PREPARATION OF PREFORMED YARN TO SYNTHESIZE CARBON-CARBON COMPOSITES

Thesis

Submitted in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

by

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DECLARATION

by the Ph.D. Research Scholar

I hereby *declare* that the Research Thesis entitled **Preparation of preformed yarn to synthesize carbon-carbon composites** which is being submitted to the **National Institute of Technology Karnataka, Surathkal** in partial fulfillment of the requirements for the award of the Degree of **Doctor of Philosophy** in Mechanical Engineering is a *bonafide report of the research work carried out by me.* The material contained in this Research Thesis has not been submitted to any University or Institution for the award of any degree.

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Department of Metallurgical and Materials Engineering

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CERTIFICATE

This is to *certify* that the Research Thesis **Preparation of preformed yarn to synthesize carbon-carbon composites** submitted by **Padmayya S. Naik.** (Register Number: **MT07P02**) as the record of the research work carried out by him, is *accepted as the Research Thesis submission* in partial fulfillment of the requirements for the award of degree of **Doctor of Philosophy.**

Research Guide
(Dr. A. O. Surendranathan)
(Signature with Date and Seal)

Chairman – DRPC
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ABSTRACT

The carbon fibre-reinforced carbon composites, popularly known as the carbon-carbon (C/C) composites, are of interest because of their ability to retain strength and structural integrity till 3000^{0}C either in vacuum or in inert environment. C/C composites also possess outstanding specific strength and stiffness. Hence, the C/C composites are suitable for application in thermal protection systems used in nose cones and leading edges of hypersonic and re-entry type vehicles, which are exposed to elevated temperatures. Due to impressive high temperature properties, the applications of C/C composites in the aerospace industry have increased significantly in recent decades.

Significant research has been carried out on the conventional C/C composites manufacturing methods and their properties. The conventional C/C composites synthesis requires many steps and consumes more time which leads to comparatively more cost. This research is mainly focused on a new method of C/C composite-synthesis by giving importance on preform preparation which includes reinforcement and matrix materials.

In order to simplify the manufacturing of C/C composites, it was decided to develop a new production method, using preformed yarn, which contains the carbon fibre filaments as well as the matrix materials (coke and pitch binder).This method is named as preformed yarn (PY) method. This PY enables us easily to fabricate primary workpieces such as unidirectional (UD) sheets, cloth sheets, tapes and chopped yarns. Further, it is possible to prepare pipe and tube shaped standby preforms. From these workpieces and preforms, the C/C composites can easily be produced by hot press molding method.

This investigation presents the construction of preformed yarn machine, and to produce preformed yarn bundles. In the beginning, it was decided to prepare proto-type PY model and three carbon fibre weight percentages of PY were prepared. Using this PY, unidirectional C/C composites were synthesized and various properties were analyzed. The C/C composites were prepared by varying the weight percentage of carbon fibre. The PY was prepared with carbon fibre (reinforcement) filaments surrounded by coke and pitch (matrix materials), which were enclosed in nylon-6 as sleeving material for easy handling and processing. Three types of PY samples were prepared with carbon fibre fractions of 30wt%, 40wt% and 50wt% respectively and accordingly the composites were synthesized. In each case, the PY was chopped and filled unidirectional into a die of required shape and hot pressed at 600°C. Subsequently, impregnation of the carbon fibre preforms by pitch was carried at 250^{0}C under a pressure of 0.1 MPa for 2-3 hrs. Then the pitch impregnated carbon fibre preforms was heated to 800°C and hot isostatic-pressed at 90–100 MPa pressure for 24 hrs. This was followed by carbonization at 900^{0}C for 48 hrs and graphitization at 2500^{0}C for 24 hrs, each under 1 atm. pressure. The process cycle from pitch impregnation to graphitization was repeated 2 to 3 times, until a density of 1.7 g/cc could be obtained in the investigated C/C composites. The characteristics such as microstructure (SEM images), XRD analysis, hardness, compressive strength, impact energy, creep, wear property, thermal degradation, have been studied. It is observed that, as the carbon fibre weight percentage increased the properties have also improved. The C/C composite made by the proposed PY technique is superior to any other conventional method. Among the three C/C composites containing different weight percentages of the fibre content, the one with 50wt% carbon fibre showed better properties. After developing a prototype model, it was further decided to design automatic (using PLC) machine for producing 50wt% carbon fibre-PY. The design is presented with relevant drawings. The sleeve weight percentage was reduced considerably in automatic PY technique as sleeve is not much important in composites except for holding and handling purposes. Due to this the weight percentage of matrix is proportionately increased against the reduction of weight percentage of the sleeve and weight percentage of carbon fibres would remain same.

Key words: C/C composites; Preformed Yarn; Carbon fibre; Proto-type; Sleeve.

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NOMENCLATURE

b	Width of the beam	mm
d	Depth of the beam	mm
D	Track diameter	m
e	Elongation at failure	mm
E	Flexural modulus	MPa
F_N	Normal load	Newtons (N)
J	Joules	Nm
L	Sliding distance	m
L’	Support span of beam	mm
m	Slope of load-deflection curve	kN/mm
N	Speed of the disc	Revolutions per minute (rpm)
P	Maximum load	kN
p	Normal pressure	MPa
S	Flexural strength	MPa
T	Time	minutes (min)
ρ	Measured density	g/mm^3
W_s	Specific wear rate	mm^3/Nm
W_1	Initial weight	g
W_2	Final weight	g
δ	Deflection	mm
μ	Coefficient of friction	unit less
v	Sliding velocity	meters/second (m/s)
Δm	Loss of weight	grams (g)

ABBREVIATIONS

C/C	Carbon-Carbon
CFRP	Carbon Fibre Reinforced Polymers
CVD	Chemical Vapor Deposition
CVI	Chemical Vapor Infiltration
DTG	Differential Thermo Gravimetric
HIP	High Impregnation Pressure
LVI	Liquid Vapor Infiltration
LS	Longitudinal Strength
LVDT	Linear Variable Differential Transformer
MS	Mild Steel
PAN	Poly-Acrylo-Nitrile
PID	Proposnal Integral Derivative
PP	Poly-Propylene
PLC	Programmable Logic Controller
PY	Preformed Yarn
SEM	Scanning Electron Microscope
SS	Structural Steel
TGA	Thermo Gravimetric Analysis
TS	Transverse Strength
UD	Uni-Directional
XRD	X-Ray Diffraction

CHAPTER-1

INTRODUCTION

1.1 **The Carbon**

Carbon an ‘old and new material’ is known to mankind from times immemorial in a variety of forms. Carbon has an atomic weight of 12.011 and is the sixth element in the periodic table. Carbon is a truly remarkable element existing in four allotropes in general, viz. diamond, graphite, carbynes and fullerenes, each having significant scientific and technological importance. Its most abundant allotrope, graphite, can take many forms with respect to microstructure, amorphous to highly crystalline structure, highly dense with density 2.2 g/cc to highly porous with density 0.5 g/cc and different shapes. These types of graphite are called synthetic carbons and in technical terms, engineered carbons. Examples are coke, graphite electrodes, mechanical carbons, glassy carbons, carbon black, porous carbons, activated carbons, carbon fibres and composites etc. Major improvements in the properties of carbon used in ‘old’ applications such as electrodes for arc furnaces resulted in ‘new’ carbons such as synthetic diamonds and carbon-based composites. Carbon materials are used in many diverse engineering applications, where properties such as lightweight, strength, high thermal conductivity, and high temperature stability are important. Crystallographic carbon namely graphite, consists of tightly bonded, hexagonally arranged carbon layers that are held together by week Vander Waal’s forces. The atoms in the basal plane have covalent bond strength of 525kJ/mol while the bond energy between basal planes (c-direction) is 10kJ/mol is shown in Fig. 1.1. The result is a crystal that is markedly anisotropic in which it is almost isotropic within the basal plane, but with the c-direction, properties differ by more than an order of magnitude. The single crystal graphite structure is shown in Fig. 1.2 along with some other structural form of carbon i.e. diamond [Edie D. D. 1998].

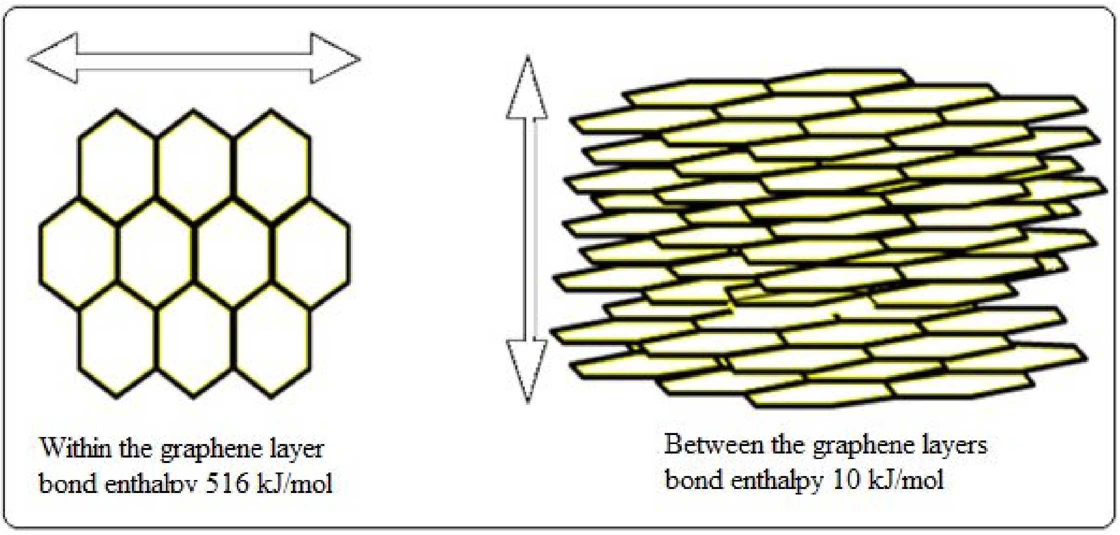


Fig. 1.1 Bond strengths in graphite [Edie D. D. 1998]

Figure 1.3 is a schematic diagram showing the atomic arrangements in poorly crystalline forms. The remarkable variations in properties that accompany variations in structure have allowed carbon to be used with outstanding success in numerous, seemingly contradictory applications. For example, in various states of aggregation, the graphitic form can be used as a lubricant or as a high strength component in high temperature applications. The beauty of diamond makes it highly regarded as a precious stone; its hardness makes it technologically important as a grinding or cutting material. In the near amorphous state, poorly conducting lampblacks are used as thermal insulator or fillers for rubber.

Aerospace applications utilize the highly directional thermal conductivity of quasi-crystalline pyrolitic carbon to distribute heat and to insulate rocket nozzles or nose cones for ballistic reentry vehicles. The low cross section for neutron capture, the ability to efficiently moderate neutrons, and the refractory nature makes carbon a natural choice as a reactor material in various forms. Biocompatible nature of the carbon with both tissue and blood, coupled with superior wear properties, non-corroding characteristics, heparin sorptivity, and resistance to thrombus formation make it the most sought after of all the current biomaterials. Interesting applications include prosthetic knee joints, Steinmann pins, dental implants, and heart valves.

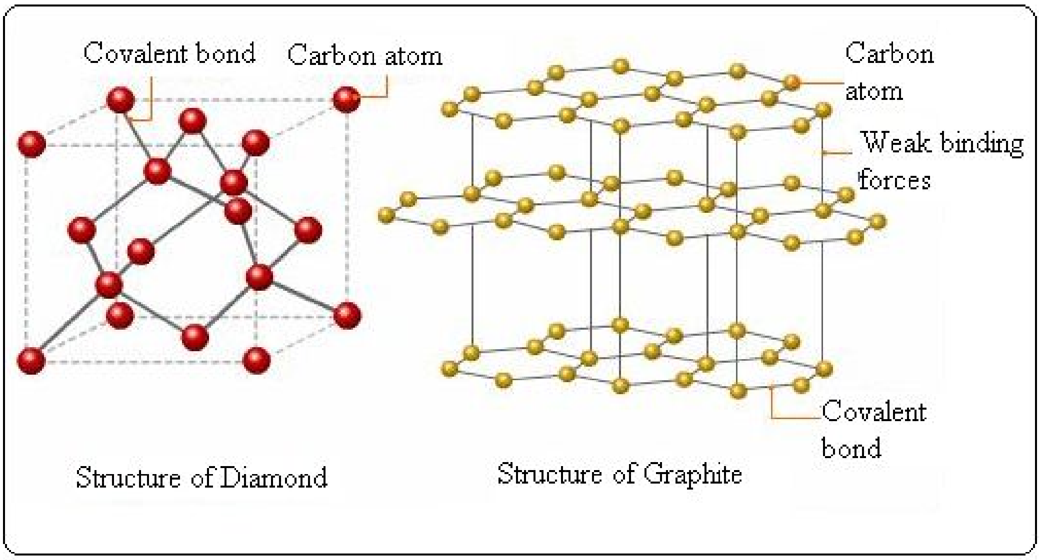


Fig. 1.2 Schematic diagrams of diamond and graphite structure [Carl Zweben, 1998]

Carbon is well known absorber of radar waves (throughout the entire microwave radar spectrum) and its stealth like properties did not escape the attention of radar experts and engineers in the aerospace community. The wings of the Northrop's B-2 bomber is believed to be made of carbon-carbon composites, to enable it to withstand the extremely high temperatures developed when the exhaust gas vents directly over the wing so as to preclude detection from the infra-red, heat seeking guided missiles. Comprehensive reviews comprising of the properties, applications and recent developments of carbon-carbon may be found in literatures.

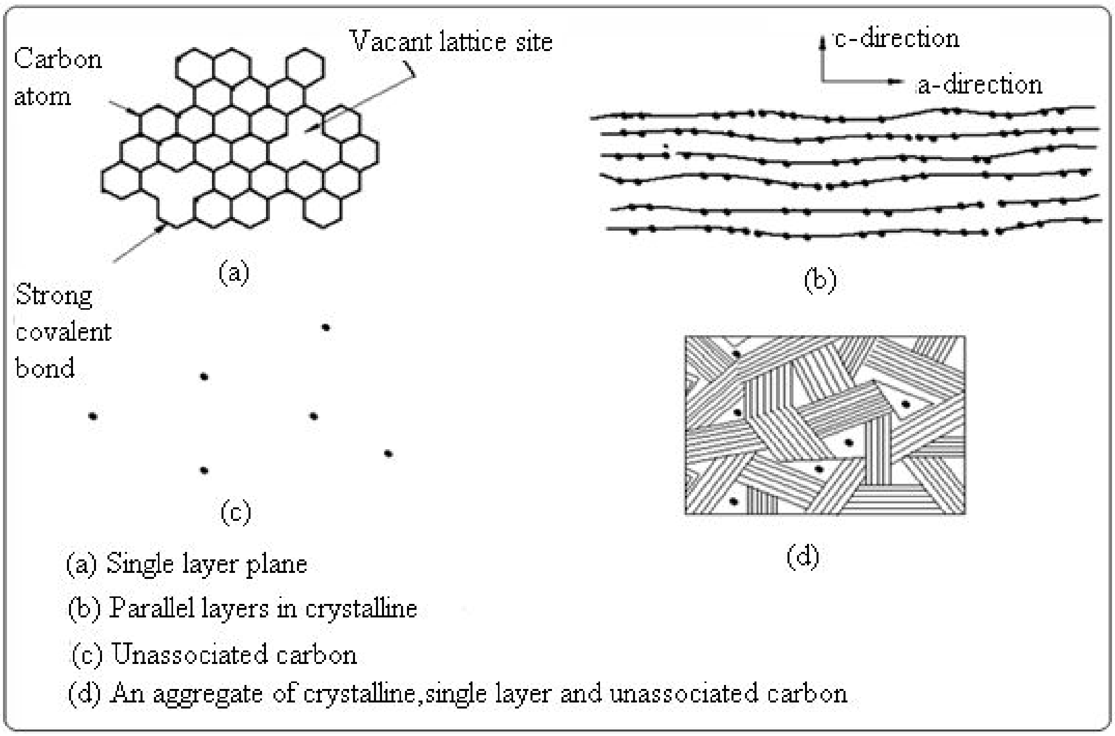


Fig. 1.3 Schematic diagram showing the atomic arrangements in poorly crystalline forms [Deborah D. et al, 1994]

1.2 Carbon Fibres

Carbon fibres are the best known and most widely used reinforcing fibres in advanced composites. Although there are many reasons for this situation, two factors predominate. Firstly, the manufacturing technology for carbon fibres, although complex, is more amenable to large-scale production than are those of many of the other advanced fibres. Secondly, carbon fibres have very useful engineering properties that, for the most part, can be readily translated into usable composite physical and mechanical properties [Carl Zweben, 1998].

The production of carbon based composites begins with the production of the carbon fibres. Carbon fibres are long bundles of linked graphite plates, forming a layered crystal structure parallel to the fibre axis. This crystal structure makes the fibres highly anisotropic, with an elastic modulus of up to 500GPa on-axis versus only 35GPa off-axis. Fibres can be made from several different precursor materials, and the method of production is essentially the same for each precursor; a polymer fibre undergoes pyrolysis under well controlled heat, timing and atmospheric conditions, and at some point in the process it is subjected to tension. The resulting fibre can have a wide range of properties, based on the orientation, spacing, and size of the graphite chains produced by varying these process conditions.

Precursor material is drawn or spun into a thin filament. The filament is then heated slowly in air to stabilize it and prevent it from melting at the high temperatures used in the following steps. The stabilized fibre is placed in an inert atmosphere and heated to approximately 1500°C to drive out the non-carbon constituents of the precursor material. This pyrolysis process, known as carbonization, changes the fibre from a bundle of polymer chains into a bundle of "ribbons" of linked hexagonal graphite plates, oriented somewhat randomly through the fibre. The length of the ribbons can be increased and their axial orientation improved through further heating steps up to 3000°C, a process called graphitization. Because the graphite ribbons are bonded to each other perpendicular to the fibres only by weak Vander Waals bonds, the ribbons must be reoriented to increase the tensile strength of the fibre to a useful level. This is accomplished through the application of tension at some point in the stabilization or pyrolysis phases, the exact time depending on the precursor material. Increased axial orientation increases the fibre's tensile strength by making better use of the strong covalent bonds along the ribbons of graphite plates.

There are many principal precursor materials for carbon fibres, of which polyacrylonitrile (PAN), pitch and rayon are the most common. PAN is stretched during the stabilization phase, and heated to 250°C in air. The tension is then removed, and the fibre is heated slowly in an inert nitrogen atmosphere to 1000-1500°C. Slow heating maintains the molecular ordering applied by tension during the stabilization phase. Graphitization at temperatures up to 3000°C then follows **(**processes shown in Fig. 1.4).

