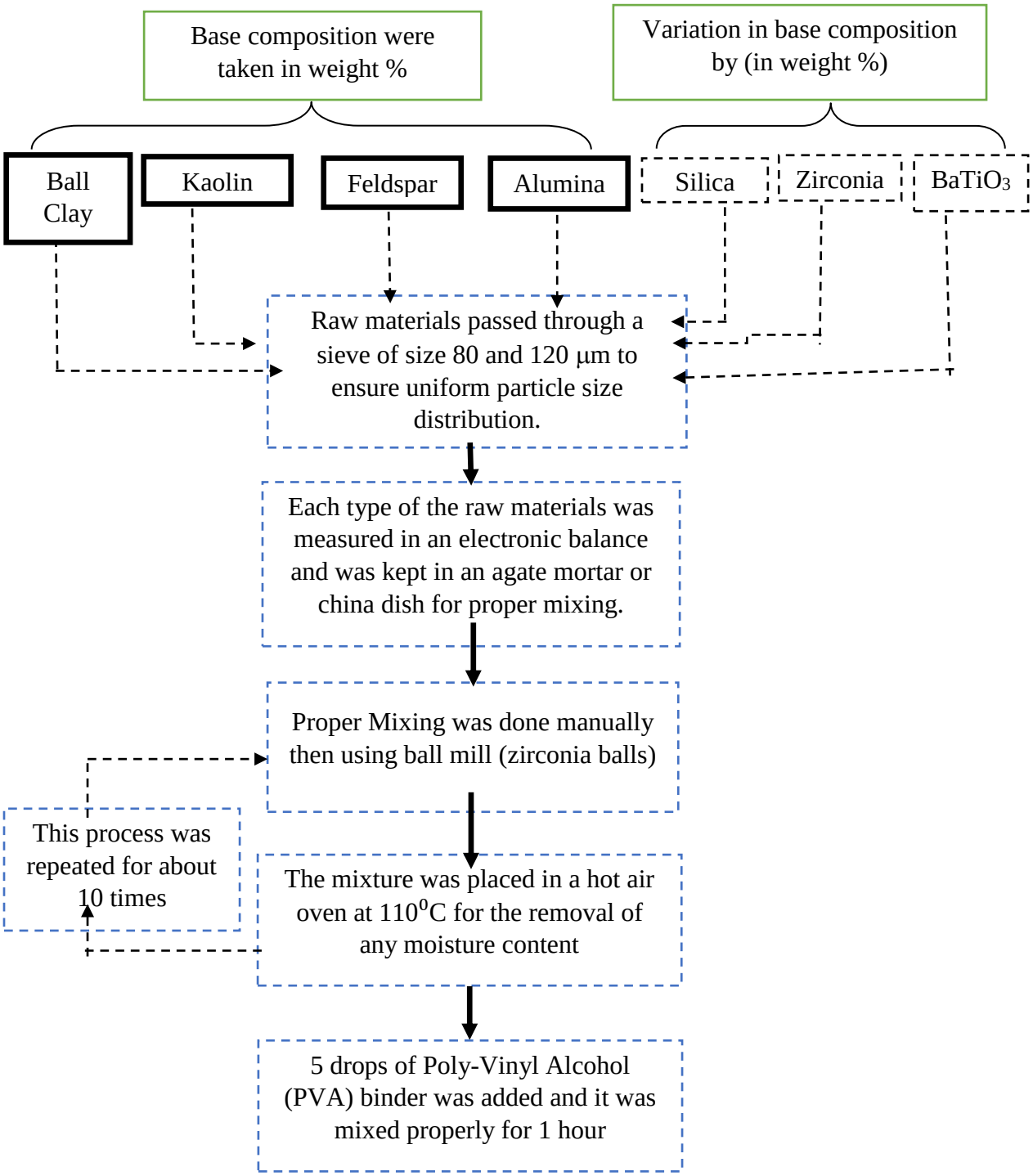
