CHAPTER II

EXPERIMENTAL
Chapter II
Experimental

2.1 INTRODUCTION
The present research work involved
(a) Development of silicon containing resins through co-polymerization of Inorganic and
Organic compounds containing silicon and carbon elements.
(b) Development of Si, C, O, N containing matrices for Ceramic Matrix Composites.
(c) Evaluation of properties and structure of the pyrolyzed materials from above resin systems.
(d) Fabrication of Ceramic- matrix composites using above resin systems.
(e) Characterization of ceramic matrix composites.

Various important materials/chemicals used in the present studies, techniques and equipments employed in synthesis, fabrication and characterization are described in this chapter.

2.2 INTRODUCTION TO MATERIALS USED
2.2.1 Reinforcements
The studies were performed with glass fibers and carbon fibers.
Glass fibers - E-glass fiber in the form of cloth and Roving were procured from
M/s UNNATI CORPORATION, Ahmedabad. Staple glass fibers of various lengths were cut from rovings.
Carbon fiber- Indcarb 6K High strength carbon fibers from Indian Petrochemicals Corporation Limited (IPCL), Baroda, India were used.
- Carbon cloth used was Toroyaca 8 HS high strength type from M/s Toray Industries, Japan.
2.2.2 MATRIX PRECURSORS

2.2.2 [A] Inorganic gels from hydrolysis of Tetraethyl orthosilicate (TEOS)

(a) Materials used

(i) Tetraethyl orthosilicate (TEOS), National Chemicals, Baroda
  Density : 0.933 gm/cm$^3$
  Boiling Point : 163-167°C

(ii) Ethyl alcohol, Alembic Chemicals, Baroda
  Dehydrated Alcohol
  Boiling Point : 78-80°C

(iii) Hydrochloric acid, Merck
  35% GR

(b) Synthesis of resin

The flow chart for the preparation of monolithic silica gels is shown in Figure 2.1. The inorganic silica gels were prepared by hydrolysis of organometallic compound Tetraethyl orthosilicate (TEOS) with acidified water, both in presence and absence of common solvent ethyl alcohol. The pH of water was adjusted ≈ 2 using hydrochloric acid. Molar ratio of water/TEOS was varied from 2 to 10 for obtaining monolithic gels with molar ratio of ethyl alcohol/TEOS = 2. The mixture of TEOS and alcohol was stirred well in closed conical flask using magnetic stirrer and then acidified water was added drop by drop to it at room temperature. After stirring for required time, the solution was casted into glass, plastic and teflon petridishes for gelling. The gels obtained were kept in an oven at 60°C for aging and drying. The dried gels were further post cured to 180°C.

(c) Pyrolysis

Post cured samples were pyrolyzed at 1000°C in pure nitrogen atmosphere. The pyrolysis was carried in a quartz reaction vessel (35 mm in diameter) kept in a furnace as shown in Figure 2.2. The reaction vessel had inlet for nitrogen gas and outlet for the
Figure 2.1 Flow chart for the preparation of Monolithic silica gels
Figure 2.2 Photograph showing Assembly of furnace used for pyrolysis
removal of byproduct gases. A Chromel-Alumel thermocouple was used to measure temperature of the furnace. Indotherm Temperature Programmer/controller MPC 500 was used to maintain linear heating and cooling rates as well as for constant temperature for soaking. The heating and cooling rate used for pyrolysis of cured samples are shown in Figure 2.3.

2.2.2 [B] Carbon/Silica based glasses using aqueous medium

(a) Materials used

(i) Tetraethyl orthosilicate(TEOS), National Chemicals, Baroda
   Density : 0.933 gm/cm³
   Boiling Point : 163-167°C

(ii) Ethyl alcohol, Alembic Chemicals, Baroda
   Dehydrated Alcohol
   Boiling Point : 78-80°C

(iii) Hydrochloric acid, Merck
   35% GR

(iv) Furfuryl alcohol (FFA), National chemicals, Baroda.
   LR grade furfuryl alcohol manufactured by National Chemicals, Baroda
   Density : 1.132 gm/cm³
   Boiling point : 162-167°C