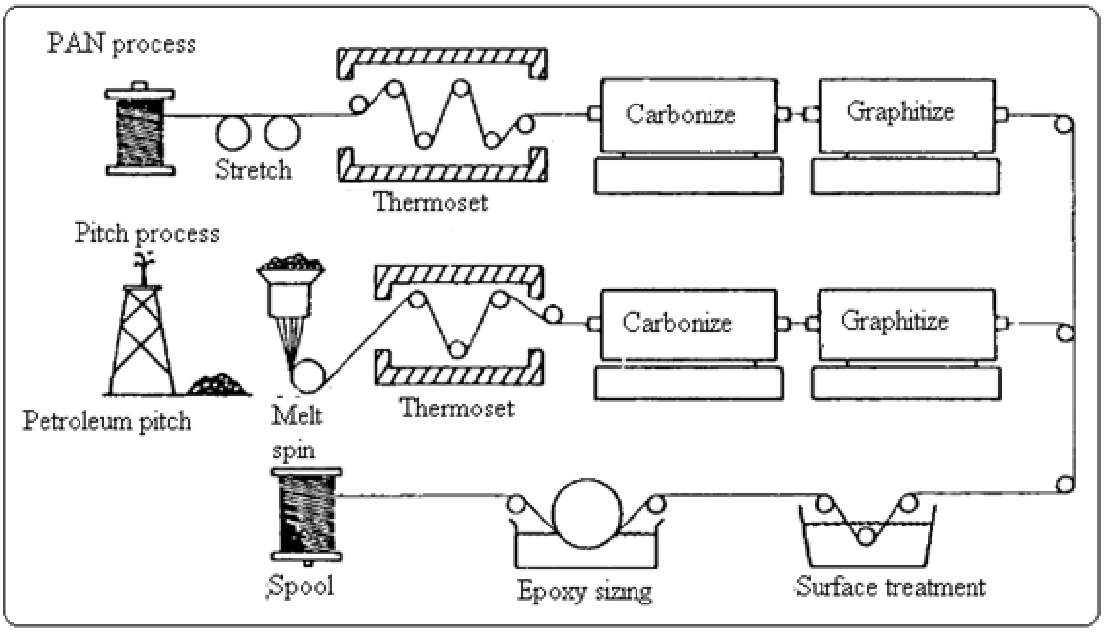


Fig. 1.4 Schematic diagram of manufacturing of PAN and Pitch based carbon fibres [John D. Buckley et al, 1993]

Applying tension at 2000°C further increases the proper ordering of graphite ribbons. Rayon, cellulose based fibre made from wood pulp, is spun into a filament from a melt, and stabilized without tension at 400°C. It is then carbonized without tension at 1500°C, and is stretched in the graphitization phase at 2500°C. The pitch material is spun into a filament from either a simple melt or from a liquid crystal "mesophase" melt (obtained by heating the melt above 350°C for an extended period). If the precursor filament is obtained from the simple melt, it must have tension applied during the graphitization phase. When a mesophase melt is used, the spinning process imparts a high degree of order to the resulting graphite, so tension need not be applied during the pyrolysis phases. Filaments from pitch are otherwise processed into fibres by the same process as other precursors. Polyvinyl alcohol, polyamides and phenolic may also be used as precursors [Deborah D. L. Chung, 1994].

1.3 Carbon-Carbon (C/C) Composites

A composite material is defined as “a multi-phase material from a combination of materials, differing in composition or form, which remain bonded together, but retain their identities and properties. The components do not dissolve or completely merge. They maintain an interface between each other and act in concert to provide improved specific or synergistic properties not obtainable by any of the original components acting singly”.

Carbon-carbon materials are generic class of composites, consisting of a fibrous carbon in a carbonaceous matrix. Although both constituents are of the same element, this fact does not simplify the composite behavior because the state of each constituent may range from carbon to graphite. In the words of Fitzer and Huttner, the principal concept of C/C composites can be seen as an attempt to develop and tailor special graphite materials that utilize the chemical bonds to enhance their mechanical and thermal bulk properties. The synergetic nature of a composite material can be best explained the work of fracture of the individual carbon fibre and that of the matrix carbon which is about 10 J/m^2, whereas that of a C/C is about14 kJ/m^2 a phenomenal increase in magnitude. The development of C/C materials began in 1958 and was nurtured under the U S Air Force’s space plane program and NASA’s Apollo projects. It was not until the Space Shuttle Program that C/C material systems were intensively researched. In C/C composites, the range of properties resulting from the anisotropy of the C-crystal can extend to both the constituents. The matrix precursor determines the type of carbon obtained and the degree to which it graphitizes, and hence forms an important variable. The reinforcement can either be cellulose-based, pitch-based or polyacrylonitrile (PAN)-based carbon fibres. The fibres in the composite can be in either the continuous or the discontinuous form. The fibre orientation can be varied ranging from unidirectional lay-ups to multi-directional weaves to produce delamination-free near-isotropic composites if required. The volume or weight fraction of the fibre constitutes another variable. A range of carbon structures can further be obtained by choosing the process route (vapor phase deposition or liquid phase impregnation and carbonization). Finally, the heat treatment of the composite at graphitization temperatures offers additional variability to the properties that can be obtained. Coupled with a variety of processing techniques that can be used in fabrication, these composites can be tailored to obtain the requisite physical, thermal and mechanical properties of an end use [B. Venkataraman et al, 2002].

The criteria that led to the selection of C/C composites for many applications and thermal protection were based on the following requirements:

i. Retention of mechanical properties such as strength and stiffness up to 3000°C,
ii. Sufficient stiffness to resist thermo-structural loads,
iii. High thermal conductivity and low coefficient of thermal expansion to minimize induced thermal stresses and thereby improving thermal shock resistance,
iv. Oxidation resistance sufficient to limit strength reduction,
v. Tolerance to impact damage and
vi. Manufacturing process within the state of the art and the ability to manufacture complex shapes.

Carbon materials have long been recognized as a potential candidate for tribological applications. For example, graphite having hexagonally arranged carbon layers separated by weak Vander Waals bond is known to be good solid lubricants. Their applications in electrical bushes and bearings are well known. The interest in the tribological response of this class of material escalated with the advent of carbon fiber reinforced carbon matrix composites (known as carbon-carbon composites) for aircraft brake applications. The primary requirements for aircraft brake materials are high thermal capacity, good strength, adequate and consistent friction characteristics in conjunction with high wear resistance and high thermal conductivity [H. W. Chang. 1982]. Lighter and refractory, with equivalent mechanical properties, thermo structural composites replace more and more metallic parts of aeronautic structures. In 1969, C/C composite were introduced in propulsion field and rapidly applied to aircraft brakes thanks to their excellent tribological properties, especially at high temperatures. With a low wear rate and a stable behavior, they are perfectly suited to all the aircraft brake applications: not only taxying and landing but also rejected take-off. In the early 1980s, their use was extended to Formula 1 brake fabrication. However, such materials have never been applied to other types of surface transport, because of too high a cost and unsuitable tribological behavior in the concerned temperature ranges [S. Fouquet M. et al. 2008].

In spite of the excellent thermo-mechanical properties, the carbon-carbon finds limited specialized applications due to the following reasons. Carbon is highly susceptible to oxidation at high temperatures and the carbon-carbon composite is no exception to it. The only major weakness of C/C composites is their extreme oxygen sensitivity above approximately 500 ºC [I. J. Davies et al. 1999]. This requires either the operative environment to be made inert, or the time of exposure to be small enough to limit oxidation. And finally, the huge infrastructural setup required and the long process cycles to attain good density leads to high manufacturing costs. However, with the adoption of efficient processes like hot isostatic pressing and selection of precursor materials with higher coking value had led to cost-effective products. Despite the limiting factors, the technical importance of the carbon-carbons is growing as evident from the increased consideration of these for long life, repeated-loading situations such as heat engines, gas ducts, hot-pressing moulds, aircraft brakes and biomedical implants. Whether one regards C/C as an improvement over a plastic composite, a pyrolyzed CFRP (in terms of heat resistance and chemical stability), or as a stronger, less brittle form of bulk graphite, albeit with some shortcomings, C/C materials are frequently the best available option for certain high performance thermo-structural applications. Especially, for short life single exposures experienced by space-vehicle reentry capsules or for rocket nozzle throats, the C/C has been the state- of-the-art material.

The classical or conventional method for fabricating carbon-carbon materials involves combining solid particles of pure carbon (filler coke) with a precursor that can be carbonized to serve as a binder. In C/C composite, carbon fibres are used as the primary carbon instead of the filler coke. Unfortunately, the mass loss and shrinkage of the matrix during carbonization result in a final material that exhibits considerable bulk porosity. The objective of repeated infiltration-pyrolysis is to densify the initial porous skeleton. Densification is achieved by impregnation with liquid or gaseous carbon precursor compounds and subsequent pyrolysis. A key factor in the selection of a matrix carbon precursor involves the ability to fully densify the preform and to achieve a high char yield. Three basic methods of fabricating carbon fibre reinforced carbon materials exist. The first two methods are based on thermally degrading a thermosetting resin or a thermoplastic pitch. The third method involves depositing carbon into a fibrous preform using CVI (chemical vapor infiltration).

As discussed in a following section, the choice of fabrication method depends to a major extent on the geometry of the part being processed. Thin sections are prime candidates for CVI processing; however, since this method tends to preferentially deposit in and on the surface layers, it is not suitable for the fabrication of thick sections. Thick sections therefore tend to be produced using resin or pitch infiltration. Thermosetting resins remain solid during carbonization; however, pitches soften and tend to flow from a preform at high temperatures; therefore, they require containment during the carbonization step. Complex shapes are difficult to fabricate using either CVI or pitch matrix materials owing to the difficulty of maintaining the dry fibre preform shape during the initial infiltration. Hybrid densifications are sometimes practiced when a rigid structure is first made using the resin prepreg-autoclave molding process. In this process after carbonization, subsequent infiltrations are made with CVI or pitch [Fitzer E. 1987]. Tokyo University in Japan and Across Co. Ltd has developed a unique manufacturing process of this C/C composite utilizing preformed yarn (PY); a preformed yarn already contains the components of the matrix within the carbon fibre bundles and this PY composite is superior to any other conventional method. Conventional C/C composite used to be expensive and time consuming to manufacture due to its complicated processing method. Both time and cost were reduced using patented PY-C/C composite manufacturing method [K. Goto H.et al. 2003 and Fitzer E. 1987].

1.4 Scope Of The Present Work

As stated above there exists a variety of precursor materials both for fibre as well as for matrix in a C/C composite. Further, the different types of processes result in a variety of carbon structures thereby affecting the properties. Hence there arises a need to understand the process-structure-property relationship in the carbons formed from different precursors as well as different process routes. Different precursor materials have been used to start with and the effect of these on the densification efficiency and microstructures formed are investigated by many researchers. C/c composites used to be expensive and time consuming to manufacture due to its complicated processing method such as CVD (chemical vapor deposition) and CVI. Institute of Production Technology of Tokyo University in Japan and Across Co. Ltd has simplified the manufacturing process by using a new method called preformed carbon fibre and matrix yarns, resulting in great reduction in time and cost. Preformed yarn preparation machine or model and even method were not yet reported in the literature. Hence, the thrust area in this field is preparation of preformed yarn, development of preformed yarn model and systematic study of various physical and mechanical properties of PY C/C composites are of more important.

1.5 Objectives Of The Research Work

The main objectives of the present investigation were to design and develop PY machine. Using prepared PY, it was planned to fabricate and characterize carbon-carbon composites. The specific objectives of the work were as follows:

i. To construct proto-type PY machine by appropriate design and development using suitable software.

ii. To prepare preformed yarn using proto type PY machine by varying the fibre content from 30 wt %, to 50 wt % in steps of 10wt%.

iii. To study the effect of fibre content on the mechanical, tribological and thermal properties of C/C composites.

iv. To compare the reported conventional C/C composites' properties with those of PY composites.

v. To construct automatic PY machine to produce at least one composition of higher properties reported among above three PY-C/C composites.

CHAPTER-2

LITERATURE SURVEY

2.1 General Background Of Carbon-Carbon Composites

Carbon-carbon (C/C) composites or more precisely, carbon fibre reinforced carbon composite consists of synthetic elemental carbon with a weight or volume fraction up to 75% of carbon fibres in a carbon or graphite matrix. Carbon-carbon composites are particularly a material made from carbon fibre reinforced carbon matrix. Carbon-carbon composites are new-type high-temperature materials and are widely used in aerospace and automobile industry for their excellent properties, such as light weight, high modulus, high specific strength, low thermal expansion, high temperature resistance and outstanding corrosion resistance [Li Rui-zhen et al. 2005, Ken Goto et al. 2005, Ken Goto et al. 2003 and Donald L.S. et al. 1999]. With the rapid development of scientific technology, the electronic equipments of thermal control systems in aerospace craft's tend to miniaturize, tend to be of lightweight, and tend to be compact and efficient. For this equipment to work safely, the accumulated heat must be conducted away, which leads to specific thermal requirements for the materials [Vitaly V.et al.2011 and Zhang Guang-jin et al.2001]. The C/C composite is a carbon fibre reinforced carbon matrix, so carbon matrix is the important part of the C/C composites. Carbon-carbon composites with different carbon matrices have different microstructures. Moreover, the thermo physical properties are comparatively very structure sensitive, and the heat transfer mechanism is governed by the atomic level structure of the composites. [Chen Jie et al. 2009 and J. Economy et al. 1992].

Carbon, as a solid, is unique solid substance that can be made to exhibit the broad variety of different structures and properties in its allotropic forms. Some carbons are extremely strong, hard and stiff while some other forms are soft and ductile; some are highly porous and exhibit large surface area, while others are impervious to liquids and gases [J.R. Gomesa et al. 2001 and Fitzer E.et al. 1987]. These variations result from structural effects and crystalline order. Carbon-fibre reinforced carbon can exhibit high fracture toughness and pseudo-plasticity. Carbon-carbon composites have important properties such as high specific strength and stiffness up to the highest temperature and excellent dimensional stability due to their very low thermal expansion coefficient. They increasingly qualify as materials for advanced and highly specified applications resulting in significant weight reduction with the structural members when compared to monolithic members [David E.et al. 1998 and Hatta H. et al. 2004].

The fabrication of such materials can be manipulated to alter the type and the distribution of the carbon fibres which themselves can be modified according to structural need and also to alter the carbon or graphite matrices that binds these fibres. Such manipulations would enable the designer's specific requirements to be met. When selecting a resin, metal, ceramic, or carbon-matrix composite for a given application, three criteria are generally used: the composite must have the desired physical and mechanical properties; it must be capable of being processed or manufactured into the desired shape; and it must be economical to produce [Urs I.et al.2004 and J. E. Sheehan et al. 1994]. For most composites, the primary consideration for a given application concerns the properties of the reinforcing fibres. The mechanical properties (strength and modulus) of carbon fibres are also related to the physical properties (thermal and electrical conductivity and coefficient of thermal expansion). Because of the interrelationships that exist between the microstructure and the elastic and physical properties the choice of a fibre based on one property usually determines the value of the other properties. For instance, the microstructure of very high modulus fibres usually consists of long fibrils that are almost perfectly aligned parallel to the fibre axis. As a result, the transverse modulus will be relatively low, the thermal and electrical conductivity will be high in the longitudinal direction, and the thermal expansion coefficient will be small or negative [B. K. Pudhi et al. 1992]. A composite matrix usually serves to protect the reinforcement fibres from damage or reaction with the environment, to provide some measure of support in compression, to provide adequate matrix-dominated properties, and to provide a continuity of material. This last property is important in electrical and thermal applications and is particularly important in mechanical applications since load must be transferred to the fibres through the matrix. In this respect, a load can be transferred to the fibres across a chemically or physically bonded interface or across a mechanically interlocked one formed by the matrix shrinking onto and thereby gripping the fibre surface. The properties of the matrix dominate the properties of composites in any direction in which fibres are not aligned. Such properties include the transverse tensile strength and modulus, interlaminar shear strength, thermal expansion coefficient, and electrical conductivity. Composite materials reinforced with continuous fibres are complex materials that, for a given weight, exhibit specific mechanical properties almost always superior to those exhibited by conventional metals and alloys. Three basic composite types can be conveniently discussed in terms of maximum temperature of use: reinforced polymers, which are restricted to use at relatively low temperatures; reinforced metals, which can be used at intermediate temperatures; and reinforced carbons and ceramics, which can be used up to very high temperatures. In order for the mechanical properties of a continuous fibre-reinforced composite to be superior to an unreinforced material, the modulus and strength of the fibre reinforcement must be greater than the matrix. In addition, there must be chemical, physical, or mechanical bonds formed between the fibres and matrix that are strong enough to transfer load between individual fibres and between fibre layers [John D. Buckley et al. 1993].

Ogasawa and Ishikawa studied thermal response and oxidation behavior of commercial metal silicon-infiltrated carbon-carbon composites in a high-enthalpy convective environment using an arc jet facility (an arc wind tunnel). Composite specimens were put into a supersonic plasma air stream having a gas enthalpy of 12–18 MJ/kg for 50–600s. Excellent oxidation resistance was obtained by the formation of a porous SiC layer at the surface of the C/C composite. Goto et al. and others examined [Goto Ken et al. 2005 and Goto Ken et al. 2003] the tensile fatigue behavior of a cross-ply C/C laminate composites at room temperature. Tension–tension cyclic fatigue tests were conducted under load control at a sinusoidal frequency of 10 Hz to obtain stress–fracture cycles (S-N) relationship which shows good fatigue resistance. The residual tensile strength of specimens that survived fatigue loading was enhanced with increase in fatigue cycles and applied stress. Formation of micro-cracks at the fibre–matrix interfaces was observed during fatigue loading. Fatigue behaviors of unidirectional reinforced, symmetric cross-ply laminated and symmetric quasi-isotropically laminated C/C composites were examined and obtained very high fatigue resistance [Toshio Ogasawara et al. 2001]. An experiment on measurement of tensile strength of carbon-carbon composites which was made by hot press mould method was reported as slight improvement over usual conventional C/C composites synthesis method. Tensile fracture behavior of carbon-carbon composites with $0^0/90^0$ laminations were examined using double end-notched specimens. In their experiment, the specified weight fractions of the 0^0 and 90^0 layers were systematically varied to observe the variation of fracture patterns as a function of the shear strength and observed good fracture resistance [Nago H. et al.1998].

Advanced composites, particularly carbon fibre-reinforced composites, are being increasingly used in modern aircraft because of their superior strength, weight and stiffness properties and because they offer 25 to 30% savings in structural weight which is of strategic importance in a military aircraft [Magali Rollin et al. 2008]. They enable the designer to tailor- make the strength and stiffness in the desired direction as well as cut down drastically the number of parts to be assembled. As basic materials of composites are intrinsically different, so also their manufacturing processes, structural design procedures, in-service performance, methods of testing, inspection and evaluation. Further, composites are susceptible to moisture, temperature, inflammability, lightning, erosion, fatigue and galvanic corrosion [Tanabe Y. et al. 2009]. Also, the properties of the advanced composite aircraft structures are generated during the process of its manufacture. Carbon fibres are one of the important reinforcing materials used in composites. In continuous fibre composites, the tensile strength and modulus of the composites can be realized nearer to the predicted values using rule of mixtures [R. Janardhan et al. 2000]. Besides, the high production cost of continuous fibre- reinforced carbon matrix composites limits their application to a few critical areas where cost is secondary. The emerging new class of discontinuous fibre-reinforced composites concept has opened up the application for short fibre composites in other engineering areas also [Mel M. Schwartz, 1992]. This is because discontinuous fibre-reinforced composites are less expensive and easy to manufacture. Design of advanced composite structures requires much more enhanced analytical capabilities while its manufacture calls for new handling and processing techniques like preformed yarn method. Preformed yarn method would reduce the cost further, due to lower production time and less pitch impregnation cycles compared to any other conventional methods.

