Chapter 4
Experimental set-up and procedure

4.0 INTRODUCTION:
The deterioration of stator winding insulation under accelerated electrical and thermal ageing are investigated. Two types of Class F insulation (155° C class) system viz. Epoxy–Mica: Resin rich and Epoxy-Mica: Resin poor systems are studied. Sample Generator bars and laminates made of resin poor epoxy mica and motor coils and laminates made from resin rich epoxy mica insulation system are evaluated in the present investigation. The dielectric diagnostic techniques like IR/PI, tan δ and Capacitance measurements both in time domain & frequency domain are carried out. Also PD measurements are made for insulation condition assessment. In addition spectroscopic techniques like FTIR, x-ray energy spectrum analysis for the study of structural / chemical changes, Recovery voltage measurements for dielectric response were used for better understanding of the dielectric processes. To supplement the results, Thermo analytical techniques like DSC, TGA, TMA are used for the understanding of chemical kinetics.

Measurements are also conducted on a few class B (Bitumen/Asphalt–Mica) and class F (epoxy–mica) insulation of electrical machines which are in service. Based on Insulation theory and laboratory results using electrical techniques, studies were also carried out on machines like generators and motors which are in service.

This chapter presents the details of sample preparation, experimental setup and type of electrodes and experimental arrangement used along with measuring instruments and their accuracy of measurements.

4.1 DETAILS OF EXPERIMENTS
Experiments are conducted on model Generator stator bars and Motor coils. In addition, experiments were also carried out on epoxy mica–glass laminates and stator winding of in-service high voltage generators and motors.
4.1.1 LABORATORY INVESTIGATION
The stator winding insulation prepared under resin poor and resin rich process is used for investigation. The model bars of generator stator winding (resin poor), stator winding of motor coils (resin rich) and Epoxy mica laminates (both resin rich and resin poor) systems are subjected to accelerated electrical, thermal and electro-thermal stress ageing. The insulation curing status and behavior on ageing was studied by thermo analytical techniques like differential scanning calorimeter (DSC), thermo gravimetric analysis (TGA) and thermo mechanical analysis (TMA). Dielectric properties like dissipation factor, capacitance, partial discharge, polarization currents etc. were measured at regular intervals during ageing. The structural and chemical changes that could have occurred during ageing were examined using techniques like Fourier Transform Infrared Spectrometer (FTIR), Scanning Electron Microscope (SEM), and X-ray energy spectral analysis. The glass transition temperatures of fresh (unaged) and thermally aged samples are determined by DSC and TMA. Dielectric response studies using low frequency techniques like Dielectric spectroscopy and Recovery voltage measurement are conducted on motor coils and laminates.

4.2 DETAILS OF SAMPLES USED FOR EXPERIMENTS
4.2.1 Resin poor Epoxy-Mica Generator sample bars
The experimental investigations are carried out on stator winding bars of generator each of length 1000 mm. The stator bars rated 11 kV and insulated with class F ground wall insulation comprising of mica, inorganic reinforcing, epoxy bonding and impregnating materials were manufactured at one of the leading manufacturers according to resin poor Vacuum Pressure Impregnation process. The B-stage pre-polymer tape comprising uncalcined muscovite mica paper bonded to glass cloth with epoxy resin is about 0.18 mm thick. The use of uncalcined muscovite mica paper bonded with adequate resin produces a tape of sufficient porosity for complete impregnation of insulation layer within air pockets after curing. The composition of tapes is glass fabric: 32 ± 2 gm/m² and resin content 12 ± 3 gm/m², thickness 3 mm and details are given in Table 4.1. The B stage insulation wrapped on the conductor is hot pressed, vacuum impregnated and
cured. The dimension of generator stator bars manufactured for the present study is shown in Figure 4.1. The Metallic bar has length of 1000 mm, width of 45 mm and height of 15 mm.

Table 4.1 : Specification of Resin Poor Epoxy-Mica

RESIN POOR : MICA : (CALCINED MICA PAPER + EPOXY RESIN)
Thickness: 3 mm
Composition: MICA paper 120 ± 7 (g/m²)
Glass fabric 32 ± 2 (g/m²)
Resin content 12 ± 3 (g/m²)

<table>
<thead>
<tr>
<th>Thickness</th>
<th>MICA paper</th>
<th>Glass fabric</th>
<th>Resin content</th>
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<tbody>
<tr>
<td>3 mm</td>
<td>120 ± 7</td>
<td>32 ± 2</td>
<td>12 ± 3</td>
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A total number of 42 sample bars were obtained from the generator manufacturer, a leading PSU. These samples were used for ageing studies under electrical, thermal and electro thermal stresses and the structural changes due to ageing were examined using spectroscopic & thermo analytical techniques and dielectric tests.

