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Assessment of Stator Winding Insulation by Spectroscopic and Thermo-Analytical Techniques

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1.0 INTRODUCTION

Over the past few decades, the progressive deterioration of high voltage machine insulation has been assessed through non-destructive techniques like measurement of Insulation Resistance, Polarization Index, Dissipation Factor, Loss Angle and Capacitance, Partial Discharge (PD) measurements, mainly for trend analysis [1-4]. The stator winding insulation deteriorates under the conditions of thermal, electrical, vibration and thermo-mechanical stresses during service. Aging process is complicated and takes place under stresses simultaneously or sequentially. Thermal aging is a chemical process leading to molecular decomposition and oxidation of organic materials resulting in decrease adhesive strength of epoxy to mica surface and also to delamination at the interface between mica and epoxy [5,6]. Delamination further aggravates under thermo-mechanical force. Small cracks are likely to be generated in epoxy rich areas due to thermal aging which could trigger electrical trees. Electrical stress causes Partial Discharge in defects, across the surface which erode stator insulation [7].

Insulation condition can also be affected by various factors such as thermal decomposed byproducts, moisture absorption, oxidation, electrolytic effects of leakage currents, attack by electrical discharges and their chemical byproducts [8]. However, the study of structural

*Central Power Research Institute, Bangalore, INDIA **PES College of Engineering, Mandya, INDIA email: nagesh@cpri.in and chemical changes that insulation undergoes during aging is limited and not fully explored and is of absolute necessity to understand the deterioration mechanisms. This paper reviews the various spectroscopic and thermo-analytical techniques that are used for characterization of materials and presents the laboratory investigations carried out to understand the structural changes that stator winding insulation under goes during aging.

2.0 REVIEW OF SPECTROSCOPIC AND THERMO-ANALYTICAL TECHNIQUES

There are several spectroscopic and analytical techniques which are used for characterization of materials and as a diagnostic tool to study the structural changes of aged/deteriorated insulation. Some of the techniques which are used for the study of structure and chemical analysis include scanning electron microscope, transmission electron microscope, X-ray diffractometer, optical microscope, optical emission spectrometer, nuclear magnetic resonance techniques, etc. Equipments used for physical and chemical properties include, differential scanning calorimeter, differential thermal analyzer, etc.

2.1 Dielectric spectroscopy

Dielectric relaxation spectroscopy is increasingly being recognized as a tool for material characterisiation. Dielectric spectroscopy measurement is essentially a dissipation factor measurement performed at multiple frequencies ranging from few milli Hz to kHz.

2.1.1 Principle of measurement

During the process of thermal aging of insulation or due to ingress of moisture, polar species are generated in the insulation as byproducts due to increased rates of chemical reactions that occur at high temperatures. The polar species, which vary in size and density with the aging, orient or move in different directions under the influence of electric field and thus contribute to the losses in the insulation. Dissipation factor tests below 1 Hz are responsive to these polar species and this response can be studied using the dielectric functions [9]. The frequency dependence has been found to be different in the different functions [10] and are based on basic equations

$$Z^* = Z' - j Z''$$
(3)

$$Y^* = Y' + j Y''$$
(4)

where ε^* is the complex permittivity, M* is the complex modulus, z* is the complex impedance, Y* is the complex admittance and ω is the angular frequency. Insulation diagnostics is based on material characterization and therefore material models are often used. Impedance models are represented in polar and rectangular co-ordinates. More often a complex capacitance model describes insulation impedance as a complex capacitance, where the imaginary part of the capacitance represents the losses. The complex capacitance model is defined as follows:

$$Z = 1/j\omega C$$
 where
 $C = C' - jC''$ (7)

Complex C:

 $C' = Re\{1/j\omega Z\}$ (8) $\Delta C' = C' + k$ (9)

 $C'' = -Im \{1/j\omega Z\}$ (10)

Where $\Delta C'$ is defined as the capacitance, C', with an arbitrary constant k.

Parameters such as capacitance, tan δ and $\cos \phi$ (Power Factor) are calculated from the measured impedance as follows:

$$C' = \text{Re} \{1/j\omega Z\}$$
(11)

$$PF = \cos \varphi = Re\{Z\} / |Z|$$
(12)

Tan $\delta = -\text{Re}\{Z\}/\text{Im}\{Z\}$ (13)

2.2 Fourier transform Infrared (IR) spectroscopy

Fourier transform infrared spectroscopy is material analysis technique which provides the structural information, compound identification. The use of infrared spectrometry for depth analysis of compositions and functional groups like carboxyl groups, carbonyl groups, hydroxyl groups and epoxy groups in stator winding insulation is increasing. IR method has depth resolution as low as 1 micrometer order and often has been used for the estimation of degraded polymeric materials.

