CHAPTER 3
EXPERIMENTAL

This chapter deals with materials, testing equipments and experimental methods adopted for the present study. For the sake of convenience this chapter is divided into two parts. Part A covers the materials used in this research programme such as polymers, fibres and others along with their chemical structure, physical properties and a brief discussion of their applications. In Part B theory and techniques adopted for characterization of reinforced polymer composites are briefly highlighted. It also includes method of fabrication of SGFR composites.

PART A

3.1 MATERIALS

The details of materials such as polymers and fibres used in this research work are now presented.

3.1.1 POLYMERS

Five types of polymers have been used in the investigation they are (a) Nylon 6 (b) Polypropylene (c) Polybutylene terephthalate (d) Polycarbonate and (e) Polyurethane.

3.1.1.1 NYLON 6

Polyamide is the technical name given to nylon 6; polycaprolactum; \(-\text{NH-} (\text{CH}_2)_5 \text{ CO-}\)\textsubscript{n}. The water absorption of nylon 6 at 23°C and 50 % relative humidity, fall in the range of 3 ± 0.4 %.
In 1907, J. Von Braun at Gottingen University in Germany discovered that the self-condensation of ε-aminocaproic acid led to a caprolactam polymer, probably of low molecular weight, as it appears today. In 1938, Schlack at I.G. Farbenindustrie in Berlin polymerized caprolactam to high-molecular-weight nylon 6. The first applications included melt-spun bristles and textile filaments; melt-cast tapes used as transmission belts; and injection-moulded bushing, bearings and gears.

![Figure 3.1. Chemical Structure of nylon 6](image)

Nylon 6 is formed by ring-operating polymerization (either hydrolytic or anionic) or ε-caprolactam, represented by the formal equation.

Nylon 6 is manufactured in a three step, continuous process. First, caprolactam is polymerized at 240°C - 280°C then, the residual monomer is extracted from the chips with water at 100°C; lastly, the material is dried at 100°C - 150°C with nitrogen or under vacuum. In an alternative process, the hot water extraction step is replaced by vacuum demonomerization of the polymer melt.

Nylon 6 is a crystalline thermoplastic with a low thermal coefficient of linear expansion. The reinforced resins, in particular, undergo very little dimensional change when the temperature changes. However, the linear expansion of the glass-reinforced resins depends on the orientation of the glass fibre. Nucleated products show a higher heat-distraction because of their higher crystallinity. This effect is more significant with nylon 6. By reinforcing nylon with glass fibre, the heat distraction temperature also increases. Nylon 6 is tough and hard. It is the best material for parts that must be shockproof, even at sub-zero temperatures.
The most common reinforcing fillers for nylon resins are glass fibres. When reinforcing fillers are added to nylon, the resulting toughness decreases with a significant increase in stiffness, impact, tensile strength and hardness. Nylons are thermoplastics. They possess the greatest hardness and rigidity and the highest resistance to abrasion and heat deformation. Nylons are creamy, off-white, smooth appearance. Nylons are inherently resistant to lubricants, engine fuels, hydraulic fluids, coolants, refrigerants, paint solvents, cleaness and aliphatic and aromatic hydrocarbons. They are also resistant to aqueous solutions of many inorganic chemicals (such as salts and alkalies). Nylon is gradually attacked over time by hot, oxygenated and hot water, Strong acids, phenols, cresols and some heavy metal slot solutions attack nylons. Certain oxidizing agents and a few chlorinated hydrocarbons at elevated temperatures will also attack. Nylon resins begin to decompose at temperature above 299°C; combustible gases are formed at temperature between 449°C - 499°C that will continue to burn after injection.

(i) Properties of Nylon 6

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cc)</td>
<td>1.136</td>
</tr>
<tr>
<td>Surface hardness (shore-D)</td>
<td>M 86</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>80</td>
</tr>
<tr>
<td>Tensile modulus (MPa)</td>
<td>3000</td>
</tr>
<tr>
<td>Impact strength (J/M)</td>
<td>53</td>
</tr>
<tr>
<td>HDT (°C)</td>
<td>54-90</td>
</tr>
</tbody>
</table>

(ii) Applications

The most important fields of application for nylon include electrical engineering (that is power transmission and telecommunication), automotive engineering, mechanical and chemical engineering in general, materials handling, instrumentation, plumbing and sanitary ware and packaging.
(iii) Advantages and disadvantages

Nylons will share good mechanical strength, good toughness at low temperature and high thermal stability. Their resistance to chemical is excellent and their dielectric properties are very good. The chief disadvantage of nylon is water absorption. When water is absorbed, a change in properties takes place. The values for tensile strength, tensile modulus and hardness will decline. But the toughness of nylon significantly increases while resistance to creep decreases.

3.1.1.2 POLYPROPYLENE (PP)

Polypropylene (PP) is a versatile thermoplastic offering a useful balance of heat and chemical resistance, good mechanical and electrical properties and processing ease.

\[ \text{CH}_3 \]

\[ \quad \mid \]

\[ \text{[-CH}_2 - \text{CH} - \text{]}_n \]

Figure 3.2. Chemical Structure of Polypropylene

(i) Properties

Polypropylene (PP) has resistance to deformation at elevated temperature, high stiffness, tensile strength, surface hardness and good toughness at ambient temperature. They are resistance to impact. Since PP is essentially a crystalline polymer its morphology and the nature of its crystalline structure plays a large part in the physical properties of homopolymers, principally with respect to flexural modulus, surface hardness and transparency [100]. Surface treated mica provides higher stiffness and approaching data obtained with glass fibre reinforcement [145].
<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity</td>
<td>D-792</td>
<td>1.03 - .10</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>D-638</td>
<td>31 MPa</td>
</tr>
<tr>
<td>Tensile Modulus of Elasticity</td>
<td>D-638</td>
<td>114 – 6895 MPa</td>
</tr>
<tr>
<td>Elongation</td>
<td>D-638</td>
<td>200 – 700 %</td>
</tr>
<tr>
<td>Flexural Modulus of Elasticity</td>
<td>D-790</td>
<td>118 – 6895 MPa</td>
</tr>
<tr>
<td>Shear Strength</td>
<td>D-732</td>
<td>40 MPa</td>
</tr>
<tr>
<td>Compressive Strength</td>
<td>D-1822</td>
<td>46 MPa</td>
</tr>
<tr>
<td>Heat Deflection Temp. @ 1.82 MPa</td>
<td>D-648</td>
<td>43.34°C</td>
</tr>
<tr>
<td>Melting Point</td>
<td>D-789</td>
<td>168.3°C</td>
</tr>
<tr>
<td>Continuous Service Temp., Air, Max.</td>
<td></td>
<td>82.2°C</td>
</tr>
</tbody>
</table>

(ii) Applications

Polypropylene (PP) is utilized in non-woven applications disposable diaper and incontinence pad cover stocks. Other application includes clothing inner liners, wiping cloths, drapes and gowns, tea bags, sleeping bags and wall coverings. Furniture and automotive [162] upholstery fabrics are produced from both continuous multifilament and staple fibres. PP is having good market for packaging specifically closures and containers. In medical applications PP can be used as disposable syringes, hospital trays and laboratory wares. Washing machine, agitators, tub liners, bleach and detergent dispensing unit valves and control assemblies, drain tubes and pump housings are same examples.

