STUDY ON ELECTRO-THERMAL AGEING CHARACTERISTICS OF EPOXY ALUMINA NANO-COMPOSITE INSULATION USED IN HIGH VOLTAGE EQUIPMENTS

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by

CHANDAN MAHATO

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Under the guidance of

Prof. KESAB BHATTACHARYA

and

Dr. ARPAN KUMAR PRADHAN

DEPARTMENT OF ELECTRICAL ENGINEERING
FACULTY OF ENGINEERING AND TECHNOLOGY
JADAVPUR UNIVERSITY
KOLKATA - 700032

2022

JADAVPUR UNIVERSITY

KOLKATA- 700032, INDIA

FACULTY OF ENGINEERING AND TECHNOLOGY

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KBhattacharya

Prof. Kesab Bhattacharya
Professor,
Dept. of Electrical Engineering,
Jadavpur University
Kolkata – 700032

Professor
Electrical Engineering Department
JADAVPUR UNIVERSITY
Kolkata - 700 032

Dr. Arpan Kumar Pradhan
Assistant Professor,
Dept. of Electrical Engineering,
Jadavpur University
Kolkata – 700032

Dr. Arpan Kr. Pradhan
Assistant Professor,
Electrical Engineering Department
Jadavpur University
Kolkata-700032

Saswati Mazumdar 28/6/22

Prof. Saswati Mazumdar
Head, Dept. of Electrical Engineering
Faculty of Engineering and Technology
Jadavpur University
Kolkata – 700032

Head
Electrical Engineering Department
JADAVPUR UNIVERSITY
Kolkata - 700 032

28/6/2022

Prof. Chandan Mazumdar
Dean
Faculty of Engineering and Technology
Jadavpur University
Kolkata – 700032

DEAN
Faculty of Engineering & Technology
JADAVPUR UNIVERSITY
KOLKATA-700 032

JADAVPUR UNIVERSITY

KOLKATA- 700032, INDIA

FACULTY OF ENGINEERING AND TECHNOLOGY

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Name : **CHANDAN MAHATO**

Class Roll no. : **002010802020**

Registration no. : **154010 of 2020-2021**

Name of the thesis : **STUDY ON ELECTRO-THERMAL AGEING CHARACTERISTICS OF EPOXY ALUMINA NANO-COMPOSITE INSULATION USED IN HIGH VOLTAGE EQUIPMENTS**

Chandan Mahato
28/06/2022
....

Signature with date:

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CONTENTS

ABSTRACT

In this work, an experimental investigation is carried out on pure epoxy resin and epoxy with alumina (Al_2O_3) nano-composite. The main objective of this work is to estimate the ageing status of the epoxy based nano-composites insulation. Effects of ageing, the condition of the insulation degrades. Activation energy is a parameter which changes with the degradation of the insulation. Therefore, activation energy can be a good ageing sensitive marker. Hence, the ageing state identification of the epoxy based insulation are performed based on the activation energy. Initially, the epoxy and epoxy-alumina nano-composites samples are prepared. After that, the prepared samples are allowed for electro-thermal stress upto 600 hours in step of 100 hours duration. After every 100 hours instant the electrical measurement (polarization and depolarization current) is performed at different ambient temperatures (30^oC, 40^oC, 50^oC, 60^oC, 70^oC and 80^oC). After that, the conductivity of the test insulation samples has been estimated from the PDC. The activation energy has been calculated from the conductivity versus temperature plot. Based on the activation energy, an empirical relationship has been established for estimation of the ageing state of the epoxy based nano-composites insulations. Finally, the proposed relationship has been verified with the newly prepared test samples. The validation of the proposed technique shows good correlation with the actual ageing state. Therefore, the proposed method can be applicable to estimate the ageing state of the epoxy based insulation of the high voltage equipment.

NOMENCLATURE

D	Electric flux density
E	Electric field
ε_0	Permittivity of vacuum
ε_r	Relative permittivity of an insulating material
P	Polarization vector
χ	Electrical susceptibility of a dielectric material
U_0	Step voltage source
$\delta(t)$	Dirac function/delta function
ε_s	Static relative permittivity
ε_∞	High frequency relative permittivity
χ_s	Static electrical susceptibility
χ_∞	High frequency susceptibility
J	Displacement current density
σ_0	DC conductivity
C_0	Geometric capacitance
$f(t)$	Polarization function
i_{pol}	Polarization current
i_{depol}	Depolarization current
ω	Frequency of applied voltage
$\hat{\chi}(\omega)$	Complex susceptibility of the sample
$\chi'(\omega)$	Real part of complex susceptibility
$\chi''(\omega)$	Imaginary part of complex susceptibility
$C'(\omega)$	Real part of complex capacitance
$C''(\omega)$	Imaginary part of the complex capacitance
ε'_r	Real part of relative permittivity
ε''_r	Imaginary part of relative permittivity
$\tan\delta$	Dielectric dissipation factor

CHAPTER-1

INTRODUCTION

Power transformer becomes an important part of the power system network. It requires regular condition monitoring and maintenance for providing trouble free service. In dry type transformers, solid insulating materials are used as insulation. The condition of these insulating materials are related to life span of power transformer. A good insulating material must have following dielectric properties - like low dielectric dissipation factor (tanδ), high dielectric breakdown strength and very low conductivity. After a certain operating time (a few years) the insulation system of the transformer deteriorates. This degradation occurs due to the temperature, moisture and some chemical particles, present in the environment [1]. Another factor is the ageing of the transformer. Overall degradation of insulation leads to the failure of the transformers [2].

The solid insulation costs more than the liquid insulation [3]. Due to the high insulation cost, the cost of the dry-type transformer is more than oil cooled transformer of the same power rating. The main disadvantage of the dry-transformer is that while operating, the temperature of the transformer rises and if the temperature of the transformer exceeds a certain limit, a hotspot gets create in windings of the transformer [4]. The surface of the insulation gets degrade and some tiny voids get create inside the insulation, due to this hotspot. The main cause of partial discharge is the presence of this void, which causes breakdown of the insulation [5]. The insulation system of the transformers is affected by the moisture, present in the air due to wet weather condition [6].

There are so many methods for monitoring and diagnosis of the high voltage equipment. With the help of these methods, the information about the transformer insulation condition can be known. Time domain spectroscopy (TDS) and frequency domain spectroscopy (FDS) are the two methods for analysis the dielectric response of any insulation, employed for high voltage equipment [7]. Polarization and depolarization current (PDC) measurement and recovery voltage measurement (RVM) – these two methods are the part of time domain for measuring dielectric response [8]. Frequency domain spectroscopy (FDS) gives the information about power loss coefficient or dielectric dissipation factor (tanδ) [9].

1.1 BRIEF INTRODUCTION OF DRY-TYPE TRANSFORMER

The dry-type transformer also known as cast resin transformer, is a power transformer which needs less maintenance to provide the services for many years. It can be used for longer time, under normal conditions. If cooling system is considered for the dry-type transformer, no liquid or mineral oil is required rather its core and windings are kept inside a sealed tank with pressurized air [10]. Dry type transformer acts as safe and reliable electrical instrument. The Dry-type transformer core needs CRGO or cast iron for core formation as similar to an ordinary transformer. The dry-type transformer windings are impregnated by resin or varnish under high vacuum pressure [11].

1.1.1 CONSTRUCTION BASED CLASSIFICATION OF DRY-TYPE TRANSFORMERS

Depending on the construction method for manufacturing and working environment, the dry-type transformers are classified into four different types [12] - (a) Open Wound, (b) Vacuum Pressure Impregnated, (c) Vacuum Pressure Encapsulated and (d) Cast Coil.

A brief description of above mentioned four different dry type transformers is given as -

1.1.1.1 OPEN WOUND DRY-TYPE TRANSFORMER

These dry type transformers are constructed with dip and baked method. Conductor coils are heated and then dipped into the varnish under high temperature. Then the coils are baked to cure the varnish [13]. Open wound transformers (shown in figure 1.1) are fire-proof, dustproof and has better overload capacity. It does not emit any hazardous gas into the atmosphere below the temperature of 750 ° C.

Fig. 1.1 Open wound Dry-type Transformer

1.1.1.1.1 SPECIFICATION OF OPEN WOUND TRANSFORMER

Depending on size, voltage rating, application area and capacity of the transformer, it has following specifications -

1. **Capacity:** 50 kVA to 30 MVA
2. **Voltage level:** up to 33 kV
3. **Class of insulation:** F and H
4. **Cooling system:** AN (Air Natural) / ANAF (Air Natural Air Forced) cooling
5. **Impregnation:** Varnish

The second classification is vacuum impregnated dry type transformer.

1.1.1.2 VACUUM PRESSURE IMPREGNATED (VPI) DRY-TYPE TRANSFORMER

This type of transformer is made of components that can bear high temperature to 220^0C [14]. Class H insulating epoxy resin is employed to avoid risk of fire. If any fault occurs in winding, it gives better chance for repairing that portion. VPI type transformers (shown in figure 1.2) are installed in moisture prone areas.

Fig. 1.2 Vacuum Pressure Impregnated Transformers

1.1.1.2.1 SPECIFICATION OF VACUUM PRESSURE IMPREGNATED TRANSFORMER

The specification of this type of transformer has given below -

1. **Capacity:** 5 kVA to 3.5 MVA
2. **Voltage level:** Up to 11 kV
3. **Classes of insulation:** H/C

iv. **Cooling System:** AN (Air Natural) cooling
v. **Impregnation:** Polyester Varnish

The third classification of dry type transformer is vacuum pressure encapsulated transformer. A brief description is given below -

1.1.1.3 VACUUM PRESSURE ENCAPSULATED (VPE) DRY-TYPE TRANSFORMER

It is similar to VPI type transformer. They are manufactured with silicon based resin instead of polyester [15]. This resin makes thick varnish which is resilient to moisture, high humidity. It gives a unique feature to operate even in adverse atmosphere. The normal withstand temperature of silicon varnish insulation of VPE transformers (as shown in figure 1.3) is in the range of 220 °C to 250 °C.

Fig. 1.3 Vacuum Pressure Encapsulated Transformer

1.1.1.3.1 SPECIFICATION OF VACUUM PRESSURE ENCAPSULATED TRANSFORMER

According to manufacturing process, service areas, capacity, this type of transformer has following specification -

i. **Capacity:** 5 kVA to 30 MVA
ii. **Voltage level:** 33 kV
iii. **Classes of insulation**: F and H
iv. **Cooling System:** AN (Air Natural) / AF (Air Forced) cooling
v. **Impregnation:** Silicone Varnish

The fourth classification is cast coil dry type transformer. Brief description about this transformer is given below -

1.1.1.4 CAST COIL DRY-TYPE TRANSFORMER

the primary and secondary windings are completely encapsulated with epoxy resin to prevent moisture from atmosphere [16]. This cast resin provides good protection for adverse condition. Also, the cast coil dry-type transformer (as shown in figure 1.4) produces less noise. The insulation can withstand temperature from 155 ^{0}C (class F) to 180 ^{0}C (class H) [17].

Fig. 1.4 Cast Coil Transformer

1.1.1.4.1 SPECIFICATION OF CAST COIL TRANSFORMER

It has following specifications depending on its application -

i. **Capacity:** 3.5 MVA
ii. **Voltage level:** 6 kV to 33 kV
iii. **Classes of insulation:** F and H
iv. **Cooling System:** AN (Air Natural) / AF (Air Forced) cooling
v. **Impregnation:** Cast Resin Varnish

1.1.2 ADVANTAGES AND LIMITATIONS OF DRY-TYPE TRANSFORMER

Dry type transformer is more advantageous than oil type transformer due to absence of oil in tank, rather it uses solid insulation for winding protection.

1.1.2.1 ADVANTAGES

The advantages of the dry-type transformer are as following

i. Safe operation for people.
ii. Easy to install.
iii. Excellent quality to support overloads.
iv. No fire hazard.
v. Excellent resistance to short circuit currents.
vi. No chemical wastage comes out of transformer tank.

1.1.2.2 LIMITATIONS

Having a lot of advantages, these type of transformers have some limitations also. That is – on the occurrence of fault in oil type transformer, the faulty part will have to be replaced. But in case of Dry-type transformer, the whole system will be replaced. So the operation cost is more for dry type transformer.

1.1.3 APPLICATION OF DRY-TYPE TRANSFORMER

Dry type transformers do not have flammable liquids and air is used as coolant. So it can provide very safe operation. Due to these reasons, it has been used in following areas -

i. Chemical, oil and gas industry.
ii. Environmentally sensitive areas.
iii. Fire-risk areas (i.e. forest zone).
iv. Indoor and underground substation.
v. Renewable power generation (i.e. off shore wind turbine).
vi. High residential areas.

1.2 SCOPE OF THE THESIS

In this thesis the dielectric response of epoxy resin Nano-composite solid insulation has been analyzed. It helps to find out the ageing duration of epoxy resin based solid insulation system of dry type transformer from activation energy. This technique is applicable for on-site transformers also.

1.3 CONTRIBUTION OF THE THESIS

In high voltage laboratory, pure epoxy resin and epoxy resin with 1 wt.% alumina and 2 wt.% alumina Nano composite solid insulations are made manually. Experiments are performed on these samples to fulfil the objective of the thesis.

1.4 THESIS OUTLINE

i. **Chapter 1** provides the information about dry-type transformer, different types of dry-type transformer, the scope and contribution of the thesis.
ii. **Chapter 2** narrates different dry-types insulating samples.
iii. **Chapter 3** describes about condition monitoring techniques of Dry-type transformer.
iv. **Chapter 4** explains time and frequency domain analysis of dielectric response.
v. **Chapter 5** gives information about the experimental procedure.
vi. **Chapter 6** describes. about the results, discussions and validation of the experiment
vii. **Chapter 7** describes the conclusions and future scope.

CHAPTER-2

DIFFERENT DRY-TYPE INSULATING SAMPLES

2.1 BRIEF INTRODUCTION OF TRANSFORMER INSULATION

Insulation is the most important material of transformer that can separate the windings of different voltage ratings and also the core from the metal tank. Insulation system of transformer consist of fluid (in liquid or gaseous form) and solid material [18].

In oil type transformer, the oil (mineral oil, mostly naphthenic) itself works as insulation as well as coolant. On the other side, the solid insulation can be classified into two groups based on their application – major and minor insulation system. Spacers, clamps fall under the major insulation and winding insulation comes in minor insulation [19]. Solid insulation serves mainly three functions – firstly, it can store electrical charge as dielectric medium. Secondly, it provides insulation for transformer equipment. Thirdly, it gives mechanical support to the transformer windings.

Few solid insulating materials and their applications are described below -

2.2 NOMEX PAPER

The materials used for manufacturing the insulation of transformer, Nomex paper is one of them. Nomex is meta aramid fibres made from condensation reaction of m – phenylenediamine and isophthaloyl chloride (as shown in Fig. 2.1) [20]. It provides high dielectric strength, resilience and mechanical toughness [21]. Due to its mechanical strength and electrical properties, nomex is used as insulation in electrical equipment [22]. Operating temperature for nomex is 220^0C. After 220^0C, the electrical and chemical properties get change.