2.2 Conventional Carbon-Carbon Composites Production Methods

Conventional production methods of manufacturing C/C composites include chemical vapor deposition (CVD) in which a thermosetting resin binder is carbonized; further this will be graphitized. In the CVD method, a preform of carbon fibres having a specified shape is heated in a furnace to a high temperature, while a hydro-carbon gas is fed to the furnace. The gas is thermally cracked to form carbon, which then deposits uniformly on the fibre surfaces [W. Kowbel et al. 1995]. In the other process, fibre yarns or woven or nonwoven fabrics of carbon fibres are shaped into various structural shapes, with the use of a thermosetting binder such as phenolic or epoxy resin. The structure is subsequently heated in an inert gas atmosphere to carbonize the resin and further heat treatment will be carried to obtain crystalline structure [N. P. Radimov et al. 1994].These conventional techniques have reportedly encountered problems because the resulting C/C composites lack uniformity in properties such as bending strength and density. The processes are also complicated and time consuming.

The two popular conventional processing methods are CVD and Liquid Phase Impregnation [Jinyong Lee et al. 2005). Gas phase impregnation or CVD technique uses volatile hydrocarbons such as methane, propane, benzene and other low molecular weight units as precursors. Thermal decomposition is achieved on the heated surface of the carbon fibre substrates resulting in a pyrolitic carbon deposit. This technique can be employed to deposit carbon on to dry fibre preforms or to densify porous carbon-carbon structures produced by the liquid impregnation route, in which case it is referred to as chemical vapor infiltration. This process route was widely used by the western countries for the production of thinner parts like aircraft brake discs and nozzles. Carbon-carbon process technology using CVD technique is old but yet to be established in our country. The Liquid Phase Impregnation Process involves impregnation with liquid impregnates like coal tar or petroleum pitches and high char-yielding thermosetting resins. The criterion for selection of impregnates is based on the characteristics like viscosity, carbon yield, matrix microstructure and matrix crystalline structure which are considerably influenced by the time-temperature pressure relationships during the process. Carbon-carbon manufacturing process involves thermosetting resins such as phenolic, furans and advanced resins like coal tar pitches, petroleum pitches and their blends. Fig. 2.1 shows the carbon-carbon manufacturing process using the multiple impregnations, carbonization at 1000°C and high pressure of 100 MPa which will be further heat-treated to carbonization and graphitization at 2750^{0}C. In atmospheric pressure carbonization, the carbon yields obtained from pitch are only around 50 per cent i.e. approximating those from high yield thermosetting resins yields as high as 90 per cent can be obtained by carbonizing the pitch under high pressure of 1000 bars, thus making the process more efficient [Christ K. et al. 1993]. Pressure applied during pyrolysis also affects the matrix microstructure. The higher the pressure the more isotropic will be the microstructure due to the suppression of gas formation and escape. High impregnation pressure (HIP) also helps in lowering the temperature of mesophase formation in pitch, resulting in highly oriented crystalline structure. The HIP process is the only practical route to lower the production cost of carbon-carbon composites [G. Rohini Devi. et al. 1993]. The second phase in carbon-carbon composite production is the building up of the carbon matrix around the graphite fibres. There are two common ways to create the matrix; through chemical vapor deposition and through liquid impregnation of a resin, the process flow chart is shown in Fig. 2.2.

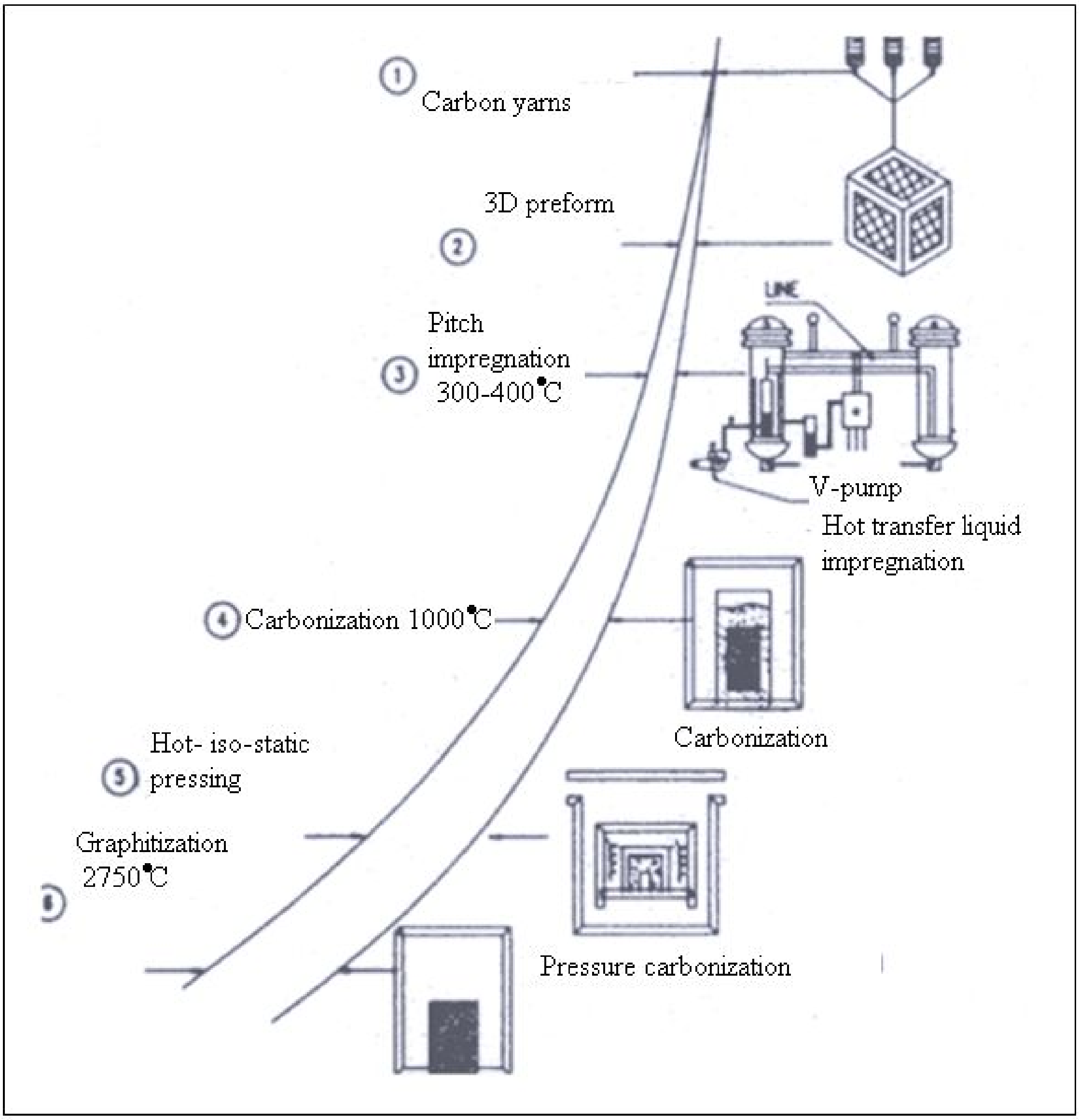


Fig. 2.1 Schematic representation of carbon-carbon production route [G. Rohini Devi et al. 1993]

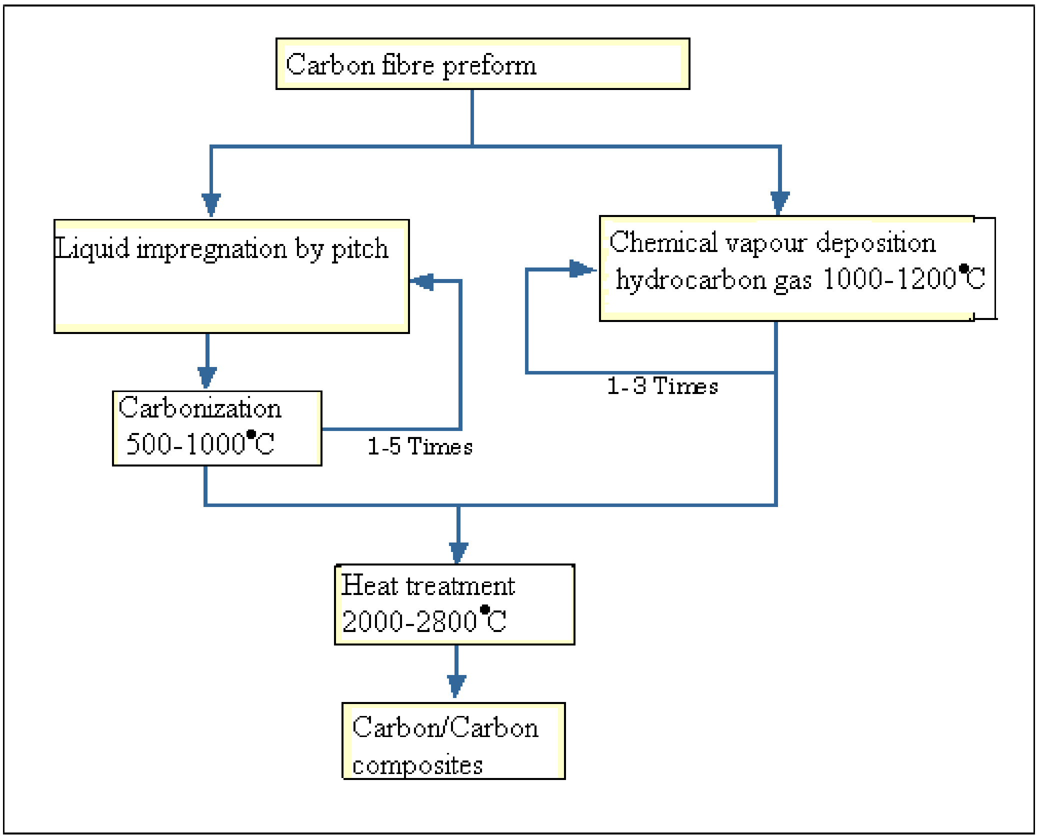


Fig. 2.2 Process flow chart for production of carbon-carbon composites [Christ K. et al. 1993]

2.3 Microstructure Of Carbon-Carbon Composites

Microstructure plays an important role in the performance of carbon-carbon composites. The study of microstructure gives vital information about distribution of fibres and matrix, pore distribution, isotropy and anisotropy of matrix formation and filling of cracks and extent of damage during either processing or testing. Microstructure of carbon-carbon composites has been studied using optical and scanning electron microscopes (SEM). Composite samples made with different types of fibres and matrices are studied at various stages of processing and also after subjecting to friction testing. Figures 2.3 (a) and (b) show the optical micrograph of carbon-phenolic sample in transverse (TS) and Longitudinal (LS) directions respectively. Bulk phenolic resin matrix is seen between the layers and between carbon fibre tows, which can be seen in Fig. 2.3 (a) bulk matrix has micro pores which are produced during the curing of phenolic resin because of volatile evolution. Fig. 2.3 (b) shows the accumulation of resin near the tows crossover points of fabric weave.

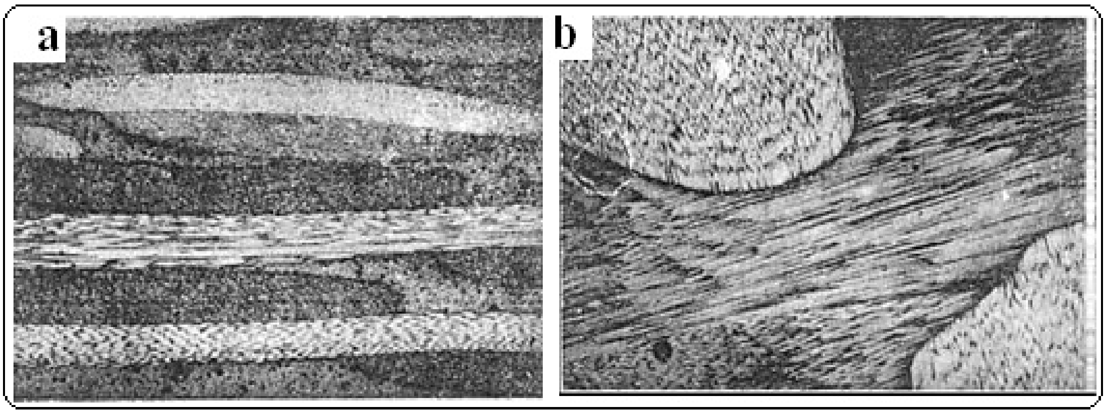


Fig. 2.3 Optical micrograph of carbon-phenolic composites [(a) TS, b) LS].

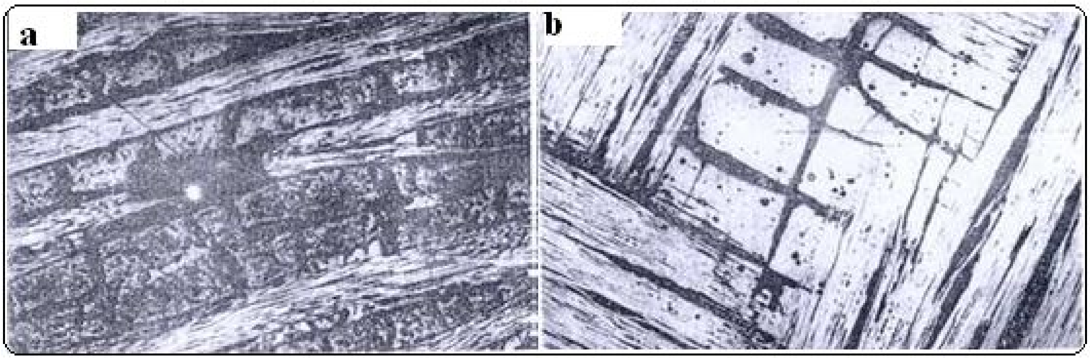


Fig. 2.4 Optical micrograph of carbon-carbon composites [(a) TS and (b) LS]

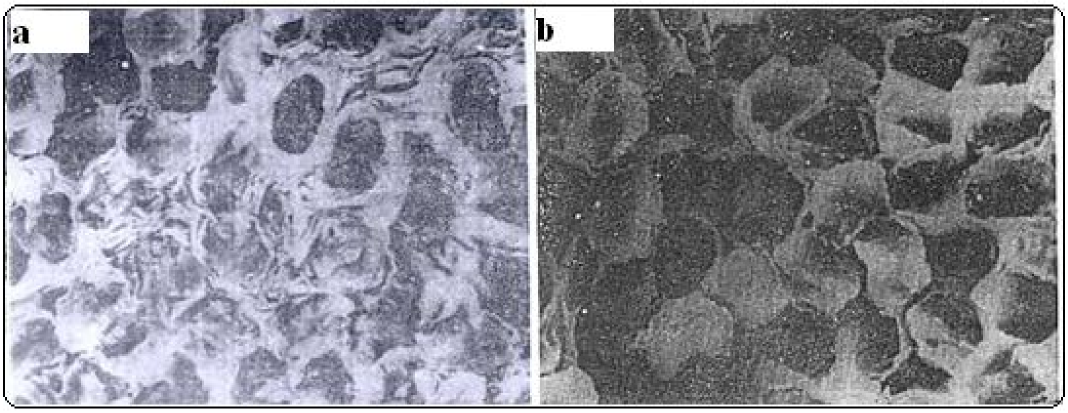


Fig. 2.5 SEM micrograph of carbon-carbon composites with total pitch derived Matrix [(a) P+P and (b) R+P] [G. Rohini Devi et al. 1993]

Figures 2.4 (a) and 2.4 (b) show the optical micrographs of carbon-carbon skeleton formed from carbon-phenolic composite after carbonization. Gaps or cracks are seen in the transverse tows. No cracks are seen in the longitudinal tows. This indicates that the fibre is not getting damaged during skeleton formation but in transverse tows filaments are getting separated. Generally, cracks occur during pyrolysis as shrinkage of resin is restricted by the surrounding fibres. Strong chemical adhesion between fibre and resin has to be avoided to prevent the damage of carbon fibre during carbonization. A study of different matrix precursors namely resin (R) and pitch (P) for skeleton formation showed that the porosity is slightly higher in composites totally processed with pitch material possibly due to the higher density of carbon derived from pitch. The final density of the composite can be achieved faster with pitch matrix. During the graphitization process, the carbon derived from pitch readily undergoes graphitization and the extent of graphitization depends upon the heat treatment temperature. Carbon derived from thermosetting resin is generally glassy in nature and becomes graphite only under stress-induced graphitization. The pitch carbon is anisotropic as seen under polarized light. Figures 2.5 (a) and 2.5 (b) show the SEM micrograph of the composite totally processed with pitch. Bulk matrix and matrix around the filament show lamellar structure indicating good graphitization; and the SEM micrograph of the composite where skeleton was made using resin and subsequent densification was carried out by pitch. The bulk matrix (pitch) is in lamellar structure. But the matrix around the filament shows the lamellar structure to a limited extent only [G. Rohini Devi et al. 1993].

2.4 General Properties And Characteristics Of Conventional Carbon-Carbon Composites

The most important class of properties of carbon-carbon composites is their thermal properties. Carbon-carbon composites have very low thermal expansion coefficients, making them dimensionally stable at a wide range of temperatures, and they have high thermal conductivity. Carbon-carbon composites retain mechanical properties even at temperatures above 2000°C in non-oxidizing atmospheres. They are also highly resistant to thermal shock, or fracture due to rapid and extreme changes in temperature.

Table 2.1 Carbon-carbon composites compared to other materials [G. Savage 1993]

Comparisons	Carbon-carbon Composites
Compared to metal	High heat resistance over 2000℃ Low thermal expansion Light weight (1/5 of metal) Excellent resistance to corrosion and radiation
Compared to graphite	High strength and rigidity High resistance to fracture
Compared to ceramic	High resistance against fracture Does not bond Can be made into complex shape
Compared to plastic	Heat resistance over 2000℃ Excellent resistance against corrosion and radiation

The material properties of a carbon-carbon composite vary depending on the fiber fraction, fibre type selected, textile weave type and similar factors, and the individual properties of the fibres and matrix material. Fibre properties depend on precursor material, production process, degree of graphitization and orientation, etc. The tensioning step in fibre formation is critical in making a fibre (and therefore a composite) with any useful strength at all. Matrix precursor material and manufacturing method have a significant impact on composite strength. Sufficient and uniform densification is necessary for a strong composite. Generally, the elastic modulus is very high, from 15-20GPa for composites made with a 3D fibre felt to 80-120GPa for those made with unidirectional fibre sheet (along fibre axis). Other properties include low-weight, high abrasion resistance, high electrical conductivity, low hygroscopicity, non-brittle failure, and resistance to biological rejection and chemical corrosion. Carbon-carbon composites are very workable, and can be formed into complex shapes. Compared to other materials such as ceramics, metal, and plastic as listed in Table 2.1 it is light and strong and can withstand temperatures over 2000℃ without any loss in performance. Some of mechanical properties listed in Table 2.2.