(iii) Advantages and disadvantages

Low specific gravity excellent chemical resistance, high melting point, good stiffness toughness, adaptability, excellent dielectric properties and low costs. PP also has flammability, low-temperature brittleness, difficult to printing, painting and gluing, low UV resistance etc.

3.1.1.3 POLYBUTYLENE TEREPTHALATE (PBT)

Polybutylene Terephthalate (PBT) is crystalline, high molecular weight polyester that has an excellent balance of properties and processing characteristics. PBT
is produced by the transesterification reaction between 1, 4-butanediol and dimethyl terephthalate (DMT). PBT resin is available in both resin and compound form.

![Chemical structure of Polybutylene terephthalate](image)

Figure 3.3. Chemical structure of Polybutylene terephthalate

The matrix material used is commercially available PBT Duranex Grade 2002 manufactured by polyplastics, with a density of 1300 kg m\(^{-3}\). Type E-glass fibres with a mean diameter 12 \(\mu\)m and a density of 2540 kg m\(^{-3}\), supplied by Eurochem (M), are used as reinforcement.

Polybutylene Terephthalate (PBT) granules and various loading (10, 20 and 30 % by wt.) of 4 mm SGF are compounded in a Betol single screw extruder having a barrel temperature profile ranging from 220\(^{\circ}\)C - 240\(^{\circ}\)C and at a screw speed of 25 rpm. A granulator subsequently palletized the extruded strands. The reinforced granules are then injection moulded on a Battenfeld BA 350 CD Plus with a Unilog 400 control system. Mouldings are prepared using 90 bar injection pressure, barrel temperature profile ranging from 210\(^{\circ}\)C - 240\(^{\circ}\)C, mould temperature and cooling time of 80\(^{\circ}\)C and 20 s, respectively. A test specimen in accordance with ASTM D638 Type 1 is used. PBT and reinforced granules are dried for 4hr. at 100\(^{\circ}\)C under vacuum prior to extrusion and injection moulding.
(i) Properties of PBT

<table>
<thead>
<tr>
<th>Property</th>
<th>SI/Metric</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Conductivity</td>
<td>2.6 W/mK</td>
</tr>
<tr>
<td>Thermal Diffusivity</td>
<td>0.014 cm²/sec</td>
</tr>
<tr>
<td>Heat Capacity</td>
<td>1.31 J/g°C</td>
</tr>
<tr>
<td>Tensile Modulus</td>
<td>6.0E3 Mpa</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>42 MPa</td>
</tr>
<tr>
<td>Nominal Strain @ Break</td>
<td>1.45 %</td>
</tr>
<tr>
<td>Flexural Modulus</td>
<td>3.4E3 Mpa</td>
</tr>
<tr>
<td>Flexural Strength</td>
<td>61 MPa</td>
</tr>
<tr>
<td>Charpy Notched</td>
<td>2.8 kJ/m²</td>
</tr>
<tr>
<td>Charpy Unnotched</td>
<td>24 kJ/m²</td>
</tr>
</tbody>
</table>

(ii) Applications

Polybutylene Terephthalate (PBT) resins are used in many types of products in automobiles, electrical and electronic parts, industrial components, and consumer items. Their resistance to heat and chemicals, plus their wide range of color possibilities, dictate their use in appliances where they have been found easier to mold and less expensive than thermosetting plastics. Iron and toaster housings, cooker/fryer handles, hair drier nozzles, and food processor blades are all made of PBT.

In the industrial world, PBT resins are used for pump housing, impellers, valves, brackets, water meter components, tool housing, casings and replacements for metals in many types of load-bearing parts. Their high strength-to-weight ratio and resistance to corrosion and chemicals make them ideal choices for these and other industrial components.

3.1.1.4 POLYCARBONATE (PC)

General-purpose polycarbonate is one of the toughest, most versatile engineering polymers.

Polycarbonate (PC) is a most important technical thermoplastic because of its excellent heat resistance, outstanding impact strength and good dimensional stability.
Acetyl resin and PC both resulted from research on pure formaldehyde. Bisphenol A, which is produced for the epoxy resins, opened the door for acetyl resin and polycarbonate in 1959 and Bayer, in West Germany, is first to produce and market polycarbonate. Polycarbonate is the first rigid thermoplastics to offer good temperature stability up to 130°C and impact strength.

![Chemical structure of Polycarbonate](image)

Figure 3.4. Chemical structure of Polycarbonate

(i) Properties

Polycarbonate resin is generally stable to water, mineral and organic acids. However, crazing and embrittlement may occur if a part molded from polycarbonate resin is highly stressed and exposed to water or a moist, environment at elevated temperatures. For this reason, a temperature limit of 60°C - 70°C is recommended. Also, it is necessary to design to the worst abuse and environmental conditions. PC’s tensile strength, flexural strength and flexural modulus decreases steadily as the temperature increases. On the other hand, the effect or temperature on impact strength is the opposite. In fact, its notched izod impact strength drops dramatically near -18°C. Still, a notched izod value of 107 J/m is a very respectable number for many plastics, even at room temperature.

Although stress levels are low in PC the shapes of the resin stress/strain curves simulated those of aluminum and steel. Outstanding properties of PC is impact strength, PC also exhibits a property known as “critical thickness” at a particular thickness, the notched impact strength will suddenly drop from a high-energy-observing ductile failure to a low-energy observing brittle failure. The optical properties of PC are excellent. It is for this reason that the product is used extensively for many types of lens
and glazing application. The refractive index of PC is 1.586 and that a scratch resistant cooling is recommended where these products are subject to abrasion.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity</td>
<td>1.20</td>
</tr>
<tr>
<td>Tensile strength, Ultimate</td>
<td>62 MPa</td>
</tr>
<tr>
<td>Elongation at break</td>
<td>130 %</td>
</tr>
<tr>
<td>Rockwell hardness</td>
<td>R118</td>
</tr>
<tr>
<td>Flexural strength</td>
<td>98 MPa</td>
</tr>
<tr>
<td>Melting point</td>
<td>154°C</td>
</tr>
<tr>
<td>Heat deflection at 1.82 MPa</td>
<td>132°C</td>
</tr>
<tr>
<td>Thermal expansion</td>
<td>93°C</td>
</tr>
<tr>
<td>Water absorption - 24 hours</td>
<td>0.15 %</td>
</tr>
<tr>
<td>Water absorption - saturation</td>
<td>0.35 %</td>
</tr>
</tbody>
</table>

(ii) Applications

Polycarbonate (PC) lends to higher performance to rear and forward lighting lens applications. Opaque grades are widely used for thin wall mechanical parts and large exterior parts. PC offers impact properties that can meet requirements for instrument panels with out the need for padding. Coated mark resistant glazing provides an extra margin of durability in mass in transit vehicles. In construction, PC is used for door and window components. Hardware application includes drapery fixtures, furniture, plumbing, and sophisticated electrical and electronic products.