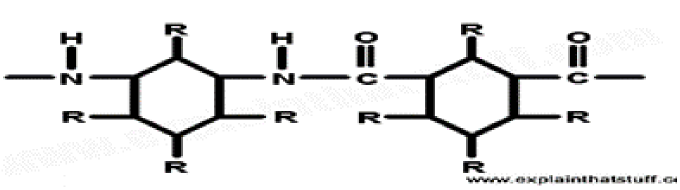

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Fig. 2.1 Chemical structure of Nomex

It has good dielectric strength of 18 kV/mm to 34 kV/mm and dielectric constant is about 1.6 to 4.1.

2.1.1 APPLICATIONS OF NOMEX PAPER

As an insulation, the nomex paper is used in following cases -

i. Nomex paper is used for electrical lamination in transformer core and circuit board.
ii. It is used for insulation purpose while manufacturing the windings of transformer and generator.
iii. Nomex paper is required for the production of hybrid electric vehicles (HEV).
iv. It is used in computers, LCD, mobile phones, Televisions etc.

Another important solid insulation is epoxy resin. Due to possessing of higher mechanical strength and chemical resistance, it becomes the most useful insulation materials.

2.2 EPOXY INSULATION

Epoxy resins are also known as poly-epoxides. It contains epoxide group. The epoxide functional groups are collectively called epoxy [23] . Bisphenol A-based epoxy resins are the most commercially used resin. Another type of resins available in market is of Bisphenol F-based epoxy resins. Fig. 2.2 shows the chemical structure of epoxy resin.

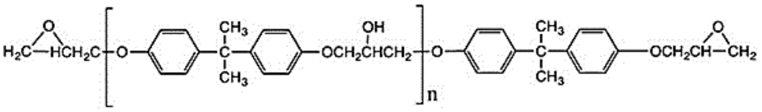

Fig.2.2 Chemical structure of epoxy resins

2.2.1 APPLICATIONS

Epoxy resin shows low shrink while curing and has excellent moisture resistance. Reinforcements of different combination of epoxy resin results in wide range of properties. Due to this, epoxy resins are used in following areas -

i. Epoxy resins are excellent electrical insulation used in motors, generators, transformers, bushing, printed wiring boards and protect electrical equipment from short circuit, dust and moist.
ii. By applying vacuum impregnation to uncured epoxy, winding-to-winding, winding-to-core air voids can be eliminated.
iii. Cured epoxy is better heat conductor than air.

iv. In electronics industry epoxy resins are used in semiconductor encapsulation, transistors and hybrid circuits.
v. Epoxy resin coatings are used as primers to enhance the adhesion on metal surface where corrosion resistance is important. Metal containers are coated with epoxy to prevent from rusting.
vi. In industry to produce lamination and casting, epoxy resins are used.

Another widely used insulating material is low density polyethylene. Its chemical structure, manufacturing process, dielectric strength is briefly described -

2.3 LOW DENSITY POLYETHYLENE

Low density polyethylene (LDPE), has density of 0.915-0.925 g/cm^3, is made of monomer ethylene (shown in Fig. 2.3) through the following way-

$$\mathrm{n}\ [\mathrm{CH_2 = CH_2}]\ (\mathrm{gas}) \rightarrow [-\mathrm{CH_2} - \mathrm{CH_2} -]_\mathrm{n}\ (\mathrm{solid})$$

It operates normally at room temperature. But under the influence of solar radiation, the LDPE decomposes in two gases – Ethylene (C_2H_4) and Methane (CH_4) [15]. For continuous operation, it can withstand temperature of 80^{0}C. Its melting temperature is about 110^{0}C. Aldehydes, vegetable oils have no effect on LDPE. It has tensile strength about 0.20-0.40 N/mm^2 and dielectric strength lies in between 20 kV/cm to 160 kV/cm [16].

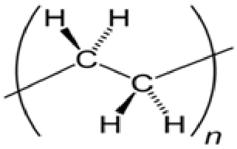

Fig. 2.3 Chemical structure of LDPE

2.3.1 APPLICATIONS

Low density polyethylene is flexible and has low tensile strength. It can withstand a large amount of stretching before breaks. Due to these properties, they are used -

i. for manufacturing of water pipes, cable jacketing.
ii. in computer hardware, screen cards and optical disc drives.
iii. for insulation purpose in wires and cables.

Fibre glass tape is another solid insulation used in household purposes. It serves also as an insulating material.

2.4 FIBRE GLASS TAPE

Fibre glass tape is made of twisted glass filament strands. These strands are woven at right angles, look like fibre glass cloth. It has selvedge edges to save the cloth from continuous unravelling. Fibre glass tape provides water resistance. The image of fibre glass tape has been shown in Fig. 2.4.

Fig. 2.4 Image of fibre glass tape

2.4.1 APPLICATIONS

Fibre glass tape can prevent the crack formation. It has the following applications -

i. Joint fibre glass tape is used in the junction of door and window frames to the wall.
ii. It has dielectric strength of 2 kV/cm, ensures safety from electrical hazards.

2.5 SAMPLES USED IN THIS THESIS

In this thesis, the used samples are – pure epoxy resin, epoxy resin with 1 wt.% alumina nanoparticles, epoxy resin with 2 wt.% alumina nanoparticles. All three samples are in solid while experiment is being carried out.

CHAPTER - 3

CONVENTIONAL DIAGONSTIC TECHNIQUES FOR TRANSFORMER INSULATION

3.1 INTRODUCTION

Transformer has been playing an important role in electrical industry since nineteenth century. The transformer is used for Transmission and Distribution of Electrical Power [25]. The power rating of the transformer varies from few kVA to several of MVA. The replacement of transformer is a cost-effective job. The cost of the transformer sometimes goes up to several million dollars, it depends on the rating of the transformer. So a sudden failure in a running transformer may lead to a huge amount of economic losses. The factors which accelerate the ageing of transformer insulation is moisture, oxygen and heat [26].

Diagnostic techniques are applied to transformer based on physical, chemical and electrical parameters [27]. Physical test includes temperature, vibration test of transformer. But the chemical and electrical tests – these two diagnostic techniques can give useful information about electrical insulation [28].

3.2 ELECTRICAL DIAGNOSTIC TECHNIQUES

When a transformer operates under high electrical and thermal stress, the degradation rate of insulation increases with transformer ageing [29]. Due to this degradation, the surface of the insulation becomes brittle. The mechanical stress occurs due to -

i. thermal expansion and contraction
ii. vibration of transformer

The most commonly used electrical diagnostic techniques are-

i. Insulation Resistance (IR) measurement and Polarization Index (PI) Test
ii. dissipation factor (tan δ) measurement

3.2.1 INSULATION RESISTANCE (IR) AND POLARIZATION INDEX (PI) TEST

Insulation Resistance (IR) is measured to know the condition of the insulation of motors, generators, transformers and switchgear [29]. The measurement of IR must be done very frequently from the new condition of the insulation and always be compared the new values with the previous values of IR. This comparison gives information about the insulation quality like - voltage withstand capability, the breakdown voltage of insulation.

When a high voltage dc source is applied across any insulation system, a high valued current starts flowing through it [30]. This current gradually decreases and finally it reaches to a stable value. The insulation resistance at any instant of time is measured from dividing the applied voltage by the current flowing at any instant of time. Initial current value is high because of high capacitive charging current. But this current reduces and reaches to negligible value with time (within 15 s) due to the voltage developed across the geometric capacitance of insulation reaches to the applied voltage. High initial current is also caused by the high initial dielectric absorption current [24]. This current flows until all the dipoles align themselves in the direction of electric field. This current needs 10 min to few hours to reach very low value. The dipoles present inside the insulation system get polarize by the applied field.

So if megger value is taken for 1 minute, the effect of capacitive charging current on measurement of IR can be eliminated. Similarly, if megger value is taken for 10 minutes, the result will be free from both capacitive charging current and polarising current on IR measurement [24]. Now, the polarization index is the ratio of two measured values of IR taken for 10 minutes and 1 minute respectively.

$$\mathbf{PI} = \frac{\mathbf{R_{10}}}{\mathbf{R_1}} \qquad 3.1$$

Where, R_{10} = Insulation resistance at 10 minute, R_1 = Insulation resistance at 1 minute; If PI value of an insulator is greater than 2, its insulation condition is good. It works as poor if PI is less than 1.5. To measure insulation resistance, following methods are used – (a) time v/s resistance test, (b) stepped voltage test [24].

3.2.1.1 TIME V/S RESISTANCE TEST

Keeping applied voltage fixed, the reading of insulation resistance is recorded successively at specified intervals. If insulation resistance continuously increases with time, it shows good insulation property. It happens so because of the absorption current in good insulation gets decrease with time due to increase in insulation resistance.

3.2.1.2 STEPPED VOLTGE TEST

The applied voltage is being continuously increased at one-minute interval and the corresponding insulation resistance is noted down at every voltage level. Any reduction in resistance while measuring indicates poor insulation quality.

3.2.2 DISSIPATION FACTOR ($tan\delta$) MEASUREMENT

When an alternating electric field is applied across an insulation, a current starts flowing through it. This current has two components - (i) Resistive component and (ii) Capacitive component. Basically, all insulation systems are capacitive in nature [31]. For the analysis purpose, any insulator is modelled as the parallel combination of the resistance ($\mathrm{R_p}$) and capacitor ($\mathrm{C_p}$). Figure 3.1 (a) shows the insulation model used in transformer. From fig. 3.1 (b) the dielectric loss angle (δ) is the angle between the capacitive current (Ic) and the resultant current (I).

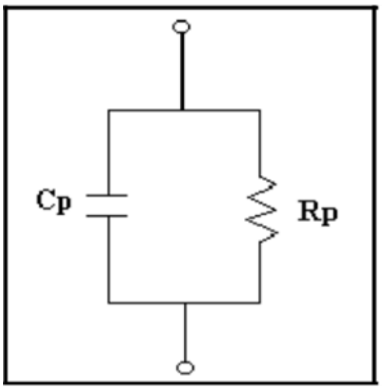

Fig. 3.1 (a) R-C model of Insulation

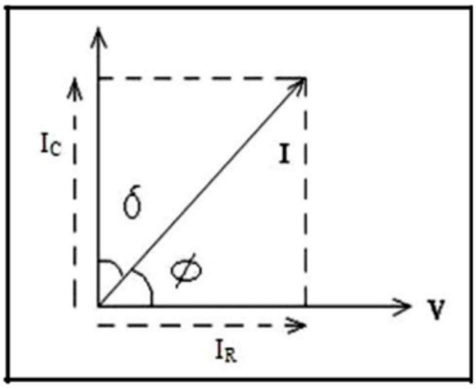

Fig. 3.1 (b) Phasor Diagram

tanδ is dissipation factor and indicates the dielectric loss occurs in insulation system. Complex permittivity of an insulating material is expressed as –

$$\varepsilon = \varepsilon' - j\varepsilon'' \qquad 3.2$$

tanδ is the ratio of imaginary part (ε'') and real part(ε') of complex permittivity. The permittivity and the dissipation factor depends on the molecular size, orientation of dipoles within the insulation [24]. From permittivity or dissipation factor, the quality regarding contamination level of insulation is known. Alternating voltage is applied by varying its range from 10-30 kV in power frequency and tanδ values are recorded. tanδ values and corresponding applied voltages are plotted. If the nature of the graph is steady, the insulation quality is good and If graph is increasing in nature, the insulation gets deteriorate. The increment of resistive component is caused by the ageing of transformer insulation in hygroscopic atmospheric condition. From fig. 3.1 (b)

$$tan\delta = I_R/I_C = \frac{1}{\omega RC} \qquad 3.3$$

Where ω denotes supply frequency in rad/sec, C presents the capacitance of the insulation in farad (F) and R is resistance in ohm. High value of tanδ indicates the high energy loss within insulator. So, for a good insulating material, tanδ value should be low.

3.3 OVERHEATING MONITORING SYSTEM

Many dry type transformers are designed with a PTC (Positive Temperature Coefficient) temperature fuse connected to low voltage coils. It gives signal when temperature increase due to high operating current. Local overheating also takes place in high voltage coils, because partial discharge damages of solid insulation [30]. PTC cannot detect these types of overheating.

The new developed system protects both high and low voltage windings along with the core from the overheating. This overheating monitoring system (shown in Fig. 3.2) consists of a fiber optic sensor and a control unit. An optical transmitter injects a light signal into one side of optic sensor, from other side the optical receiver can detect the signal [31].

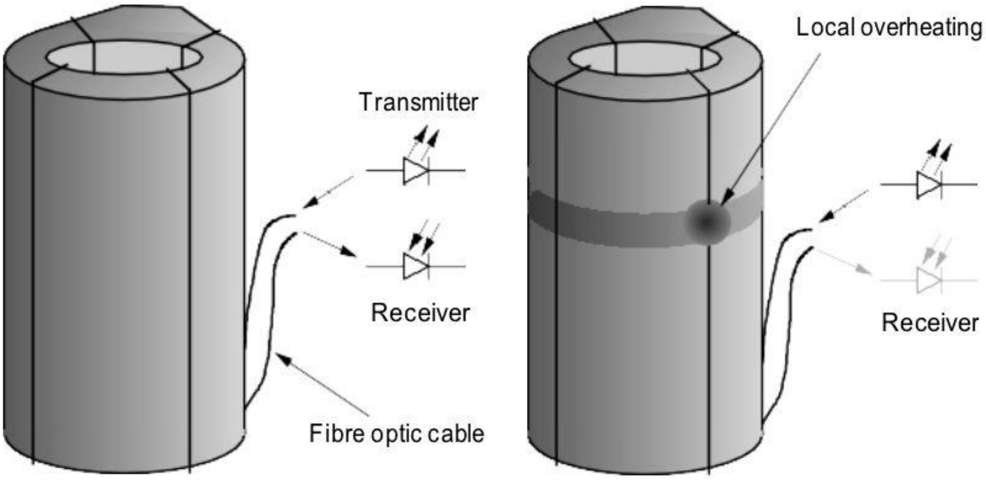

Fig.3.2 Scheme for overheating system

If surface temperature rises and reaches to a certain limit, the light transmission gets interrupted [32]. When such scenario happens, automatically a bell alarms and the transformer is disconnected by monitoring system.

CHAPTER - 4

BASIC THEORY OF DIELECTRIC SPECTROSCOPY

4.1 INTRODUCTION

The demand for transformer is continuously increasing around the world and has resulted in the growing interest on condition assessment and monitoring of transformer insulation [30]. Nowadays for maintenance of a transformer, two distinct maintenance approaches have been adopted. These are time-based maintenance (TBM) and condition-based maintenance (CBM) [31]. Time based maintenance (TBM) is to be performed regularly on calendar schedule and condition based maintenance (CBM) is performed only when the data shows a decline in performance or the warning sign of failure of equipment [32]. Dielectric response is obtained from Time Domain Spectroscopy (TDS) and Frequency Domain Spectroscopy (FDS) [33]. Polarization and Depolarization Current (PDC) and Recovery Voltage (RVM) measurements are the two techniques in Time Domain Spectroscopy [34].