Table 2.2 Mechanical properties of carbon-carbon composites [Donald LS. et al. 1999]

Property (50wt% fibre)	Fine Grained Graphite	Unidirectional Fibres	3-D Fibres
Elastic Modulus (GPa)	10 -15	36 - 40	25 -35
Tensile Strength (MPa)	40-60	200-250	100-150
Compressive Strength (MPa)	95-105	100-115	108-115
Fracture Energy (Charpy) (kJ/m^{-2})	0.07-0.09	1.4-2.0	5-10
Oxidation resistance	very low	poor	better than graphite

Many users have found that the carbon-carbon composites have a longer life than stainless steel in some applications. The major applications for this new material are parts for furnaces, ovens, and other industrial heating equipment. Aircraft brakes utilize carbon-carbon composites for its frictional characteristics and thermal shock resistance; while prosthetic devices may use carbon-carbon composites in future (carbon has been demonstrated to have biological compatibility). The inertness of carbon-carbon composites in most space environment gives them potential for space structures, and their corrosion resistance makes them ideal for use in chemical plants and other aggressive environments. By controlling the directional arrangement and the length of carbon fiber reinforcement that supports the carbon matrix, it is possible to change the characteristics of the carbon-carbon composite so that it can be used for different kinds of applications as listed in Table 2.3.

Table 2.3 Characteristics of the carbon-carbon composites used for different applications [John D. Buckley et al. 1993]

Directional Arrangement of fibre	Length of fiber	Application	Characteristics
Unidirectional	Long	Rods, Springs, Pins	Maximum rigidity
Bi-directional Layer	Long	Plates, Heat trays	High resistance against heat High resistance against corrosion
Bi-directional Woven	Long	Pipes, Angles, Sheets	Can be made into complex shape
Multi-directional	Short	Brakes, Clutches, Bolts/Nuts	For precision processing and high technicality material
Custom/specially designed	Long	Rocket, aircraft material	Available for special application

2.5 Creep Behavior And Stress Relaxation Of Carbon-Carbon Composites

Creep is the time-dependent deformation or strain exhibited by a material under the action of a constant stress. Creep is characterized as a function of time by measurement of the creep compliance which is determined by dividing the time-dependent strain by the level of constant stress. Similarly, stress relaxation is the time-dependent stress exhibited by a material under the action of a constant strain. The relaxation modulus is determined by dividing the time-dependent stress by the constant applied strain. Creep and stress relaxation are different manifestations of the same underlying mechanisms of molecular mobility. At low levels of applied stress or strain these time-dependent effects may be completely recoverable when the forcing function is removed, but at higher levels irrecoverable deformations may occur under load. Irrecoverable strain, sometimes called permanent set, may be accompanied by time-dependent damage development such as the formation and growth of transverse matrix cracking. Viscoelastic effects should be considered if the end use involves high stresses in a matrix-dominated direction, high temperature or exposure to a harsh chemical environment. Composite structural designs should be evaluated for potential time-dependent effects if the working load involves significant shear loading. Since high shear loads can be generated near a structural discontinuity, these are areas of potential concern. It should be noted that viscoelastic effects can be beneficial in some of these instances, since stress relaxation in high stress regions can help prevent catastrophic failure. When a thermoplastic matrix is employed, time-dependent behavior may be a problem, especially if the service temperature is at or near T_g. The extent of creep should be smaller in thermoset composites due to cross-linking in fiber-reinforced plastic composites; one can assume that creep will be more important when the composite is loaded in a matrix-dominated manner than in a fiber-dominated manner. For instance, the creep of a unidirectional specimen tensile loaded in the fiber direction is expected to be small and hence only of secondary importance. However, loading a specimen in a matrix-dominated manner is not as straight forward as one would expect. Testing a unidirectional specimen in a transverse tensile manner one would think must essentially load the matrix, and this is not so. There are several explanations. One of the explanations is that loading a transverse specimen puts the matrix in a bidirectional state of stress (tensile) because the fibers prevent the matrix from laterally contracting (i.e., Poisson's effect) and thus the amount of creep response is restricted. Another argument for low creep response in transverse specimens is that the specimens are weak and the strains are small, so the change in strain would also be small. Another way to load the matrix is in shear where the creep response should be large. The most convenient way to load the matrix in shear is to load a [+45^0] specimen in tension. Although there is some argument that this test does not produce pure intra-laminar shear, it at least produces some shearing and can be thought analogous to loading a unidirectional laminate in shear. Experience has shown that the resulting creep is significant. Other loadings that would be interesting to examine with respect to creep response would be compression of unidirectional specimens in the fiber direction and three-point bend loading of unidirectional specimens (in both of these methods shear plays a role). A common experimental procedure is to apply a dead weight tensile load to a (+45^0) specimen at 35 MPa, 70 MPa, or 105 MPa and monitor the strain as a function of time. The strain reading at the first application of the full load is designated as the strain at zero time. Subsequent measurements are timed from that zero time reading. Readings are taken at 1, 2, 3, 10, 20, 30, 60, 100, and 200 minutes, and then as convenience dictates. Strain as a function of time is plotted on semi-log axes and the test continued for at least 30,000 minutes (or 3 weeks). Testing should be done at controlled (constant) humidity and temperature conditions [J. Schijodt et al 2000]. Generally, specimens are 25 mm wide, 150 mm long and 1-1.5 mm thick [ASTM Practice E 139-83. 1984]. These dimensions are open to question; there is some evidence that wider samples will creep less than narrow samples.

2.6 Wear Properties

The main definitions concerning tribology are summarized as follows. The term tribology is derived from the Greek word tribo, which means rubbing. It is defined as the science and technology of interacting surfaces in relative motion and of related subjects and practices. Tribology embraces the scientific investigation of all types of friction, lubrication and wear and also the technical application of tribological knowledge. Resistance against the lateral reciprocal motion of two surfaces is relative to each other. The friction force is the resisting force tangential to the common boundary between two bodies, under the action of an external force, when one body moves, or tends to move, relative to the surface of the other [Czichos H. 1986, Czichos H. et al. 1992].

The progressive loss of material from the surface of a solid body is due to mechanical action, i.e. the contact and relative motion against a solid, liquid or gaseous counterface. Friction and wear are not intrinsic material properties but are characteristics of the engineering system. Friction and wear are respectively serious causes of energy dissipation and material dissipation. Damage occurring when two bodies are in contact and in relative motion produces debris [Toby J.et al. 2001, Haytam Kasema et al. 2010]. Wear is produced when this debris is removed from the contact. The rate of expulsion of the debris depends on the contact geometry. Confined or conforming contact will not allow debris to displace. The wear particles may act as weak layers which reduce friction, i.e. as lubricants. They have a much more complex effect such as making the counterpart smoother. The contact zone is seen as having changing morphology, structure, chemistry, particle size and aspect ratio. It will be stretched, compacted and rolled by the repeated deformations in the contact [T. Policandriotes. et al. 2011, Briscoe B. J. et al. 1990]. Wear is defined as damage to a solid surface, generally involving progressive loss of material, due to relative motion between that surface and contacting substance or substances. The five main types of wear are abrasive, adhesive, fretting, erosion and fatigue wear, which are commonly observed in practical situations. Abrasive wear is the most important among all the forms of wear because it contributes almost 63% of the total cost of wear [B. Suresha et al. 2009]. Wear mechanisms are divided into four basic categories under the headings of adhesion, abrasion, surface fatigue and tribochemical reactions [Czichos H. 1986]. These are presented in Table 2.4.

Table 2.4 Wear mechanisms [Czichos H. 1986].

Adhesion	Formation and breaking of interfacial adhesive bonds
Abrasion	Removal of material due to scratching
Surface fatigue	Fatigue and formation of cracks in surface regions due to tribological stress cycles that result in the separation of material
Tribochemical reactions	Formation of chemical reaction products as a result of chemical interactions between the elements of a tribo-system initiated by tribological action

The adhesive wear processes are initiated by the interfacial adhesive junctions formed if solid materials are in contact. Different adhesive junctions may result depending on the nature of the solids in contact. The properties of the contacting solids influence the adhesive wear mechanisms. Since both adhesion and fracture are influenced by surface contaminants and the effect of the environment, it is quite difficult to relate adhesive wear processes with elementary bulk properties of materials. The tendency to form adhesion junction depends on physical and chemical properties of the materials in contact, the mode and value of loading and properties of the contacting surfaces such as contamination or roughness. The main contributions to adhesion that may be expected are for metals; primary bonds, metallic and covalent, and secondary bonds such as Vander Waals. For polymers: Vander Waals bonds, electrostatic bonds due to electrically charged double-layers, and hydrogen bonding by polar molecules.

Many theories of adhesion have been proposed in the literature. Kinloch A. J. in the year 1987 presented five main groups of mechanisms of adhesion. The effect of abrasion occurs in contact situation, in which direct physical contact between two surfaces is given, where one of the surfaces is considerably harder than the other one. The harder surface asperities penetrate into the softer surface with plastic flow of the softer surface occurring around the asperities of the harder surface [Kinloch A. J. 1987 and Czichos H. 1986]. When a tangential motion is imposed, the harder surface removes the softer material by combined effects of micro-ploughing, micro-cutting and micro-cracking. Abrasion is the wear by displacement of materials from surfaces in relative motion caused by the presence of hard protuberances or by the presence of hard particles either between the surfaces or embedded in one of them [Lee L. H. 1985].

Wear by surface fatigue can be characterized by crack formation and flaking of material caused by repeated alternating loading of solid surface. Localized fatigue may occur on a microscopic scale due to repeated sliding contact of asperities on the surfaces of solids in relative motion. Under repeated tribological loading, surface fatigue phenomena may occur leading finally to the generation of wear particles. These effects are mainly based on the action of stresses in or below the surfaces without needing a direct physical solid contact of the surfaces under consideration. The mechanisms of surface fatigue and abrasion can be described mainly in terms of stress interactions and deformation properties whereas, tribochemical wear has a third partner, the environment [Czichos H. 1986]. The wear process proceeds by continual removal and formation of new layers on the contacting surfaces. In the presence of oxygen, worn debris consists largely of oxides which have been formed upon the surfaces and have been removed by rubbing. The dynamic interaction between the material components and the environment determine the wear processes. These interactions may be expressed as cyclic stepwise processes. First, the material surface reacts with the environment. In this process, reaction products are formed on the surfaces. Secondly, the reaction products wear away as a result of crack formation and abrasion in the contact process. When this occurs fresh i.e. reactive surface parts of the materials are formed, step 1 continues [Czichos H. 1986].

Due to their low density (≤ 2g/cc) combined with remarkable thermo mechanical properties, carbon-carbon composites are recognized as excellent materials for use as aircraft brake discs. They are effective in the different types of aircraft braking operations, since they preserve their mechanical and thermal properties even at high temperatures over 2000^0C [Haytam Kasem.et al. 2010, S. Fouquet. et al. 2008]. Several studies reported in the literature are focused on the tribological behavior of carbon-carbon composites. Carbon–carbon composites have widened the scope of application of carbon-based materials in wear-related applications from bearing seals and electrical brushes to brake pads for heavy duty vehicles such as military, supersonic and civilian aircrafts to trucks and railways. This has been due to basic tribological properties of carbon with additional high strength and thermal conductivity contribution from the reinforcing fibers. Carbon-carbon composites exhibit low coefficient of friction in the fiber direction (0.33-0.53 and 0.55-0.80 in the perpendicular direction and wear rates also follow similar trends. [Lalit M. Manocha 2003]. The friction and wear mechanism of carbon-carbon composites on application of brakes is quite complex and various factors like peak temperature, formation of debris and films on sliding surfaces further affect the coefficient of friction etc. which is too vast to cover here. In general, for fabricating brake discs for different vehicles, different types of fibres (PAN, rayon and pitch-based high strength and high modulus fibre) are used in a number of configurations, either alone or in combinations with carbon matrix derived from different types of precursor, pitch or chemical vapor deposition. In commercial carbon-carbon brake pads, carbon fiber fabric plies are used with fiber tows inserted in the third direction and pitch or chemical vapor deposition route for densification. However, choice of the constituents and processing parameters is at the discretion of the manufacturers [Kia-Moh Teo et al. 2004]. Carbon/carbon composites are extensively used as aircraft brake pad materials on account of their excellent tribological behavior at high sliding speeds. It is well known that the tribological behavior of carbon-carbon composites is dependent on the nature of the fibre layup, heat treatment condition employed, etc. More importantly, carbon-carbon composites also exhibit a transition from a low friction coefficient (μ), normal wear regime, to a high μ dusting wear regime, when the PV value (P is normal pressure and V is sliding velocity) exceeds a critical value. It is also generally accepted that the above transition is associated with the attainment of a critical temperature at the interface between the two carbon-carbon composite bodies sliding against each other [B. Venkataramen et al.2001]. Because of their low density, low wear and high temperature stability, carbon-carbon composites have emerged as advanced friction materials. Many recent military aircraft employ carbon-carbon composites in their brake systems. Future applications in commercial aircraft and automobiles are being considered [Sheehan J. E. et al.1994]. Carbon fiber reinforced carbon composites are a family of materials which possess much of the properties of polymer composites and monolithic carbon materials. They are usually made in a variety of fiber preform architectures which are impregnated either by liquid precursors (pitch or resin) prior to carbonization, or by a carbon bearing gas precursor (e.g. methane) till an accepted level of porosity is achieved. These combinations result in a variety of materials that have high specific strength, stiffness and toughness, self-lubricating capability, low thermal expansion coefficient and outstanding refractory properties. Therefore, a number of applications are available ranging from prosthetics to aero spatial vehicle structures. Carbon-carbon composites have emerged as a strong friction material in the late sixties and since then, their use as brake disc materials for military aircraft, such as the US F-14, F-15, F-16 and F-18, the French Mirage 2000, as well as in commercial aircraft as Boeing 747, Boeing 787, Airbus and Concorde, have this disc material. For brake applications, a primary requirement is the maintenance of a stable coefficient of friction and a low wear rate over a wide range of sliding conditions, particularly temperature, that can exceed 1500°C [J.R. Gomesa et al. 2001]. Brake applications require the sliding interfaces to convert about 4500 J cm^{-2} of kinetic energy per unit area to heat in approximately 30 seconds. In addition, the thermal cycling promotes cracking which can result in accelerated wear and oxidation.

2.7 Oxidation Behavior Of Carbon-Carbon Composites

So far, the main problem with carbon-carbon composites is the tendency to oxidize at high temperatures (500 to 700^0C, in air); particularly in the presence of atomic oxygen, also micro cracks may develop in the matrix and at matrix-fiber interfaces after processing and during thermal cycling particularly in multidirectional reinforced composites. Some research has shown that such cracks have little effect on bending strength; although they may reduce the elastic modulus and co-efficient of thermal expansion. Some evidence also indicates that micro cracks may reduce tensile strength, fatigue strength, fracture modulus and thermal conductivity. A protective coating usually silicon carbide must be applied to prevent high-temperature oxidation, adding an additional manufacturing step and additional cost to the production process. The high electrical conductivity of airborne graphite particles creates an unhealthy environment for electrical equipment near machining areas. Carbon-carbon composites are currently very expensive and complicated to produce, which limits their use mostly to aerospace and defense applications. Carbon-carbon composites are widely used as aeronautic and astronautic materials for their excellent performance in mechanical properties at high temperature, such as heat shields, nose-tips for reentry vehicles, leading edges and airplane brakes [Jun Li. et al. 2008, R.Y. Luo et al. 2002]. However, several technological barriers limit a wider application of carbon-carbon composites. One of the major technological obstacles is the susceptibility of these composites to air oxidation at temperature above 400^0C, which would result in the erosion of the structure and eventually the degradation of the properties that the material originally possesses [T. Damjanovic et al. 2005]. Over the past 60 years, many researchers have been conducted on oxidation resistance for carbon-carbon composites, and the application of refractory materials coated on the surface of carbon-carbon composites is considered to be the best choice. Consequently, most of the researchers focus on the silicon-containing ceramic coatings. However, the cracks formed by the coefficient of thermal expansion mismatch of the coatings and the carbon-carbon substrate are an inevitable problem, which results in limited oxidation protection. Recently, the advanced coatings, with a functionally gradient layer to minimize the influence of the coefficient thermal expansion mismatch, are designed to be multilayer coatings. A number of different oxidation protection mechanisms have been explored to improve the oxidation resistance of carbon-carbon composite also. The techniques developed can be categorized as surface coatings; single layer-multilayer's, using chemical vapor deposition, pack cementation, physical vapor deposition and plasma spray. However, the interfacial cracks and the high cost still limit the application of such coatings. On the other hand, the oxidation kinetics of multilayer coatings has not yet been discussed in depth [W. Kowbel et al. 1995].

The attractive mechanical and thermal properties of carbon-carbon at elevated temperatures and some of the potential applications like turbine structural components which require long term exposure and high temperatures are restricted by the inherent reactivity of carbon towards oxygen beyond 400^0C. In depth protection includes sol gel process, impregnation with inorganic salts for limited temperature range and melt impregnation or in depth deposition of SiC matrix. With the external protection methods, the thermal expansion mismatch between carbon material and possible refractory coatings is the main problem to be overcome [F. Smeacetto et al. 2007]. Micro cracks developed in refractory layers have to be sealed with glassy coatings is another method of protection. The best oxidation resistance was achieved in which chemical vapor deposited surface coatings were formed in addition to in depth protection. Internal protection methods include first direct removal or deactivation of catalytic impurities, and second incorporation of oxidation inhibitors and total or partial substitution of matrix material. A successful protection system comprises a coating, internal inhibitor and a compatible substrate since carbon-carbon composites constitute a diverse class of materials with a wide range of mechanical, thermal and morphological properties. Selection of appropriate fibre, preform fabrication technique, matrix precursor and densification processing method is essential if good oxidation resistance as well as physico-chemical compatibility between substrate and coating is to be achieved. The progress of research on oxidation protection is illustrated by the bar chart shown in Fig. 2.6 [Yanhui Chu et al. 2011 and G. Rohini Devi et al. 1993].