4.2.2 Resin Rich Epoxy-Mica Laminates

The details of Resin rich Epoxy-Mica insulation used in the present study is given in the Table 4.2

Table 4.2 : Specification of Resin rich Epoxy - Mica

RESIN RICH : MICA : (CALCINED MICA PAPER + EPOXY RESIN)
Thickness: 1 mm, 2 mm and 3 mm
Composition: MICA 230± 15 (g/m²)
Glass fabric 35 ±3 (g/m²)
Resin content 145 ±15 (g/m²)
Volatile content % ≤ 0.8

<table>
<thead>
<tr>
<th>Thickness</th>
<th>MICA</th>
<th>Glass fabric</th>
<th>Resin content</th>
</tr>
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<tbody>
<tr>
<td>1 mm</td>
<td>230± 15</td>
<td>35 ±3</td>
<td>145 ±15</td>
</tr>
<tr>
<td>2 mm</td>
<td>230± 15</td>
<td>35 ±3</td>
<td>145 ±15</td>
</tr>
<tr>
<td>3 mm</td>
<td>230± 15</td>
<td>35 ±3</td>
<td>145 ±15</td>
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</table>

Samples are made using B grade, pre polymer tapes consisting of mica paper thoroughly impregnated with electrical grade modified epoxy resin. The muscovite uncalcined mica paper is about 0.26 mm thick. The composition of tapes is glass fabric: 35± 3 gm/m², resin content: 145±15 gm/m² and volatile content ≤ 0.8%. The uncured tapes were layered to get the required thickness.
by machine hot pressing under tension of 40 to 60 N and cured at 180 °C for 2 hours. Samples of dimension 150 mm x 150 mm were cut from sheet of thickness 1 mm, 2 mm and 3 mm.

4.2.3 Resin Rich Epoxy-Mica 6.6 kV Motor coils

6.6 kV, Class F motor coils (10 coils) shown in Figure 4.2 are used for studies for low frequency technique on both unstressed and thermally stressed coils. 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 coils are subjected to accelerated thermal aging using thermal oven and its response to low frequency (dielectric spectroscopy) are studied on both unstressed and thermally aged samples.

The samples studied, ageing scheme, techniques and measured parameters on resin rich and resin poor samples are shown in the flow chart 4.1.
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Flow chart 4.1: Samples, ageing scheme and properties investigated under present research studies
4.3 MEASURING SETUP & PROCEDURE

4.3.1 Electrode configuration and arrangement for stator bars and coils.

The slot portion of generator / motor coil stator was wrapped with thin aluminum foil half overlapped tightly ensuring that there are no air gaps to the possible extent. It is firmly held with a thin fuse wire and insulation tape wrapped around it (Figure 4.2). The copper conductor of the stator bars were connected to high voltage point during measurements and aluminum foil was connected to measuring lead or firmly earthed.

4.3.2 Test setup for Dielectric response studies

The experimental investigations are carried out on epoxy-mica glass laminates having dimension 150 mm x 150 mm and thickness of 3 mm. The laminates comprised of mica with organic reinforcing, bonding and impregnating materials. The sample laminate surface was coated with fine semi conducting paint in the shape of a circular disc at the centre. This circular coating acts as electrode ensuring uniform contact with the sample surface. The laminate sample is placed between two round shaped Bruce profile [79] brass electrodes of 100 mm diameter as shown in figure 4.3.

4.3.3 Polishing and cleaning of electrodes;

In the present study, the electrodes have been polished using zero grade Carborundum paper. They are then highly polished by buffing after applying metal polish. The electrodes are then degreased, washed with acetone & distilled water and dried in a dessicator.