4.2 TIME DOMAIN SPECTROSCOPY (TDS)

It is considered that the used dielectric is homogenous and isotropic. When an external electric field is applied to the dielectric material, polarization process starts. In a vacuum insulated electrode arrangement, the electric displacement vector or the electric flux density D is proportional to electric field vector E,

$$D = \varepsilon_0 E \qquad 4.1$$

If the vacuum is exchanged by an isotropic dielectric medium, the polarization P must be added with the above electric flux density D [31]. The relation will be -

$$D = \varepsilon_0 E + P \qquad 4.2$$

Where,

$$P = \chi \varepsilon_0 E \qquad 4.3$$

Here, χ is the electric susceptibility of the material. A time-varying voltage source generates time varying electrical field, then equation 4.3 can be written as,

$$P(t) = \chi(t) \varepsilon E(t) \qquad 4.4$$

Similarly, the Electric flux density D(t) can be expressed as,

$$D(t) = \varepsilon_0 E(t) + P(t) = \varepsilon_0 \big(1 + \chi(t)\big) E(t) \qquad 4.5$$

The polarization process P(t), within the dielectric medium is the contribution of all polarization mechanisms. The electronic polarization is very fast among all the polarization. As all the polarization processes have finite magnitude, they will settle after a long time and finally become static i.e. $P(t \to \infty) = P_s$ (shown in figure 4.1).

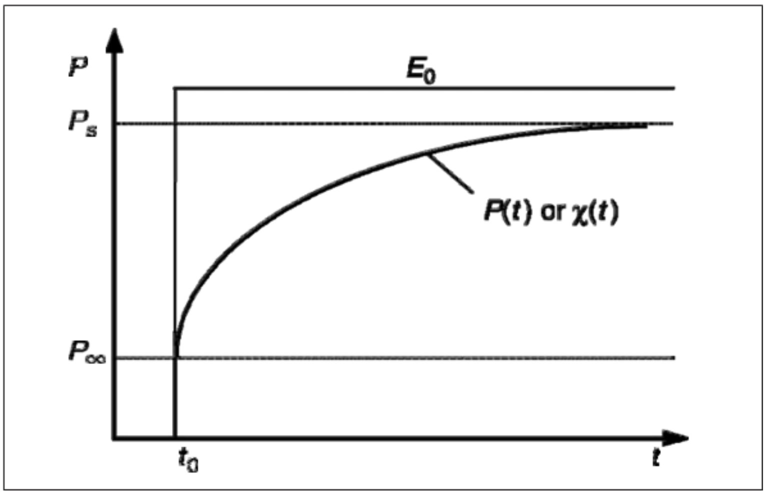

Fig.4.1 Polarization of dielectric material

Electric field E_0 is applied to the material at time t_0 and remains constant for $t \geq t_0$ and the polarization vector P(t) monotonically increasing. This polarization vector P(t) can be expressed as by the following equation,

$$P(t) = (P_S - P_\infty)\, g\,(t - t_0) + P_\infty \delta(t - t_0) \qquad 4.6$$

Where $\delta\ (t\ \text{-}t_0)$ is Dirac delta function. It the fast polarization processes and g(t-t_0) is monotonically increasing function with time, represents the interfacial polarization process. Where, $g(t - t_0) \geq 0, \frac{dg(t-t_0)}{dt} \geq 0\ for\ all\ t_0 < t < \infty$

$$g(t - t_0) = 0\ for\ all\ t \leq t_0$$

$$g(t - t_0) = 1\ when\ t \to \infty \qquad 4.7$$

$$P(t) = \varepsilon_0[\chi_\infty + (\chi_S - \chi_\infty)g(t - t_0)]E(t) \qquad 4.8$$

Since susceptibility is related to the permittivity of the dielectric medium so the equation (4.8) can be expressed as, $P(t) = \varepsilon_0\ [\chi_\infty + (\chi_S - \chi_\infty)\ g\ (t - t_0\ )]E(t)$ 4.9

In general, a material cannot get polarize instantaneously in response to an applied field. The polarization is a convolution of the electric field at previous times with time dependent susceptibility. So this P(t) can be expressed by,

$$P(t) = \varepsilon_0(\varepsilon_\infty - 1)E(t) + \varepsilon_\infty \int_{-\infty}^{t} f(t - \tau)E(\tau)d\tau \qquad 4.10$$

Where *f(t)* is the dielectric response function. It is monotonically decreasing function. It is expressed as, $f(t-\tau) = (\varepsilon_0 - \varepsilon_\infty)\frac{dg(t-\tau)}{dt}$ 4.11

Therefore, under an electric field E(t) total current density J(t) in the dielectric medium can be expressed as,

$$J(t) = \sigma_0 E(t) + \varepsilon_0 \varepsilon_\infty \frac{\partial E(t)}{\partial t} + \frac{\partial P(t)}{\partial t} \qquad 4.12$$

Where σ_0 is dc conductivity of the dielectric medium, first term presents conduction current, second term is vacuum displacement and the third term is polarization displacement current, ε_0 is permittivity of free space [31]. For a homogenous and isotropic medium, the total current through the dielectric medium can be written as,

$$i(t) = C_0[\frac{\sigma}{\varepsilon_0} U(t) + \varepsilon_r \frac{dU(t)}{dt} + \varepsilon_0 \frac{d}{dt}\int_0^t f(t-\tau)U(\tau)d\tau \qquad 4.13$$

Where, C_0 is the geometric capacitance of that insulating material. Equation (4.13) is applicable for both single and multiple dielectric materials in series or in parallel.

4.2.1 POLARIZATION AND DEPOLARIZATION CURRENT (PDC) MEASUREMENT

For the measurement of polarization and depolarization current (PDC), a dc voltage is applied to the dielectric material (as shown in Fig. 4.2(a)). Due to this applied voltage or electric field, the dipoles present inside dielectric medium, try to align themselves in the direction of the applied voltage or field [24]. Then polarization process occurs. During polarization, the current which flows through the dielectric material, has one of the components, known as 'Polarization Current'. This current decreases continuously. The polarization process gets stop when all the dipoles of the insulating material orient themselves in the direction to the applied electric field. Then only the conduction current flows through the insulating material and polarization current reaches to zero [24]. The conduction current depends on dc resistance of the insulating material. For the measurement of depolarization current, terminals of the insulating material are short-circuited and the voltage source is removed. At this time, the energy (or charge) stored by the dipoles during polarization, starts to release and the dipoles try to restore its initial position. The nature of depolarization current gets reverse direction of the polarization current also decreases continuously and finally, it reaches to zero after a long time (as shown by Fig. 4.2(b)). If a ripple and noise free dc. voltage is applied to an insulating material, polarization current (shown in fig. 4.2 (b)) starts flowing through the body of the material, it can be expressed as, For $0 < t < t_c$,

$$i_{pol}(t) = C_0 V_{dc}[\frac{\sigma_0}{\varepsilon_0} + \varepsilon_\infty \delta(t) + f(t)] \tag{4.14}$$

During the time period t_c in the equation (4.14) the insulating material is under an electric field and the material gets charge from the field. This time is known as charging time. The first part of the equation is conduction current. The part f(t) represents the dielectric response function [31].

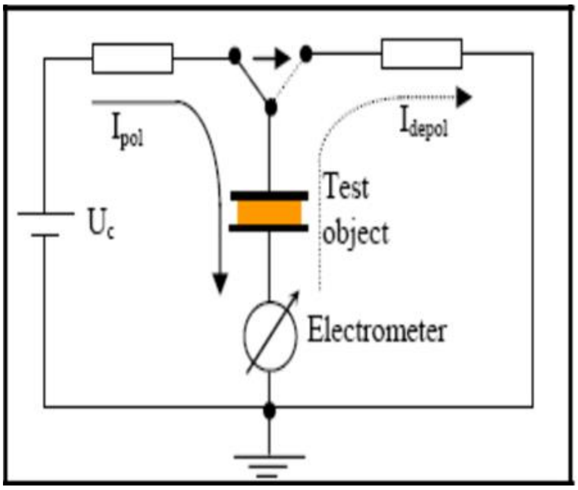

Fig. 4.2 (a) PDC measurement circuit

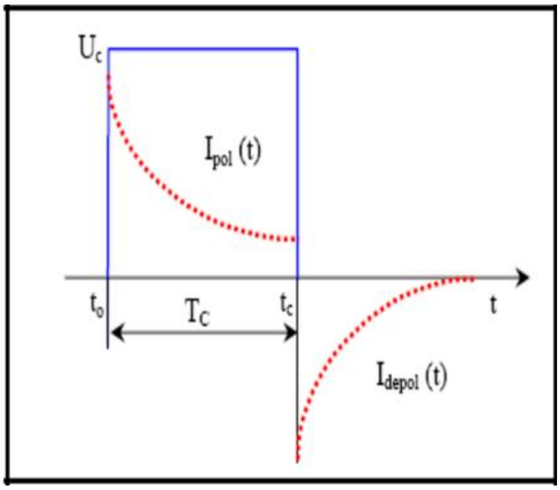

Fig. 4.2 (b) waveform of PDC currents

If a ripple and noise free dc. voltage is applied to an insulating material, polarization current (shown in fig. 4.2 (b)) starts flowing through the body of the material, it can be expressed as,

For $0 < t < t_c$,

$$i_{pol}(t) = C_0 V_{dc}[\frac{\sigma_0}{\varepsilon_0} + \varepsilon_\infty \delta(t) + f(t)] \tag{4.14}$$

During the time period t_c in the equation (4.14) the insulating material is under an electric field and the material gets charge from the field. This time is known as charging time. The first part of the equation is conduction current. The part f(t) represents the dielectric response function [31].

After the time period tc the terminal of the insulating material is under short circuited and the voltage source is removed . Therefore, after the time t_c the insulation material begins to release the stored energy then the dipoles of the material try to move their original position. The dipoles get re-orient when the depolarization current is flowing through the body the insulating material. The depolarization current express by –

$$i_{depol}(t) = -C_0 V_{dc}[f(t) - f(t + t_c)] \tag{4.15}$$

For $0 < t < \infty$

Therefore, the value of $f(t + t_c)$ is less as compared to f(t) for a long charging time t_c and can be neglected. So, the equation for depolarization current is rewritten as,

$$i_{depol}(t) = -C_0 V_{dc} f(t) \tag{4.16}$$

The dielectric response function $f(t)$ can be obtained from equation (4.16) once the value of depolarization current measured [35].

4.2.1.1 CONDUCTIVITY (σ_0) MEASUREMENT

Now the dc conductivity σ_0 of the insulating material is found out from PDC curves by subtracting the depolarization current from the polarization current at larger values of time. It is expressed as –

$$\sigma_0 = \frac{\varepsilon_0}{C_0 V_{dc}}[\, i_{pol}(t) - i_{depol}(t) \,] \tag{4.17}$$

4.2.1.2 ACTIVATION ENERGY (Eg) MEASUREMENT

Activation energy is the energy barrier which must be overcome by the trapped charges to start conduction in the dielectric material. The conductivity of material is related with temperature as well as activation energy. From Arrhenius equation, conductivity (σ) can be expressed as-

$$\sigma = Aexp[-\frac{Eg}{KT}\,] \tag{4.18}$$

Eg is the activation energy

K is the Boltzmann constant. Its value is $1.38064852 \times 10^{-23}$ m^2 kg s^{-2} K^{-1}

A is a constant

T is absolute temperature in Kelvin

The equation can be written as –

$$\ln(\,\sigma\,) = -\frac{Eg}{KT} + \ln(\,A\,) \tag{4.19}$$

This equation is similar to the equation of straight line i.e.

$$Y = mx + c \tag{4.20}$$

The graph is plotted against the logarithm of conductivity [$ln(\sigma)$] versus the reciprocal of temperature [1/T ($k^{\wedge -1}$)]. In MATLAB, the slope of the curve is known from curve-fitting technique. Then, the activation energy is obtained by multiplying slope to Boltzmann constant ‘k’ [35], as shown in equation (4.21).

$$Eg = -k * (-slope) \tag{4.21}$$

4.2.1.3 FLOWCHART FOR MEASUREMENT OF PDC

The flow chart (shown in Fig. 4.3) describes the method for the measurement of polarization and depolarization current to get the dielectric response.

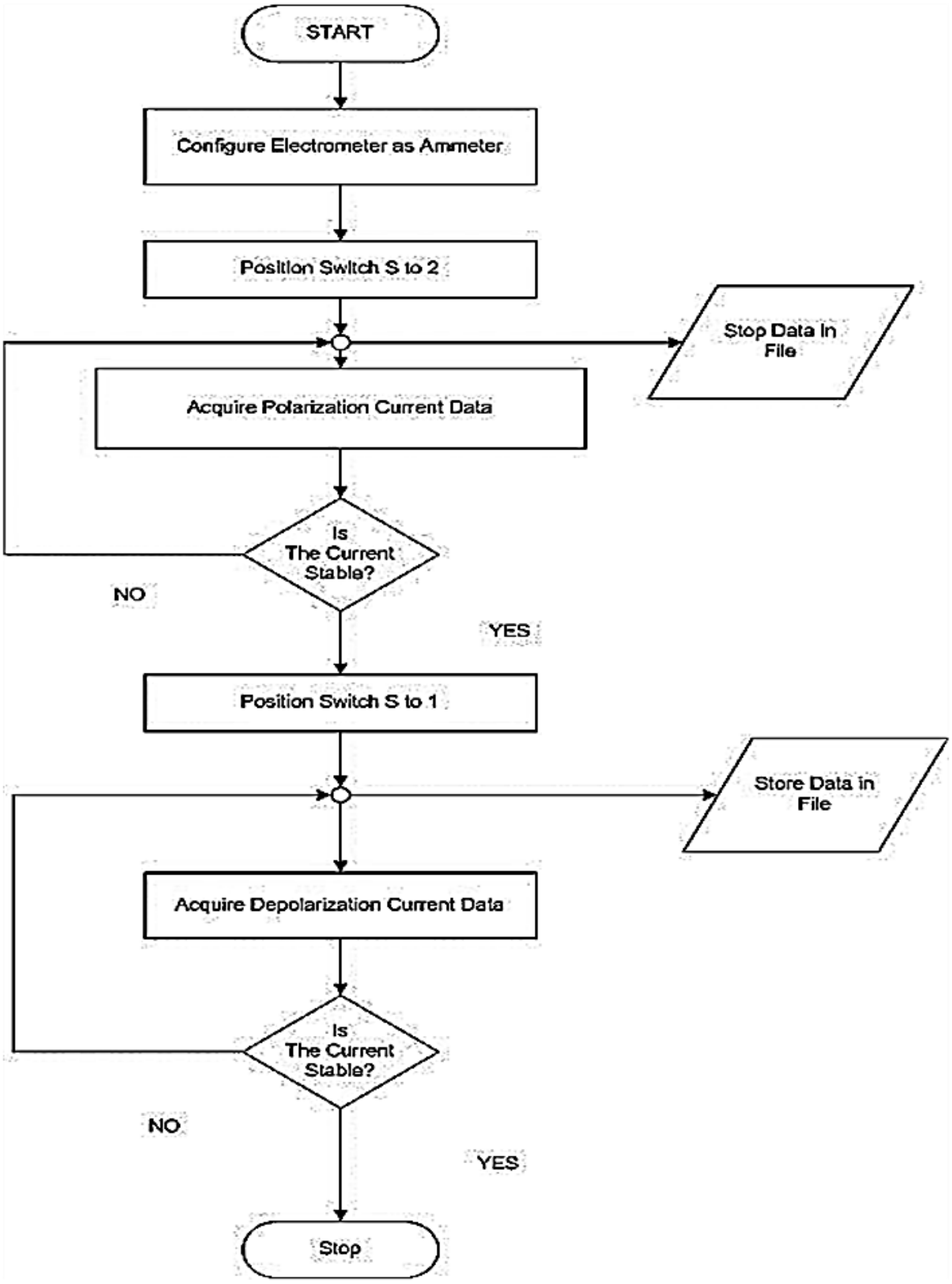

Fig. 4.3 Flow chart for PDC measurement

From fig. 4.2 (a) when the switch is connected to position 1, the electrometer shows polarization current. When this current becomes stable, the switch position is shifted from 1 to 2. Now, the electrometer shows the depolarization current. When this current becomes stable, the process is stopped.