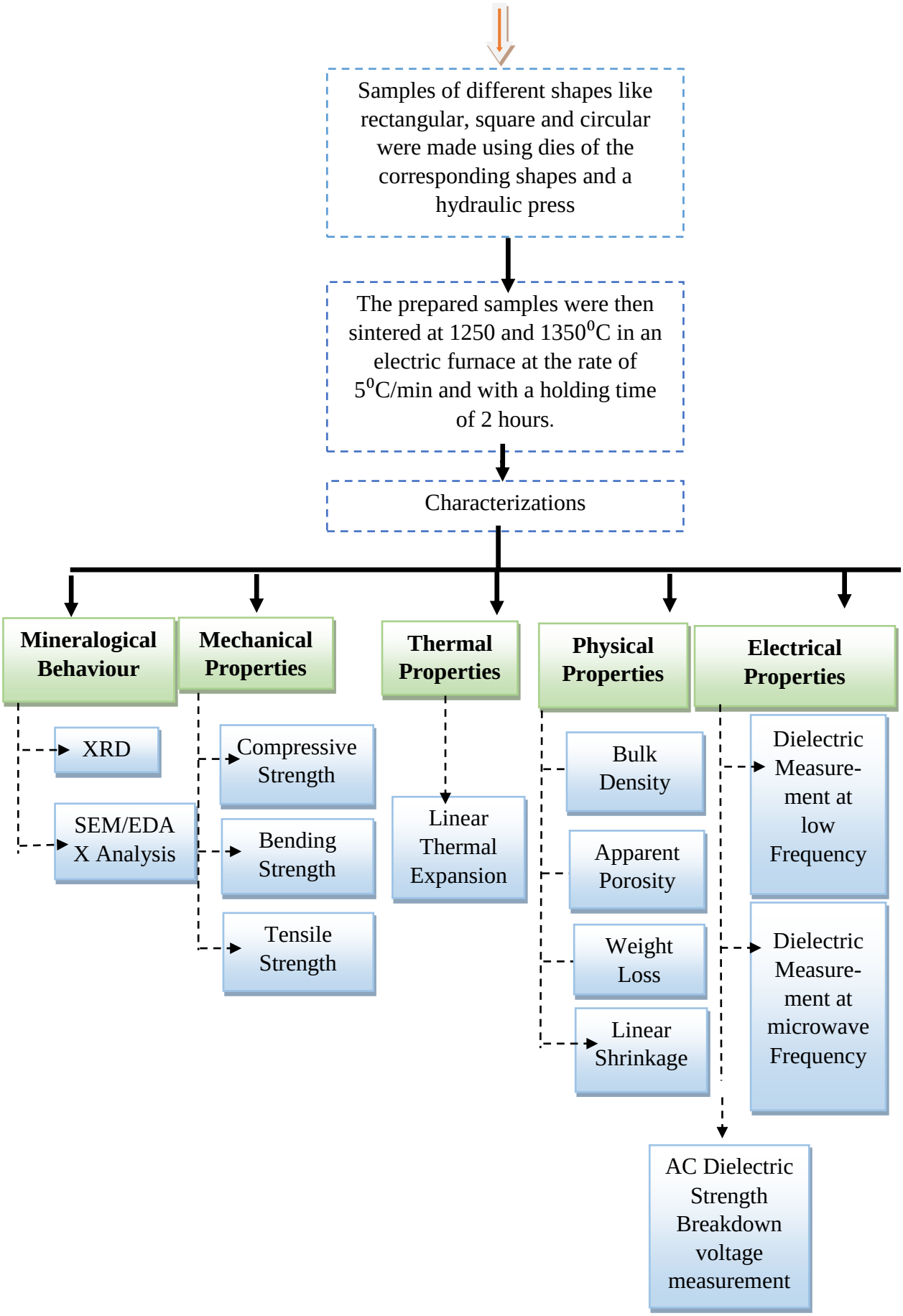
CHAPTER 2