4.4 HIGH VOLTAGE AC SOURCE:

The accelerated ageing can be achieved by stressing the sample at higher voltages or by applying the rated voltage at higher frequencies. The second method results in rapid failure and is more useful for accelerated ageing studies. In the present ageing studies, an high voltage ac source of 30 kV, frequency of 915 Hz available at research centre of M/s BHEL, Haridwar was used for conducting accelerated electrical ageing studies. Another test
transformer rated 30 kV, 100 mA and partial discharge free up to 25 kV (Figure 4.4) available at CPRI, Bangalore was used for measuring the dielectric parameters. The measured PD level of this transformer was below 5 pC at rated voltage. The test voltage was gradually raised or lowered using a suitable dimmerstat. The test voltage was gradually increased from 20% of rated voltage (2.2 kV) to 120% of rated voltage (13.2 kV) and at each step dielectric parameters were obtained. The test voltage was recorded by using a calibrated voltage divider and voltmeter. The transformer was calibrated by measuring the ratio of output and input voltage with calibrated meters.

![Epoxy glass laminate between Bruce profiled electrodes](image)

**Figure 4.3** Epoxy glass laminate between Bruce profiled electrodes

### 4.5 MEASURING APPARATUS:
Central Power Research Institute is an ISO 9000 and ISO/NABL 17025 accredited laboratory and hence all the measuring instruments are periodically calibrated and calibration has traceability to international standards. The calibration details of the instruments used in the present investigation are given in annexure ‘A’.
4.5.1 **Insulation analyser**
An AVO make insulation analyzer shown in the Figure 4.5 was used for the measurement of Insulation resistance and Polarisation index. The analyzer has provision to vary voltage in steps of 500 volts up to 5000 volts. The test voltage selected was 2.5 kV dc as per IEEE 43 standard guidelines [7]. The polarization index (which gives ratio of Insulation resistances at 10 minute and 1 minute) is graphically displayed on the screen.

4.5.2 **Schering Bridge**
Dissipation factor measurements was made on all the stator winding bars at power frequency 50 Hz using Tettex Schering Bridge shown in Figure 4.6. The stress grading treatment on the coil end windings was avoided using guard electrode during measurements. Tan δ and capacitance measurements were recorded in steps of 2.2 kV up to 11 kV for generator sample bars and in steps of 1 kV up to 5 kV for epoxy mica laminates. The dissipation factor measurements were carried out in accordance with IEEE-286-2000 [8] guidelines.

4.5.3 **Partial discharge measuring system**
Partial Discharge detector (Lemke diagnostic, GmbH, Type LDD 5U) used in this study is shown in Figure 4.7. It is used in conjunction with a standard capacitor of 35 nF, 50 kV. The PD measurements were conducted at voltages
8.8 kV, and 11 kV on generator sample bars as per IEEE 1434 – 2000 [10] guidelines. The parameters like peak PD magnitude (Qm), inception and extinction voltages were recorded.

4.5.4 Dielectric Response studies
To study the dielectric response of thermally degraded epoxy – mica resin rich and resin poor systems, both dielectric spectroscopy and recovery voltage measurements were adopted.

4.5.4.1 Dielectric spectrometer
Dielectric spectrometer Programma make, model IDA 200 shown in Figure 4.8 was used for dissipation factor measurement at multiple frequencies ranging from a few mHz to kHz. The instrument measures impedance at a specific frequency and amplitude. Parameters such as capacitance, tan δ and cosϕ (power factor) can be measured at various frequencies. The IDA 200 Instrument for diagnostics of Insulation system has following features

- Test signal: 0-200 Vpeak 0 – 50 mApeak
- Frequency: 1000 Hz – 0.001 Hz
- Sample range: 10 pF – 100 µF
- IDA 200 software running on Windows NT 4.0 Embedded operating system

4.5.5 Recovery voltage meter
Automatic Recovery voltage meter, model RVM 5462 Tettex make shown in Figure 4.10 was used. The instrument has a built in test voltage source which can be varied from 50 volts to 2000 volts dc with an accuracy of ± 0.2 %. In the present investigation a test voltage of 2000 volts dc was selected (as per Instrument specification). The charging (tc) and discharging time (td) ranged from 10 ms to 19,999 secs. The charging times selected in each RVM cycle were 0.02, 0.05, 0.10, 0.2, 0.5 .....upto 2000 seconds.

4.5.6 Thermo analytical instruments
Thermo analytical instruments like DSC (Mettler Toledo Star system make) shown in Figure 4.11, TGA (TA instruments, Model Q 500) and TMA (TA
instruments, Model Q 400) shown in Figure 4.12 were used to study the curing status and ageing behaviour for monitoring heat, weight change, Glass Transition temperature (Tg) and decomposition temperatures.