2.3 Scanning electron microscope:

This technique is used to observe the pattern of interface during the aging process. The variation of components in insulation can be analysed by X-ray energy spectrum. The surface morphology, shape, size and arrangement of the particles making up the object that are lying on the surface of the sample can be examined.

2.4 Differential Scanning Calorimeter (DSC):

Differential Scanning Calorimetry is a technique which can be used to study the thermal transitions in crystalline, nanocrystalline and amorphous materials. DSC is a well known analytical tool widely used for the study of kinetics of curing process, reactivity and reaction mechanism of a variety of thermosetting resin systems, molding compounds, prepregs etc for the purpose of development of optimum processing parameters and optimum formulations. DSC is also used as a diagnostic tool for the detection of the cause of non-cure of mica filled epoxy during manufacturing process.

2.5 Thermo-Gravimetric Analysis (TGA)

Thermo-Gravimetric Analysis (TGA is a simple analytical technique that measures the weight loss (or weight gain) of a material as a function of temperature. As materials are heated, they can lose weight from a simple process such as drying, or from chemical reactions that liberate gasses. Some materials can gain weight by reacting with the atmosphere in the testing environment. A derivative weight loss curve can also be used to determine precisely the point at which weight loss is occuring.

2.5.1 Principle of operation

A sample of the test material is placed into a high alumina cup that is supported on, or suspended from an analytical balance located outside the furnace chamber. The balance is zeroed, and the sample cup is heated according to a predetermined thermal cycle. The balance sends the weight signal to the computer for storage, along with the sample temperature and the elapsed time. The TGA curve plots the TGA signal, converted to per cent weight change on the Y-axis against the reference material temperature on the X-axis.

2.6 Thermo-Mechanical Analysis (TMA)

Thermo-Mechanical Analysis is one of the important characterization techniques in the field of thermal analysis. Using this technique one can measure the dimensional properties of a sample when it is heated, cooled or held under isothermal conditions. The technique can be used to assess properties like softening temperatures or $T_g(s)$, melting temperatures, stress relief effects at T_g , coefficients of thermal expansion etc. TMA offers a higher degree of sensitivity as compared to DSC for the detection of T_g of highly filled materials like composites.

3.0 EXPERIMINETAL DETAILS

A study was undertaken in the laboratories of Central Power Research Institute to understand the structural and chemical changes that stator winding insulation undergoes during ageing. Spectroscopic and thermo-analytical techniques were used along with electrical diagnostic test methods to understand the structural changes. The details of experimental investigations and results are discussed in the succeeding sections.

3.1 Experimental details

The insulation investigated is a complex dielectric consisting of three components, viz. Glass tape, mica paper and binding resin. Every component has its own volume fraction, permittivity and conductivity. The experimental investigations were carried out on three types of samples viz:

- a) Resin rich epoxy mica laminates of 100 mm x 100 mm size and 2 mm thick
- b) epoxy-mica based 6.6 kV motor coils and on
- c) model stator winding bars of generator each of length 1000 mm. The sample bars insulated with 3 mm composite comprising of mica, inorganic reinforcing, epoxy bonding and impregnating materials were manufactured according to resin poor VPI process.

3.1.1 Resin rich epoxy mica laminates/ motor coils

The motor coils/laminates were subjected to accelerated thermal ageing at 200° C for 2000

hours using a thermal oven and its response to low frequency (dielectric spectroscopy) was studied on both unstressed and thermally aged samples.

3.1.2 Resin poor stator windings

The resin poor stator windings subjected to accelerated electrical, thermal and combined stress aging. A group of six bars were chosen for each stress level. The electrical aging was conducted at stress levels of 5 kV/mm and 6 kV/mm using high voltage (30 kV), high frequency (915 Hz) source. Thermal aging experiments were conducted at 170° C, 180° C and 200° C. The number of samples and duration of aging is given in Table 2.

Dielectric spectrometer model IDA 200 was used to study the low frequency response of thermally degraded laminates and motor coils. Fig. 1 shows the picture of the experimental setup that was used in this investigation. Dielectric spectrometer model IDA 200 is seen on the left while M4000 Doble make bridge (extreme right) was used for measurement of tan d at power frequency. Thermo-analytical instruments like DSC (Mettler Toledo Star system make), TGA (TA instruments, Model Q 500) and TMA (TA instruments, Model Q 400) were used to study the curing status and aging behaviour by monitoring heat and weight change, Glass Transition temperature (T_{a}) and decomposition temperatures. Properties like tan delta, capacitance, PD characteristics, etc were determined at regular intervals during aging. To study the structural/molecular changes of sound and degraded specimens FTIR spectrometer (Perkin Elmer make, Model: Spectrum 2000) instrument was used. Further, to study the surface morphology and elemental analysis of the aged specimens, SEM technique and X-ray energy spectral analysis were employed. A similar study was conducted on resin rich and resin poor system laminates.