Housing and internal parts of printers, copiers terminal, business equipments, as well as telephone connectors, circuit boards, wiring blocks and other industrial components are often molded of PC.

Polycarbonate (PC) sheet manufactures have developed special products for the solar, agriculture, security and graphics markets. Coated thin-gauge sheet is growing in popularity. PC films also offers potential in the metalizing, packaging and tape markets. A widening spectrum of security needs laminated PC sheet a good choice for applications requiring bullet resistance and the ability to with stand forced entrance.
3.1.1.5 POLYURETHANE (PU)

\[
[ - O - (CH_2)_x - O - CO - NH - (CH_2)_y - NH - CO - ]_n
\]

Figure 3.5. Chemical Structure of Polyurethane

Polyurethane (PU) elastomers have a wide range of industrial applications, and they are well known for their mechanical performance. The starting materials of these elastomers are a long chain polyol and a diisocyanate. A lot of work has been carried out to develop PU for different applications using renewable resource like castor oil.

Fibre reinforced plastic composites (FRPC's) are of tremendous importance both in end-use applications and in the areas of research and development. These composites can be designed to exhibit both soft and stiff behavior. Textile composites are superior to other materials on strength to weight ratio and stiffness-to-weight basis, making them especially suitable for the applications where weight saving is an important issue.

3.1.2 Glass fibre

Glass has defined as an inorganic product of fusion which, when cooled, becomes rigid without crystallizing (i.e., its atoms never arrange themselves an ordinary crystalline pattern). Glass fibre is commercially part of the plastics and electrical industry as it is used either as a reinforcement in reinforced plastics or as yarn/fabric in electrical insulation. E-glass is lime-alumina borosilicate glass first developed for producing continuous fibres is designed for electrical and mechanical applications. It proved effective in a variety of processes and is now standard glass reinforcement. It has high bulk and surface electrical resistivity as well as excellent fibre forming characteristics. E-glass fibre the form of chopped strands, chopped strand mat, rovings, woven rovings and own cloth is commonly used as reinforcements in the manufacturing of the GRP.
The glass fibres have following characteristics:

- The very high tensile strength to weight ratio.
- High thermal conductivity, low thermal expansion and retention of strength at high temperatures.
- Excellent moisture resistance.
- Outstanding dimensional stability and excellent resistance to solvents and acids.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity</td>
<td>2.54-2.56</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>260-360 KN/m x 10</td>
</tr>
<tr>
<td>Modulus of elasticity</td>
<td>7.0-7.3 KN/m x 10</td>
</tr>
<tr>
<td>Index of refraction</td>
<td>1.547</td>
</tr>
<tr>
<td>Softening point</td>
<td>486°C</td>
</tr>
</tbody>
</table>

Composites reinforced with glass fibre or characterized by high tensile strength over a wide temperature range, high stiffness, improved creep behaviour. They may be short or long fibres. The forever imparts and high toughness with good stiffness.

E-glass is a popular fibre made primarily of silica oxide, along with oxides of aluminium, boron, calcium and other compounds. This is characterized by low alkali composition. Exhibits excellent electrical insulation properties and used in textile fibre glass production. This is given prime consideration because of excellent resistance to attack. E-glass shows lesser degradation of properties with time, high tensile strength and low dielectric constant. E-glass is strong at low in cost and accounts for over 90% of all glass fibre reinforcements, especially in air-craft, radomes, antennae and applications where radio signal transparency is desired. E-glass is also used in computer circuit boards where stiffness where electrical resistance are required.

### 3.2 EQUIPMENTS

The various equipments used to characterize the properties of the test specimens is discussed.
3.2.1 DENSITY

Density of nonwoven fabric reinforced-polymer composites are measured according to ASTM D 792-86 (displacement) method using Metler electronic balance. This technique is applicable for solid samples such as film, sheet or powder. The accuracy of density obtained by this method is ±0.0001 g/cc. The Metler electronic balance should be standardized as per standard procedure.

3.2.2 SURFACE HARDNESS TESTER

Hand operated Hiroshima durometer is used to measure the surface hardness (shore D) of the fabricated composites according to ASTM D 2240. The relative hardness scale is in the range 0-100 shore D.

3.2.3 UNIVERSAL TESTING MACHINE

The tensile properties such as tensile strength and percentage elongation of reinforced polymer composites are performed according to ASTM D 638 using Hounsfield Universal Testing Machine at sundaram ind., (UTM) H50 KM, model 4302, UK. Minimum of six samples are tested at room temperature for each composition and the average value is reported. Based on the density and tensile strength values, the specific tensile strength and reinforcement factor of the composites have been calculated.

3.2.4 IZOD IMPACT

The impact properties of the test specimens are carried out according to ASTM D 256 using Instran, Dynatup® 9200 Series which are suitable for a wide variety of applications requiring low to high impact energies. Designed for testing both raw material specimens and finished components. The machine has impact energy up to 1010J, impact velocity up to 5m/s and the maximum drop height 1.25m. Minimum of
four samples are tested at room temperature for each composition and the average value is reported.

### 3.2.5 RADIAL DRILLING MACHINE

The drilling operations are carried out on a BATLIBOI make radial drilling machine BVR3 with 12 speeds ranging from 56 to 2800 rpm, there are 6 numbers of feed available in ranges 0.03-0.30 mm/rev with 50 Hz frequency.

### 3.2.6 SURFACE ROUGHNESS

Surface roughness of the fabricated composites is performed according to ASTM using Carl Zeiss, Model: Surfcom130A, Sl. No: KA1307DA, Made in: Japan, traverse: 50 mm Max, Least Count: range/64000, Accuracy: 0.3Mic. / 50 mm band.

### 3.2.7 WEAR TEST

The wear test equipment used in this investigation is WINDUCOM, TR-2L, pin-on-disc type setup.