4.5.6.1 Differential scanning calorimeter
The differential scanning calorimeter is an instrument for measuring glass transition temperatures (Tg). Small samples are cut from generator bars or epoxy-mica laminates and powdered. The powder sample of 5 to 10 mg is mixed with sodium bromide to make small pellets of 1 cm dia and thickness of 1 mm. The sample is then placed in the purge cell of the test chamber. The cell is purged with inert gas at 10 to 50 ± 5 mL/min. Preliminary thermal cycle of heating is performed at the rate of 10 °C/min under inert purge gas atmosphere from 50 °C below to 30 °C above melting point. The Tg is determined as per the guidelines given in the standard ASTM 3417 [29].

4.5.6.2 Thermo gravimetric analysis
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 adjusted to zero 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 percent weight change on the Y-axis against the reference material temperature on the X-axis.

4.5.6.3 Thermo mechanical analyser
This test method used a thermo mechanical analyzer to determine Tg and also the linear thermal expansion of solid materials at a constant heating rate. The initial specimen length in the direction of the expansion test to ± 25 micro metre is measured at 20 °C to 25 °C. The specimen is placed in the specimen holder under the probe. The temperature sensor is placed in contact with the specimen as closely as possible. The specimen is heated at a constant heating rate of 5°C/min over the desired temperature and the changes in the specimen length and temperature are recorded. The
evaluation is done as per the guidelines given in ASTM E 831-03 standard [31].

4.5.7 Spectroscopic techniques
To study the structural / molecular changes of sound and degraded specimens, FTIR spectrometer (Perkin Elmer make, Model: Spectrum 2000) instrument shown in Figure 4.13 was used. Further, to study the surface morphology and elemental analysis of the aged specimens SEM technique and X-ray energy spectral analysis was done using Scanning Electron microscope shown in Figure 4.14. Similar study was conducted on resin rich and resin poor system laminates. For FTIR & SEM studies samples in the form of powder samples or cut pieces taken from the bar samples are used.

4.6 AGEING STUDIES:
The samples were subjected to electrical, thermal and electro-thermal ageing. The sample bars were divided into different groups for thermal, electrical and combined electro-thermal stress ageing studies. A group of six sample bars (minimum 5 samples as per IEEE Standard 483 guidelines) [80] were chosen for each stress level to conduct accelerated electrical, thermal and combined electro-thermal ageing separately. All ageing studies were conducted at M/s BHEL research laboratory, Haridwar.

4.6.1 Ageing scheme
The actual electrical stress of Class F insulation is typically about 3 kV/mm (rms) at 50 Hz. Higher stresses are necessary for any study of long term ageing and life time of practical insulation systems whose working life at normal stresses is a matter of years. Studies carried out by Izeki et al [81, 82] at frequencies 50 Hz, 500 Hz, 1000 Hz, 1500 Hz and 3000 Hz have revealed that linearity of the frequency acceleration is almost valid till a frequency level of 3000 Hz. However Arnold Wichmann [83] has experimentally concluded that with insulation thickness of 2 to 3 mm, the test frequency should not exceed 1000 Hz to avoid the undesirable side effect like local overheating. In formulating ageing scheme and experimental schedule, the important
considerations are the duration of the experimentation, cost of running ageing experiments and the stress acceleration factors. Also care is taken to choose the accelerating factors in such a way as to prevent violation of the condition that the Weibull parameter becomes invariable with respect to stress. The normal operating stress of stator winding of high voltage machine is 3 kV/mm. In the present study, 5 kV/mm, 915 Hz and 6 kV/mm, 915 Hz were chosen for electrical stress ageing studies. As the thickness of insulation was 3 mm, actual voltages applied were 15 kV & 18 kV. Three fixed temperatures of 170 ºC, 180 ºC and 200 ºC were chosen for thermal ageing studies. Combined electrical-thermal ageing was conducted at 5 kV/mm, 915 Hz and 100 ºC.

The electric stress levels of 5 kV/mm, 915 Hz and 6 kV/mm, 915 Hz were chosen on the grounds that changes in deterioration mechanism occurred beyond 6 kV/mm. Other researchers Kim Y.J, and Nelson J.K [35] have chosen similar stress levels like 5.4 kV/mm (50 Hz) and 6.5 kV/mm (50 Hz) in their ageing studies. Ageing temperatures in the range of 120 – 200 ºC are recommended as per IEEE 434 [80] guidelines which are sufficient to cause accelerated ageing for epoxy –mica stator winding insulation. A minimum of three temperatures are recommended [80, 81]. In the present study three temperatures 170º C, 180º C and 200º C are considered and the duration of the thermal ageing was restricted to 2000 hours over the range of temperatures used considering the likely effect of considerable ageing. The number of samples and duration of ageing are given in Table 4.1.