Experimental Techniques, Material and Equipment Requirement

2.1 Flow Chart




Samples of different shapes like rectangular, square and circular were made using dies of the corresponding shapes and a hydraulic press
The prepared samples were then sintered at 1250 and 1350^{0}C in an electric furnace at the rate of 5^{0}C/min and with a holding time of 2 hours.
Characterizations
Mineralogical Behaviour
Mechanical Properties
Thermal Properties
Physical Properties
Electrical Properties
XRD
SEM/EDAX Analysis
Compressive Strength
Bending Strength
Tensile Strength
Linear Thermal Expansion
Bulk Density
Apparent Porosity
Weight Loss
Linear Shrinkage
Dielectric Measure-ment at low Frequency
Dielectric Measure-ment at microwave Frequency
AC Dielectric Strength Breakdown voltage measurement

2.2 Procedure

- For the preparation of ceramic porcelain insulator (CPI) composition the different raw materials say base composition like ball clay, kaolin, feldspar were taken and doing a variation in base composition by Silica (SiO_2), Alumina (Al_2O_3), Zirconia (ZrO_2) and Barium Titanate ($BaTiO_3$) were considered.
- They were passed through a sieve of 80 and 120 μm microns size so that the particle size is homogeneous and the larger particles are separated out.
- Prepare 100 gms of each composition was made by measuring each of the raw material with an electronic balance.
- The raw materials were taken in a china crucible, and it was mixed properly with a crushing stone for nearly 1 hour so that all the particles are homogeneous. It was then kept inside a hot air oven at a temperature of 110^0C for 1 hour so that any remaining moisture and volatile matter can be discarded from the composition and there is a minimum impurity.
- This process is repeated for around 8 to 10 times to ensure the proper mixing of the particles.
- Homogeneous mixing is achieved by yttrium stabilized zirconia ball of 2:1 weight ratio was used for mixing the materials for 30 minutes in a ball mill to reduce the particle size. To obtain the homogeneity in particle sizes, above procedures had been repeated up to 8 to 10 times.
- Four drops of 5% polyvinyl alcohol (PVA) is used as a binder in prepared composition and again mixed up to 30 minutes.
- Prepared compositions were compacted by hydraulic press machine with a pressure load of 160 MPa to make testing pellets (samples).

- Moulds of Different shapes were used to make different shaped samples using a hydraulic press. Four types of dies were taken: rectangular pallets having a dimension (40 mm X 10 mm X 10 mm) b) Square (25 mm X 25 mm) c) Circular (15 mm and 10 mm diameter).
- Each prepared pellets are having different compositions sintered at 1250°C and 1350°C, using sintering rates of 5°C per minute with a soaking period of 2 hours.
- After sintering the samples, different characterization and measurements were done. Various techniques and instruments were used to investigate the physical, mechanical, thermal, and morphological characteristics of sintered samples, (shown in section 2.1).

2.3 Equipment Requirement

Various equipment has been used to performing various tests, i.e., Physical, Mechanical and Electrical properties of the manufactured ceramic porcelain insulator.

2.3.1 Electronic Balance

An electronic balance is used to measure small masses range shown in Figure 2. 1. The balance which we used in the laboratory for weighing purposes has a maximum range of 200 gram. The balance has a round platform or pan on which samples are to be kept for weighing. Electronic balances can weight sample to very high precision, up to three or four decimal places. The pan or platform is inside a transparent enclosure with doors made up of fiberglass so that dust does not collect and air current inside the room does not manipulate the balance's operation. This enclosure is called a draft shield.

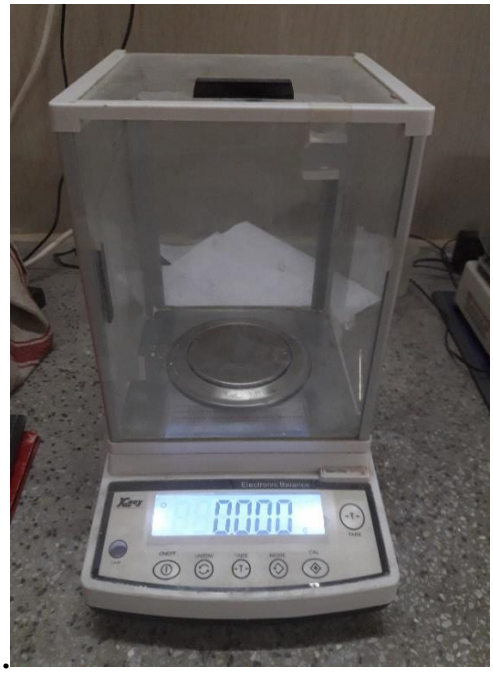


Figure 2. 1: Electronic balance for weight measurements of the powder materials.