4.2.2 RECOVERY VOLTAGE MEASUREMENT

For recovery voltage measurement, the charging and the discharging process is performed on the same circuit as PDC measurement. But, the difference is in time where discharging time is lesser than charging time according to requirement for this experiment [36]. The important characteristics of recovery voltage waveform are its maximum value of $V_{recovery}$ and the initial slope [37]. Moisture content in insulation system is calculated from RVM data [38]. The following sequence is followed while performing RVM. These are –

i. Source voltage of 1kV dc is applied to the test sample for definite time. Polarization gets start.
ii. The switch position is changed and kept it for a time, half of the charging. The dielectric material discharges itself in similar way like depolarization in PDC measurement.
iii. The electrometer is reconstructed as high valued input impedance voltmeter.
iv. The recovery voltage data are plotted in voltage v/s time graph.

The waveform of recovery voltage has been shown below (in Fig. 4.4)

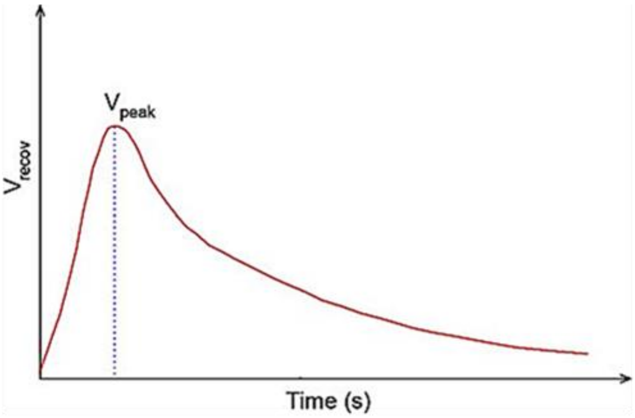

Fig. 4.4 Waveform of recovery voltage

4.2.2.1 PRINCIPLE OF RECOVERY VOLTAGE MEASUREMENT

In RVM, the discharging time is not enough for polarized material to cause full relaxation [39]. These unrelaxed charges release energy during their relaxation and the geometric capacitance gets charge. Energy re-distribution causes the rise of voltage (shown in fig. 4.5) across the geometric capacitor [40].

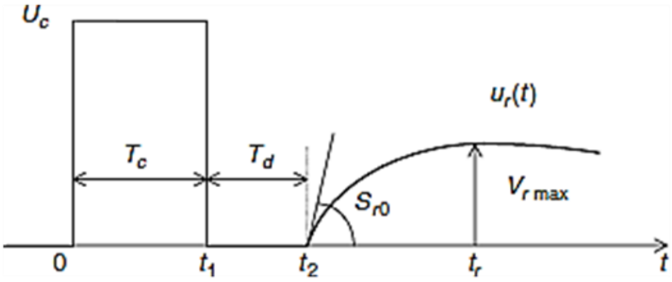

Fig. 4.5 Schematic principle of recovery voltage measurement

Here, U_c is the applied dc voltage

T_c is charging time

T_d is discharging time

U_r is recovery voltage waveform

Vr_{max} is maximum value of recovery voltage

While performing RVM test, a constant dc voltage of magnitude U_c is applied across the insulation for a time T_c. The voltage U_c^iacross the capacitor C_i of$i$ −th branch is given by–

$$U_c^i = U_c\left(1 - e^{-\frac{t}{\tau_i}}\right), i = 1,2, \dots n; 0 \le t \le T_c \qquad 4.22$$

here, $\tau_i = R_i C_i$

In next step, the insulation is shorted for a time T_d . the voltage U_c^i is –

$$U_c^i = U_c\left(1 - e^{-\frac{t}{\tau_i}}\right) e^{-\frac{t}{\tau_i}} \, i = 1,2, \dots .n; 0 \le t \le T_d \qquad 4.23$$

In last step, the insulation is kept open and the recovery voltage U_r is obtained because of residual charges inside the insulation [37]. The parameters of equivalent circuit (as shown in fig. 4.6) are calculated from the RVM data using the following Equations – 4.24 and 4.25.

$$C_0 \frac{dU_r(t)}{dt} + \frac{U_r(t)}{R_0} + \sum_{i=1}^{n} C_i \frac{dU_c^i(t)}{dt} = 0 \qquad 4.24$$

$$\frac{U_r(t) - U_c^i(t)}{R_i} = C_i \frac{dU_c^i(t)}{dt} \;;\;\; i = 1, \dots, n \qquad 4.25$$

The equivalent circuit looks like –

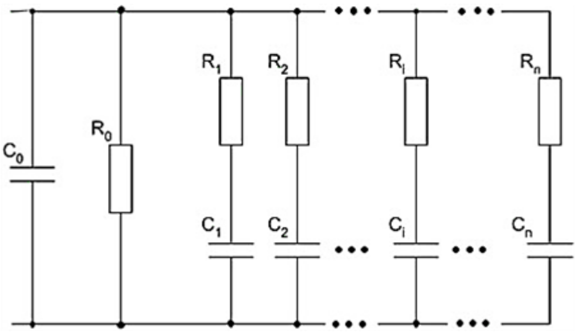

Fig. 4.6 Equivalent circuit of transformer insulation

R_0 having very high value, indicates better insulation condition. C_0 is the geometric capacitance [41].

4.3 FREQUENCY DOMAIN SPECTROSCOPY (FDS)

Although the time domain spectroscopy (TDS) is widely used for condition monitoring of insulation of transformer, it shows some limitations while applying it. The main limitations are-

i. high valued DC voltage source is needed and
ii. it is not free from noise.

To avoid these limitations nowadays, frequency domain spectroscopy (FDS) is used to get dielectric response of the insulation of transformer. In this test, AC voltage is applied to dielectric material. The insulation gets energy from pure sinusoidal voltage. Then polarization process starts and a current flow through the insulating medium (shown in fig. 4.7). The test (FDS) is performed with variable frequency in the range between 1 mHz to 10 kHz [42]. This measurement helps to estimate power factor, capacitance, dissipation factor, permittivity [43].

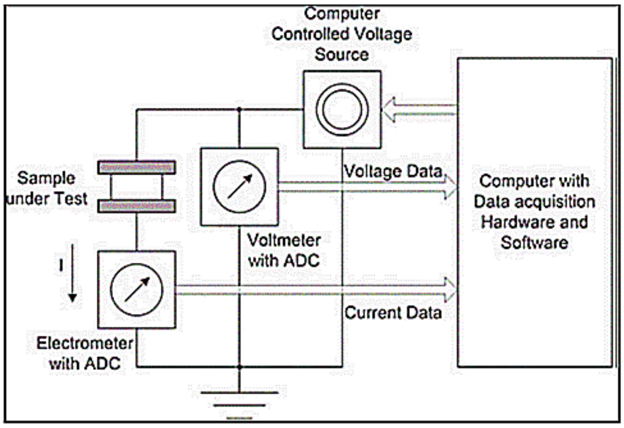

Fig. 4.7 FDS Measurement Circuit

From equation (4.12), a step response for current density $J(t)$, is given by –

$$J(t) = \sigma_0 E(t) + \varepsilon_0 \frac{dE(t)}{dt} + \varepsilon_0 \frac{d}{dt} \int_0^t f(t-\tau)E(\tau)d\tau \qquad 4.22$$

Taking Laplace transform of equation (4.22) and replacing s by jω, it is written –

$$J(\omega) = \sigma_0 E(\omega) + j\omega\varepsilon_0 E(\omega) + j\omega\varepsilon_0 F(\omega)E(\omega) \qquad 4.23$$

In equation (4.23), $F(\omega)$ indicates the Fourier transform of dielectric response function $f(t)$. $F(\omega)$ gives the complex susceptibility $\chi(\omega)$ of the dielectric material. So,

$$\chi(\omega) = \chi'(\omega) - j\chi"(\omega) = F(\omega) = \int_0^\infty f(t)e^{-j\omega t}dt \qquad 4.24$$

Putting the value of $\chi(\omega)$ in place of $F(\omega)$ equation (4.23),

$$J(\omega) = [\{\sigma_0 + \varepsilon_0\omega\chi"(\omega)\} + j\omega\varepsilon_0\{1+\chi'(\omega)\}]E(\omega) \qquad 4.25$$

Equation (4.25) can be re written in terms of complex relative permittivity ε_r . It is –

$$J(\omega) = j\omega\varepsilon_0\varepsilon_r(\omega)E(\omega) \qquad 4.26$$

Here, $\varepsilon_r(\omega) = \varepsilon_r'(\omega) - j\varepsilon_r"(\omega) = [\,1+\chi'(\omega)] - j[\,\chi"(\omega) + \frac{\sigma_0}{\omega\varepsilon_0}\,]$ 4.27

From equation (4.27), $\varepsilon_r(\omega)$ is frequency dependent complex permittivity of insulating material. The energy loss happens due to interaction among the dipoles under sinusoidal excitation, is represented by the imaginary part $\varepsilon''(\omega)$. The energy storage in the dielectric medium during polarization is represented by the real part $\varepsilon'(\omega)$. The dielectric dissipation factor is expressed as the ratio of the energy loss to the energy storage in dielectric media, can be represented as –

$$\tan\delta(\omega) = \frac{\frac{\sigma_0}{\omega\varepsilon_0}+\chi''(\omega)}{1+\chi'(\omega)} \qquad 4.28$$

So, dielectric loss factor ($tan\delta$) is dependent on frequency of applied voltage. Here, frequency varies from 0.1 m Hz to 10 kHz [42]. So it is possible to estimate relationships between different dipolar groups.

4.3.1 ADVANTAGES OF FDS METHOD

In few aspects, FDS is more advantageous than TDS. The following advantages are available while performing FDS-

i. Frequency domain spectroscopy (FDS) measures insulation capacitance, permittivity, conductivity (and resistivity) and dissipation factor depending on frequency.
ii. The real and imaginary part of the complex capacitance and permittivity can be differentiated.
iii. This non-destructive technique gives information about the moisture content in the solid insulation material.
iv. FDS has better performance against noise and separates the polarizability (χ') and losses (χ'') of a dielectric medium.

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CHAPTER - 5

EXPERIMENTAL PROCEDURE

5.1 INTRODUCTION

Sample preparation is very important feature to get maximum benefits from nano-composites. Uniform dispersion of fillers is necessary for showing dielectric properties in polymer [43]. If non-uniform dispersion or agglomerations of Nano particles occurs, it leads to weak the spots & reduces electrical, mechanical & thermal properties of Nano-composites [44]. So, appropriate processing technique must be adopted that makes uniform dispersion of nanoparticles in base polymer.

5.2 SAMPLE PREPARATION

5.2.1 MATERIALS USED FOR SAMPLE PREPARATION

The base materials used for this study are bisphenol - A based epoxy resin & hardener HY 951 [45]. Available Al_2O_3 nanoparticles, with average particle size of 65 nm, are used as the fillers. Since, they are well known materials with excellent dielectric property.

5.2.2 SAMPLE MANUFACTURING PROCESS

To prepare the sample, epoxy resin and hardener are degassed at 40^0C for 2 hours to separate the moisture and air bubbles. Similarly, the alumina Nano-composites are also heated at 150^0C for 24 hours to dry before using it. Al_2O_3 nanoparticles have density of 4 gm/cc. Then these nanoparticles are mixed with epoxy resin through mechanical mixing. This mechanical mixing follows two steps.

Step 1: mixing is performed at high speed of 750 rpm for 1 hour.
Step 2: ultrasonic agitation is given at a frequency of 24 kHz for 1 hour.

After that, hardener is poured into the mixture, mixed thoroughly and then degassed. This mixture is cast in mould. The mould is placed inside the controlled oven at 60^0C for 4 hours to cure and kept it for desiccation. Through similar way, other samples with weight percentage of 1 and 2% alumina nanoparticles are made. The thickness and diameter of the samples are 0.5 mm and 75 mm respectively [46].

5.2.3 ELECTRO-THERMAL AGEING OF EPOXY ALUMINA NANO-COMPOSITE SAMPLES BEFORE EXPERIMENT

After preparing these samples, they are kept inside of a controlled heat chamber, where thermal ageing is done at a fixed temperature of 100^0C for each Nano-composite sample under 6 kV/mm, 50 Hz electric field stress. Thermal ageing is carried out for 600 hours. Every instant of 100 hours of ageing, each sample is taken out of heat chamber and experiment is performed in ambient temperature at 25^0 C [47].

5.3 EXPERIENTAL SETUP

So many equipment has been used for this experiment. The High Voltage Laboratory of Jadavpur University hosts all the equipment and instruments for this experiment. The major types of equipment that are used for this experiment has been elaborated below.

Equipment used in this thesis, are shown below –

5.3.1 DIGITAL SIGNAL OSCILLOSCPOE

RIGOL DS1102E digital oscilloscope is used for data acquisition and store of all polarization and depolarization current shown in Fig. 5.1. The bandwidth of this digital oscilloscope is 100 MHz. The maximum sampling rate of this digital oscilloscope is about 1G Samples/s. The memory depth of RIGOL DS1102E is 1Mpts. This digital oscilloscope is fitted with a 5.6” TFT QVGA (320X240) with 64K color LCD backlit display.

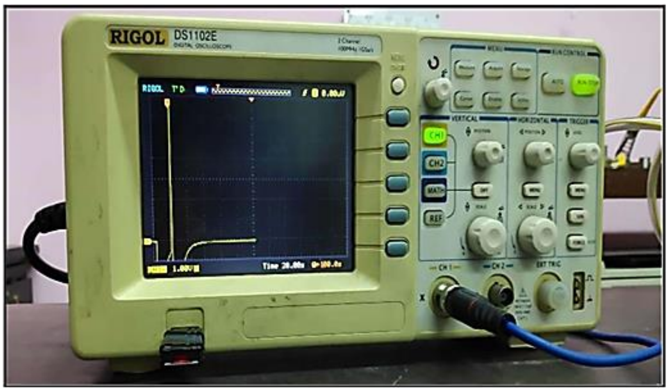

Fig. 5.1 RIGOL DS1102E Digital Oscilloscope

The used second instrument is -

5.3.2 DIELECTRIC RESPONSE ANALYZER

Dielectric Response Analyzer (or an amplifier) is used to check the condition of high voltage insulation systems like in power transformers, cables, bushings, and generators shown in Fig.5.2. In this present work, it is used for Polarization Depolarization Current (PDC) measurement. One resistance box also has been attached. It can successfully measure current range of Nano-ampere.