(b) Synthesis of Resin

Series of experiments were carried out to prepare inorganic gels (silica system described in section 2.2.2[A]) and silica glass. These results revealed that water/TEOS molar ratio from 4 to 8 gives better results in terms of monolithicity of gels. But for gel to
Figure 2.3 The heating and cooling cycles used during pyrolysis
glass transition, higher water to TEOS molar ratio gives better results in terms of size of pieces. Therefore, the optimum conditions were taken as

\[ \text{TEOS} : \text{H}_2\text{O} = 1 : 8 \text{ and } \text{pH} = 2 \]

Synthesis of Organic - Inorganic hybrids through sol-gel-reactions from FFA and TEOS involved

(i) Hydrolysis of TEOS and

(ii) Subsequent copolymerization of hydrolyzed TEOS with the organic network precursor, i.e., FFA.

The TEOS was hydrolyzed with water keeping the \( \text{H}_2\text{O}/\text{TEOS} \) molar ratio = 8. The time of hydrolysis was kept about 12 hrs., which yields homogeneous, transparent sols. Ethyl alcohol was used as solvent for obtaining better homogeneity with Ethyl alcohol/TEOS molar ratio = 2. After hydrolysis furfuryl alcohol was added dropwise into the hydrolyzed solution. The molar ratio of furfuryl alcohol to TEOS was varied from 0.5 to 10. The polymerization of FFA was observed in fifteen minutes with the change in colours with time from light yellow to greenish to brownish to dark brown. The subsequent copolymerization was carried out till the colour changed to dark brown and the viscosity seemed to increase. The sols were then casted and cured at different temperatures for different time intervals up to 180°C (post curing).

Samples were also prepared by removing excess solvent after hydrolysis and polymerization by vacuum at room temperature.

(c) Pyrolysis of the cured resin

The post cured samples so prepared were pyrolyzed at 1000°C at controlled heating rate under an inert atmosphere. The pyrolysis was carried out in a quartz reaction vessel kept in a furnace as shown in Figure 2.2. The reaction vessel had inlet for nitrogen gas and outlet for the removal of byproduct gases. A Chromel-Alumel thermocouple was used to measure temperature of the furnace. Indotherm Temperature
Programmer/controller MPC 500 was used to maintain linear heating and cooling rates as well as for constant temperature for soaking. The heating and cooling rate used for pyrolysis of cured resin are shown in Figure 2.3. To study the transformation reaction during pyrolysis, some samples of the cured resin were heated to different end temperatures of 400°C, 600°C, 800°C and 1000°C. Samples were further heated to 1400°C and 1600°C under Argon atmosphere in a commercial high temperature furnace at Graphite India Limited, Bangalore.

2.2.2 [C] Carbon/silica based glasses using non aqueous medium

(a) Materials used

(i) Tetraethyl orthosilicate(TEOS), National Chemicals, Baroda

Density : 0.933 gm/cm³

Boiling Point : 163-167°C

(ii) Carbon tetrachloride : GR grade carbon tetrachloride manufactured by Merck

Density : 1.59 gm/cm³

Boiling Point : 76-77°C

(iii) 1,4-Butanediol, LR grade, National Chemicals, Baroda.

Density : 1.015 gm/cm³

Boiling Point : 226-236°C

(b) Synthesis of resin

Organic-Inorganic gels with TEOS and dihydric alcohol were synthesized by reaction copolymerization of TEOS and 1,4-Butanediol in Carbon Tetrachloride medium. The TEOS was dissolved in CCl₄ and stirred for five minutes in closed conical flask and then Butanediol was added dropwise. The molar ratio of CCl₄/TEOS was kept at 2 and the molar ratio of Butanediol/TEOS was varied from 2 to 8. These were then stirred on
magnetic stirrer till the solution became clear. The sols were casted in plastic petridishes and kept for gelation at room temperature followed by gradually raising temperature of drying to 180°C. At higher Butanediol/TEOS molar ratio, monolithic gels were obtained.

(c) Pyrolysis of the cured Resin

Dried gels were pyrolyzed at 1000°C as described in section 2.2.2 [A](c). Samples were further heated to 1400°C and 1600°C under Argon atmosphere in commercial high temperature furnace at Graphite India Limited, Bangalore.