The sample to be weighed must be in room temperature to prevent natural convection from forming air currents inside the chamber. The balance measures the force that acts

downwards on the pan. This force is converted to an electrical signal and displayed on a digital display.

2.3.2 China Crucible

Figure 2. 2, a crucible is a container which can withstand very high temperatures, and they are also used to store, mix and crush raw material powder samples inside it with the help of grinding stone.

Figure 2. 2: China crucible.

2.3.3 Hot air Oven

Hot air ovens are an electrical device which use dry heat to sterilize. They can be operated from 50 to 200^0C using a thermostat to control the temperature, shown in Figure 2. 3. They have double-walled insulation that keeps the heat inside it and conserves energy. The inner layer is a poor conductor, and the outer layer is metallic. Between them, is an air space to the utility the insulation. Uniform heat distribution inside it is ensured by an air circulating fan. They have aluminum trays or mesh plated trays and have indicators and controls for temperature and holding time

Figure 2. 3: Electrical oven

2.3.4 Hydraulic Press

A hydraulic press is a device using a hydraulic cylinder to generate a compressive force. It uses the hydraulic equivalent of a mechanical lever. It works on Pascal's principle which states that the pressure throughout the system is constant. One part of the system is a piston acting as a pump with a mechanical force acting on a small cross-sectional area. The other part is a piston with a larger area, and it generates a correspondingly large mechanical force, shown in Figure 2. 4.

Figure 2. 4: Hydraulic press machine

A fluid, such as oil, is displaced when either piston is pushed inward. Since the fluid is incompressible, the volume that the small piston displaces is equal to the volume displaced by the large piston. Moulding dyes of various shapes like rectangular, square and circular were used to make the different sample moulds.

2.3.5 Electrical Furnace

An electric furnace is like a conventional gas forced-air furnace except that it produces heat with electric heating elements instead of gas burners. Circuit breakers that control the heating elements may be either inside or outside the cabinet. It has a blower that draws air into the cabinet through a cold-air return and then pushes the air through the heat exchanger. There, electric heating elements heat the air, and the blower pushes

the warmed air back into rooms through a system of duct work. The maximum temperature range of the electric furnace which was used in our laboratory is 1700^0C, shown in Figure 2. 5.

Figure 2. 5: Electric silicon carbide furnace.

2.3.6 X-Ray Diffraction Analysis

Figure 2. 6, X-ray measurement of the catalyst was carried out by using Rigaku Ultima IV X-ray diffractometer (Figure 3.1) for phase identification. The patterns were run with Cu-Kα radiation at 40kV and 40mA. The mean crystallite size (d) of the phase was calculated from the line broadening of the most intense reflection using the Scherrer Equation.

$$d = 0.89\lambda/ \beta\cos\theta$$

Where d is the mean crystallite diameter, 0.89 is the Scherrer constant, l is the X-ray wave length (1.54056Å), and b is the effective line width of the observed X-ray reflection, calculated by the expression $\beta^2 = B^2\text{-}b^2$ (where B is the full width at half maximum

(FWHM), b is the instrumental broadening) determined through the FWHM of the X-ray reflection at 2θ of crystalline SiO_2.

Figure 2. 6: X-ray powder diffraction (XRD) instrument.

Principle of XRD

X-rays are electromagnetic radiations of accurately the same nature as light but very much shorter wavelength lying approximately in the range 0.5–2.5Å. X-rays are emitted from a source to the sample and interact with electrons in matter. Matter absorbs X-rays in two different ways, by scattering, and by true absorption. When a beam of X-rays impinges on the material, it was scattered in various directions by the electron cloud of the atoms. X-ray diffraction peaks are created by the constructive interference of monochromatic light scattered by each set of lattice planes at specific angles. From the scattering, a pattern of the intensity as a function of the scattering angle is obtained and can be compared to known patterns to identify the crystal structures of elements. X-ray diffraction is most commonly use and the least ambiguous method for the precise determination of the

positions of atoms in all kinds of matter ranging from fluids and powders to perfect crystals. It was a non-destructive technique applied for the characterization of crystalline materials. It provides information about the structure, phases, preferred crystal orientation and other structural parameters such as lattice parameters, crystallite size, crystallite strain and crystal defects.