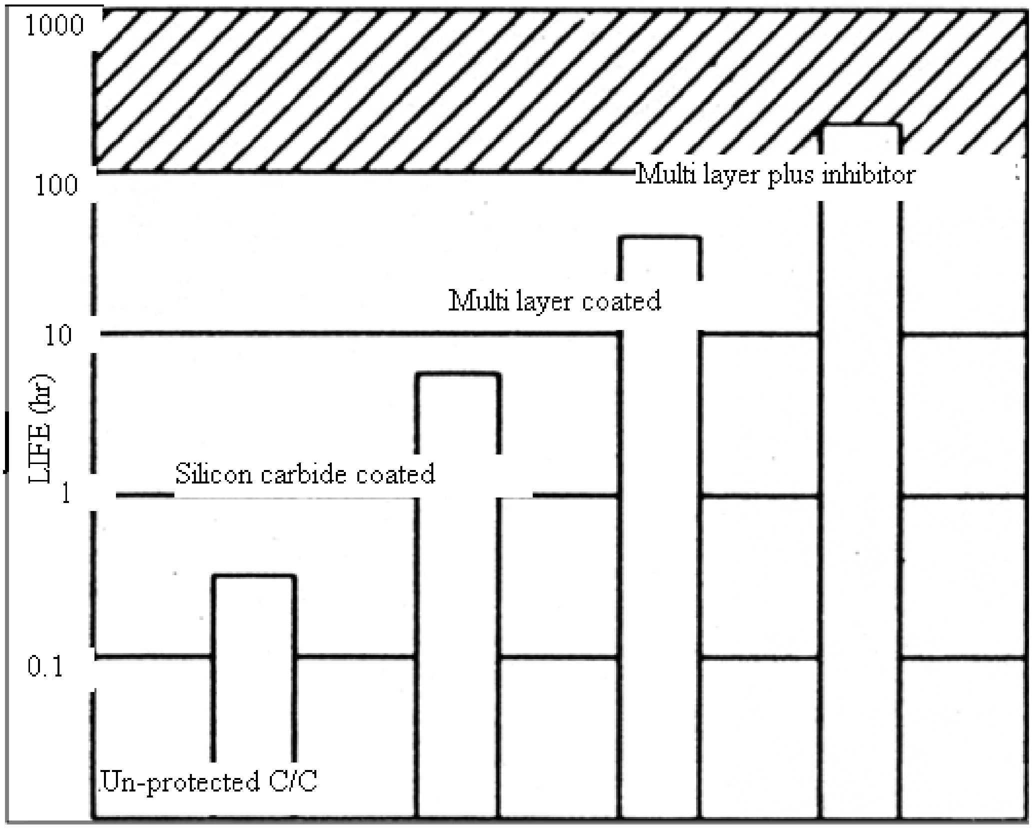


Fig. 2.6 Oxidation protection life versus types of coating [G. Rohini Devi et al. 1993]

Anti-oxidation is a key requirement for carbon-carbon composites to be used as high-temperature structural materials in an oxygen-containing environment [N.S. Jacobson et al.2006]. To prevent carbon–carbon composites from oxidation, Si–Mo–Cr alloy has been extensively used as the coating material because a compound glass layer involving SiO_2 and Cr_2O_3 would be formed, thus effectively preventing carbon-carbon composites from oxidation due to its high resistance to both volatilization and oxygen diffusion [A. Tomasi.et al.1997]. In addition, provided that the coating materials could infiltrate into carbon-carbon composites deeply through the cracks or holes, the favorable inlaid structure and intensive interfacial bonding would be formed at the carbon-carbon coating, which could further improve the oxidation resistance and mechanical properties of coated carbon-carbon composites. Low-density carbon-carbon composites have been considered as potential candidates into which could be readily infiltrated by the coating materials through the simple and low-cost techniques.

By summarizing, many articles on conventional carbon-carbon composites are of interest because of their ability to retain strength and structural integrity till 3000°C either in vacuum or in inert environment. Not only the mechanical properties are retained, but also the tensile strength is found to increase with increase of temperature [Sourav Sarkar et al. 2010]. These composites also possess outstanding specific strength and stiffness. Hence, the carbon-carbon are suitable for application in jet engine components, as well as in thermal protection systems used in nose cones and leading edges of hypersonic and re-entry type vehicles, which are exposed to elevated temperatures [K. Goto H. et al.2003]. Due to impressive high temperature properties, the applications of carbon-carbon in the aerospace industry have increased significantly in recent decades. Carbon-carbon composites are lightweight materials that can perform structurally at extreme temperatures and have superior thermal shock, toughness, ablation, and high-speed friction properties. Carbon-carbon composites have been emerged in response to sustained aerospace and defense needs [K. Goto H.et al.2003]. However, the extremely high price of carbon-carbon composite due to production characteristics made it difficult to the industrial application. The Institute of Production Technology of Tokyo University in Japan stated that they have developed innovative Preformed Yarn (PY) technology, which improved manufacturing process drastically from existing impregnation method (conventional) and resulted in reduction of manufacturing cost and time considerably [Nagao H. et al. 1999]. Substantial investigations have been carried out but there is lot of scope for further research, on the design and development of PY production model and synthesis of carbon-carbon composites by preformed yarn method. The systematic study of PY preparation method and synthesis are still underway. A comprehensive review on all those works is beyond the scope of this article; however, a few works which are related to the current study are summarized here.

2.8 Preformed Yarn Carbon-Carbon Composites

Carbon-carbon composite is compound materials of reinforced carbon fiber and carbon matrix, made utilizing preformed yarn. It has an excellent physical characteristic of oxidation resistance, strong hardness and high elasticity etc. This unique method has enabled Across Company to drastically shorten processing time. This improvement allows this company to satisfy various kinds of customers needs for reasonable time periods. The new method has also resulted in dramatic cost reductions for the manufacturing of carbon-carbon composites. Conventional carbon-carbon composites used to be expensive and time-consuming to manufacture due to its complicated processing method such as chemical vapor deposition.

The Authors [Toshoku C. et al. 2004 and M. G. Jenkins 1998] have simplified the manufacturing process by using a new patented method called preformed carbon fibre yarn, resulting in great reduction in time and cost as compared in Fig. 2.7 below. In order to simplify the manufacturing of carbon-carbon composites the authors have developed a new production method, using preformed yarns, which contain the carbon fiber bundle as well as the matrix (coke and pitch). These PY enable us easily to fabricate primary work pieces such as unidirectional sheets, cloths, tapes and chopped yarns. Further, it is possible to prepare pipe and tube shaped standby preforms. From these work pieces and preforms, the carbon-carbon composites can easily be produced by hot press molding method.

The authors [T Chang et al. 1988 and Nakagawa et al. 1988] have worked up an experimental production of carbon fiber-carbon matrix composites by hot press molding method. They developed a simplified molding method allowing to form a lamination of unidirectional or cloth type carbon fibers with a mixture of coke powders and a carbonaceous binder, the latter is used to form the matrix of the new developed carbon-carbon composites.

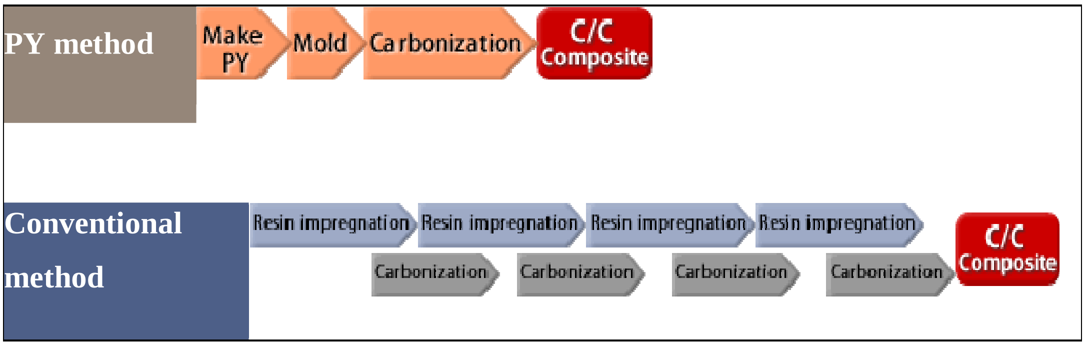


Fig. 2.7 Manufacturing process compared [K. Goto H. et al. 2003]

The binder, used for the simplified manufacturing process of carbon-carbon composites, has the following characteristics; the binder in matrix softens with raising of temperature in the hot press process and penetrates into the fiber bundles. In this simplified process, the binder is prevented from the complete penetration because of its high viscosity, from which certain voids are formed in the matrix and therefore, carbon-carbon composite shall not be able to obtain a much stronger bond. In this process a preformed yarn already contains the components of the matrix within the carbon fiber bundles. The purpose of the present work is to explain the newly developed manufacturing process of preformed yarns and the mechanical properties of new carbon-carbon composites thus manufactured from the preformed yarns.

The preformed yarns are made by including the matrix powders in the endless carbon fibers bundle stand by coating its circumference with a thermoplastic resin. The matrix powder used for producing the preformed yarns consists of coke powders and the binder; the former being petroleum coke with the particle size of 4 to 5mm and the latter being a petroleum pitch with the volatile matter of 17 wt%. The carbon fibre used was the Torayka 300 (T300) having 6,000 filaments, and the sleeve was made of Nylon. The petroleum coke used for the matrix was selected in consideration of its better affinity with the binder made from petroleum pitch. The patented preformed yarn method involves producing yarn consisting of a bundle of carbon fibres in a matrix precursors of coke and pitch binder powders. The yarn is encased in a flexible thermoplastic sleeve to contain the powders during handling and subsequent processing. This simplified manufacturing process ensures better penetration of the binder into the carbon fiber bundle, thereby resulting in higher strength than conventional carbon-carbon composites. Furthermore, the matrix powders are often derived from the residue of petroleum processing, which can have beneficial environmental effects. Research has shown that unidirectional carbon-carbon composites manufactured by this simplified method achieve strengths of between 214 and 270 MPa, compared with 70 MPa for conventional carbon-carbon composites. They also show significantly higher flexural modulus [T Chang et al. 1988]. The preformed yarn may be woven into sheet or chopped to fill a mold, and is then hot pressed to form the composite product. Since the reinforcing fibers are homogeneously dispersed in the matrix, properties of the resulting composite are quite uniform. Carbon-carbon composites are composed of carbon or graphite fibre reinforcements in carbon or graphite matrices. They maintain high strength and low co-efficient of thermal expansion at temperatures above 2000^0C (3600F), and exhibit excellent thermal shock resistance. They also have superior toughness, excellent thermal and electrical conductivity, and resistance to corrosion and abrasion as in the case of conventional composites. In spite of this outstanding combination of properties, high cost has limited their applications primarily to aerospace and defense. However, this new processing method involving performed yarns has been developed by the Across Company, Japan that is said to considerably reduce both cost and manufacturing time.

The patented preformed yarn method involves producing yarn consisting of a bundle of carbon fibres in a matrix precursor of coke and pitch binder powders. The yarn is encased in a flexible thermoplastic sleeve to contain the powders during handling and subsequent processing. This simplified manufacturing process ensures better penetration of the binder into the carbon fiber bundle, there by resulting in higher strength than conventional carbon-carbon composites. Furthermore, the matrix powders are often derived from the residue of petroleum processing, which can have beneficial environmental effects.

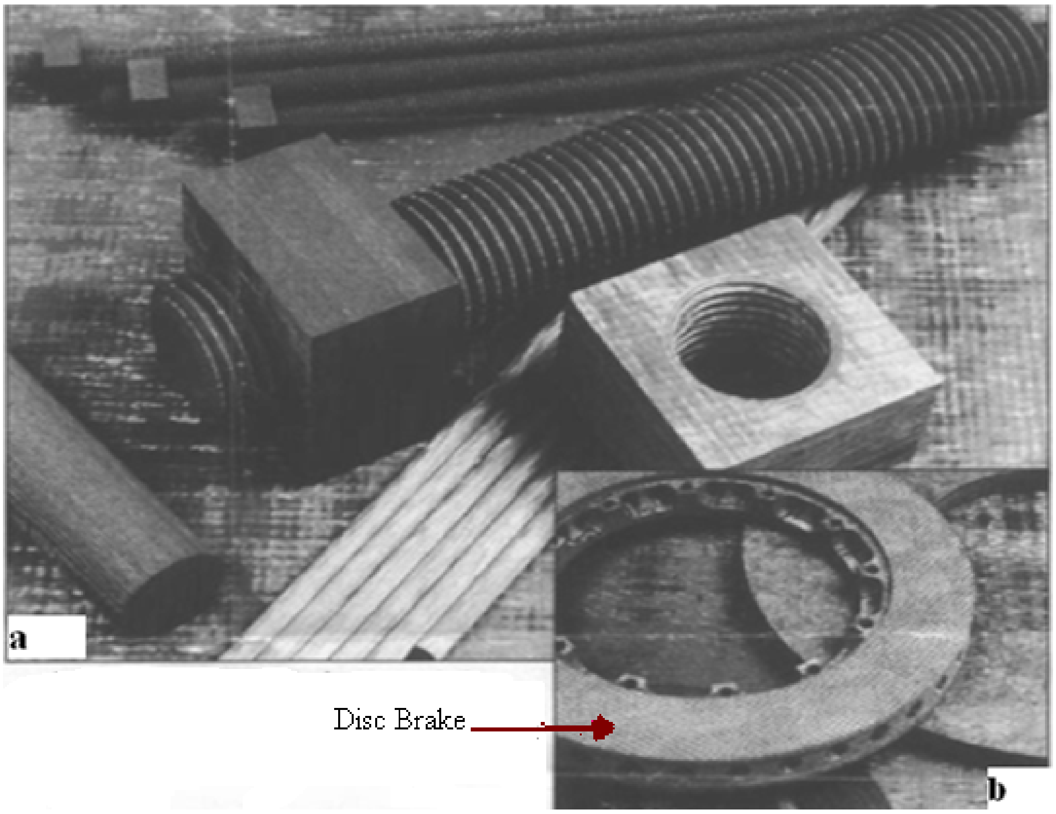


Fig. 2.8 (a) Carbon-carbon composite nuts, bolts, rods and (b) carbon-carbon composites brake [K. Goto H.et al. 2003]

The preformed yarn may be woven into sheet or chopped to fill a mold, and is then hot-pressed to form the composite product. Since the reinforcing fibres are homogeneously dispersed in the matrix, properties of the resulting composites are quite uniform. The preformed yarns have excellent workability and processability. For example, pipe-, rod- and crucible-shaped preforms as well as unidirectional sheets, cloth sheets, thick textiles, tapes, and chopped yarns may be readily fabricated. Workability of the preformed yarns allows the material to be tailored to meet various design loads. This method is also well-suited for the production of complex shapes and components having small radii of curvature. The composites are available in un-machined form, as well as finished products such as coil springs, rods, shelves, bolts and nuts, link conveyor belts, roller conveyor parts, and furnace parts; Fig. 2.8 (a) and 2.8 (b) show some of the applications.

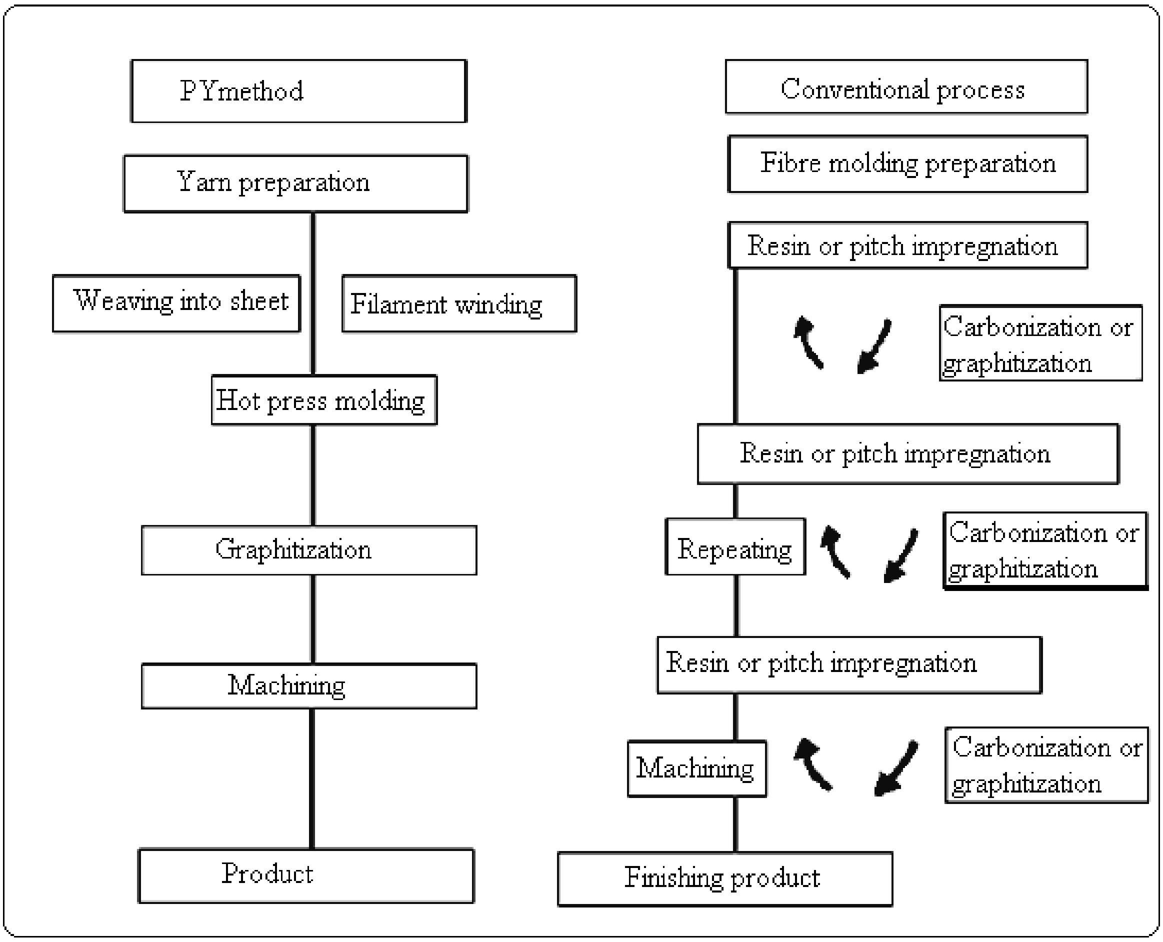


Fig. 2.9 The preformed yarn process reduces the number of steps required to produce carbon-carbon composites, compared with conventional methods [Hatta H. et al. 2004]

A Japanese steel company found that racks made of the new carbon-carbon composite allowed the construction of lighter-weight platforms for annealing of steel powder than conventional graphite racks. Use of the material resulted in a 22.6% savings in heating energy as well as an 80% improvement in heating speed; Fig. 2.9 shows comparison between preformed and conventional methods of manufacturing. Nippon and Denso, Japan's largest manufacturer of automotive electrical components used the material for shelving and coil springs in their continuous heat treating equipment, and also significantly reduced their number of impregnation cycles and costs.