### 3.2.8 MACHINABILITY (ACOUSTIC EMISSION)

The instrument AET 550 (Plate II) consists of a piezoelectric transducer with a resonant frequency of Hz, which senses the AE wave and converts it into electrical voltage, which is sent to a mainframe for processing through a pre amplifier. The pre amplifier used is a 160 model, which has a gain of 60 dB. It has got an AET filter with a pass band 30 KHz to 2 MHz (wide bands). The AET mainframe with its 16-bit microprocessor performs the entire signal processing for the AE signal. It has got eight channels for monitoring and processing AE data. The common system module include signal processing units (SPU 2) which is a dual channel, adjustable gain post amplifier that begins the digitization of the AE signal, voltage control gate and the model 207 audio unit.
3.2.9 X-RAY

The x-ray diffraction results are carried out by using D/max 2200 x-ray diffractometer supplied by M/s Rigaku International Corporation, Japan. Cuk α with graphite monochromatic, scintillation counter and diffractotmeter are used.

3.2.10 SCANNING ELECTRON MICROSCOPE (SEM)

Surface morphology of fractured surfaces of composites is performed using SEM, (LEICA 5440i, model-7060), Oxford. Surface of the samples are gold coated before analysis.

PART B

3.3 THEORY AND TECHNIQUES

The fabricated composites have been characterized by tests namely tensile, impact, shear strength and sliding wear. Composites have been studied for machinability test for tool wear. Physical methods such as density and surface hardness and thermal properties like HDT and VST. The tensile strength of composites has been theoretically calculated by FEM and the results are compared with the experimental results. The morphology for both surface and tensile fractured composites by SEM and crystalline parameters by x-ray method has been studied. The composites are also been studied for x-ray spectrogram. The following paragraphs provide a brief theory, technique and experimental procedure adopted for each of the above methods.
3.3.1 PHYSICAL PROPERTIES

The following physical properties have been characterized for the test specimen at room temperature.

(i) Density of composites

"Archimedes Principle" performs specific density determinations. This principle states that every solid body when immersed in a fluid apparently loses weight by an amount equal to the fluid it displaces.

Density of composites is measured according to the ASTM D 792-86 (displacement method) using Mettler electronic balance. The instrument is calibrated before use. This technique is applicable for solid samples such as film, sheet or powder. The accuracy of density obtained by this method is ± 0.0001 g/cc.

A specimen of solid plastic is weighed in air. It is then immersed in a liquid of known density loss in weight is determined, and its density is calculated. The specific density of a solid is a property that can be measured conveniently to define a material, to follow physical changes in a sample, to indicate degree of uniformity among different sampling units or to indicate the average density of a large item.

(ii) Surface hardness test

Hardness is the property of the material showing resistance to surface indentation. Surface hardness of composite is performed according to ASTM D 785 on Shore D tester. The sample is kept under indenture of the Shore D tester and the deflection on the scale is noted. The indentation value reflects the resistance to local deformation, which is a complex property related to modulus, strength, elasticity, plasticity and dimensional stability. It also gives an idea about degree of cross linking/interlocking.
The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and viscoelastic behaviour of the material. The shape of the Indentor and the force applied to it influence the results.

The test is carried out by first placing a specimen on a hard, flat surface. The pressure foot of the instrument is pressed onto the specimen, making sure that it is parallel to the surface of the specimen. The Durometer hardness is read within 1 second after the pressure foot is in firm contact with the specimen.

(iii) Ash content

The following procedure adopted to determine ash content of the reinforced samples. The empty crucible is cleaned well and dried. The crucible is weighed in an analytical balance with an accuracy of 0.0001 gm ($W_1$g). The crucible is filled with a small quantity of the sample. Once again its weight is recorded ($W_2$g). Now, the crucible is kept in the furnace maintained at 600 - 800°C for 4 - 5 hours. After the completion of pyrolysis of the specimen, the crucible is cooled well with desiccator and the weight ($W_3$g) is recorded. The ash content of the specimen is due to the inorganic filler in the specimen. The percentage of ash content of the sample can be calculated using the following equation

\[
\text{Percentage of ash content} = \frac{(W_3 - W_1)}{(W_2 - W_1)} \times 100
\]

where, $W_3 - W_1$ is the weight of the residue and $W_2 - W_1$ is weight of the sample taken.

(iv) Surface roughness test procedure

1. Probe calibration is done before measurement
2. Individual operations are carried out as per Surfcom 130A Instruction manual.
3. Clean the component before measurement
4. Component has to place parallel to the axis of measurement
5. Set all the comments and defaults before starting measurement.
6. Set the off length by using Cut off length selection chart displayed near equipment
7. After measurement take the print outs in the required formats
8. Enter the Roughness Register after measurement.
3.3.2 MECHANICAL PROPERTIES

The following mechanical properties have been investigated and the data are useful for qualitative characterization, engineering design and research.

(i) Tensile test

This test method is designed to produce tensile properties such as tensile strength, modulus of elasticity (Young's modulus), and percentage of elongation for control and specification of composite materials.

Tensile test is a measure of the ability of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking. Tensile properties such as tensile strength and percentage elongation at break are determined according to ASTM D 638. The specimens from the flexible sheets are prepared with the help of a punching die, whereas for rigid sheets, dumb bell-shaped specimens are prepared by first cutting 165 mm long and 15 mm wide rectangular pieces and then shaping them using filing. A gauge length of 35 mm full scale, cross head speed of 50 mm/min and load of 100 kg, is adopted. Minimum of four samples are tested for each composition and an average is taken. Mathematical expressions used to calculate tensile behavior are as follows;

\[
\text{Tensile strength (MPa)} = \frac{\text{Load at break (N)}}{\text{Cross sectional area (mm}^2\text{)}}
\]  

\[
\% \text{Elongation} = \text{Strain} \times 100
\]

Based on the density and tensile strength values, the specific tensile strength and reinforcement factor have been calculated using the relations;

\[
\text{Specific tensile strength (Nm/g)} = \frac{\text{Tensile strength (N/m}^2\text{)}}{\text{Density (g/m}^3\text{)}}
\]
Tensile test, in a broad sense, is measurement of the ability of a material to withstand forces that trend to pull it apart and to determine to what extent the material stretches before breaking. Tensile modulus, an indication of the relative stiffness of a material, can be determined from a stress-strain diagram.

Tensile measurements of reinforced samples are carried on Universal Instron testing machine model 4302, with standard and dumbbell shaped specimens prepared according to ASTM D638. The dimensions of the specimen are specified according to M-I type in this standard. The draw rate is 5 mm/mm.

(ii) Izod impact test

The machine components, which are subjected to sudden applied loads, are called impact blow and hence this test enables to estimate the property of toughness of a material.

Standardized pendulum type hammers, mounted in standardized machines, in breaking standard specimens with one pendulum swing.