2.3.7 Scanning Electron Microscopy (SEM)

Scanning electron micrographs (SEM) and SEM-EDX were recorded on Zeiss EVO 18 scanning electron microscope (SEM) instrument as shown in Figure 2. 7. An accelerating voltage of 15 kV and magnification of 5000X was applied.

Principle of SEM

SEM consists of an electron gun and electromagnetic lens system. A well-defined electron beam focused to a small spot, 50-100Å in diameter, on the surface of the sample. Electron beams which have been accelerated through a voltage lying between 1 and 50 kV, was used for the most applications. Accelerated electrons from electron gun carry significant amounts of kinetic energy, and this energy is degenerate as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signal lead to the generation of secondary electrons (that produce SEM image), back scattered electrons (BSE), diffracted backscattered electrons (EBSD that are used to determine crystal structures), photons (characteristic X-rays that are used for elemental analysis and continuum X-rays), visible light and heat. The secondary electrons can be detected by suitable detectors. These are the most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples (i.e. for rapid phase discrimination).

Figure 2. 7: SEM/EDAX Analysis instrument.

Morphological characterization is performing by using scanning electron microscopy (SEM). Topographical images in a SEM are produced from back-scattered primary or low-energy secondary electrons. The best resolution is about 2-5 nm but many routine studies are satisfied with a lower value and exploit the case of image interpretation and the extraordinary depth of field to obtain a comprehensive view of the specimen. With non-crystalline catalysts, SEM is especially useful for examining the distribution and size of mesopores.

2.3.8 Water Bath Machine

A water bath is made from a container filled with heated water. It is used to incubate samples in water at a constant temperature over a long period. They have a digital or an analog interface to allow users to set the desired temperature, shown in Figure 2. 8. It is a preferred heat source for heating flammable chemicals instead of an open flame to prevent ignition.

Figure 2. 8: Water bath machine.

Bulk Density of a material is the mass per unit volume other than the pores. If all the pores of the body are removed, then the actual density of the material can be found out. In this experiment, we use boiling Method for measurement of suspended weight. The samples are suspended with a thread into a boiling water bath for a period of up to 5 hours. They are then removed from the bath, and a tissue paper is used to remove the water on the surface only, then the weight that is measured called wet weight or soaked weight of the sample. The water absorption, apparent porosity and bulk density of prepared sintered samples are were calculated by using the following relation:

$$W.A\ (\text{in }\%) = \frac{w_w - D_w}{D_W}\ X\ 100$$

$$A.P\ (\text{in }\%) = \frac{w_w - D_w}{w_w - S_W} x\ 100$$

$$B.D\ (\text{in } gcm^{-3}) = \frac{D_w * \rho}{w_w - S_W}$$

Where ρ density of water, w_w the wet weight which is measured after is kept the samples in a water bath for 5 hours at 80°C, D_w is the dry weight of the samples, and S_W is the suspended weight of samples in a water bath.

2.3.9 Universal Testing Machine

Figure 2. 9, a universal testing machine is used to test the bending strength, tensile strength and compressive strength of materials. The Universal Testing Machine consists of two units.