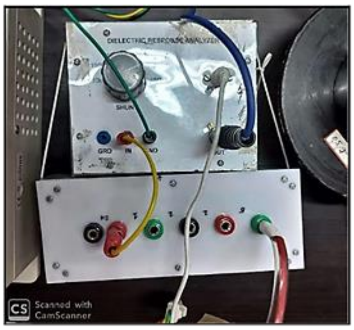

Fig. 5.2 Dielectric Response Analyzer

The third instrument is -

5.3.3 HEATING OVEN

The heating oven is used for maintaining a fixed temperature of an isolated chamber, shown in Fig. 5.3 (a) & (b). It removes the moisture present in the insulation. Thermal ageing is also done with this Oven.

Fig. 5.3 (a) Heating Oven

Fig. 5.3 (b) Heating Oven

5.3.4 EPOXY RESIN

This epoxy resin (as shown in Fig. 5.4) is used for sample preparation available in High Voltage Laboratory.

Fig. 5.4 Epoxy resin

5.3.5 SAMPLE MIXTURE (MAGNETIC STIR)

This mixture (in Fig. 5.5) is used to mix up the epoxy resin and the hardener. It is available in Jadavpur University Laboratory.

Fig. 5.5 Sample mixing device

5.3.6 EXPERIMENTAL CIRCUIT

After collecting the samples, PDC measurement was performed with the help of manually controlled measuring device named as Dielectric Response Analyzer. All PDC experiment has been done in a heating oven with different temperature. In Fig. 5.6 it is seen the experimental setup for PDC measurement.

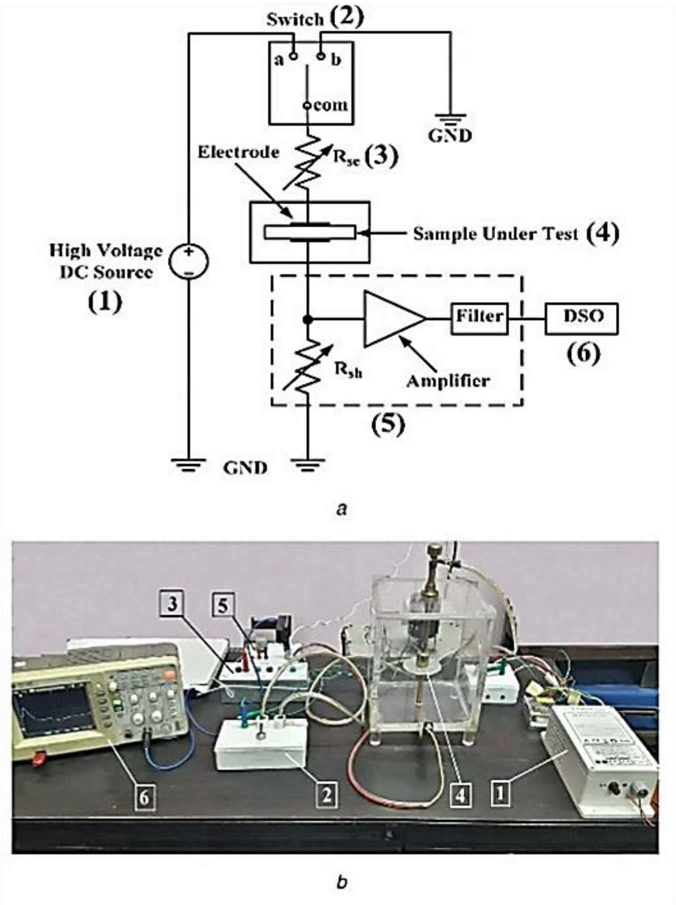

Fig. 5.6 Experimental Setup for PDC measurement.

a. Schematic experimental setup
b. Hardware setup

[(1) HVDC supply, (2) switch, (3) series resistance box, (4) test sample, (5) amplifier with resistance box, (6) DSO]

An adjustable high dc voltage source (1) of range between 200V to 4000V is connected to the switch (2). A resistor Rse (3), in the range of 1 to 10 GΩ is connected in series with the test sample (4) to limit the current for protecting the sample under test and also to increase overall time constant of the circuit. The test sample is kept in between Rse and shunt resistance Rsh (5), in the range of 1KΩ to 10 GΩ. An OP-AMP is connected from shunt resistance to a low pass filter circuit. This low pass filter circuit eliminates the unwanted signals and noise. The required data is recorded in DSO (6).

5.3.7 THEORY FOR THE CIRCUIT

Under the influence of electric field, the PDC measurement of solid dielectric material is difficult because of lower conductivity of solid material [48]. The measured current is in the range of pico-ampere (pA) for solid material, but for oil- paper insulation this current is in range of mili-ampere (mA). Two types of problem arise while measuring PDC. One is – (a) to differentiate the actual signal from noisy signal & other is – (b) to find the system time constant [49].

The polarization & depolarization process occurs very quickly in solid dielectrics in the range of nanoseconds. So, for collecting the important data, costly instruments are required. To overcome this situation, a high value resistor (Rse) is connected in series with the sample so that the overall time constant ($\mathrm{T} = \mathrm{RseC}$) for data acquisition gets increase, in the range of milliseconds. Even low valued ADC (Analog to digital converter) having sampling rate of milliseconds, can show the current waveform [50].

5.4 EXPERIMENTAL PROCEDURE

The following procedure is followed while performing the experiment -

i. At first, the sample of pure epoxy resin is heated up in oven chamber at an interval of 30^0C and continued till 90^0C. After each interval, the sample is connected to dc voltage source (switch is in position ‘a’ of schematic diagram), the polarization current flows through the test sample. When this current reaches at steady state value. Corresponding polarization waveform is recorded in DSO. Then switch is placed in position ‘b’ of schematic diagram, the test sample gets shorted. Depolarization current flows through the test sample. The corresponding depolarization current waveform is recorded in DSO.

ii. In second case, three samples are kept in heat chamber keeping temperature fixed at 30^0C. They are kept to undergo an electro-thermal ageing, duration of 600 hours at an interval of 100 hours. In each interval (including unaged condition also), the PDC measurement procedure is followed as mention in the above paragraph (i).

iii. After collecting the PDC data from the sample, their conductivity is measured by the equation (4.17) from chapter 4. The table no. 1, 2 and 3 show the conductivity of each sample at different temperature.

iv. Then, the logarithm of conductivity along y-axis versus the inverse of absolute temperature along x-axis is plotted. From there, the corresponding slope of each figure is calculated.

v. The obtained slope is multiplied by Boltzmann constant (as shown in equation 4.21 from chapter 4) to get the Activation energy of each sample.

vi. The Activation energy is plotted against ageing duration in MATLAB. From there, the ageing of each sample is known.

CHAPTER – 6

RESULTS AND DISCUSSIONS

6.1 INTRODUCTION

In this chapter, the experimental results are shown. While performing this experiment, the ageing is done for a duration of 600 hours. Every 100 hours of interval, the thermally aged sample are taken out of the oven and PDC measurement is performed. The same procedure is followed when temperature is varied. Every interval of 10^0C, PDC data is collected using the experimental setup of each sample. Polarization current, depolarization current, conductivity, Activation energy and the ageing duration are respectively the outcome of this experiment. These are shown below -

6.2 NATURE OF POLARIZATION CURRENT

Polarization current has been measured by two ways. Firstly, by varying the temperature of the samples placed in oven, keeping the ageing duration as fixed (unaged condition shown in fig. 6.5). Secondly, by varying the ageing duration of the samples, keeping temperature as fixed (shown in fig. 6.2, 6.3 and 6.4). The below figures are obtained -

Fig. 6.1 shows the variation of polarization current of pure epoxy resin with different temperature. It can be seen that with the increase of temperature, polarization current increases. It happens due to the thermal energy which decreases the resistance. Thus, the polarization current increases with temperature.

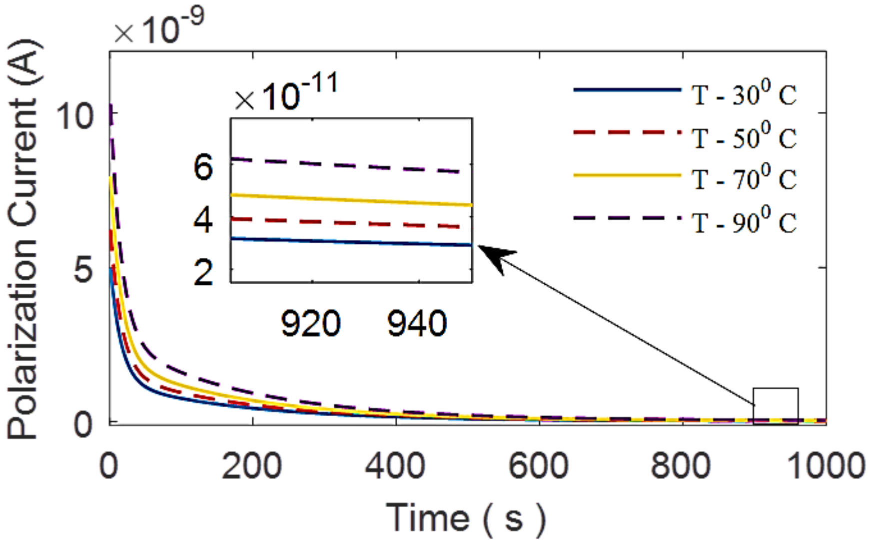

Fig. 6.1 Polarization current of pure epoxy at different temperature.

Fig. 6.2 shows variation of polarization current with ageing duration of pure epoxy resin. Polarization current gets increase with increase in ageing duration. The chemical bonding inside the pure epoxy molecules become weak due to temperature, moisture present in atmosphere. Availability of free electron increases.

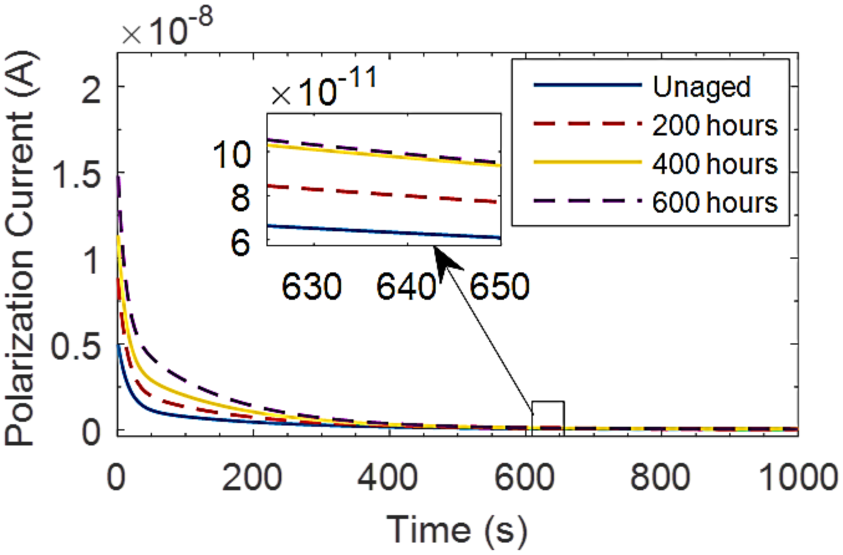

Fig. 6.2 Polarization current of pure epoxy resin at temperature at 30^0C with different ageing duration.

Fig. 6.3 shows the variation of polarization current of epoxy resin with 1 wt.% alumina. Polarization current magnitude increases with increase in ageing duration. But, it has the current magnitude lower than the pure epoxy resin. Because, the dipoles get entrap by the alumina Nano-fillers inside the molecules.

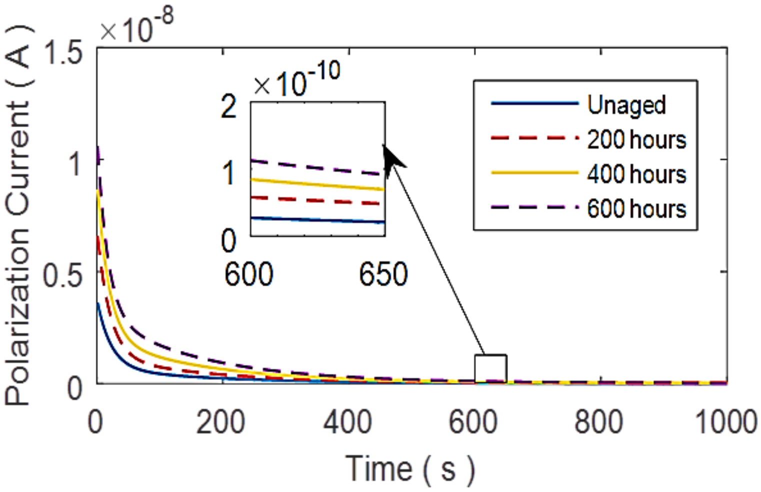

Fig. 6.3 Polarization current of epoxy resin with 1 wt. % alumina at 30^0C with different ageing duration.

Fig. 6.4 shows the variation of polarization current of epoxy resin with 2 wt.% alumina at different ageing duration. Like the above two figures, polarization current also increases with increase in ageing duration of this sample. It can be observed that for a particular ageing duration, polarization current decreases with increase in alumina Nano-fillers concentration among all three samples.

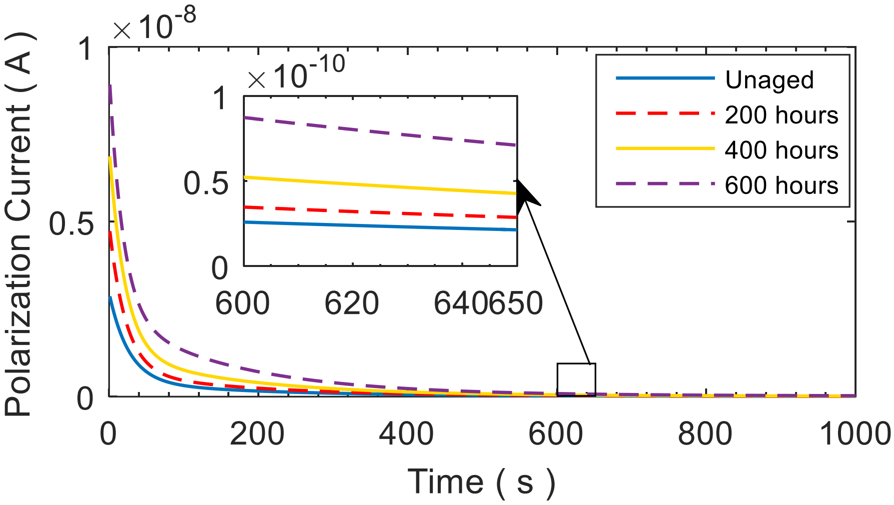

Fig. 6.4 Polarization current of epoxy resin with 2 wt. % alumina at 30^0C with different ageing duration.

