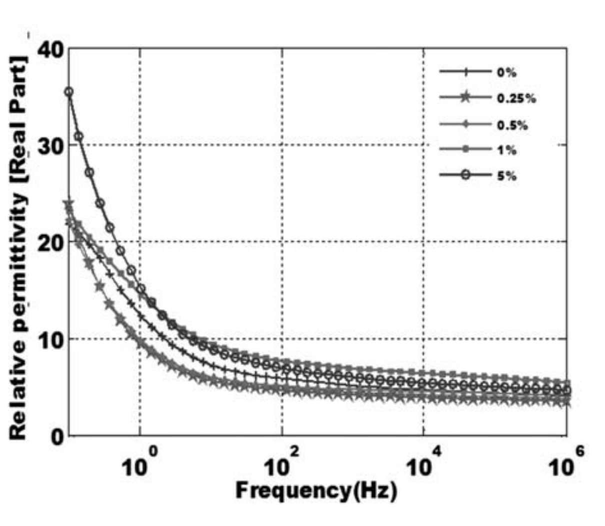

FIG. 8. DIELECTRIC CONSTANT VARIATION WITH RESPECT TO CONCENTRATION OF MOS_2AT 900C.

In general, the Inorganic nano fillers have high permittivity. When it is added with the base resin material, the effective permittivity of the material can be tuned to the required value. Also when percentage of nano filler is increased, the chances for reduction in permittivity of the material at low filler loadings [10,11]. Figure 8 shows variation in permittivity of the material with different frequencies. Permittivity of epoxy/MoS_2 nanocomposite is less than the unfilled epoxy up to 05wt%, after which marginal increase in permittivity is observed. The cause for reduction in permittivity could be the formation of strong interaction of nanoparticles with the polymer chain causing reduction in chain mobility. At higher filler loadings, increment in permittivity can be attributed to influence of filler permittivity. In short, characteristic variation in permittivity with the level of loading of nano fillers is observed.

3.5.2 Tan Delta

It can be observed from Figure 9, Nanocomposites show lower tan (δ) values than that of unfilled epoxy up to 100 Hz. The reduction in tan delta values can be attributed the reduction in conductivity of the composites. Polymer chains forming strong bonds with nanocomposites could be reason for reduction in conductivity. The cause for reduction in tan(δ) further need to be analyzed at different temperature and frequency of supply voltage.

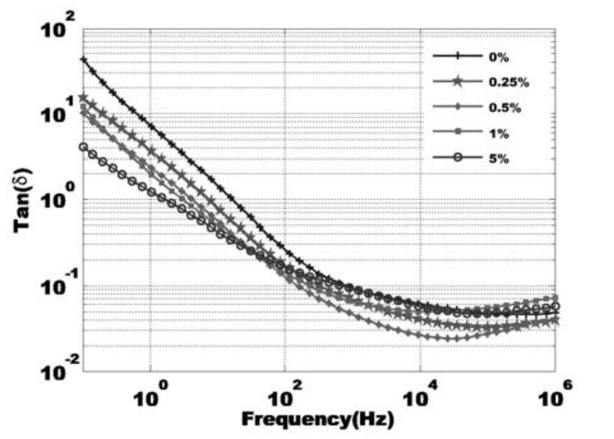

FIG. 9. TAN DELTA VARIATION WITH RESPECT TO CONCENTRATION OF MOS_2 AT 900C.

4.0 CONCLUSIONS

The important conclusions acquired based on the present study are the following.

- It is observed that surface discharge inception voltage of epoxy MOS_2 nanocomposite is high under DC voltage compared to AC voltages. It is also observed that SDIV increases up to 0.5wt% of MOS_2 added to epoxy resin and above which a marginal reduction in SDIV is observed.
- Surface charge accumulation is less with epoxy nanocomposite compared with epoxy resin. Increase in percentage of MOS_2, complete reduction in charge retention time occurs.
- Surface roughness study indicates that the erosion due to surface discharges is less with epoxy nanocomposites. Above certain

weight percentage of MOS_2 content, the surface roughness is almost same.

- Contact angle of Epoxy nanocomposites marginally increases with increase of weight percentage of molybdenum disulphide.
- Dielectric constant of epoxy nanocomposites reduces with increase in frequency of the supply voltage. Also increase in weight percentage of MOS_2 increases the dielectric constant of the material after 0.5wt% of MoS_2.

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