![Izod impact test specimen](image)

A=30mm  B=63mm  C=126mm  W=3mm

Figure 3.6. Izod impact test specimen

The specimen is held as a vertical cantilever beam and is broken by a single swing of the pendulum with the line of initial contact at a fixed distance from the specimen clamp and from the centerline of the notch and on the same face as the notch. The notch produces a stress concentration, which promotes a brittle rather than a ductile fracture.
(iii) Sliding wear behaviour

The tribological properties of polymer composites are studied using a pin-on-disc thermometer (wear and friction monitor DUCOM TR-2L developed according to ASTM G99. the counterface disc is made of stainless steel. the centre line average surface roughness (Ra) of the ground disc is 0.6 μm. The friction force is measured using a force transducer fixed on the loading lever arm the temperature of the disc is measured using a noncontact type infrared sensor. Friction force and disc are measured and data are stored using a personal computer based data acquisition system.

Abrasive wear studies are carried on a dry sand rubber wheel abrasive test rig as shown in Figure 3.7. The abrasive (silica sand of AFS 60 grade) is fed at the interface between the rotating rubber wheel (200 rpm) and the test sample measuring (75 × 25 × 5) mm³. The weight of cleaned and dried composites is recorded in an analytical balance (0.1 mg accuracy) before it is subjected to wear analysis. The particles of silica sand used are angular in shape with sharp edges having hardness of 7.0 Moh’s scale. The abrasives are introduced between the test specimens and rotating abrasive wheel composed of chlorobutyl rubber tyre (hardness: Durometer-A60). The test specimen is pressed against rotating wheel at a specified force by means of lever arm while a controlled flow of abrasives abrades the test surface.

The rotation of the abrasive wheel is such that its contact face moves in the direction of sand flow. The pivot axis of the lever arm lies within a plane, which is approximately tangent to the rubber wheel surface and normal to the horizontal diameter along which the load is applied. At the end of set test duration, the specimen is removed, thoroughly cleaned and again weighed (final weight). At least three tests are performed and the average is used in the data representation. The abrasive wear studies are carried out at two different loads (22 and 32 N) at a constant sliding velocity of 2.15 m/s.
For varying abrading distances (150 - 600 m in steps of 150 m) samples are abraded for the appropriate time intervals. Weight loss measurements are made at regular test intervals using digital electronic balance. The wear is measured by the loss in weight, which is then converted into wear volume using density data. Before and after wear testing, samples are cleaned with acetone in an ultrasonic cleaner and then dried. The specific wear rate ($K_s$) is calculated from the equation:

$$K_s = \frac{\Delta V}{L \times d} \ (m^3/N \ m)$$ (1)

Where, ‘$\Delta V$’ is the volume loss in $m^3$, ‘$L$’ is the load in Newton and ‘$d$’ is the sliding distance in meters.

3.3.3 MACHINABILITY

Machinability is the property of a material, which governs the ease or difficulty with which a material can be machined using cutting tools. The term machinability is in
wide use by those concerned with engineering manufacture and production. Enormous amount of literature available on machinability reveals that a measure of vagueness about its precise definition or even its general meaning.

It is difficult to identify generally accepted parameters, which would define machinability comprehensively. Also, the meaning attributed to the term machinability tends to show the immediate interest of the user. Machinability becomes finish ability for the engineers who are especially concerned with surface finish problems of the components produced. Others may be of the opinion that the term machinability should be used to indicate the consistency with which a material behaves in a particular machine tool set-up under a specified set of machining conditions.

In an industrial situation, useful life of cutting tool may indicate machinability. Hence, attempts to define machinability have to consider the characteristic of the machining process such as cutting tool life, the tool wear rate, the energy required to remove the unwanted material and the quality of the machined surface that can be produced.

Modern trend towards defining machinability has been the measurement of vibration and noise of cutting tools in a machining operation [124]. Cutting noise can be measured accurately by computer based Acoustic Emission Sensing (AES) [47]. The primary and the most important advantage of an AES based sensing is that the output of AES is related to machinability and online measurement of it and storing is possible.

(i) Historical development

An important step towards the adequate appreciation of the undertaking of this research study lies in the consideration of attempts to quantity machinability from past of the present and how it may develop in future.

The earliest attempts to quantify machinability date back to the industrial revolution. It is not until Taylor classified in 1906 that the term machinability began to be addressed analytically [157]. Significant contributions towards the understanding of
machining process are made about 35 years later by Ernst and Merchant [51]. They used thermodynamic approach and proposed a specific cutting energy $P_s$, which they used to describe the cutting efficiency of the cutting process. One proposal is that it should therefore be possible to define machinability in terms of a thermal number “$R$”, which takes relevant properties into account such as density, heat capacity, velocity related to cutting speed, chip area and work piece thermal conductivity.

Later, simple ranking systems are constructed to quantify machinability. These are based on single mechanical property such as hardness. Sophisticated attempt to relate physical and mechanical properties with machinability has been made by Boulger et al. [17, 18, 77, 87]. Henkin and Datsko [76] have made considerable contribution towards the relatively adequate definition of machinability regarding a particular class of materials, such as the leaded steels. However, these tests do not permit the valid comparison of machinability of different classes of materials, e.g., between steels and non-ferrous metals, including polymer (thermoplastic) composites.

A significant contribution towards the definition of “Utilitarian machinability” is made by the publication of ASTM E618-771 in the year 1977. ASTM 918 is designed to simulate mass- production conditions in a controlled environment-using single or multiple spindle automatic screw machines. Method E618 is a ranking type production oriented test. But many researchers have shown that it can also be used to generate broadly applicable quantitative data.

From the year 1980 to the present day, researchers have constantly strived to identify more and more efficient ways to quantify machinability. This is the consequence of a very rapid technological growth during this period. The order of the day is the need for the machining systems to operate at optimum efficiency, which requires high production rate at low unit cost. This automation optimization scheme calls for reliable detection of tool wear and fracture, on line, to reduce machine downtime, product rejects and improve personnel safety. It is further expected that the machining operations take place with a minimum of human attention. This calls for the use of Acoustic Emission (AE) in defining the machinability. One should also note that these characteristics render themselves to be measured on line when machining
operations are being performed. In the year 1990 Blum and Inasaka [16] have demonstrated the usefulness of AE in the determination of tool wear which is an important characteristic used to define machinability.

(ii) Acoustic emission (AE) measurement

Some important digital modules of the signal processor unit include ring down counts, event duration module, and amplitude rise time module. The AET 5500 Systems Intelligent Graphics Terminal (IGT) is a complete microcomputer using MS-DOS operating system. It shows the desired AE data on the display in a format we specify before the test using specific software command. The IGT has a hard disc drive on which the AE data to be saved can be stored. The IGT keyboard can be used to enter all of the software commands to setup sensors and displays to monitor, measure and show the desired AE data. The AET 5500 software is the most important component of the system. Using this software we can setup the system to perform various types of tests and process the data to produce the desired display output.