From above three figures it can be concluded that Al_2O_3 creates hurdle during polarisation of the material. The presence of nanoparticles may cause entanglement of long polymer chains, which will lead to hindrance in the movement of the chains. This will restrict the polarisation of epoxy–alumina nano-composites.

Fig. 6.5 makes comparison of the polarization current of three different samples at unaged condition. Pure epoxy resin has highest magnitude and the epoxy resin with 2 wt.% alumina has least magnitude of polarization current. It is due to presence of alumina Nano-fillers which reduce the charge mobility. But, there is no such event occur in case of pure epoxy resin. So, it has high valued polarization current. That has been shown in below fig. 6.5.

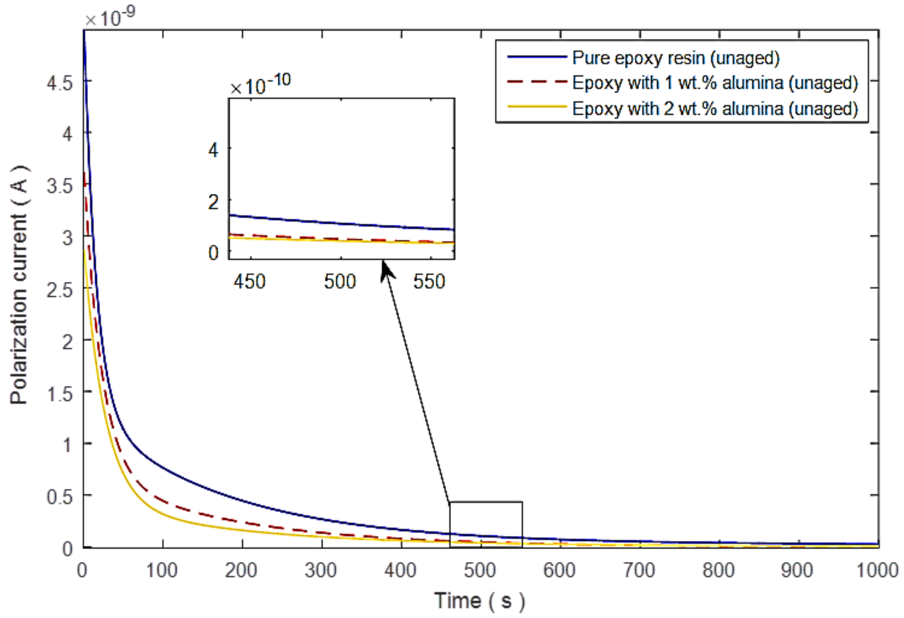

Fig. 6.5 Polarization current of all three samples at unaged condition at 30^0C.

6.3 NATURE OF DEPOLARIZATION CURRENT

Depolarization current is measured immediately after the measurement of polarization current of every sample. The following observations are obtained –

For unaged samples the variation of depolarization current with time is shown in Fig. 6.6 . From the above figure, keeping temperature fixed, pure epoxy resin has higher depolarization current as compare to other sample. Because 1 wt.% and 2 wt.% alumina fillers oppose the charge movement of these sample which is not present in case of pure epoxy resin.

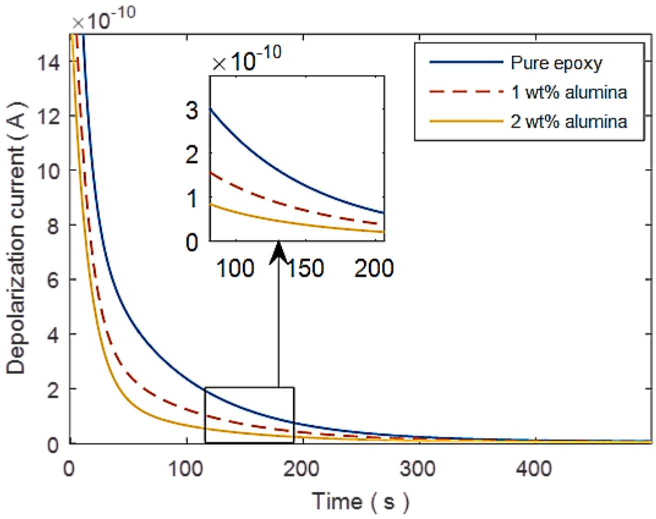

Fig. 6.6 Depolarization current of different samples (unaged) at temperature 30^0C.

Here, depolarization current is measured at ageing duration of 300 hours. Fig. 6.7 shows the variation of depolarization current of different sample. Depolarization current magnitude of all samples is less as compare to unaged condition. Due to ageing, all samples are gradually losing their charge stored during polarization process.

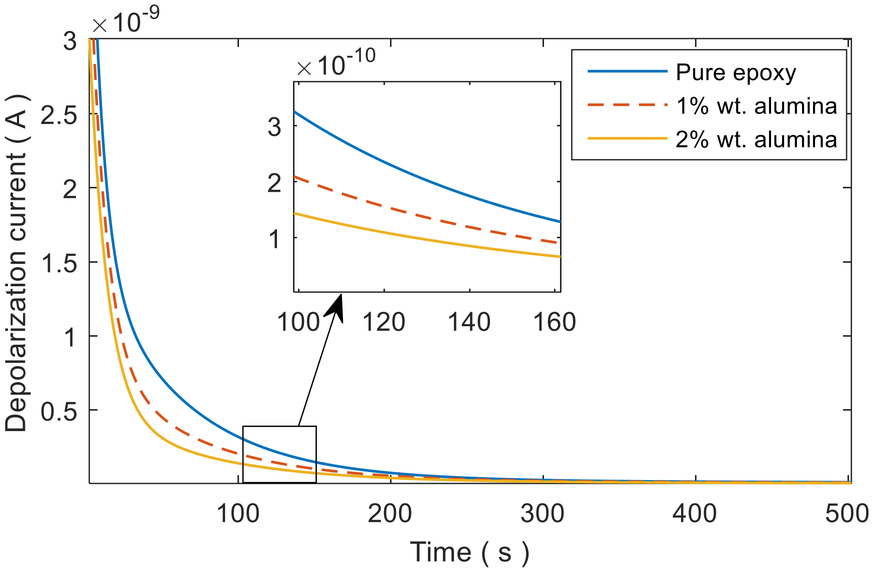

Fig. 6.7 Depolarization current of different samples (ageing duration of 300 hours) at temperature 30^0 C.

The depolarization current is measured after ageing of 600 hours, shown in Fig. 6.8. This current decreases more as compare to the ageing of 300 hours. Because, all the dipoles within the material, restore the initial position by releasing their energy. Pure epoxy has the highest value and the 2 wt.% alumina sample has the lowest value of current.

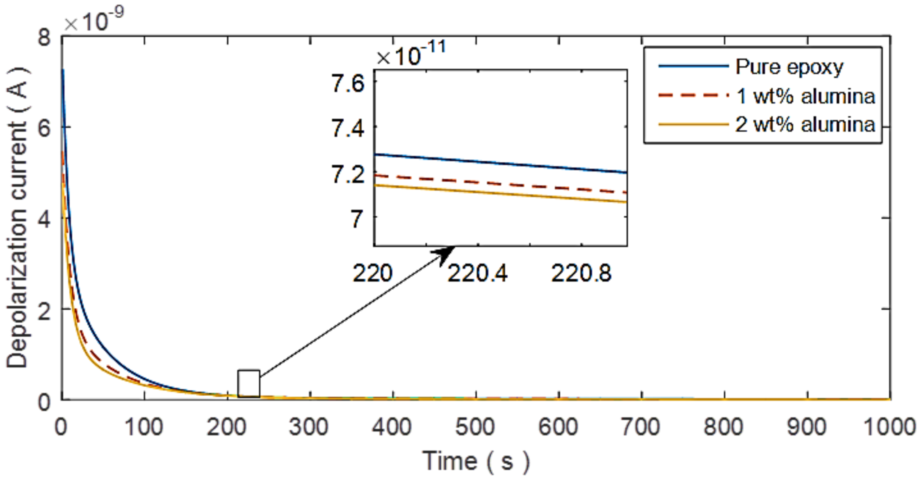

Fig. 6.8 Depolarization current of different samples (ageing duration 600 hours) at temperature 30^0 C.

Below three figures - 6.9, 6.10 and 6.11 show the effect of temperature on depolarization current.

Fig. 6.9 shows the variation of depolarization current with different temperature of pure epoxy resin. With increase in temperature, the depolarization current magnitude gets increase of that sample. External thermal energy causes to increase the charge mobility during temperature variation.

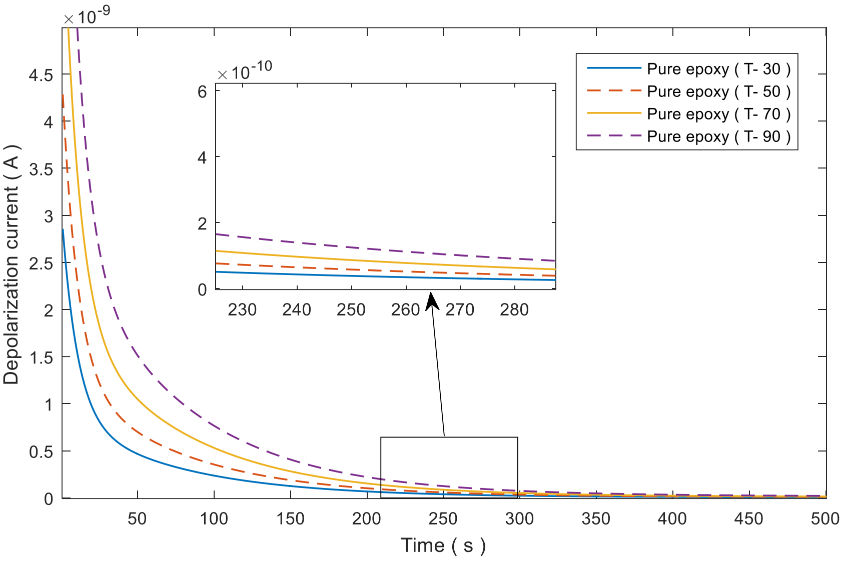

Fig. 6.9 Depolarization current of pure epoxy resin at different temperatures.

For the sample of epoxy resin with 1 wt.% alumina, Fig. 6.10 shows the variation of depolarization current with temperature. Alumina filler addition decreases the current magnitude as compare to pure epoxy resin. Because, alumina fillers oppose the charge movement.

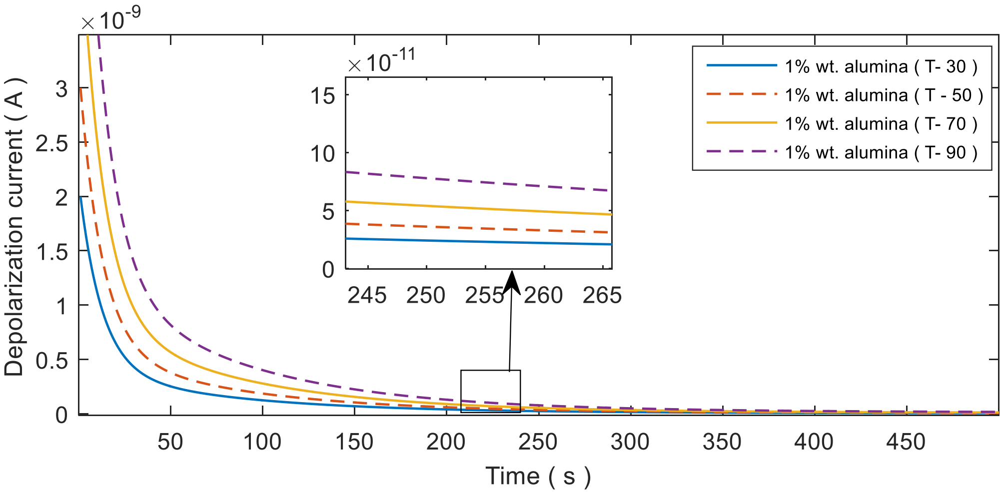

Fig 6.10 Depolarization current of epoxy resin with 1 wt.% alumina at different temperatures.

The variation of depolarization current of epoxy resin with 2 wt.% alumina is shown in Fig. 6.11 at different temperature. The more addition of alumina fillers, the lesser magnitude of depolarization current. Because, alumina fillers are insulting material in nature. They do not allow charge carrier to flow through it. For same operating temperature, pure epoxy resin has highest value of current and 2 wt.% alumina sample has lowest value.

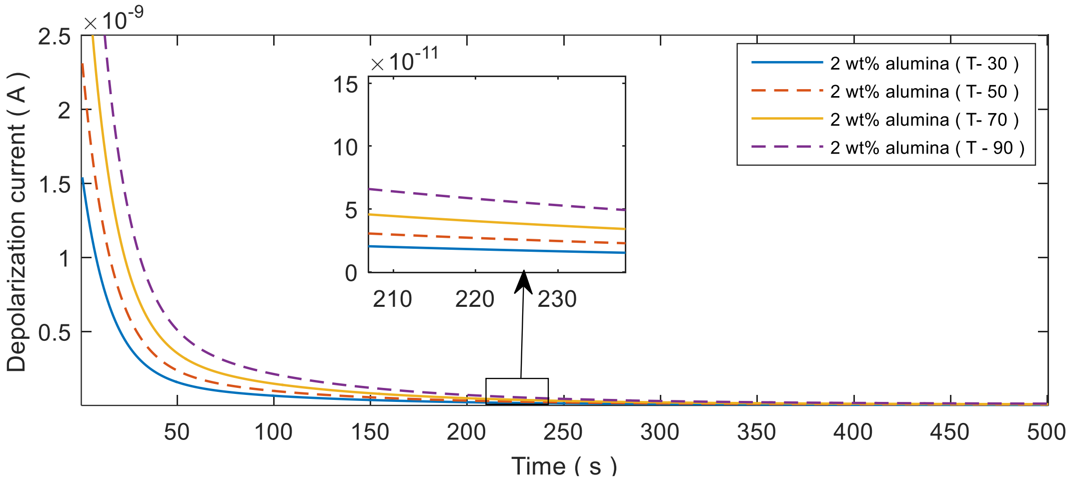

Fig. 6.11 Depolarization current of epoxy with 2 wt.% alumina at different temperatures

the depolarization current at different ageing duration of pure epoxy resin is shown in Fig. 6.12. With increase in ageing duration, this current increases. The molecular bonds get break due to temperature variation, moisture and carbonisation which results in increase of depolarization current. Highest value of depolarization current produce at ageing duration of 600 hours and lowest at unaged.