2.2.2 [D] Nitrogen Containing Ceramics.

(a) Materials used

(i) Ammonia solution, s.d.fine-chem Ltd.
   Specific gravity : 0.91
   25 % Extra pure

(b) Nitridation of Silica gels

The inorganic gels obtained in section 2.2.2[A] by the hydrolysis of TEOS with acidified water with a molar ratio \( \text{H}_2\text{O}/\text{TEOS} = 8 \) and Ethyl alcohol as a solvent with molar ratio Ethyl alcohol/TEOS = 2 were dried at 60°C in an oven. To incorporate nitrogen in the gels, the gels were kept in quartz crucibles and heated to required nitridation temperature in a mixture of nitrogen gas and ammonia vapours, held at the final nitridation temperature for different time. The nitrogen gas is used as a carrier gas. Figure 2.4 shows the set-up for nitridation. Figure 2.5 shows the heating and cooling cycle used for nitridation. The mixture of nitrogen gas and ammonia vapours was passed during the cooling cycle as well.
Figure 2.4  Schematic of the Nitriding arrangement used for Nitridation of silica samples
Figure 2.5 The heating and cooling cycles used during Nitridation
2.3 FABRICATION OF CERAMIC MATRIX COMPOSITES

The composites were fabricated by using different combinations of ceramic matrices and fibers. Silicon Oxycarbide types of matrices were prepared by (i) Aqueous route and (ii) Non aqueous routes. Two types of fibers used were
- Glass Fibers
- Carbon Fibers

2.3.1 FABRICATION OF FIBER REINFORCED COMPOSITES

(a) Staple fiber composites

Continuous glass fibers and carbon fiber rovings were cut to length of 1-2 mm to get staple fibers. FFA/TEOS resin system prepared as described in section 2.2.2[B] were gelled at room temperature. After gelling, the solid mass was grounded manually using Agate pestle mortar. Powder and fibers were mixed thoroughly in the mortar. With different fiber weight % the mix was then pressed in the cylindrical form of diameter 5 cm and height of 3-4 mm and cured to temperature 180°C in circular die. The material used for the fabrication of die was EN16 (fabricated at Microtools, Vithal udyognagar, Anand). For non aqueous system, described in section 2.2.2[C] the corrosion of die is observed. Therefore pressing of composites and curing was avoided.

Composites were also prepared by first laying the fibers in the plastic rectangular moulds (95 mm by 65 mm) and then impregnating the fibers with the resin system. More fibers were added to the cast. After casting, the composites were pressed with a flat plastic plate so that there is uniform distribution of fibers and excess of resin is drained out.
(b) Unidirectional composites

Unidirectional composites were prepared by filament winding technique. Figure 2.6 shows the schematic diagram of filament winding process. The fiber tow was first passed through the resin system and then wound over the teflon coated flat mandrel. The tows were post cured at a temperature of 180°C. These tows were then stacked in a circular glass tube closed at one end and then repeatedly impregnated with powder added resin system. These were post cured & pyrolyzed to a temperature of 1000°C.

(c) Bi-directional composites

Similar to unidirectional composites the carbon cloth was first impregnated with resin system and post cured. This makes the handling of the cloth easy. The cloth was cut in required dimensions of glass mould (length 105 mm, width 15 mm and height of 95 mm). Figure 2.7 shows the photograph of the glass moulds used for making unidirectional and bi-directional composites. The cloth plies were stacked in mould whose inner side is covered with Teflon sheet. The repeated impregnation and pyrolysis resulted in composites of required dimensions and fiber volume.

2.3.2 PYROLYSIS OF THE COMPOSITES

The pyrolysis of the preceramic polymer matrix composites was carried out in an inert atmosphere at different temperatures as described in section 2.2.2 [A](c). The same heating and cooling rate were used for pyrolysis of composites as for the matrix alone (Figure 2.3).