<table>
<thead>
<tr>
<th>Table 4.1</th>
<th>Ageing scheme</th>
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<td>Stress</td>
<td><strong>Electrical stress</strong></td>
</tr>
<tr>
<td>Stress</td>
<td>5 kV /mm, 915 Hz</td>
</tr>
<tr>
<td>Number of samples</td>
<td>6</td>
</tr>
<tr>
<td>Sample bar nos.</td>
<td>1 - 7</td>
</tr>
<tr>
<td>Duration (915 Hz)</td>
<td>5000 Hrs</td>
</tr>
<tr>
<td>Equivalent no. of 50 Hz ageing hours</td>
<td>90,000 Hrs</td>
</tr>
</tbody>
</table>
Figure 4.5 Insulation Tester
Figure 4.6 Schering Bridge
Figure 4.7 Partial discharge detector
Figure 4.8 Dielectric spectrometer
Figure 4.9 Dielectric loss analyser
Figure 4.10 Recovery voltage meter
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Figure 4.11  Differential scanning calorimeter

Figure 4.12  Thermo mechanical analyser

Figure 4.13  Fourier transform infrared spectrometer

Figure 4.14  Scanning Electron microscope
4.6.2 Electrical ageing

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. Figures 4.15 and 4.16 show experimental setup for ageing studies on generator bars.

4.6.3 Thermal ageing

Ageing temperatures are to be in the range of 120 - 200 °C and minimum three temperatures are recommended [80, 84]. In the present investigation thermal ageing were conducted at 170° C, 180° C and 200° C. As mentioned in the previous section a group of six coils were subjected to thermal ageing using ovens at pre set temperatures of 170° C, 180° C and 200° C respectively for a duration of 2000 hours. Figure 4.16 shows the general view of the test set up for measurement of dielectric response.

Figure 4.15 . A view of laboratory setup at M/s BHEL, Haridwar for ageing studies of high voltage generator bars
4.6.4 Combined electrical & thermal stress ageing:
The combined electro- thermal ageing studies were carried out using high voltage high frequency (915 Hz) ac source with thermal ageing ovens. The sample bars were placed in ageing oven which has provision for applying high voltage to the samples through the bushings fitted to the oven. The samples were taken out of the oven during measurements. The stress level chosen was 5 kV/mm, 915 Hz and 100°C. This temperature was chosen to provide a representative elevated temperature ageing environment but was not high enough to generate significant thermal ageing in the absence of synergetic effect. Combined ageing studies were carried out for a duration of 1,000 hours (equivalent to 18000 hours of 50 Hz ageing) and reaching the end point criteria. This is normal practice adopted among the researchers world wide [35,71,76].

4.7 Summary of data acquisition
The experimental investigations were carried out on model stator winding bars of generator each of length 1000 mm. The sample bars were insulated with 3 mm composite comprising of mica, organic reinforcing, bonding and impregnating materials. They were manufactured according to resin rich process and subjected to accelerated electrical, thermal and combined stress aging. A group of six coils were used for each stress level. The electrical aging was conducted at stress levels of 5 kV/mm, 915 Hz and 6 kV/mm, 915
Hz using high voltage (30 kV), high frequency (915 Hz) source. Thermal ageing experiments were conducted at 170° C, 180° C and 200° C. Properties like tan δ, capacitance, PD characteristics, etc were determined at regular intervals during ageing. Thermo analytical instruments like Differential Scanning Calorimeter, Thermo Gravimetric Analyser and Thermo Mechanical Analyser were used to study the chemical kinetics: weight change, Tg, and decomposition temperatures. To study the structural / molecular changes of sound and degraded specimens Fourier Transform Infra Red spectrometer instrument was used. Further, to study the surface morphology and elemental analysis of the new and aged specimens, SEM technique and X-ray energy spectral analysis were used. The evaluation techniques like tan δ and capacitance measurements in frequency domain and recovery voltage measurements were adopted for the study of dielectric response. The results are presented and discussed in chapter 5.