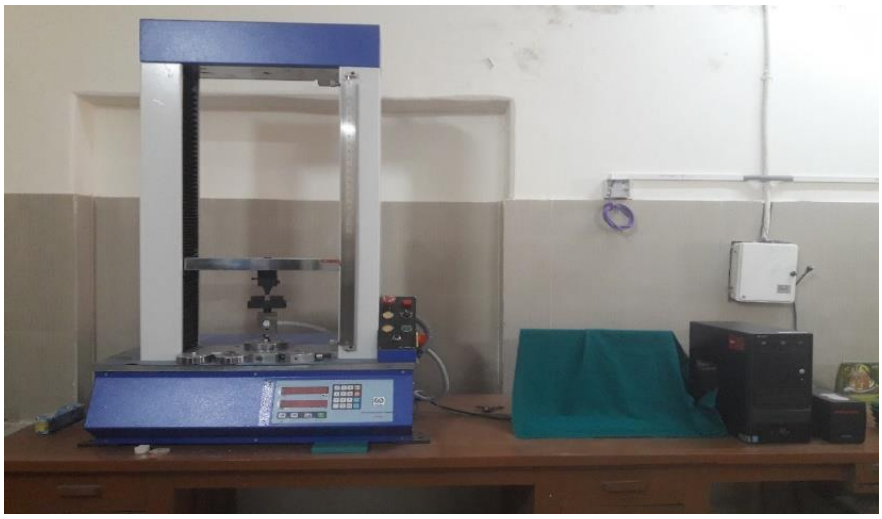


Figure 2. 9: Universal testing machine

(a) Loading unit

It consists of main hydraulic cylinder with the robust base inside. The piston which moves up and down. The chain is driven by an electric motor which is fitted on the left-hand side. The screw column maintained in the base can be rotated using the above arrangement of the chain. Each column passes through the main nut which is fitted in the lower crosshead. The lower table connected to the main piston through a ball & the ball seat is joined to ensure axial loading. There is a connection between the lower table and

upper head assembly that moves up and down with the main piston. The measurement of this assembly is carried out by some bearings which slide over the columns.

(b) Control panel.

It consists of an oil tank having a hydraulic oil level sight glass for checking the oil level. The pump is a displacement type piston pump having free plungers. Those ensure for the continuation of high pressure. The pump is fixed to the tank from the bottom. The suction & delivery valve is fitted to the pump near tank electric motor driven the pump is mounted on four studs which are fitted on the right side of the tank. There is an arrangement for loosing or tightening of the valve. The four valves on control panel control the oil stroke in the hydraulic system. The loading system works as described below. The return valve is closed, oil delivered by the pump through the flow control valves to the cylinder & the piston goes up. Pressure starts developing & either the specimen breaks or the load having maximum value is controlled with the base dynameters consisting in a cylinder in which the piston reciprocates. The switches have upper and lower push at the control panel for the downward & upward movement of the movable head. The on & off switch provided on the control panel & the pilot lamp shows the transmission of main supply.

2.3.10 Compressive Strength

Figure 2. 10, the Compressive Strength (CS) represents the ability of a product to resist failure under compressive load at room temperature. It has an indirect relevance to refractory performance and is used as one of the indicators of abrasion resistance.

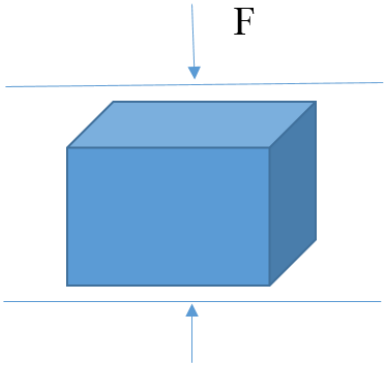


Figure 2. 10: Force applied to a sample from both sides.

The higher the CS of material is, the greater should be the resistance to abrasion. Refractories with high CS are also expected to have higher resistance to slag attack. The determination of Compressive Strength (CS) is also highly important in case of refractory insulating bricks where bricks must be porous as well as strong. For the measurement of the CS, a steadily increasing compressive load is applied to the refractory sample by a compressive machine until the sample fails.

$$\text{Compressive Strength (CS in MPa)} = F/A$$

2.3.11 Bending Strength

Flexural strength, also known as modulus of rupture, or bend strength, or transverse rupture strength is a material property, defined as the stress in a material just before it yields in a flexure test. The transverse bending test is most frequently employed, in which a specimen having either a circular or rectangular cross-section is bent until fracture or yielding using a three-point flexural test technique, Figure 2. 11. The flexural strength represents the highest stress experienced within the material at its moment of yield.