2.3.3 IMPREGNATION OF THE COMPOSITES

Carbonized composites had low density and highly porous structure. In order to increase the density of composites, impregnation of the composites with the matrix material was carried out. The impregnation setup used in the present study is shown in Figure 2.8.
Figure 2.6 Schematic of Filament winding technique
Figure 2.7 Photograph showing the glass moulds used for making composites
(a) Unidirectional composites     (b) Bi-directional composites
Figure 2.8 Experimental setup for impregnation
Pyrolyzed composites were placed in the impregnation chamber which was then evacuated to a pressure of $10^{-2}$ torr for two hours using two stage vacuum rotary pump, so that the air trapped in pores of the composites is completely removed. Resin was introduced through the opener no.1. The composites were then cured in presence of excess resin followed by carbonization to different temperatures. The densification cycles were repeated successively many times to reduce the porosity.

2.3.4 PYROLYSIS OF THE COMPOSITES TO HIGHER TEMPERATURES

Pyrolysis of the composite was carried out by wrapping the composites in graphite foils then placing the composites on flat graphite plate and placing flat plate on upper side. The plates were tied with panox fibers. The samples were pyrolyzed at different temperatures i.e. 1000, 1200, 1400 and 1600°C and holding at a maximum temperature for half an hour. The pyrolysis was carried out at Graphite India Ltd., Bangalore.

2.4 CHARACTERIZATION OF RESIN AND COMPOSITES
2.4.1 DENSITY MEASUREMENTS OF CURED AND/OR PYROLYZED SAMPLES

Bulk density of composite was calculated by ratio of weight of the sample to geometrical volume of the Composite. Density of cured and pyrolyzed resin was determined by sink and float method using the mixture of n-Haptane and Carbon tetrachloride up to a density range of 0.68 to 1.592 gm/cc and also used Carbon tetrachloride plus 1,4-dibromobenzene up to density range 1.592 - 2.24 gm/cc.

In sink and float method, solutions of different densities were prepared by using n-Haptane plus Carbon tetrachloride and Carbon tetrachloride plus 1,4-dibromobenzene in different proportions so as to obtain solutions of different densities. These Compositions are given in Table 2.1 and Table 2.2. A small piece of sample was taken and put in different bottles containing the solutions. The sample will sink in a liquid if its density is
Table 2.1

Densities of various mixtures of n-Haptane and carbon tetrachloride

<table>
<thead>
<tr>
<th>n-Haptane in ml</th>
<th>Carbontetrachloride in ml</th>
<th>mass per 10 ml</th>
<th>Density gm/cc</th>
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</thead>
<tbody>
<tr>
<td>0.0</td>
<td>10.0</td>
<td>15.920</td>
<td>1.5920</td>
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<td>0.5</td>
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<td>15.466</td>
<td>1.5466</td>
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<td>9.0</td>
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<td>1.5012</td>
</tr>
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<td>1.4558</td>
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<td>1.1834</td>
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<td>5.6</td>
<td>4.4</td>
<td>10.835</td>
<td>1.0835</td>
</tr>
</tbody>
</table>

Table 2.2

Densities of various mixtures of carbon tetrachloride and 1,4-dibromobenzene

<table>
<thead>
<tr>
<th>Carbontetrachloride in ml</th>
<th>1,4-Dibromobenzene in ml</th>
<th>mass per 10 ml</th>
<th>Density gm/cc</th>
</tr>
</thead>
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<td>8.0</td>
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<td>6.0</td>
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<td>1.9808</td>
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<td>5.0</td>
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<td>1.9160</td>
</tr>
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<td>6.0</td>
<td>4.0</td>
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<td>1.8512</td>
</tr>
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</tr>
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<td>2.0</td>
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<td>1.7216</td>
</tr>
<tr>
<td>9.0</td>
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<td>1.6568</td>
</tr>
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<td>9.5</td>
<td>0.5</td>
<td>16.244</td>
<td>1.6244</td>
</tr>
<tr>
<td>10.0</td>
<td>0.0</td>
<td>15.920</td>
<td>1.5920</td>
</tr>
</tbody>
</table>
higher and it will float if density of sample is lower than that of liquid. The sample will remain in the center of the liquid, if they have matching densities.