2.8.1 Preformed yarn composite synthesis method

Figure 2.10 shows the manufacturing process of the plate-type carbon-carbon composite in particular, which is composed of three blocks, the Block 1 ranging from the carbon fiber material to the preformed yarn (PY), the Block 2 ranging from PY to the primary workpiece, and the Block 3 ranging from the primary workpiece to the primary carbon-carbon composite. In Block 1, carbon fibres are processed into PY. It starts with the supply of continuous carbon fibre bundles to the matrix powder-applying apparatus, where the matrix powder is applied into the fiber bundles. The matrix containing bundles is fed to the fibre coating apparatus where the bundle surfaces are covered with Nylon 6. The coated bundles are wound up on the take up reel to finish up into bobbins of the preformed yarns. In the following Block 2, the preformed yarns are woven or cut to produce a variety of primary workpieces. In the final Block 3 process, the primary workpieces are baked in the hot press to produce the primary carbon-carbon composites at a baking temperature of 600°C. Then, they would have the secondary heat treatment to obtain the carbon-carbon composites. Besides the above mentioned plate-type carbon-carbon composites, the same of special forms can be produced on this manufacturing process. The PY-bobbins (produced in Block 1) contains endless carbon fibres and have characteristics of high flexibility, which allows obtaining various special forms of standby preforms which can be baked to pipes, rods, crucibles etc.

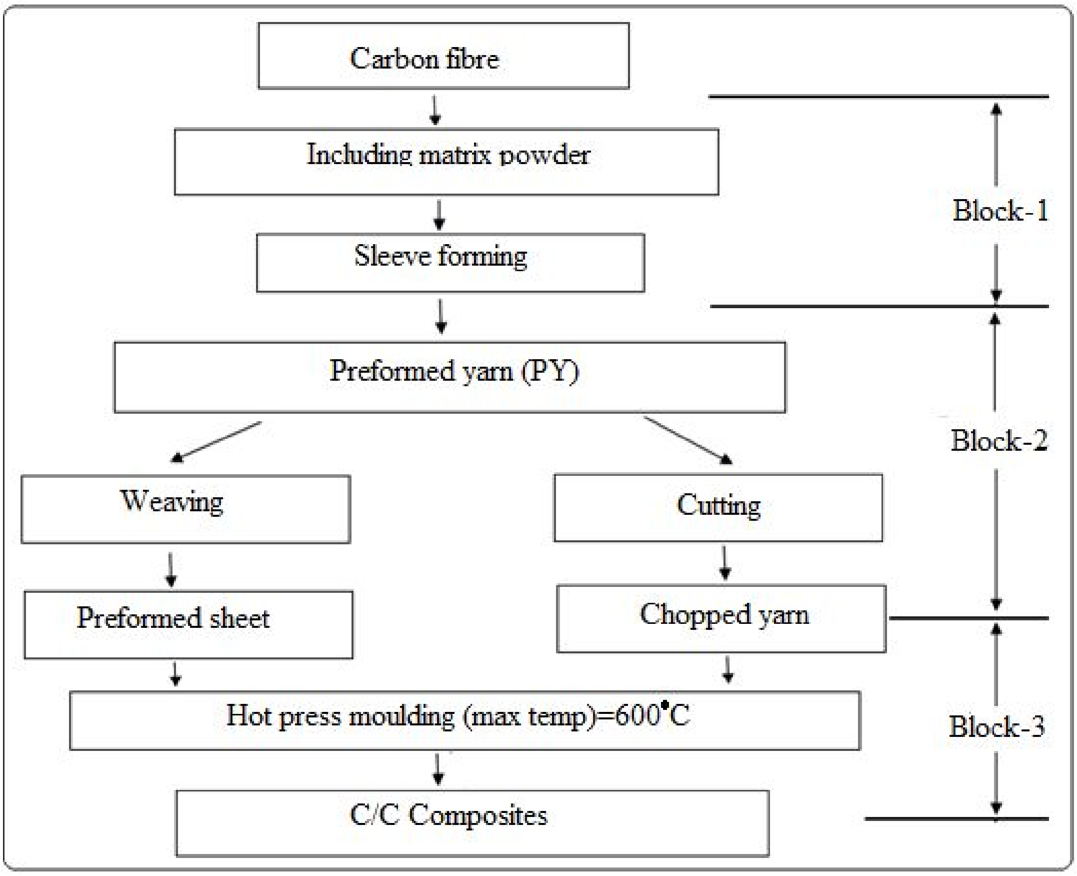


Fig. 2.10 Fabrication process of carbon-carbon composites [J. Economy H. et al. 1992]

As new materials having high temperature endurance, lightweight and high strength in various applications, carbon-carbon composite materials are undergoing examinations with great expectations. However, this material has production problems such as high cost and taking long hours to make. Also concerning its properties, it has some demerits that need improvement such as ease of oxidation at high temperatures, lack of tensile and compression strengths.

Table 2.5 Compositions of preformed yarn [Aya K. Masanao et al. 2004]

Composition	Type of yarn	Matrix (Wt %)	Fibre (Wt %)	Sleeve (Wt %)
1	PY-B50	34	51	15
2	PY-B60	50	32	18
3	PY-B75	34	42	24

2.8.2 Properties of PY carbon-carbon composites

Table 2.6 Properties of PY C/C composites using unidirectional sheets [T. Chang et al.1988]

Types of PY		PYB50	PYB60	PYB75	Conventional
Hot press condition	Max Temp.(°C)	600	600	600	-------
	Max Press(MPa)	49	49	49	-------
	V_f (vol %)	64.0	44.3	51.6	50
	ρ (g/cc)	1.638	1.56	1.594	1.760
	σ max (MPa)	214	262	270	70
	E (GPa)	96	69	77	37

As shown in Table 2.5, three different types of preformed yarn can be produced from three different components of materials. Composition 1 consists of 51 wt% carbon fibres, 34 wt% matrix (50 wt% coke powders, 50 wt% binder) and 15 wt% resin sleeve. In compositions 2 and 3, the ratio of the binder in matrix is increased to 60% and 75 wt% respectively, whereas the fiber content is decreased to some extent. The preformed yarns are named PY-B50, PY-B60 and PY-B75 respectively in accordance with the binder ratio of the matrix. The endless preformed yarns of the bobbins are used to fabricate unidirectional sheets, thicker fabrics and tapes as the primary workpieces. By cutting the endless preformed yarns, chopped yarns are obtained, which are used to produce random reinforcement. Table 2.6 shows the physical properties of different types of carbon-carbon composites made by unidirectional sheets. The maximum temperature and pressure for the hot press molding was set to 600°C and 49MPa respectively. The flexural strength of each of the carbon-carbon composite using PY-B50, PY-B60 and PY-B75 was 214MPa, 262MPa and 270MPa respectively, and this result suggests that the strength of each of experimental carbon-carbon was extremely high as compared with that of commercial carbon-carbon in table 2.6, and the flexural modulus of experimental products had high values of 96 GPa, 84 GPa and 77 GPa respectively as compared with 37GPa of the conventional carbon-carbon composites [Ken Goto. et al.2003 and Ken Goto. et al.2005]. The study on the effects of the hot pressing pressures upon the strengths of the two samples made from above experimental preformed yarn, were indicating the maximum (PY-B75) and minimum strengths (PY-B50), has results as shown in Fig. 2.11 which gives the pressure indicated on the abscissa and the flexural strength on the ordinate. This graph indicates the increasing trend of flexural strength in line with the increasing pressure when the PY-B75 having a higher content ratio of the binder in the matrix while the said strength shows a decreasing trend for the lower binder ratio PY-B50.As for the modulus; there were no significant variations (opposite trends) observed caused by the pressure difference between PY-B50 and PY-B75 but within the range of pressing pressure from 19.6MPa to 49MPa.

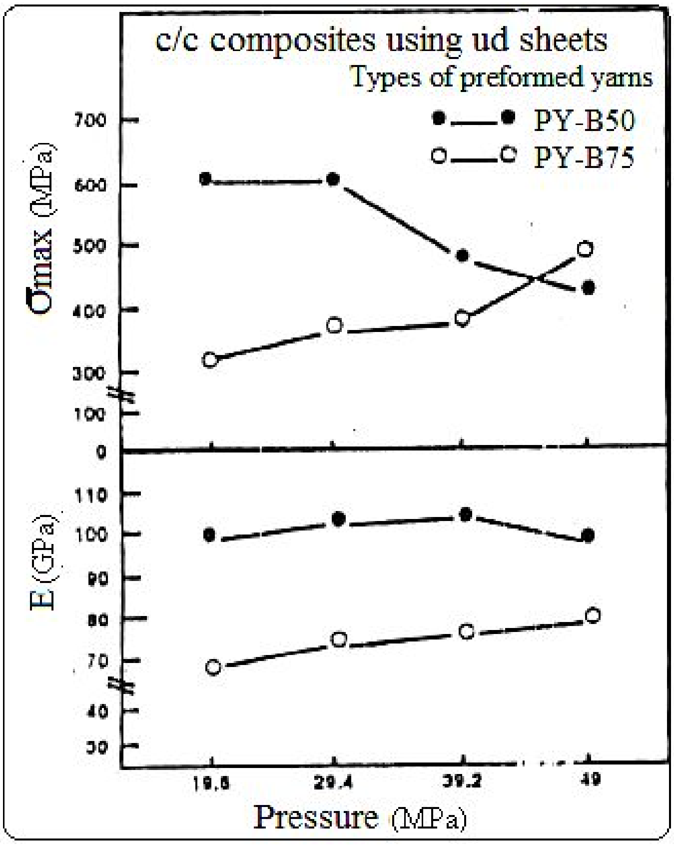


Fig. 2.11 Relation between hot pressing pressure and bending strength and Young's modulus of carbon-carbon composites using unidirectional sheets [Nago H. et al. 1999]

Figure 2.12 shows the relation between the apparent density and the flexural strength with changes in the pressure applied on the same yarn. The apparent density is indicated with an increase for each yarn along with increasing the pressing pressure. On the other hand, the flexural strength of PY-B75 increased with the increment of pressure, while PY-B50 took the reverse trend of decrease. From the fact that both the density and the flexural strength of PY-B75 increased, this increase in the flexural strength seems to have been made by its increasing density [Nagao H.et al.1999 and K. Goto H.et al.2003].

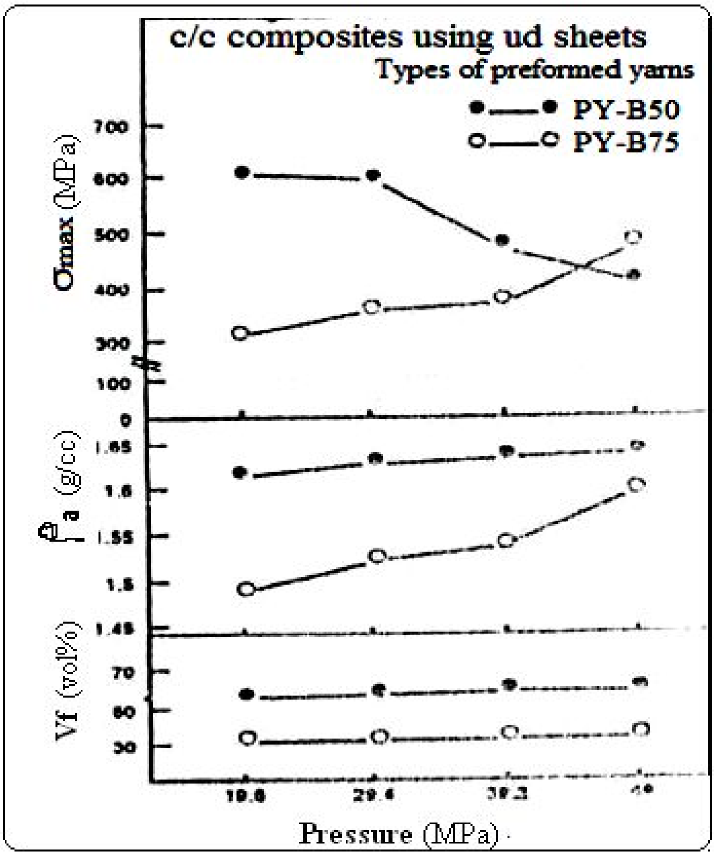


Fig. 2.12 Change of apparent density (ρ_a) and tensile strength of carbon-carbon composites using unidirectional sheets with hot-pressing pressure [Nago H. et al. 1999]

The comparison of properties of preformed yarn composites were focused at the area of ultra high temperature of 2500°C. The main objective is to clarify the properties of carbon-carbon composites, made by PY method. Looking at the micro-compositional structure point of view specific characteristic of PY carbon-carbon composite is residual stress aroused by casting and shrinkage of matrix when cooled from high to room temperature which brings cracks along the direction of the micron-size fibers. These cracks are called transverse cracks. These cracks bring out deterioration of carbon-carbon composite materials. But also, at the same time, bring out the merit of having thermal shock improvement. We will focus special attention on the effects on material properties of these cracks.

Materials tested were obtained from Across Company Ltd. The materials comprised of reinforced fiber of Toray M-40 matrix charcoal coke and bulk mesophase particle acted as binder. At casting stage, in the bulk of carbon fiber, the matrix materials are dispersed and enveloped by high polymer film and laid down in one direction to make it sheet shape. Generally these are called prepregnated materials. This is the start- ing material for the synthesis. Thermal condition of casting is pre-casting temperature of 600°C and casting temperature of 2000°C. Tested materials are prepregnated materials laid down multi-directionally and compressed with high heat. Multilayer compositions are of three kinds, unidirectional reinforced materials, perpendicular material (0°/90°) and pseudo-isotropic materials (0°/90°/±45°). Across, the manufacturer says that the carbon fiber content of all the three of these materials is 50wt% and unidirectional material shows less number of cracks compared to other two types.

Materials tested, as shown in Table 2.7 are predominantly, carbon-carbon composites made by preformed yarn, compared with characteristic of carbon composite made by conventional method from Across Company Ltd. Fig. 2.13 shows cross sectional view of preformed yarn [M. G. Jenkins et al.1988].

Table 2.7 Comparison between conventional carbon-carbon and Across PY carbon-carbon composites [Nagao H.et al.1999]

Properties-comparison between conventional C/C and Across PY C/C composites				
Specifications of Across Company Properties	PYAC100	PYAC200	PYAC250	Conventional C/C
Carbon fiber ratio by weight (%)	30	40	50	65
Processed temperature (°C)	2000	2000	2000	2000
Density (g/cc)	1.7	1.7	1.7	1.7
Flexural rigidity (MPa)	78	147	155	39
Compression strength (MPa)	78	98	170	78
Charpy impact value (kJ/m^2)	10	13	53	2
Thermal expansion (10^{-6}/°C)				
i. Along length	2	1.1	0.6	4.0
ii. Along thickness	---	8.4	8.2	4.0
Heat conductivity (kJ/m. hr .°C)				
i. Along length	62	120	242	405
ii. Along thickness	---	45	36	405
Specific heat (J/g°C).				
i. 20°C	0.74	0.74	0.74	0.72
ii. 1200°C	2.1	1.98	1.85	0.72

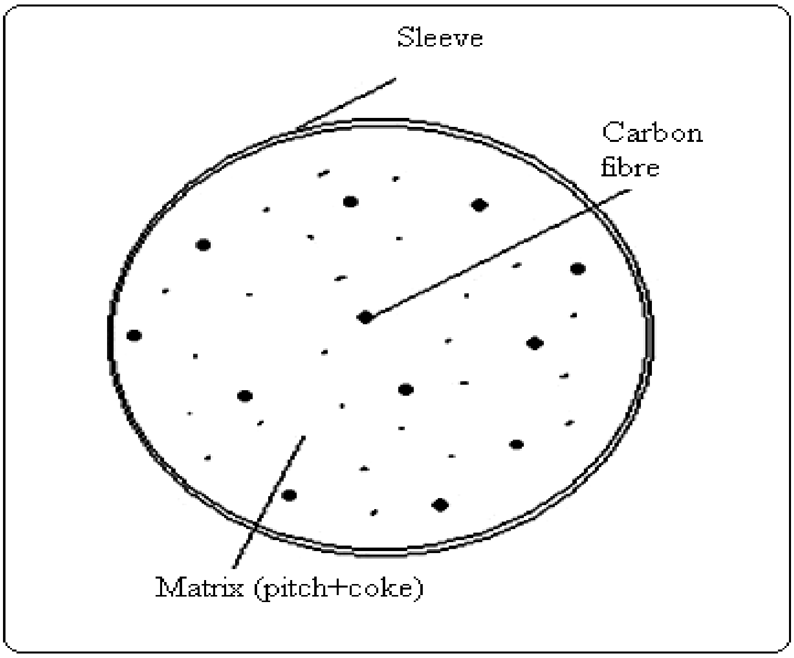


Fig. 2.13 Cross section of conceptual drawing of preformed yarn [M. G. Jenkins et al.1988]

2.8.3 Physical characteristics of preformed yarn carbon-carbon composites

The physical characteristics of preformed yarn carbon-carbon composites are listed below:

- No deterioration of strength under high temperature of more than 2000°C (in either N_2 or vacuum)
- Light weight in comparison with metal
- High tenacity and hard to break
- Excellent resistance to thermal shock, has durability during drastic increase and decrease of heat, thus good for extremely severe environment
- Small rate of thermal expansion which keeps an accuracy of dimension, volume etc under thermal change
- Good for components such as bolt, nut, springs etc for heat resistant equipment
- Excellent corrosion resistance.

- It has characteristic stability against chemicals which is the nature of carbon
- Good electric and thermal conductivity
- Good for components of furnace, ovens, electrode and pyrogenetic materials

2.8.4 Merit obtained through preformed yarn method

The merits obtained through preformed yarn method can be listed as:

- Drastically shortened lead time for production and delivery
- Flexibility for customers’ requirement
- Possibility of materials which has a thickness of more than 150mm
- Good for friction device such as brake, bearing, etc
- As the product has minimal thermal deformation characteristics, it is good for components for robotics system under high heat.

2.8.5 Major applications of preformed yarn carbon-carbon composites

The major applications of preformed yarn carbon-carbon composites are as follows:

- As heat resistant materials
    - Structural materials, components for high temperature devices such as furnaces, industrial ovens, etc.
    - Tray for furnaces and ovens
    - Various types of pyrogenetic materials
- Friction materials
    - Excellent frictional characteristics and thermal shock resistance of carbon-carbon composites makes them useful as; brake materials, industrial bearings etc.
- Anti-corrosive materials
    - Electrode and components for chemical plant.

A carbon-carbon composite coil spring was manufactured, using PY method. A bundle of carbon fibres was included in a matrix composed of mesophase pitch and petroleum coke, and coated with polypropylene, resulting in a preformed-yarn. Two kinds of carbon fibre, filament yarn and spun yarn, were used for reinforcement. The preformed-yarn was wound spirally around a metal core, and this was formed into a carbon-carbon coil spring by hot press forming. The spring manufactured by PY method using spun yarn showed a higher spring constant than that obtained using filament yarn. Subsequently, they applied similar technique to fabricate unidirectional carbon-carbon composites. Density, porosity, flexural strength and modulus of the resulting unidirectional carbon-carbon composite test pieces were measured and found to be superior by a factor of about 1.3 than the unidirectional carbon-carbon composites manufactured by conventional methods [Hiroshi Hatta et al. 1999]. The high-temperature oxidation behavior of bare and silicon carbide coated PY carbon-carbon composites were examined. Their measurements were focused on the effect of oxygen partial pressure on the oxidation behavior and the transition temperature between the passive and active oxidation regimes. It was concluded that the oxidation resistance of PY carbon-carbon composites is comparatively more over conventional composites.