(iii) Criteria for machinability

Machinability can be judged from many considerations depending on the employed machine tool, cutting tool, work material an cutting conditions and also on the preference of the user for a particular choice. The general criteria commonly adopted for evaluating machinability is tool life/tool wear rate and cutting force or surface finish produced on a job. Assessment of machinability can also be based on specific parameters like torque and thrust during machining, penetration rate, ease of chip disposal, temperature of cutting tool, work hardening, etc. From practical considerations, the criteria can be restricted to tool life or tool wear rate, cutting force and surface finish. It is evident that these relate, in general terms, to the cost of machining operations and can be expressed in quantitative terms for purposes of comparison. They are the most commonly accepted measures of machinability.
(iv) Criterion based on tool life

Tool life is usually the most important of the three main parameters used for assessing machinability. This could be conveniently expressed in terms of cutting speed; because, all the other variables being kept constant, tool life will be a direct function of the cutting speed. By increasing the cutting speed, the tool life may be decreases and by reducing the cutting speed the tool life may be increased. Thus, cutting speed for producing a predetermined value of tool life, termed as the specific cutting speed, could be made on the basis of comparison of machinability of materials. For example, if $V_s$ is the cutting speed to produce a tool life $T$ for a standard material, and $V_t$ is the cutting speed to produce the same tool life $T$ for the test material, then machinability of the test material can be expressed as $V_t / V_s \times 100$. The higher the cutting a material allows for a certain tool life, the better is its machinability.

The cutting speed is a direct indication of the cost at which a part can be produced and hence, the machinability rating, based on cutting speed or tool life, provides a firm basis for comparison of various materials.

(v) Criterion based on cutting forces

Machinability rating based on the cutting force is important, where it is necessary to limit the values of cutting force in keeping with the rigidity of the machine and to avoid vibrations during machining. If the cutting force is high and consequently the power consumption is also high, a larger machine tool may be required, thus increasing the overhead cost and unit cost of the part produced. The higher the cutting forces induced under a set of cutting conditions during the machining of a material, the lower is its machinability index.

(vi) Criterion based on surface finish

There are many situations where surface finish on the job is of primary importance. Though a given material may allow higher cutting speeds or induce lower...
cutting forces, it may not produce good surface finish. Where the finish produced on the parts is a cause for reflection this consideration has an important bearing on the cost. The higher the surface finish obtained on material under a given set of conditions, the better is its machinability.

(vii) Theoretical background for AE

Acoustic emission (AE) may be defined as the class of the phenomenon where transient elastic waves are generated by the rapid release of energy from localized sources within a material. In other words acoustic emission refers to the stress wave generated by dynamic processes in materials. Emission occurs as a release of a series of short impulsive energy packets. The energy thus released travels as a spherical wave front and can be picked from the surface of a material using highly sensitive transducer, usually Electro mechanical type, placed on the surface of the material. The mechanical waves thus picked up is converted into electrical signal which on suitable processing and analysis can reveal valuable information about the source causing the energy release. The process of generation and detection is illustrated in Figure 3.8.

![Figure 3.8. Principle of generation and detection of AE signals](image)

The source of the acoustic emission energy is the stress field in the material. Without stress there is no emission. The definition of acoustic emission given above indicates that processes that are capable of changes in the internal structure of a material such as dislocation motion, directional diffusion, creep, grain secondary sliding and twinning, which result in plastic deformation, phase transformation, vacancy coalescence, decohesion of inclusions and fracture, are sources of acoustic emission.
The amount of AE energy released depends primarily on the size and speed of the local deformation process.

(viii) Types of AE signals

There are two types of AE signals, namely (i) The high amplitude, somewhat erratic, (ii) low frequency type called the burst emission, with is generally associated with surface events, and such as slip line formation and surface micro cracks. In this the individual events are not discernible. Individual burst lasts for a few microseconds to several milliseconds.

![AE Burst Signals](image)

![AE Continuous Signal](image)

Figure 3.9. Types of AE Signals (a) AE burst signals and (b) AE continuous signal

The lower amplitude, steady and high frequency type is called the continuous emission and is generally associated with internal mechanism activity. Continuous emission refers to the emission coming from rapidly occurring events. If burst emission
occur rapidly that the burst overlap, the result is continuous emission and is shown in Figure 3.9 (a) and (b).

(ix) Features of acoustic emission

(a) Frequency range

The frequencies encountered in the acoustic emission range from the low end of the audible spectrum to the mega hertz (MHz) range. The specific material characteristics that are of importance are the size and the shape of the material and its frequency-dependent attenuation effects. The transducer and the electronic filters in the instrumentation are the principle equipment components that affect the frequency spectrum.

In actual practice, frequencies below some arbitrary limits, such as 30 KHz, are filtered to avoid interference from unwanted sources of noise such as machinery, impact sounds or electrical equipments.

Higher frequency (greater than a few MHz) are lost because of attenuation or because they are removed by the instrumentation. The frequencies that are commonly used for AE testing lie between 50 KHz and 1 MHz.

(b) Stress waves

The types of stress waves by an acoustic emission source depend on the nature of the source and its location within the material. A source below or at the surface of a solid can produce longitudinal, shear and surface waves. Waves produced below the surface radiate in all directions from the source. When they reach the surface they are reflected, and a part of their energy is converted to other types of waves including surface waves. If the source of the emission is in a plate, most of the energy will be propagated as shear waves, plate waves and surface waves. The signal i.e., detected by a typical sensor is usually a surface wave because they are attenuated less.
(c) Attenuation of AE signals

The intensity of an acoustic emission signal decreases as distance from the source increases. The reasons for this decrease are geometrical factors, mode conversion and energy absorption and scattering. The effect of geometrical factors depends on the size of the acoustic emission source, the wavelength of the acoustic emission signal and the presence or absence of nearby reflecting surfaces. Energy loss due to mode conversion can occur if an acoustic emission signal is reflected one or more times from surface before reaching the sensor. The loss mechanism due to scattering and absorption in solids are complex.

(d) Instrumentation

Equipment’s for processing AE signals is available in a variety of forms ranging from small portable instruments to large multi channel systems. The instrumentation of AE test equipment provides the necessary detection of continues emission of detectable burst type emissions. Components common to all systems are sensors, preamplifiers, filters and amplifiers to make the signal measurable. Methods used for measurement, display and storage more widely according to the demand of the application.

Acoustic Emission Sensors- When an AE wave front impinges on the surface of the test object, very minute movement of the surface molecules occurs. Sensor’s function is to detect this mechanical movement and convert it into a specific, usable electric signal. The main considerations in sensor selection are: operating frequency, sensitivity, environmental and physical characteristics.