* Density of n-Haptane : 0.684 gm/cc
* Density of Carbon tetrachloride : 1.592 gm/cc
* Density of 1,4 - dibromobenzene : 2.240 gm/cc

2.4.2 DETERMINATION OF FIBER VOLUME FRACTION OF COMPOSITES

In fiber reinforced composites, fiber being the load bearing constituents and the final properties of the composites depend largely on its fiber volume fraction. Therefore in order to correlate the properties of the composites with those of the fibers, it is essential to know precisely the fiber volume fraction in composites. These can be calculated theoretically[138] as well as measured experimentally by Acid digestion method. In the present studies fiber volume fraction was calculated using former method only, because acid digestion can be used only for polymeric composites where matrix becomes soluble in acids and not for ceramic matrix composites. For ceramic matrix composites, optical microscopy is used to determine the fiber volume content by measuring the area of the fibers per unit area of the composite sample.

Theoretical calculation of fiber volume fraction

This method is based on calculating the volume of fiber used in preparing the composite (based on number of plies, or fiber weight etc.) and dividing it by the volume of composites[139]. As described in section [2.3.1(b)], unidirectional composites are made from prepregs by stacking the tows of carbon fibers parallel to each other in a circular tube. Total number of tows was known. Weight of the fiber tow per unit length was calculated by taking the average of 20 meters of fiber tow.
The density of the fiber is known by knowing the number of tows used in composites, fiber volume fraction was calculated as follows:

Weight of fiber used in the composites = \( n \times l \times w \)
- \( n \) = No. of tows in the composites
- \( l \) = Length of each tow or composite
- \( w \) = weight per unit length of fiber

Volume of fiber used in the composite = \( n \times l \times w / d \)
- \( d \) = density of the fiber

Volume of composites = \( \pi \times r^2 \times L \)
- \( r \) = radius of composite
- \( L \) = length of composite

Fiber volume fraction = volume of fiber used in the composites / volume of composite

Fiber vol (\%) = \( n \times w \times 100 / d \times \pi \times r^2 \)

Similarly for bi-directional composites, fiber vol. % is calculated by taking number of plies in consideration. This was further confirmed by using optical microscopy.

### 2.4.3 BULK DENSITY AND POROSITY

The small rectangular composites (100 mm X 10 mm X 6 mm) were used for this purpose. Four samples were kept in boiling water for 2 h and cooled. The porosity and bulk density of the sample was obtained by taking its dry weight (\( D \)), its weight after it was fully soaked with water (\( W \)) and the weight of the soaked piece when suspended in water (\( A \)) and by applying Archimedes principle as follows[140]

\[
\text{Porosity} = \left( \frac{W - D}{W - A} \right) \times 100(\%)
\]

\[
\text{Bulk Density} = \left( \frac{D}{W - A} \right) g / \text{cm}^3
\]
2.4.4 DETERMINATION OF FLEXURAL PROPERTIES

Flexural properties of composites were measured by three point bending technique as per ASTM standard method 1966b Part 13 C93-54 74[141]. In this method, composite bar having rectangular cross section is supported on two supports and is loaded midway by a loading nose.

In the present studies, flexural tests were conducted using Instron Universal Testing Machine Model 4483. Samples of 8 mm width were cut from the composite laminate of 105 mm and 95 mm by a diamond cutter. Span length of the sample was taken about 10 times the thickness of the composite. The loading was done by downward movement of loading nose at a cross head speed of 100 mm/min as shown in the Figure 2.9.

Stress so developed was measured with the help of a load cell and recorded on the computer which on step analysis gave the necessary information and the flexural strength was calculated. Thus a load versus deflection plot was obtained. Specimens were loaded till complete fracture occurred in the composites. Flexural strength and flexural modulus were calculated as follows:

\[
\text{Flexural strength} = \frac{3 \times P_{\text{max}} \times S}{2 \times b \times t^2}
\]

\(\text{Kg/mm}^2\)

where,

\(P_{\text{max}}\) = Maximum fracture load (Kg)

\(S\) = Span length (mm)

\(b\) = Width of the specimen (mm)