$$\text{Bending strength} = 3FL/2wh^2$$

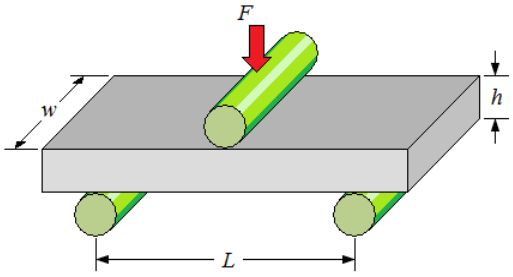


Figure 2. 11: Three-point flexural or bending test technique.

2.3.12 Linear Thermal Expansion

If the temperature of a body is increased that leads to thermal expansion giving out the change in dimension either in length, volume or area. Hence, there are three types of thermal expansion - Linear expansion, Area expansion, and Volume expansion. For linear expansion:

$$\frac{\Delta L}{L} = \alpha \Delta T$$

Where, L s original length,

α is the length expansion coefficient,

ΔT is a temperature difference,

ΔL is changing in length

Figure 2. 12, is a dilatometer is used to determine the linear thermal expansion of a solid as a function of temperature. In this dilatometer, a small load acts on the specimen. The measured expansion of the specimen can be used to determine the coefficient of linear thermal expansion. The first heating phase yields information about the actual state of the specimen, including its thermal and mechanical history. When thermoplastics soften, especially above the glass transition orientations and stresses may relax, as a result of which post-crystallization and recrystallization processes may occur. To determine the

coefficient of expansion as a material characteristic, the material must undergo reversible changes and so forth during a second heating phase that has followed controlled cooling.

Figure 2. 12: Dilatometer used for measurement of thermal expansion of the sample.

2.3.13 AC Dielectric Strength Measurement

The AC dielectric strength of prepared sintered samples is measured using a test setup supplied by NPTL Neo Tele Tronix Pvt. Ltd. Kolkata, shown in Figure 2. 13. The test set up consists of two spherical electrodes with a diameter of 10 mm. Samples (with thicknesses of 1.2 mm to 1.5 mm, and diameter of 15mm) is inserted between two spherical electrodes. The voltage applied across the specimen is raised gradually (at the rate of 1kV/s) till sample fails. To avoid any surface flashover, the sample electrode assembly is submerged in transformer oil.

Figure 2. 13: Voltage breakdown insulation test (1 to 150 KV).

2.3.14 AC Dielectric Constant and Loss Tangent Measurement at Low and Microwave Frequency

The electrical characterization namely measurement of the dielectric constant and loss are focused upon in this work. Overall dielectric and electrical properties of polycrystalline electronic ceramics have contributions from (i) bulk or grains (ii) grain boundaries and (iii) electrode specimen interface or electrode polarization. The complex permittivity and loss tangent are given as follows

$$\varepsilon^{*} = \varepsilon^{/} - i\varepsilon^{//} \qquad \tan\delta = \frac{\varepsilon^{//}}{\varepsilon^{/}}$$

The pellets were adequately polished with emery paper so that surface becomes homogeneous. They were then electrode using low-temperature silver paste and heat treated for 15 minutes at a temperature of 200 °C in an electric oven. AC dielectric and loss measurements (the value of real and imaginary parts of permittivity) of the samples were carried out on these electrodes pellets using four probe method in the frequency

range of 20 Hz to 1 MHz and at an interval of 10 °C in the range 20 – 200 °C in air using a Novo control Alpha- A High Performance Frequency Analyzer shown in Figure 2. 14.

Figure 2. 14: Novo control Alpha-A Frequency Analyzer with the furnace and the required interfaces.

Also, measure the value of AC dielectric constant and loss tangent at a microwave frequency (1 to 20 GHz). The instrument using for measuring dielectric measurement are vector impedance and vector network analyzer (Key sight E5071C ENA), shown in Figure 2. 15.

Figure 2. 15: An instrument vector network analyzer used for measuring dielectric constant and loss tangent at a microwave frequency (1-20 GHz).