One of the most sought after properties in an AE sensor is high sensitivity. Although high fidelity, flat frequency response are available, most practical AE testing employes resonant type sensor that are more sensitive, as well as less costly and of the flat frequency response type.
(e) Preamplifier

The preamplifier must be located close to the sensor. Often it is actually incorporated into sensor housing. The preamplifier provides required filtering, gain and cable drive capability. Filtering in the preamplifier is the primary means of defining the monitoring frequency for the AE test. This may be supplemented by addition filtering at the main frame. The preamplifier includes a high pass or band pass filter to eliminate the mechanical and acoustical background noise that prevails at low frequency. The choice of operating frequency is a trade off between noise and detecting range.

System mainframe—the first elements in the mainframe are the main amplifiers and thresholds, which are adjusted to determine the test sensitivity used. Main amplifier gain in the range of 20 to 60 dB is most commonly used. Instruments vary widely in form, function and price. In a small portable instrument AE events or threshold crossings may be simply counted on a chart recorder. In more advanced hardware systems, provisions may be made for energy or amplitude measurement, spatial filtering and automatic alarms.

(x) AE system accessories

Accessory items used in AE work widely include oscilloscopes, transient recorders and spectrum analyzer, magnetic tape recorder, rms voltmeters and devices for simulating AE. Specific advantages of AE as related to material cutting are

1. Changes in AE signal level occurs almost at the instance of tool fracture, whereas that in the force level occurs only after the tool has broken or chipped off.
2. The frequency range of the AE signal is much higher than that of the machine vibrations and environmental noises. Therefore a relatively uncontaminated signal can be easily obtained by the use of high pass filter.
3. AE can be measured by simply mounting a piezoelectric transducer on the tool holder. It does not interfere with the cutting operation thus allows for continues monitoring of the tool conditions. However, due to its high frequency nature and the sensitivity to micro-structural behavior of material, AE signals often have to be
treated with additional signal processing schemes so that the most useful information can be extracted.

4. Several investigators have suggested the AE signal to be sensitive to the prevailing cutting conditions, tool fracture, including internal cracking recognition prior to tool fracture. There is also evidence to suggest that there is a correlation between the AE and extent of tool wear at any given time.

5. Since AE signals are not influenced by the dynamic characteristics of the machine tool, implying that such techniques would be readily transferable one machine tool to another.

6. AE monitoring has proven to have an advantage over many other techniques because the output acoustic emission signal is directly related to the basic mechanism of the cutting process.

(xii) AE signal measurement parameters are

1. Ring down count (RDC)/(RCT): the number of times the signal amplitude exceeds the preset threshold.
2. Cumulative count: the number of times the amplitude of the signal has exceeded the threshold since the start of the test.
3. Event: a micro displacement-giving rise to transient elastic waves. The event begins when the signal pulse first crosses the threshold and ends when it last crosses the threshold. High amplitude, short duration and exponential decay characterize AE events.
4. Rise time: this is the time duration for the rise from an event’s first crossing until the signal reaches its peak amplitude. This is measured in microseconds.
5. Event duration: it is defined as the amount of time that passes between the event’s first threshold crossing and last threshold crossing.
6. Peak amplitude: it is the highest amplitude reached by the AE signal during an event. It is measured in decibel (dB). This is the very important parameter because it directly determines the detectability of the AE event.
7. MARSE: some times known as energy counts. Is the measured area under the rectified signal envelope MARSE is preferred over counts because it is sensitive
amplitude as well as duration and it is less dependent on threshold setting and operating frequency.

8. Reference threshold: a preset voltage level that has to be exceeded before an AE signal is detected and processed. The threshold may be fixed or automatic.

9. Mean rise time per event: summing the rise time value interval and dividing the same by the number of events in that interval obtain it.

The above parameters are schematically shown in Figure 3.10.

![Figure 3.10. AE Parameters](image)

(xii) Acoustic emission in material cutting

Materials undergoing deformation or fracture generate acoustic emission. Both of which occur when chips are formed in machining. Continues type of AE signals is associated with plastic deformations in ductile materials while burst type signals are observed during crack growth in the material. Additionally, chip impacts or chip tangling generates burst type AE signals. It is generally agreed that during material cutting, plastic deformation and fracture of the material are the major sources for AE waves. Operation of deformation mechanisms requires the application of stress and consequent expenditure of energy. Generally the energy applied results in elastic energy, which is stored and plastic work of deformation that is irrecoverable. The plastic work of deformation occurs mainly by the motion of dislocation and is the
source of acoustic emission. The motion of dislocation requires the supply of a certain amount of energy to overcome drag forces. As it releases the strain energy applied in overcoming the drag forces, a stress wave is produced the material which causes displacements on the surface of the material that can be picked up as AE.

(xiii) Machining

Machining is the term that covers a large collection of manufacturing process designed to remove unwanted material usually in the form of chips, from a work piece. Machining is used to convert castings; forging of preformed blocks of materials into desired shape, size and finishes specified to fulfill design requirements. Almost every manufactured product has components that require machining often to great precision.

The majority of industrial applications of machining are in materials although the material cutting process has restricted theoretical analysis because of its complexity. Machining processes are performed on a wide variety of machine tools such as lathes, milling and drilling machines.

(xiv) Tool wear

Cutting wear is a process of gradual loss of tool material from the cutting edge through interactions between the tool and the work piece. Observed tool wear can generally be classified into crater wear and flank wear. Flank wear is the land produced on the side faces of the tool below the cutting edge and the crater wear is the characteristic cavity produced on the rake face which begins at a finite distance from wear directly affects the surface finish and dimensional accuracy. It reduces the depth of cut and results in an over sized work piece. Since the worn tool surface is irregular and rough, so is surface of machined part.

(a) Mechanisms of tool wear

There are four wear mechanisms, which can operate singly or in various combinations, to produce tool wear. These four mechanisms are adhesive wear, abrasive wear, electrochemical wear and diffusion wear. At low temperatures,
mechanical wear process, such as abrasion and adhesion, are rate controlling and the wear of the tool is determined by its hardness. At high temperatures, the wear is predominantly determined by the chemical properties of the tool and work piece material; hence diffusion wear will be dominant.

(b) Tool life

The single most important element in material cutting is the tool life. It can be defined, as the cutting time required reaching tool-life criterion or when the tool no longer produces a satisfactory part. Therefore, all conditions, which lead to a shorter tool-life, are uneconomical because of the higher tool replacement cost. On the other end, low speed and feed rate, which bring about longer tool life, is uneconomical as well, because of the low production rate. Hence, a compromise is required in order to seek the optimum tool-life. Tool-life criterions can be due to:

- Gradual or progressive wear at the cutting edges, tool breakage, chipping, colorization at the cutting edges and vibration generated as wear increases.
- Contact with the work piece surface. The mechanical movement of stylus tip due of the undulations on surface is converted into digital values and stored in M4Pi profile memory.