TABLE 2									
AGING SCHEME									
	Th	ermal stress at		Electrical stress at		Combined stress			
Stress	170° C	180° C	200° C	5 kV /mm	6 kV /mm	5kV/mm, 100° C			
Number of samples	6	6	6	6	6	6			
Duration	2000 Hrs	2000 Hrs	2000 Hrs	5000 Hrs	2000 Hrs	1000 Hrs			



4

The Journal of CPRI, Vol. 5, No. 2, September 2009

4.0 RESULTS AND DISCUSSION

4.1 Resin rich epoxy mica laminates

4.1.1 Dielectric spectra of unaged specimens

In order to characterize the low frequency dielectric response of thermosetting epoxy-mica insulation, the dielectric losses were measured. Figs. 2A and 2B show the variation of tan δ and power factor (cos φ) as a function of frequency obtained on 1mm thick, 100mm x 100mm unstressed epoxy-mica laminates. It is seen from these figures that the variation of tan δ and cos φ is not significant above 1 Hz but there is a wide dispersion in values below 1 Hz. A slight increase of the dielectric loss was noticed below 1 Hz upto 0.001Hz signifying the presence of





polar species which vary in size and density contributing to the losses in the insulation. The power factor is maximum at the lowest frequency of 0.001 Hz. The maxima in the tan δ curves and the significant increase in capacitance at low frequencies (figure not shown) confirm predominant influence of interfacial polarization on the total dielectric response of epoxy-mica layer.

4.1.2 Dielectric spectra of thermally aged samples

The laminate samples were thermally stressed at 200°C for 1000 hours. Fig. 3 shows a comparative plot of the variation of loss factor as a function of frequency for selective sound and thermally aged specimens. The curves at the upper band show the variation for thermally aged specimens while lower band of curves are for that of sound specimens. The degradation can be distinctly seen with aged specimens showing higher values (tan $\delta = 1$ at 0.01 Hz) than the sound specimen (tan $\delta = 0.12$ at 0.01 Hz) and the trend similar to that of sound specimen. The dissipation factor measured at 1 kV, 50 Hz using tan δ and Capacitance bridge instrument was in agreement with the dielectric spectroscopy results. However the values measured at power frequency were much higher at higher voltages due to partial discharge activity.



Figs. 4A and 4B show plots of variation of tan δ and power factors with frequency for samples thermally stressed at 200°C for 2000 hours. From Fig. 4A it is observed that there is a significant loss in dielectric (tan $\delta = 4$ to 10 at 0.01 Hz) due to higher degree of degradation of insulation compared to samples aged for 1000 hours (tan $\delta = 1$ at 0.01 Hz).

6





4.2 6.6 kV, resin rich motor coils

Motor coils of 6.6 kV, were subjected to accelerated electrical and thermal stresses at 5 kV/mm and 200°C for a duration of 1000 hours. Dissipation factor, power factor and capacitance measurements were conducted in the frequency range 1 mHz to 1 kHz. Figs. 5A to 5C show the variation of dissipation factor, power factor and capacitance as a function of frequency. The trend observed is similar to the trend observed in case of laminates. One of the coil which failed during power frequency tan δ measurement at 3 kV, has shown a different trend when measured at a very low voltage 140 V_{rms} and variable frequency as seen from Fig. 5A to 5C. In case of a failed coil, the maxima in tan δ is observed at 10 Hz and decreases below 10 Hz.

The shape of frequency dependence of the loss factor can be interpreted as follows. The dipole processes is increasingly overlapped by the effect of conduction in the resin. At still lower frequencies, the barrier effect of the lower conductivity of the organic component manifests itself. Charge is accumulated at the interface and this causes redistribution of the field strength in the composite dielectric and decrease in the resulting conduction







4.3 Resin poor stator windings

4.3.1 Dissipation factor measurements

Dissipation factor measurements were made on all the stator winding bars using Tettex Schering Bridge. The initial base values of all the bars varied from 0.5% to 0.6% at 2.2 kV and from 0.5% to 1.0% at 11 kV, well below the specified limit. The tan δ tip-up values [(0.6 Un-0.2Un)/2] ranged from 0.01% to 0.08%. For thermally aged samples, stressed at 170°C and 200°C for 2000 hours, the tan δ tip-up values [(0.6 Un-0.2Un)/2] have increased from 0.6% to 1.0% for samples aged at 100°C and from 0.85% to 1.7% for 200°C aged samples (Details are published elsewhere) [11].

4.3.2 Partial discharge measurements

Partial discharge magnitude was measured using Lemke Diagnostic PD detector. The PD magnitudes varied from 375 pC to 15000 pC for 443 K (170°C) aged samples and from 110 pC to 20000 pC for 473 K (200°C) aged samples.