CHAPTER-3

CONSTRUCTION OF PROTO-TYPE PY MODEL AND EXPERIMENTAL DETAILS

3.1 Construction Of Proto-Type Preformed Yarn Model

Proto-type preformed yarn **(**PY) machine contained three main units. Two of them, namely the fibre splitting unit and the matrix powder spraying unit were constructed. The sleeve unit already exists in wire industries such as Paras Wire Manufacturing Unit, Kumbalagodu, Bangalore.

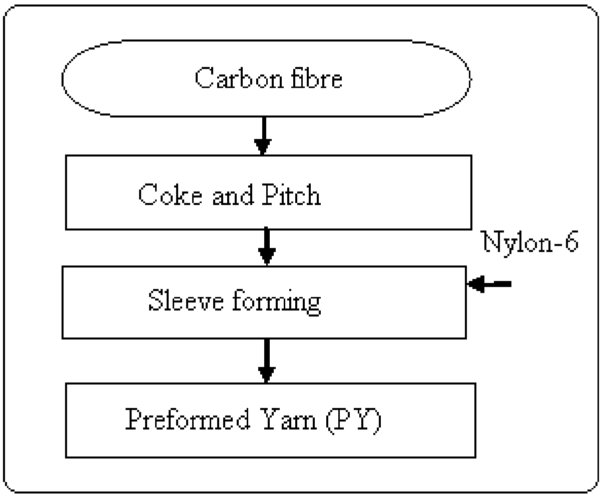


Fig. 3.1 Flow chart to produce PY

Design of these two main units had been done with the help of CATIA V5 software package. A detailed list of part name, material and dimension of parts are given in Table 3.1. The flow chart for the manufacture of PY is shown in Fig. 3.1, and a typical methodology is shown in Fig.3.2. A bobbin of carbon fibre tow is initially split into fine carbon fibres in splitting chamber, these split carbon fibres are passed to the spraying chamber where matrix material (coke + pitch binder) is sprayed over the fine carbon fibres. The sprayed carbon fibre tow is converged and slightly twisted before passing into the sleeving unit containing Nylon-6 where nylon is in molten state in order to keep the matrix material within it. A winding setup is provided to feed and wind-off the prepared preformed yarn.

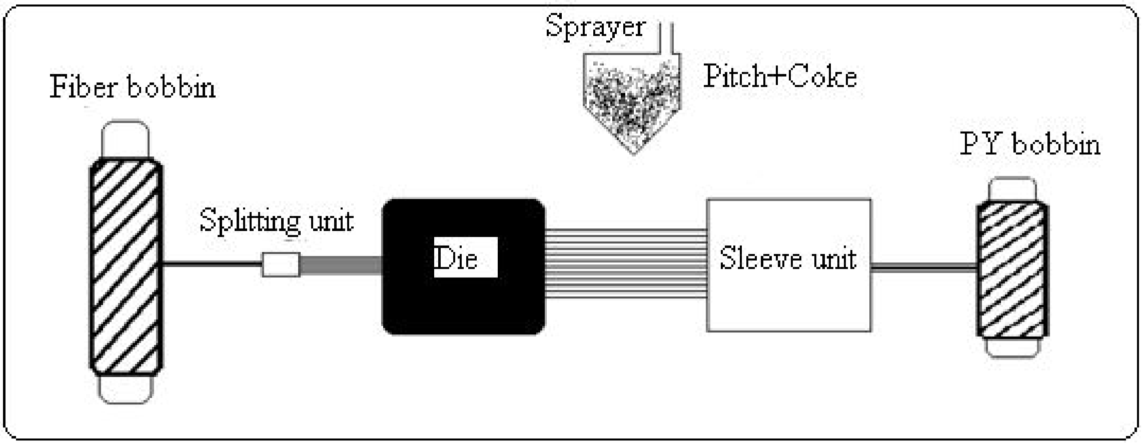


Fig. 3.2 Schematic representation of performed yarn (PY) producing arrangement

It was planned to prepare a machine to split the carbon fibre tow and spray the matrix powder around the fibres. The sleeving was done at Paras Wire Manufacturers Pvt. Ltd., Kumbalagodu, Bangalore, where hot extrusion process was used to manufacture the preformed yarn. The schematic of the hot extrusion process is shown in Fig. 3.3. It was decided to get the final diameter of yarn nearly in the range of 3-5mm. The cross sectional view of yarn is schematically represented in Fig. 2.13.

3.1.1 Major parts of PY making machine model

The parts required for constructing PY making machine model are given in Table 3.1, specifying the part name, material and quantity required. The dimensions were fixed based on the length of PY produced per minute, which is about 20 cm per min.

Table 3.1 List of main parts and materials for proto-type PY machine

Sl. No.	Part Name	Specifications (mm)	Material	Quantity
1	Converging Bracket	26x80x10	MS	1
2	Splitter Holder	50x150x20	MS	2
3	Bobbin	Ǿ100x170	MS	1
4	Matrix Tank	80x40x150	MS	1
5	Bearing House	78x165x30	MS	2
6	Drive Shaft	OD: Ǿ30x300 ID: Ǿ20	MS	1
7	Top Plate	660x660x8	MS	1
8	Matrix Tank Mounting Bracket	260x40x30	MS	1
9	Bush	Ǿ45x30	Nylon	1
10	Direct Splitter	150x103x6	MS	1
11	Parallel Splitter	150x103x6	MS	1
12	Machine Frame	660x660x1100	SS	1
13	Anti Vibration Pad	-	Nitrile	1
14	Fasteners Accessories and other Consumables	-	-	40+
15	Electromagnetic Clutch	-	Fibre-reinforced Al Composite	1
16	Electrical Components AC vibrator motor	1/6 hp	-	2

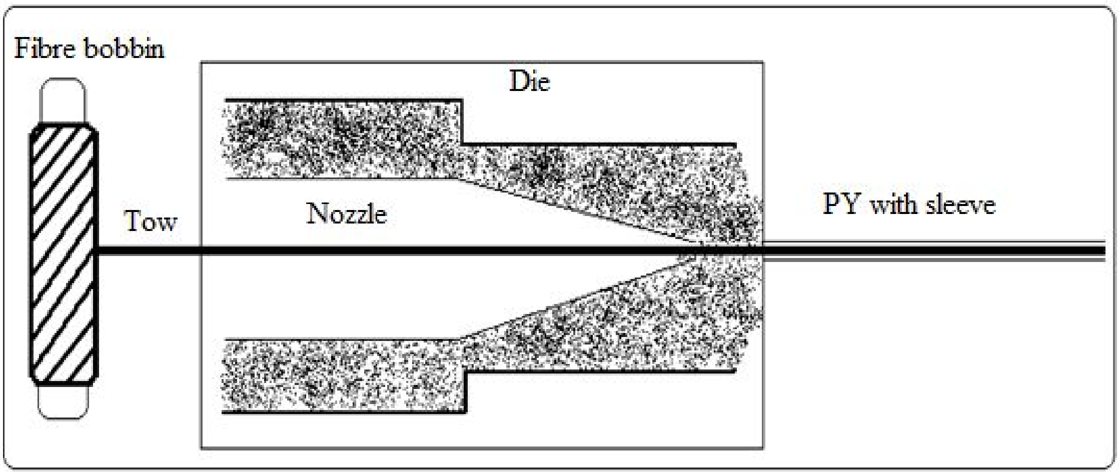


Fig. 3.3 Hot extrusion process to sleeve the split and sprayed carbon fibre tow

3.1.2 Drawings of PY machine elements (all dimensions are in "mm")

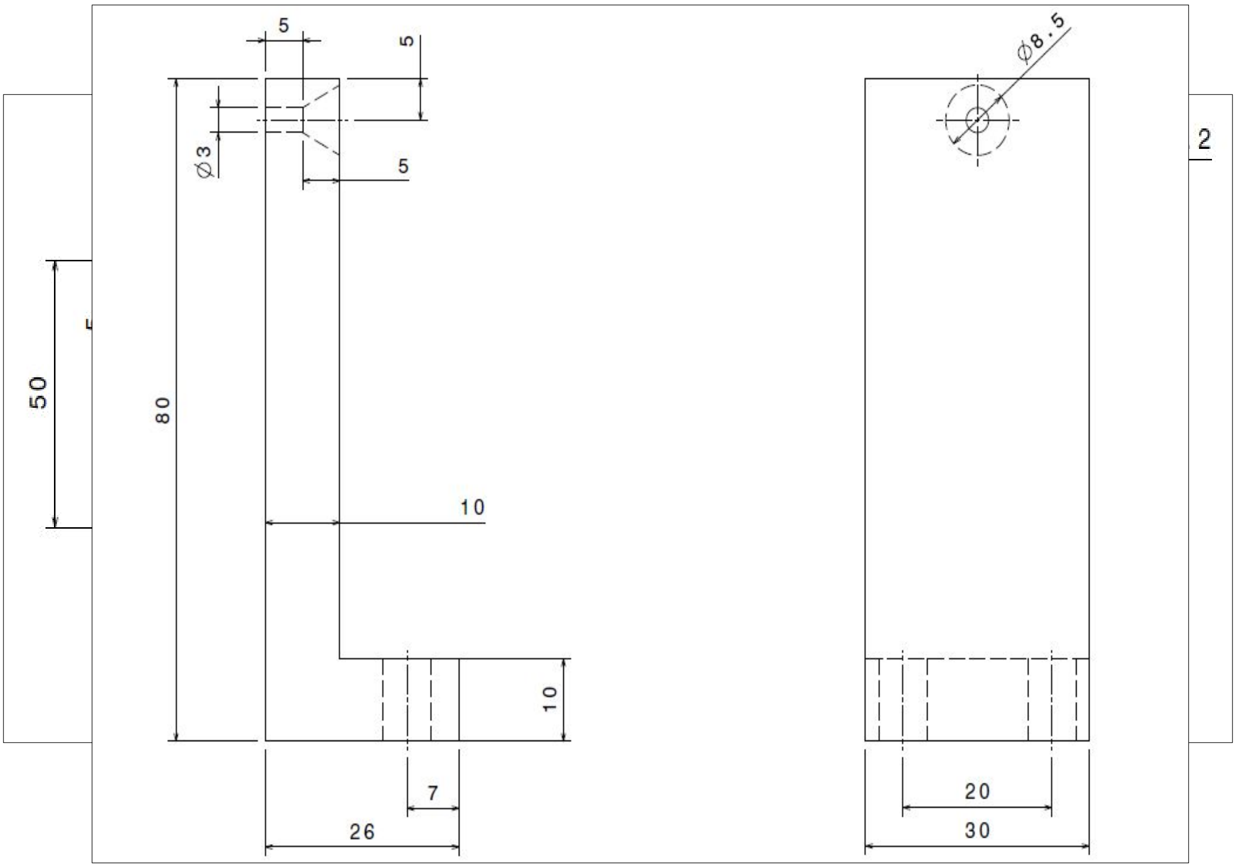
The designed dimensions of the main machine elements are shown in the following drawings and their functions are stated along with drawings. The assembled proto-type PY machine is shown in Fig. 3.4.

Converging Bracket

Material used: MS

Dimensions: 26x80x10 mm

This machine component was provided in order to converge the split fibre and it passes this fiber to the sleeving unit to perform sleeving operation on the pitch and coke sprayed carbon fibres.



Splitter Holder

Material used: MS

Dimensions: 50x150x20 mm

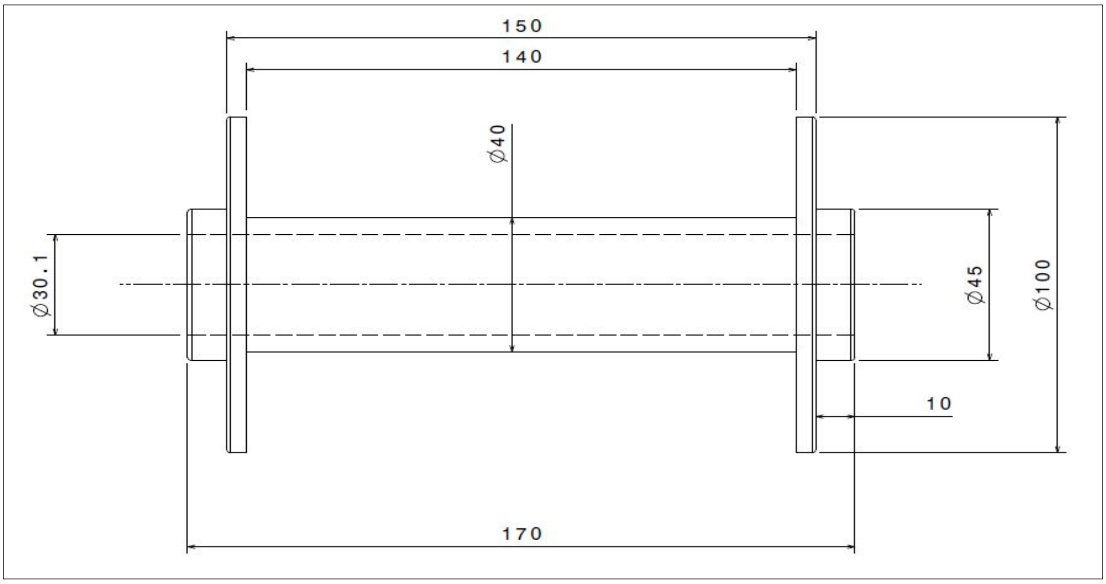
This machine component is used to hold the splitter arrangement at right alignment and vibration free; at the same time it is fixed with the help of three M8 threads.

Bobbin

Material used: MS

Dimensions: Φ100x170 mm

Bobbin contains the refined carbon-fibres; this component is a driven component. When bobbin is made to rotate, the fibres start moving forward and it will be entered into next stage which is splitting unit.

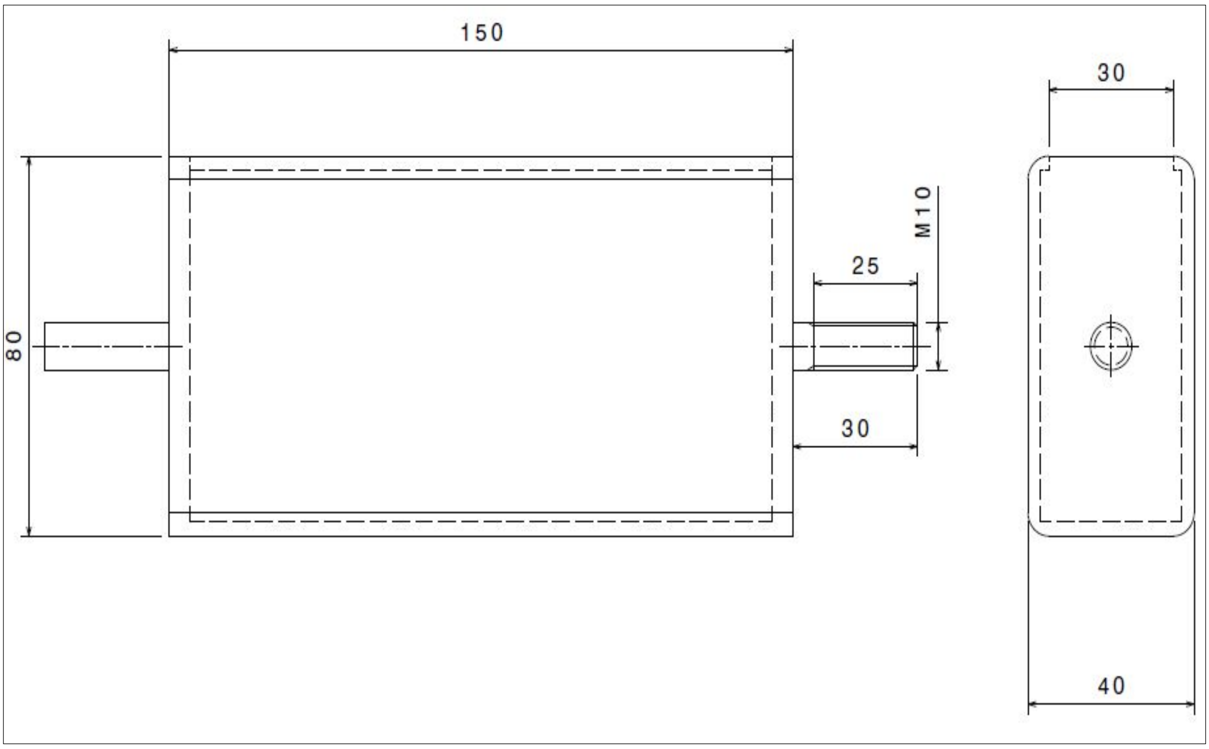


Matrix Tank

Material used: MS

Dimensions: 80x40x150 mm

Matrix tank contains the matrix material which is mixture of coke and pitch. The coke and pitch are ground to a particle size of 4 to 10µm. The right amount of pitch and coke are initially kept in the tank.

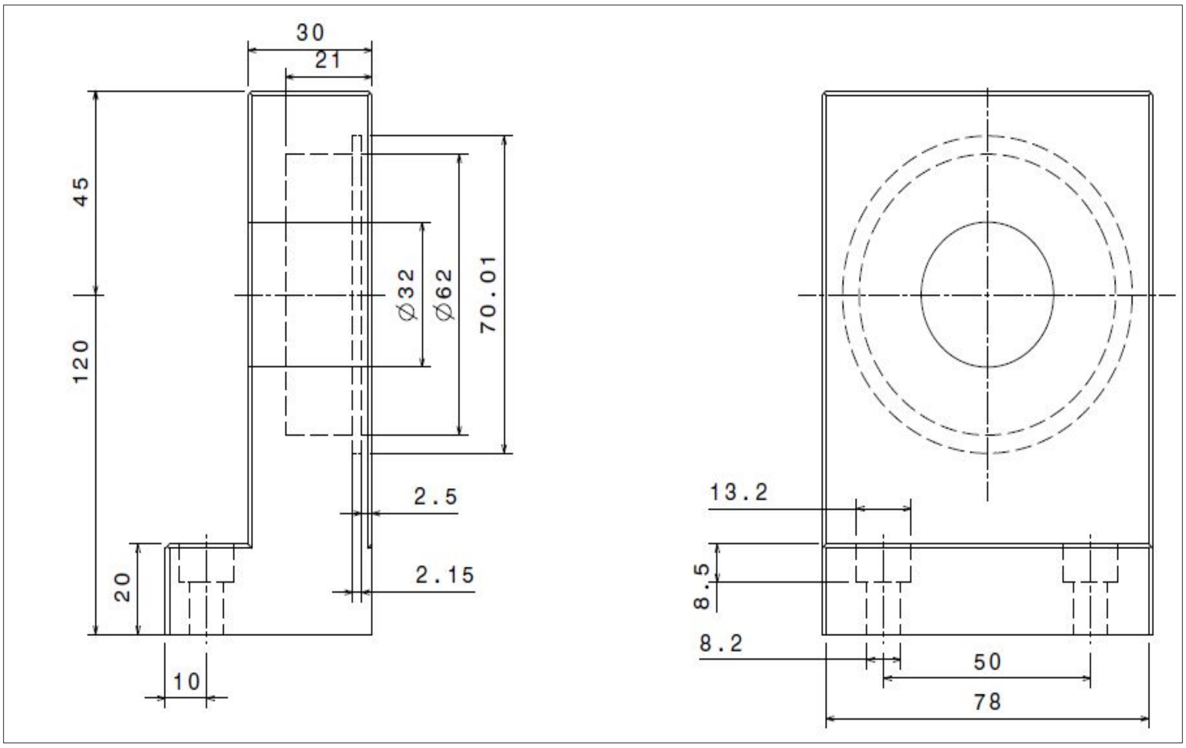


Bearing Housing

Material used: MS

Dimensions: 78x165x30 mm

Bearing housing provides the housing to bobbin, drive shaft and electromagnetic clutch which is held firmly using thread on the bench.