\(t\) = Thickness of the specimen (mm)
Figure 2.9  Three point flexural test fixture
Flexural modulus = \frac{S \times P}{4 \times b \times t^3 \times d}

where,

- $S$ = Span length (mm)
- $b$ = Width of specimen (mm)
- $t$ = Thickness of specimen (mm)
- $P/d$ = slope of initial straight line portion of load deflection curve (Kg/mm)

### 2.4.5 THERMAL CHARACTERIZATION

Mettler TA 4000 Thermal analysis system was used for studying the stability and transformations of the ceramics and composites made in the present study. Figure 2.10(a) & 2.10(b) shows the photograph of Thermal analysis system. It comprises of three modules as Thermogravimetric analyzer (TGA), Differential Scanning Calorimeter (DSC) and Thermomechanical analyzer (TMA). The weight change, shrinkage behaviour and heat flow with temperature was monitored through Mettler thermal analysis software. For thermogravimetric analysis the samples were cut in small pieces with the help of diamond cutter. About 15-20 mg of the sample was taken in an Alumina crucible and placed on the pan of balance enclosed in a furnace (25-1000°C). Sample was heated from room temperature to 950°C at a rate of 10°C/min in nitrogen as well as in oxygen. It has a attachment for change of reaction atmosphere during analysis. The oxidation resistance of pyrolyzed sample or composite was determined by measuring the weight loss of sample in oxygen atmosphere. For DSC analysis, the sample was packed in Aluminium crucible and lowered in the furnace of DSC module of TA 4000. For shrinkage behaviour and linear coefficient of thermal expansion, the flat sample of about 20 mm$^2$ was placed on quartz plat form of TMA module of TA 4000 and then probe was lowered on to it. The whole arrangement is enclosed in the TMA furnace (25-1000°C). The samples were characterized at the rate of 10°C/min.
Figure 2.10(a) Photograph showing DSC and Thermogravimetric Analyzer TA 4000

Figure 2.10(b) Photograph showing Thermo Mechanical Analyzer TA 4000
2.4.6 X-RAY DIFFRACTOMETRY

The crystalline phases of different ceramics and ceramic matrix composites were analyzed by RIGAKU X-ray diffractometer using wide angle x-ray diffraction and CuKα as a radiation source. Wide angle X-ray diffraction patterns were used to identify the material. The diffraction patterns of the samples made in the laboratory were compared with the diffraction patterns of the standard samples of silica, silicon carbide and silicon oxycarbide in the available literature. By comparison method, the amount of different species present in the sample were identified and approximated.

2.4.7 OPTICAL MICROSCOPY

Leitz LABORLUX 12 POLS optical microscope was used to study the microstructure of the ceramic matrix, distribution of matrix and macro/micro cracking in composites. Figure 2.11 shows the photograph of Optical microscope. For optical microscopy, the fibers as well as composites were embedded in epoxy resin moulds in vertical as well as in longitudinal directions. These moulds were then lapped with silicon carbide water proof papers of grade 220, 400, 800, 1000 and 1200. The lapped samples were polished on polishing machine using different sized alumina powders (BUEHLER, USA) of 1 micron and finally 0.05 micron in size. After each cycle of polishing with different grade of powder, the samples were cleaned thoroughly with distilled water in an ultrasonic cleaner (Electronic & Engineering Co., Bombay, model no. ES/250w) for five to seven minutes. These samples were then viewed under optical microscope at different magnifications using polarized light.
Figure 2.11 Photograph showing Leitz Optical microscope LABORLUX 12 POLS
2.4.8 FTIR SPECTROSCOPIC STUDIES

Infrared spectra of resin, cured resin as well as pyrolyzed samples were taken by using Hewlett Packard, Infrared Spectrometer.

Cured samples were crushed and made into powder form. These powders were kept in the oven at 100°C for 30 minutes to remove moisture. After drying the powder was mixed with KBr and crushed again to make uniform mixture. Thin and transparent pellets of these samples were prepared by compression moulding. Moisture, if any, was removed by vacuum pump attached with the die used in moulding. Pellet was fixed in the IR spectrometer sample holder and spectra was recorded. From the IR spectrum, various functional groups present on the resin samples were determined.