(c) Procedure

Preliminary works are carried out to arrive at the standard speed and fed, for the choose speed of 560 rpm and feed of 0.08 mm/rev., the time is sufficient for recording the vibration signals.

Cutting test are carried out o a BETLIBOI make radial drilling machine with a 10mm taper shank twist drill at a sped of 560 rpm and a feed of 0.08 mm/rev. The depth of cut is fixed at 20mm. Also 8mm and 12mm taper shank twist drill at a speed of 560rpm and a feed of 0.08 mm/rev, the depth of cut is fixed at 30mm. The piezoelectric transducer is fixed on the work piece using a layer of couplant to ensure good coupling. The transducer is mounted horizontally at the centre of the work piece as this position is
found to be the best during preliminary works. The sensor is held firmly on to the work piece using insulation tape.

The pencil lead break test has been used on in the calibration of AE to estimate the attenuation factor of the AE Signal when the signal is transmitted form the work piece to cutting tool.

Electrical signal produced by the transducer are first amplified with a pre-amplifier of 40 dB gains. The defected signals are amplified and filtered through band-pass filter.

The threshold voltage is set for to 1.00 V (automatic) using AET software command. The drilling operation is carried and the gain switch of the signal processing unit (SPUZ) is adjusted unit the LED on the front panel of the SPCZ started mashing indicating threshold crossing by the AE signal. The conditioned signals are received in the computer for further analysis.

Flank wear is measured after every twenty holes using toolmakers microscope. The AE signals reading wear taken for 20 holes for which considerable wear is observed during preliminary works.

3.3.4 THERMAL PROPERTIES

The thermal properties of plastic materials are equally important as the mechanical properties. Unlike metals, plastics are extremely sensitive to change in temperature.

(i) Heat distortion temperature

Heat deflection temperature (HDT) is defined as the temperature at which a standard test bar deflects 0.25mm under a stated load of 455kPa. HDT is used to determine the temperature at which an arbitrary deformation occurs when composites are subjected to an arbitrary set of testing conditions.
A bar of rectangular cross section is tested as a single beam with the load applied at its center to give maximum fibre stress of 455 kPa. The specimen is immersed in a heat transfer medium with a means of raising the temperature at 2 ± 0.2 °C/min. The temperature is recorded when the test bar has deflected 0.25 mm, which is the HDT of the test specimen.

(ii) Vicat softening temperature

Vicat softening point (VST) is the temperature at which a flat ended needle of 1sq-mm circular cross section will penetrate a thermoplastic specimen to a depth of 1 mm under specified load and uniform rate of temperature raise. Rate is 120 ± 12 °C/h.

Data obtained by this test method may be used to compare the heat softening qualities of thermoplastic materials. This test is useful in the areas of quality control, development and characterization of materials.

3.3.5 CHEMICAL RESISTIVITY

The chemical resistance of short glass fibre reinforced polymer composite is carried out according to the ASTM D543-67 (1972) method. The selections of test conditions are taken into account, the manner and the duration of contact with the
chemical reagents, the temperature of the systems and other performance factors involved in the particular applications. The limitations of the results obtained from this test method should be recognized. The selected chemical reagents or solvents for measuring the chemical resistance are 10 % sodium hydroxide (NaOH), potassium permanganate (KMnO₄), salt solution (NaCl 10 %, 20 % and 30 %), hydrogen peroxide (H₂O₂), benzene, ethyl acetate, hydrochloric acid (HCl) and nitric acid (HNO₃).

The composite of required dimensions (10mm x 30mm x 3mm) are cut and weighed accurately. The samples are immersed in a wide mouthed reagent bottle along with the reagent for 7 days in the standard laboratory atmosphere. The samples are hung so as to avoid any contact with the walls or bottom of the reagent bottle. After 7 days the specimens are removed individually from the reagent bottle and gently wiped with the tissue paper and reweighed. The difference in weights and thickness are calculated which in turn gives the idea about the resistance of samples to various chemical environments.

3.3.6 X-RAY

The x-ray diffraction results of the α composites film prepared x-ray patterns are recorded by using D/max 2200 x-ray diffractometer supplied by M/s Rigaku International Corporation, Japan. CuKα with graphite monochromatic, scintillation counter and diffractotmeter are used. Pattern is recorded for α-phase composite film, for the 2θ range of 10-60⁰, most intense peaks are observe at 2θ=20.30⁰. It shows the dominance of α-phase, which is the most stable phase of the composite, it will be in the TG conformation and non-planar form. The theoretical formulas are used to calculate 2θ values.

3.3.7 SCANNING ELECTRON MICROSCOPE (SEM)

The tensile fractured surface morphology of the various composites is well documented in the literature. The polymer composite gives an interesting SEM photographs. Since surface morphology plays a major role on the performance of polymer, it has been extensively studied using SEM.
The basic principle of the image formation on microscope is dependent on the interaction of a focused electron beam with the spectrum, which enables collection of information pertaining to topological structural nature of the specimen under examination. The electronic beam scans the specimen area with the help of deflection coils with a variable-scanning rate employed for observation and photographic recording.

Scanning a focused beam of electrons across the surface of a specimen and detecting the secondary electrons that are ejected by the specimen form conventional SEM images. The interactions of the electron beam with the sample surface cause a penetration of the electron into material up to a considerable depth before losing their energy. The amount of lost energy depends upon the thickness of the sample. Depending on the type of interaction, different types of images are formed. A suitable detector also gains the secondary electrons, which is amplified and modulated to control the brightness on cathode ray tube.

A piece of specimen about \((1 \times 2)\) mm size is fixed on the sample holder using adhesive tape and is then coated with a thin layer of gold to improve image resolution. The magnification of the sample is printed on the corresponding SEM micrographs.

3.3.8 Fabrication of SGFR composite

The short glass fibre is incorporated with different weight ratios using twin-screw extruder in the temperature range \(245^0\text{C} - 260^0\text{C}\), fabricated at M/s Brakes India Limited, Nanjangud.

Composites are pre dried at \(105^0\text{C}\) for 24 hrs prior to compounding. The composite is compounded with short glass fibre (SGF) in Berst off Extruding machine with l/d ratio 1:18 of capacity 100 - 120 kg/hr. The machine consists of 9 nozzles and temperatures zones are maintained at each nozzle are different and lie in the range \(245^0\text{C} - 260^0\text{C}\). The maintained vacuum, torque and speed of twin-screw extruder are 17 Hg/mm, 50 to 55 Nm and 280-300 rpm respectively.
The specimens are fabricated according to ASTM standards using Windsor Sp80DD, injection-moulding machine with 80 ton capacity. The screw speed is 90-100 rpm and zone temperature for composites are maintained 240°C and different zone temperatures are 230°C, 220°C and 210°C. The throat temperature is 50°C - 60°C. The pre-dried filled composites are pellets injected at pressure of 700 N/mm² for specimens.