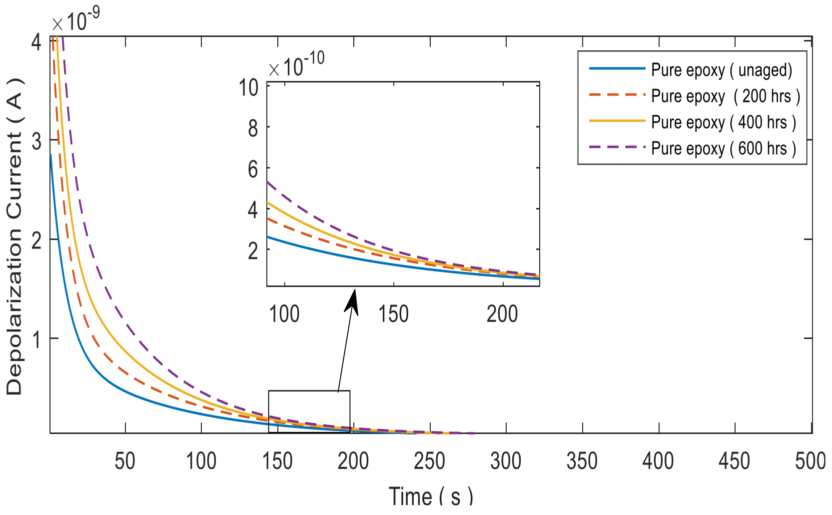

Fig. 6.12 Depolarization current of pure epoxy resin at different ageing duration

The variation of depolarization current at different ageing duration of epoxy resin with 1 wt.% alumina is shown in Fig. 6.13. Alumina fillers addition reduce the current magnitude as compare to pure epoxy resin. They offer resistance to charge movement.

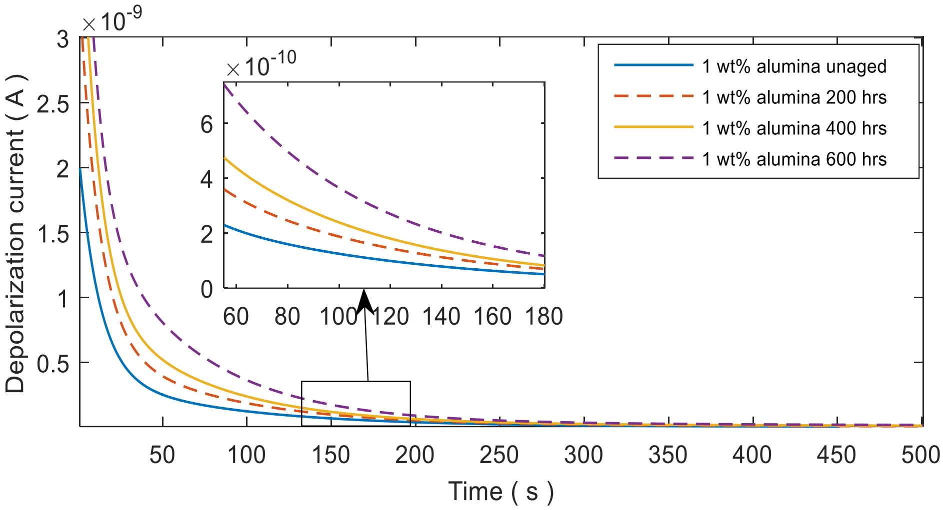

Fig. 6.13 Depolarization current of epoxy resin with 1 wt.% alumina at different ageing duration

The variation of depolarization current at different ageing duration of epoxy resin with 2 wt.% alumina is shown in Fig. 6.14. Alumina Nano-fillers concentration is higher than the former two. So, current reduction is more in this case. For same ageing duration, 2 wt.% alumina sample has the lowest value of current and the pure epoxy resin has the highest value.

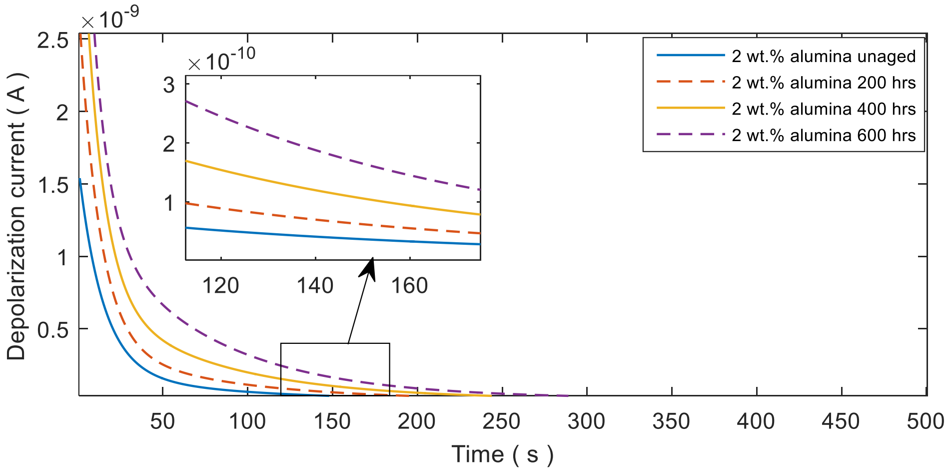

Fig. 6.14 Depolarization current of epoxy resin with 2 wt.% alumina at different ageing duration

6.4 MEASURED VALUE OF CONDUCTIVITY OF DIFFERENT SAMPLES

Conductivity is calculated from polarization and depolarization current. It is obtained by subtracting the depolarization current from polarization current when they attain steady state value respectively. It can be shown –

$$\sigma_0 = \frac{\varepsilon_0}{C_0 V_{dc}}[\ i_{pol}(t) - i_{depol}(t)\ ]$$

σ_0 is the conductivity of each sample at different temperature and different ageing duration is shown below in tables – 6.1, 6.2 and 6.3.

Table 6.1: shows conductivity of the pure epoxy resin. It is -

Table 6.1: conductivity (σ) of pure epoxy resin

Aging Duration (hours)	**Conductivity (S/cm)**						
	30ºC	**40ºC**	**50^0C**	**60^0C**	**70^0C**	**80^0C**	**90^0C**
Unaged	1.54E-16	1.3E-15	2.7E-15	8.61E-15	1.7E-14	2.69E-14	9E-14
100	2.03E-16	1.59E-15	3.13E-15	1.02E-14	2.09E-14	3.23E-14	1.01E-13
200	2.57E-16	1.91E-15	3.63E-15	1.20E-14	2.53E-14	3.83E-14	1.14E-13
300	3.13E-16	2.04E-15	3.86E-15	1.27E-14	2.68E-14	4.08E-14	1.21E-13
400	3.73E-16	2.18E-15	4.13E-15	1.36E-14	2.87E-14	4.36E-14	1.29E-13
500	4.12E-16	2.38E-15	4.48E-15	1.46E-14	3.06E-14	4.6E-14	1.35E-13
600	4.47E-16	2.57E-15	4.79E-15	1.55E-14	3.21E-14	4.79E-14	1.39E-13

The conductivity profile with different ageing duration of pure epoxy resin is shown in Fig. 6.15. It can be observed that with increase in ageing duration, the conductivity of pure epoxy resin increases. It has the highest conductivity at 600 hours and lowest at unaged. Because, the moisture production increases in the sample day by day. More number of free electron is available due to breakdown of chemical bonding between the molecules of epoxy resin. It enhances the conductivity.

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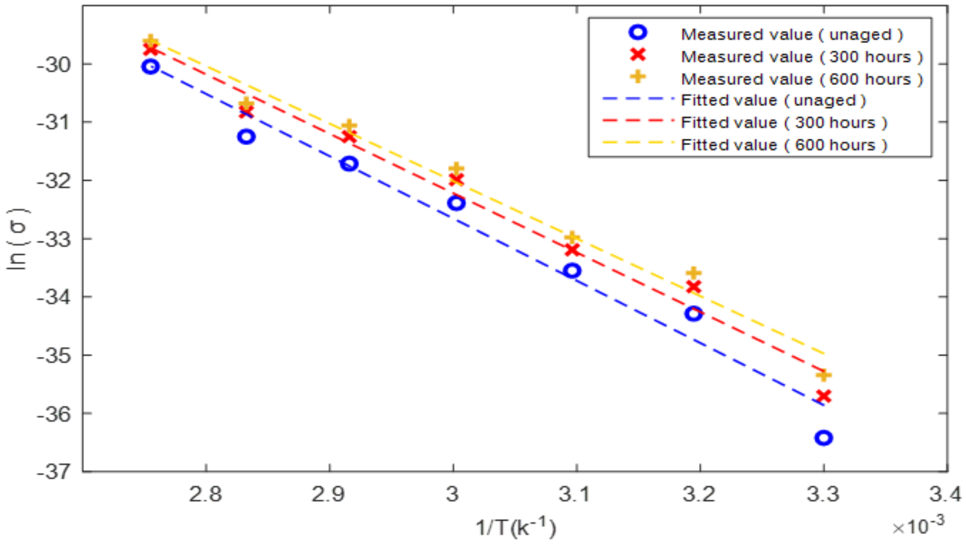

Fig. 6.15 Conductivity of pure epoxy resin at different ageing duration.

Table 6.2: shows the conductivity of epoxy with 1 wt.% alumina sample. It is -

Table 6.2: conductivity (σ) of epoxy resin with 1 wt.% alumina

Ageing Duration (hours)	**Conductivity (S/cm)**						
	30°C	**40°C**	**50°C**	**60°C**	**70°C**	**80°C**	**90°C**
Unaged	1.69E-16	7.31E-16	1.59E-15	6.69E-15	1.26E-14	2.13E-14	8.52E-14
100	1.93E-16	1.1E-15	2.27E-15	8.47E-15	1.68E-14	2.66E-14	9.40E-14
200	2.22E-16	1.48E-15	2.99E-15	1.04E-14	2.14E-14	3.24E-14	1.05E-13
300	2.58E-16	1.55E-15	3.14E-15	1.1E-14	2.24E-14	3.36E-14	1.07E-13
400	2.97E-16	1.62E-15	3.33E-15	1.17E-14	2.37E-14	3.51E-14	1.11E-13
500	3.28E-16	1.78E-15	3.61E-15	1.25E-14	2.53E-14	3.7E-14	1.16E-13
600	3.57E-16	1.92E-15	3.86E-15	1.33E-14	2.66E-14	3.86E-14	1.20E-13

For the sample of epoxy resin with 1 wt.% alumina, Fig. 6.16 shows the variation of conductivity with ageing duration. Conductivity increases with increase in ageing duration. It shows the highest conductivity at 600 hours and lowest at unaged. But, it has the conductivity lower than pure epoxy resin. Because, Al_2O_3 inserted in the epoxy resin can restrict the molecular chain movement, which is not present in case of pure epoxy resin. Charge cannot penetrate into the molecule due to presence of alumina fillers.

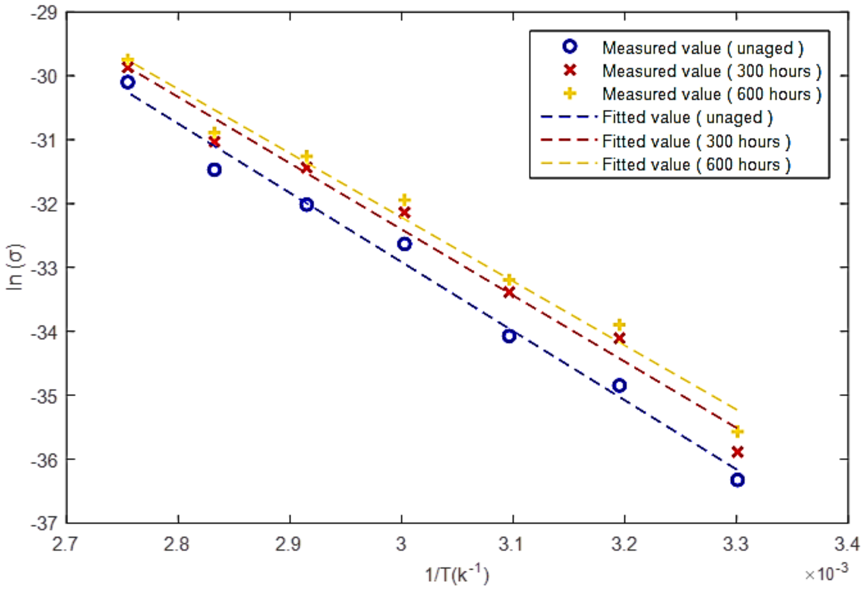

Fig. 6.16 Conductivity of epoxy resin with 1 wt.% alumina at different ageing duration

Table 6.3: shows the conductivity of epoxy with 2 wt.% alumina. It is -

Table 6.3: conductivity (σ) of epoxy resin with 2 wt.% alumina

Aging Duration (hours)	**Conductivity (S/cm)**						
	30°C	**40°C**	**50°C**	**60°C**	**70°C**	**80°C**	**90°C**
0	1.72E-16	1.07E-15	2.2E-15	8.22E-15	1.63E-14	2.58E-14	9.13E-14
100	1.79E-16	9.76E-16	2.1E-15	7.93E-15	1.57E-14	2.43E-14	8.74E-14
200	1.89E-16	9.06E-16	2.04E-15	7.8E-15	1.54E-14	2.32E-14	8.54E-14
300	2.04E-16	9.84E-16	2.27E-15	8.71E-15	1.7E-14	2.48E-14	8.91E-14
400	2.22E-16	1.07E-15	2.53E-15	9.71E-15	1.87E-14	2.66E-14	9.37E-14
500	2.57E-16	1.22E-15	2.84E-15	1.08E-14	2.05E-14	2.88E-14	9.98E-14
600	2.89E-16	1.36E-15	3.13E-15	1.17E-14	2.21E-14	3.06E-14	1.05E-13

Fig. 6.17 shows the conductivity at different ageing duration of epoxy resin with 2 wt.% alumina Nano-composite. It shows the highest conductivity at 600 hours and lowest at unaged. Among all three samples, the last one has the lowest conductivity because the molecular chain of cross-linked epoxy with alumina fillers oppose the movement of electrons or ions.

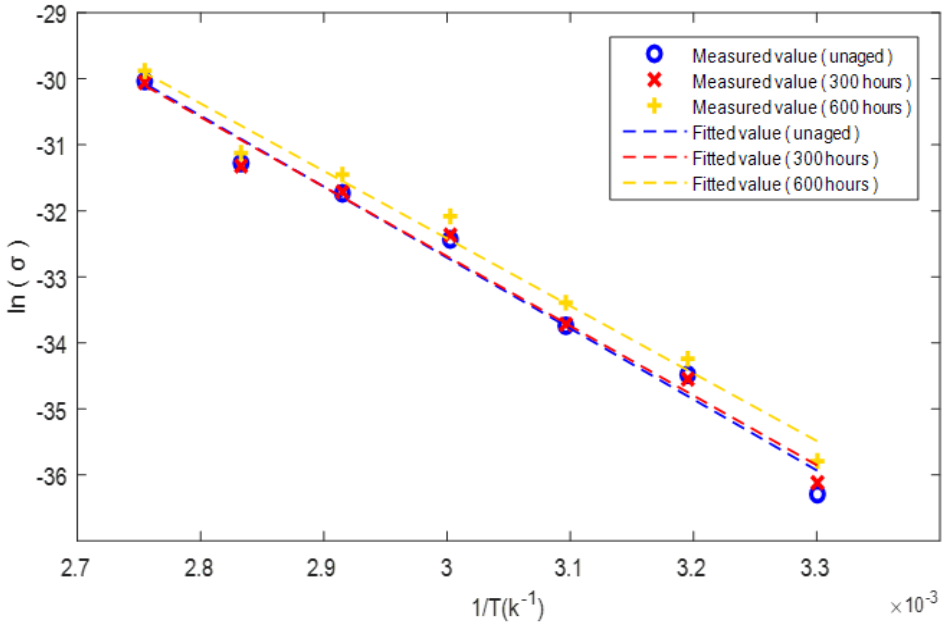

Fig. 6.17 Conductivity of epoxy resin with 2 wt.% alumina at different ageing duration.