4.3.3 Fourier Transform Infra red spectroscopy

Small specimens were cut out and used in this study. In case of VPI samples, no significant change in FTIR spectra of unaged and aged samples was observed. The FTIR spectra of resin rich samples, unstressed and thermally aged, are shown in Fig. 6. It is seen from this figure that for an unstressed specimen the dominant peaks are observed at 3449 cm⁻¹, 2926 cm⁻¹, 1610 cm⁻¹, 1509 cm⁻¹, 1454 cm⁻¹,



1245 cm⁻¹, 1026 cm⁻¹, 829 cm⁻¹. FTIR studies on epoxy reported [21], indicate that there is no carbonyl band in the epoxy spectrum, but the aromatic ring at 1510 cm⁻¹ is very strong. Also the 1610 cm⁻¹ ring breathing mode is also relatively strong. The C-O stretch is strong and appears as two bands, a broad band with a maximum near 1247 cm⁻¹ and a narrower and slightly weaker band with a maximum near 1182 cm⁻¹. Significant intensity is also seen in the aromatic C-H at 830 cm⁻¹. Comparing the spectra in the present investigations with spectra of epoxy reported in literature, the absorption peaks observed at 1609 cm⁻¹, 1508 cm⁻¹, 1242 cm⁻¹, 829 cm⁻¹ are in close agreement. Comparing the spectra of thermally stressed samples with unstressed sample, it is seen that a new peak at 1720 cm⁻¹ resulting

8

from carbonyl (C=O) group is observed. This probably would suggest that the molecular structure of epoxy on oxidation results in carbonyl (C=O) and hydroxyl (OH) group.

4.3.4 SEM investigation

The elemental study carried out using SEM and Energy Dispersive Analysis X-ray (EDAX) technique relate essentially to mica. Mica is a complex alumino silicate mineral, while Al, K and Si are the predominant constituents; the material had traces of Fe, Na, Ti, Ca, Mg etc. The element composition (%) are summarized in Table 3. Figs. 7A to 7H show the pictorial representation of sound and aged samples. It is observed that changes in status of samples before as well as after aging is more on account of heterogeneity of mica/epoxy/glass composite.







FIG. 7D THERMALLY AGED AT 200°C FOR 11 WEEKS

The Journal of CPRI, Vol. 5, No. 2, September 2009



9



FIG. 7 SEM MICRO PICTURES (MAG. 200 X) OF SOUND AND AGED SPECIMEN

TABLE 3								
ELEMENT COMPOSITION (%) OF DEGRADED SAMPLES								
Aging conditions	Element in %							
	Al	Si	K					
Unaged sample	24.28	34.06	12.88					
Thermally aged at 443 K (170 °C) for 8 Weeks	17.45	20.86	7.83					
Thermally aged at 473 K (200 °C) for 9 Weeks	11.58	12.92	3.35					
Electrically aged at 5 kV/mm for 4000 Hrs	9.56	10.91	2.32					
Electrically aged at 6kV/mm for 2000 Hrs	8.78	9.23	1.42					
Combined aging, 5 kV/mm (E) and 373 K (100 °C) (T) for 500 Hrs	9.48	9.23	1.42					
Combined aging, 5 kV/mm (E) and 373 K (100 °C) (T) for 1000 Hrs	8.75	8.98	1.54					

5.0 CONCLUSIONS

This paper presents a bird's eyeview of the various spectroscopic and thermo-analytical detection methods. The use of dielectric spectroscopic technique has resulted in a better knowledge of the electrical properties of thermally degraded epoxy-mica stator winding insulation. The dielectric response exhibited by of thermally aged epoxy-mica insulation at lower frequencies shows the predominant influence of interfacial polarization on the total dielectric response of epoxy-mica layer. This method could be possibly used to evaluate the condition of the bulk of the insulation and its degree of aging.

Some important conclusions drawn from the laboratory investigations are

- 1. FTIR spectra of unstressed and aged specimens show a new peak at 1720 cm⁻¹ resulting from carbonyl (C=O) group in case of resin rich aged specimen.
- 2. The glass transition temperature as determined by DSC and TMA indicated some post-curing effect.
- 3. From the TGA thermo-gram, it was found that there was no change in total weight loss of unaged and aged specimen.
- 4. The decomposition temperatures of the specimens though are not that distinct from the normal curves, the derivatives precisely show the peaks at which decomposition is occurring.
- 5. SEM micro-pictures show a significant difference in surface morphology of samples, attributable to heterogeneity of the composite.

The deterioration observed in electrical parameters is mainly due to presence of voids and their further enlargement during thermal and electrical aging.

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