It can be concluded that the more Al_2O_3 particles added to the epoxy matrix, the more the restriction on epoxy molecular movement, the less will be the conductivity of epoxy alumina.

6.5 ESTIMATION OF ACTIVATION ENERGY OF THE SAMPLES

Activation energy of any insulating material is the energy barrier that the molecules or atoms must possess to undergo any chemical reaction within that material. The chemical reaction rate inside the insulating medium depends on the activation energy. Higher activation energy indicates very few molecules have sufficient energy to undergo a chemical reaction. So, chemical reaction rate decreases.

The calculation for the Activation energy is shown in chapter 4, equation 4.21.

The Activation energy profile of three samples are shown below – firstly for pure epoxy resin - fig. 6.18 shows the variation of activation energy with ageing hours. It can be noted that with increase in ageing duration, the activation energy decreases. Due to the presence of moisture, temperature variation inside the material, the chemical reaction occurs automatically. No external energy needed to start chemical reaction. Thus, activation energy decreases.

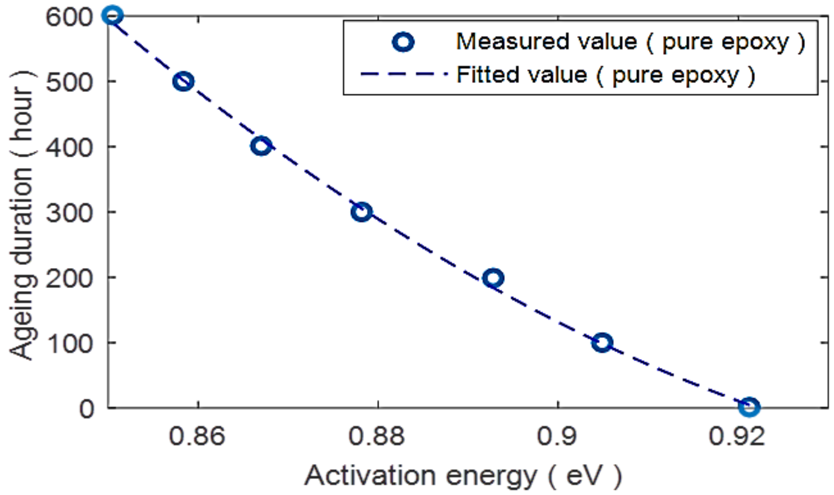

Fig. 6.18 Activation energy of pure epoxy resin at different ageing duration.

The relation between activation energy and ageing duration is f(x) – p1*(x)^2+p2*(x)+p3; 7.1

where f(x) = ageing duration, x is variable, which represents the activation energy. The coefficients are – p1 = 7.95E-08, p2 = -0.0001661, p3 = 0.9213. R-square: 0.9992, Adjusted R-square: 0.9988, RMSE: 0.0009082.

The next sample is epoxy with 1 wt.% alumina. It's Activation energy profile is shown below-Fig. 6.19 shows the variation of activation energy of epoxy resin with 1 wt.% alumina at different ageing duration. It has higher activation energy than pure epoxy resin. Because, the alumina fillers oppose the molecules or atoms to take participate in chemical reaction. Consequently, the activation energy increases in this case.

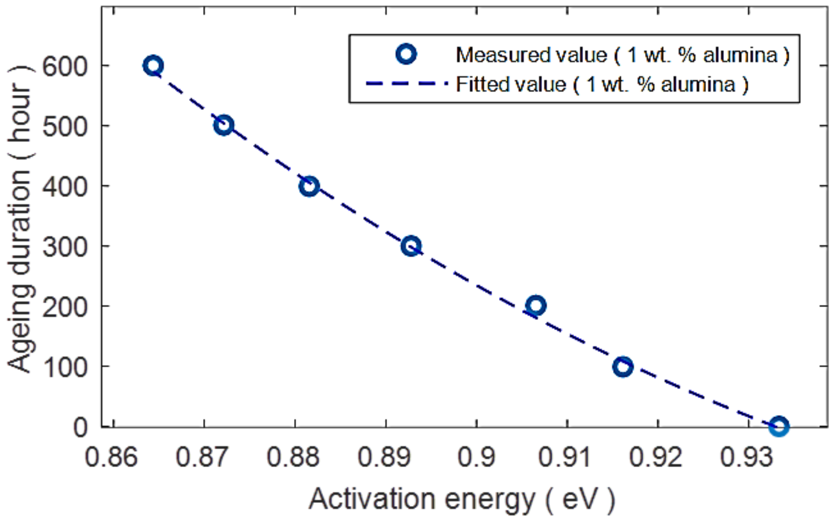

Fig. 6.19 Activation energy of epoxy resin with 1 wt. % alumina at different ageing duration

The relation between activation energy and ageing duration is f(x) = p1*$(x)^2$ +p2*(x)+p3; where f(x) = ageing duration, x is variable, which represents the activation energy. The coefficients are p1 = 6.27E-08, p2 = -0.0001518, p3 = 0.9327. R-square: 0.9978, Adjusted R-square: 0.9968, RMSE: 0.001411.

Fig. 6.20 shows the variation of activation energy of epoxy resin with 2 wt.% alumina at different ageing duration. The curve shows that it has the highest activation energy about 1 eV among all three samples. It contains more alumina fillers which strongly bound the molecules or atoms and restricts them from any type of chemical reaction. So, more energy is required for the molecules to take part in chemical reaction. Thus, activation energy increases for 2 wt.% alumina.

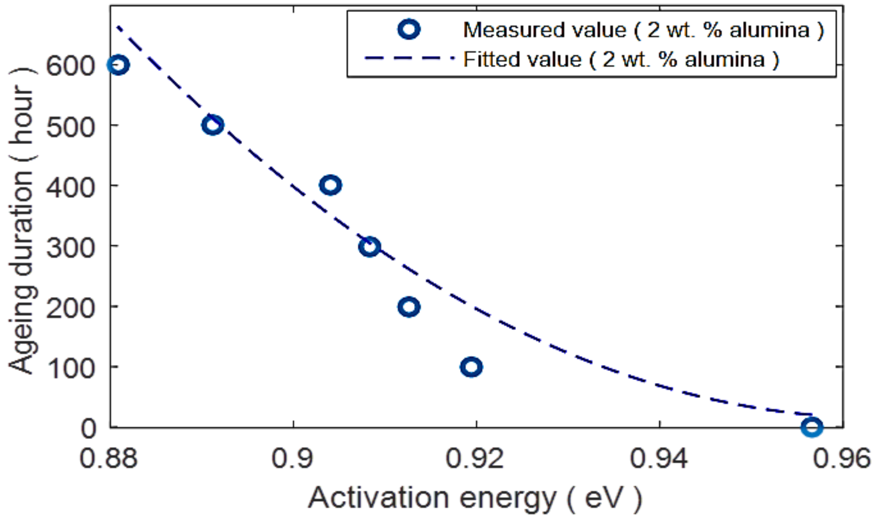

Fig. 6.20 Activation energy of epoxy resin with 2 wt. % alumina at different ageing duration.

The relation between activation energy and ageing duration is f(x) = p1*$(x)^2$ +p2*(x)+p3; where f(x) = ageing duration, x is the activation energy. The coefficients are – p1 = 1.24E-07, p2 = -0.0001789, p3 = 0.948. R-square: 0.9074, Adjusted R-square: 0.0.8611, RMSE: 0.009021.

A comparison of Activation energy among three samples is shown in table 4 as well as in fig. 6.21. Here, it is -

Table 6.4: Activation Energy of all samples

Ageing duration (hour)	Activation Energy (eV)		
	Pure epoxy resin	Epoxy Resin + 1 wt.% alumina	Epoxy Resin + 2 wt.% alumina
0	0.9213	0.9333	0.9566
100	0.9049	0.9161	0.9195
200	0.8928	0.9066	0.9126
300	0.8782	0.8928	0.9083
400	0.8670	0.8816	0.904
500	0.8583	0.8721	0.8911
600	0.8505	0.8644	0.8808

Fig. 6.21 compares the activation energies among all three samples. It shows epoxy resin with 2 wt.% alumina has higher value of activation energy than 1 wt.% alumina and pure epoxy resin for same ageing duration. For epoxy resin with 2 wt. % alumina, due to the presence of more numbers of alumina fillers, they tightly entangle the epoxy resin molecules. Alumina fillers itself are insulator. So, by adding the Alumina Nano-fillers results in increase in the energy barrier. It means the molecules require more energy as compared to pure epoxy resin and epoxy resin with 1 wt.% alumina to initiate chemical reaction in case of epoxy with 2 wt.% alumina. So, the epoxy resin with 2 wt.% alumina can offer better insulation than others two.

The graph is shown as –

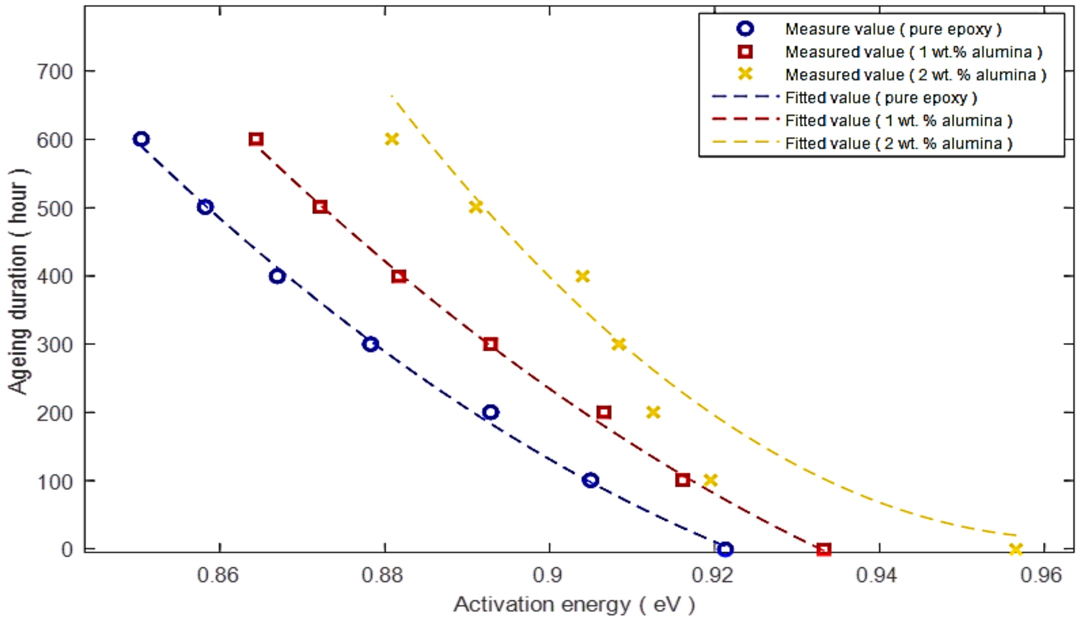

Fig. 6.21 Activation energy of the samples at different ageing duration.

6.6 VALIDATION OF DEVELOPED METHOD

In this investigation, an empirical relationship (equation 7.1) has been developed to predict the electro-thermal ageing state of the epoxy and epoxy based nano-composites material. This relationship is estimated based on the activation energy of the insulation samples. Therefore, the relation has been verified with the newly prepared test samples. For this purpose, a set of epoxy and it's nano-composites have been prepared and aged for 450 hours. Again the same PDC measurement has been performed with different temperatures. From the PDC data, the conductivity (as shown in Table 5) as well as the activation energy (Table 6) of the newly prepared test sample have been estimated. Hence, the ageing state of the test samples have been calculated from the developed empirical relationship. It can be seen from Table 7, that the calculated ageing state and the actual ageing state would be matched with an error less than 5%. Therefore, it can be considered that the proposed method has been able to estimate or sense the ageing state of the epoxy and epoxy based nano-composites.

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Table 6.5: Conductivities of newly prepared samples

Sample	**Ageing Duration (hours)**	**Conductivity (S/cm)**						
		30°C	**40°C**	**50°C**	**60°C**	**70°C**	**80°C**	**90°C**
Pure	450	3.91E-16	2.27E-15	4.28E-15	1.41E-14	2.951E-14	4.45E-14	1.31E-13
1wt% Al_2O_3	450	3.11E-16	1.69E-15	3.45E-15	1.20E-14	2.43E-14	3.59E-14	1.13E-13
2wt% Al_2O_3	450	2.37E-16	1.13E-15	2.66E-15	1.01E-14	1.94E-14	2.75E-14	9.61E-14

Table 6.6: Measured Activation energy of newly prepared samples

Ageing duration (hour)	Activation Energy (eV)		
	Pure Epoxy	Epoxy + 1wt% Al_2O_3	Epoxy + 2wt% Al_2O_3
450	0.8583	0.8720	0.8922

Table 6.7: Calculated Activation Energy and ageing state of newly prepared samples

Sample	**Ageing Duration (hours)**	**Slope**	**Activation Energy (eV)**	**Estimated Ageing Duration (hours)**	**Error (%)**
Pure	450	10010	0.8583	429	4.67
1wt% Al_2O_3	450	10170	0.8720	456	1.33
2wt% Al_2O_3	450	10410	0.8922	463	2.89

CHAPTER - 7

CONCLUSIONS AND FUTURE SCOPES

7.1 CONCLUSIONS

This experiment highlights a method to measure the Activation energy of power transformer insulation in Time Domain. Activation energy helps to evaluate the ageing duration of transformer insulation. From the results shown in this paper, the following conclusions are obtained –

i. Epoxy resin doped with Al_2O_3 improves the Activation energy, which indicates better insulation quality of the material.
ii. Conductivity of each sample reduces when alumina fillers incorporated with Epoxy resin.
iii. Polarization and depolarization current increases with ageing duration of the samples. But, their values get reduce when the concentration of alumina fillers increases.
iv. Temperature is one of the important factors during the measurement of polarization and depolarization current. It affects the conductivity and corresponding Activation energy profile also changes.

This proposed method is applied on the laboratory samples. Further, it is validated by the theoretical calculation. Therefore, it can be said that, this method can reliably be used for estimating the ageing status of the epoxy resin samples.

7.2 FUTURE SCOPES

In this experiment, alumina concentration has been taken upto 2 wt.%. This concentration may be extended to a further value and may be checked whether the increment follows the previous trends or it brings some changes from previous results.

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Dr. Arpan Kr. Pradhan
Assistant Professor,
Electrical Engineering Department
Jadavpur University
Kolkata-700032