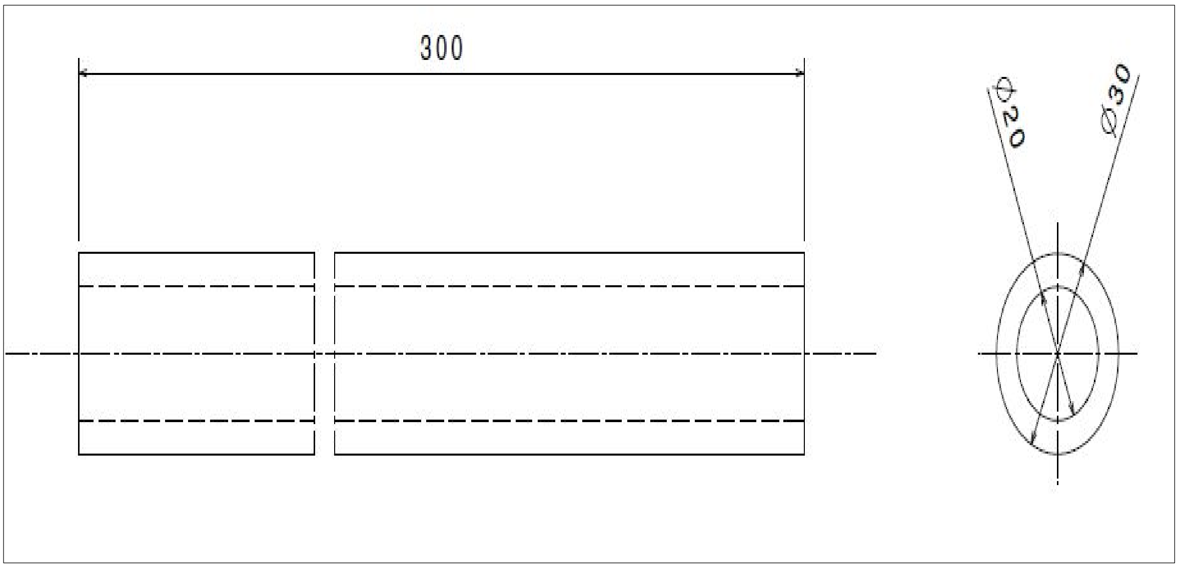
Drive Shaft

Material used: MS

Dimensions: OD: Φ30x300 mm

ID: Φ20x300 mm

This drives the bobbin and electromagnetic clutch. The bobbin is mounted on this drive shaft in order to make slow and steady movement of the fibres and magnetic clutch which splits the fibres and sends to next stage.

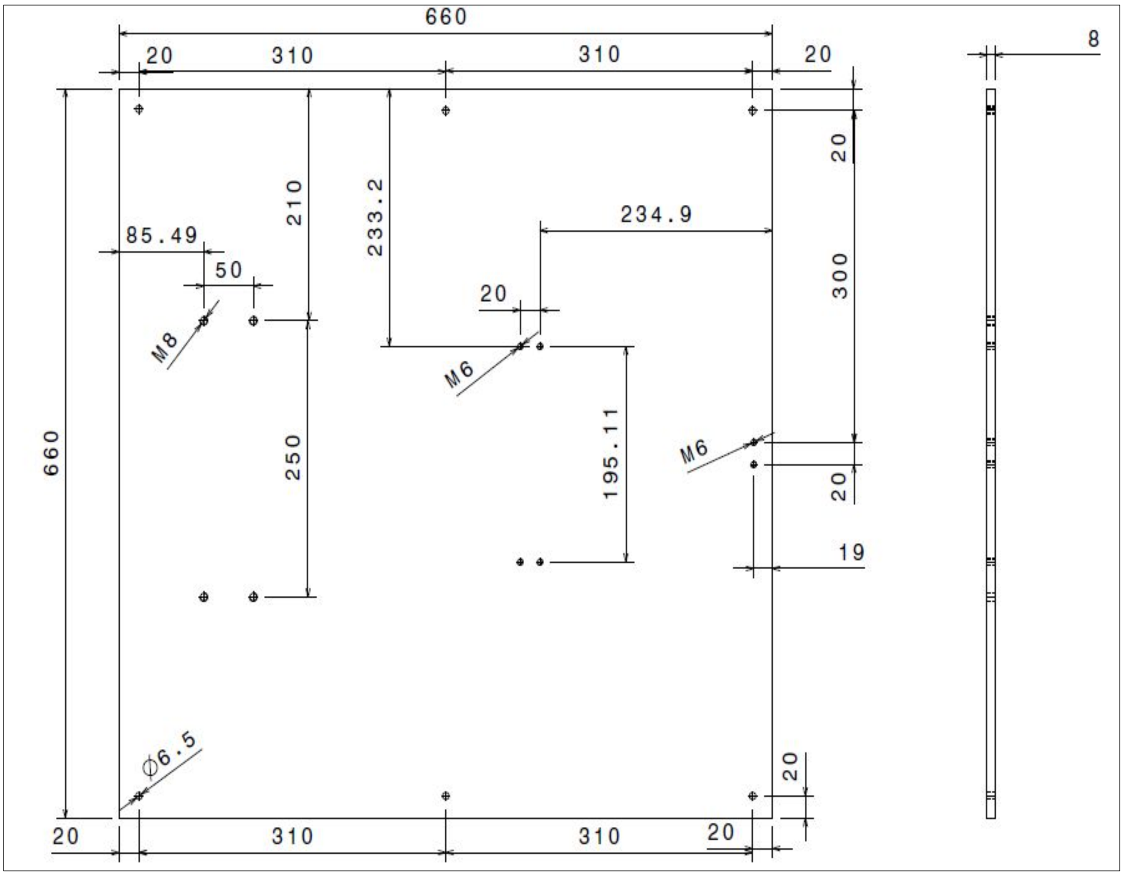


Top Plate

Material used: MS

Dimensions: 660x660 x8 mm

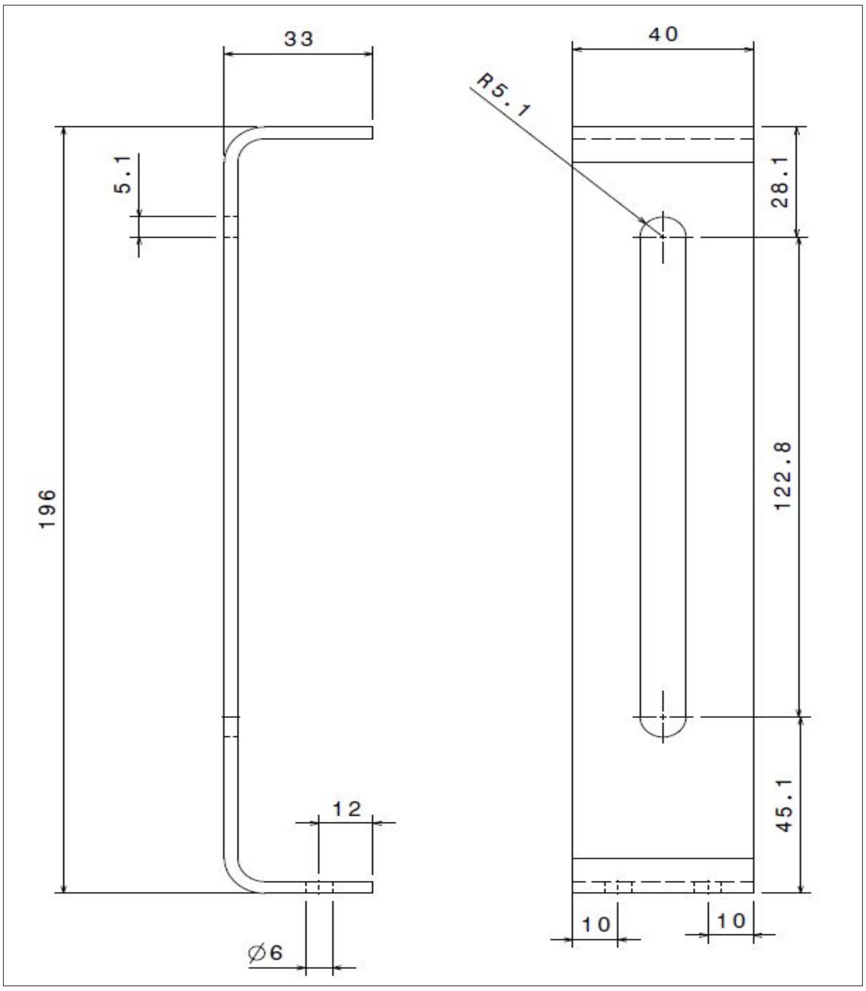
This covers the top portion of hole assembly to protect the inside assembled parts during operation and handling.



Matrix Tank Mounting Bracket

Material used: MS; Dimensions: 260x40x30 mm

This pair of machine component supports the matrix tank and this allows split carbon fiber to pass through it.

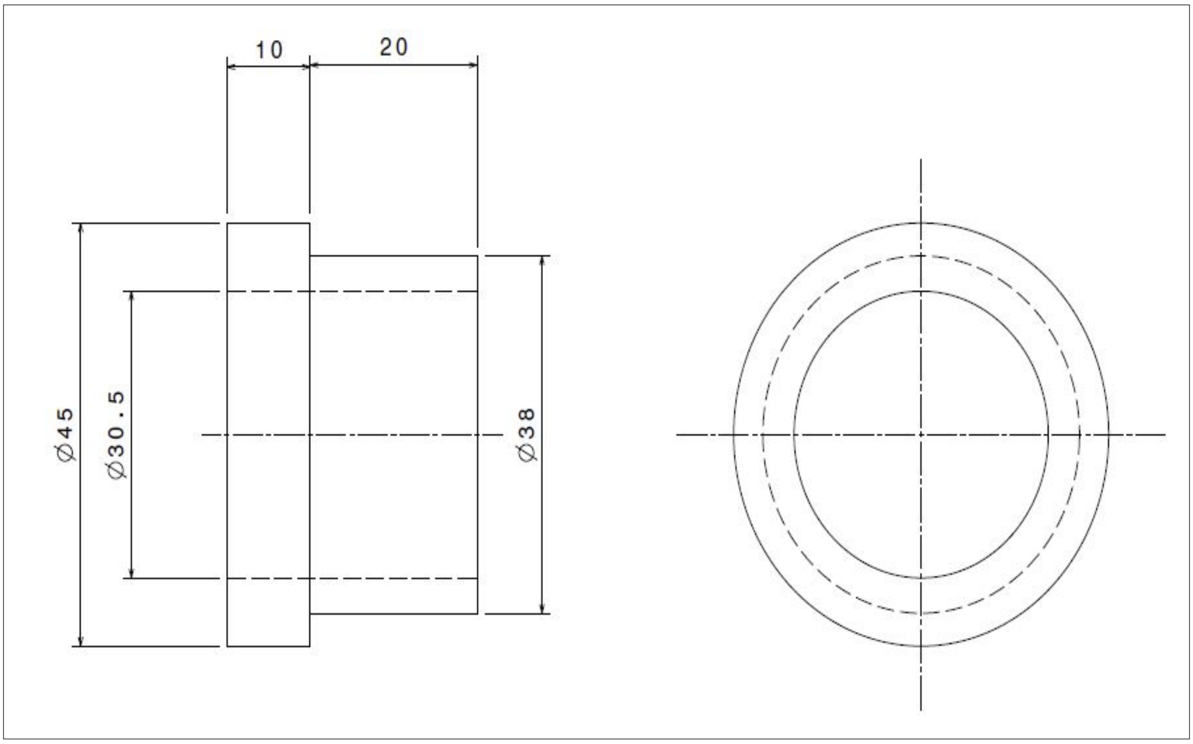


Bush

Material used: Nylon

Dimensions: Φ 45x30 mm

Bush is a machine component which is used to provide the proper coupling between the bearing housing and the drive shaft.

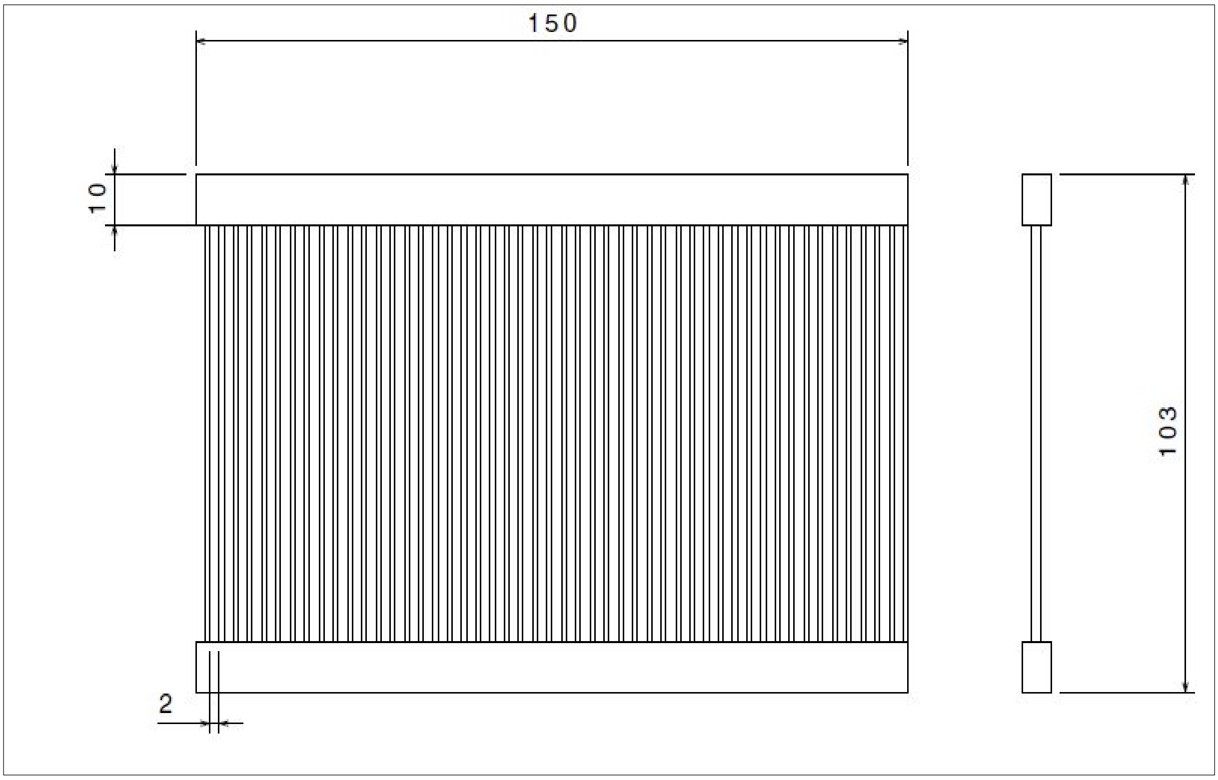


Direct Splitter

Material used: MS

Dimensions: 150x103x6 mm

Direct splitter is used initially to split the fibre bundle into fine sub-tows and to send to parallel splitter.

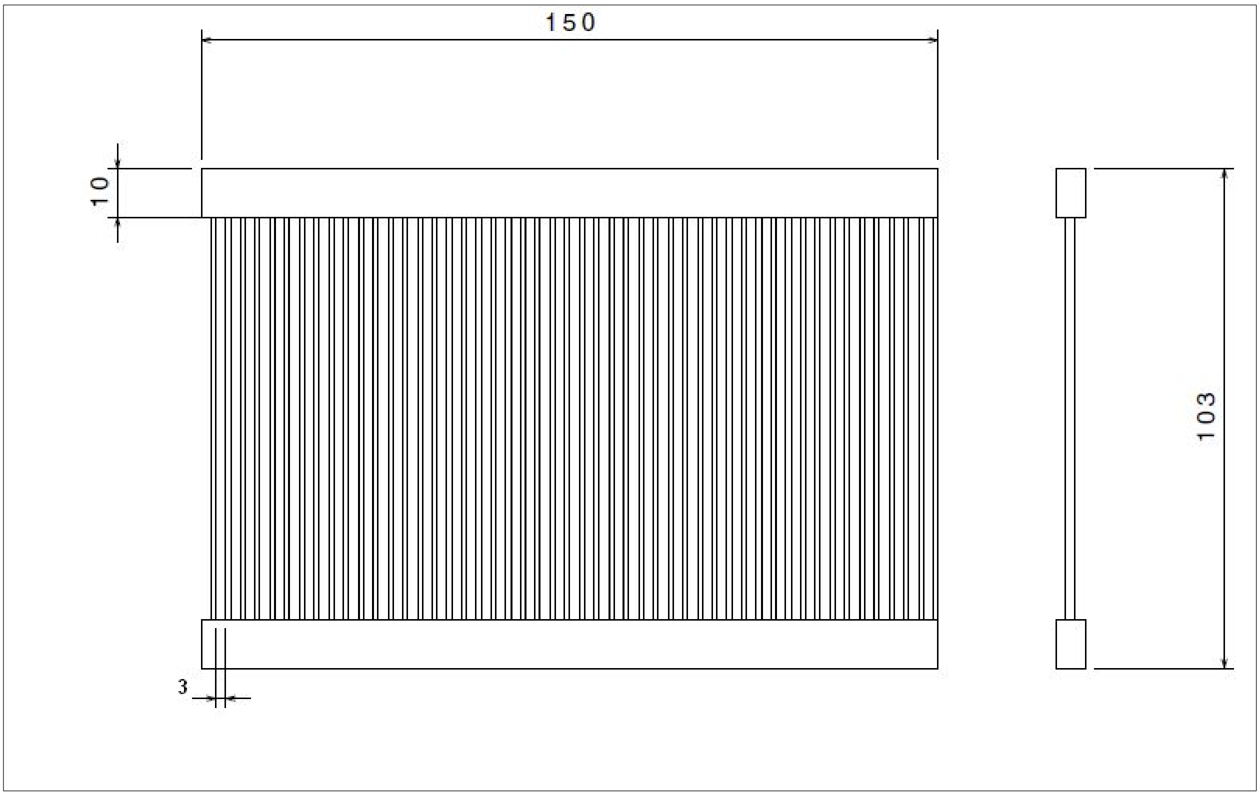


Parallel Splitter

Material used: MS

Dimensions: 150x103x6 mm

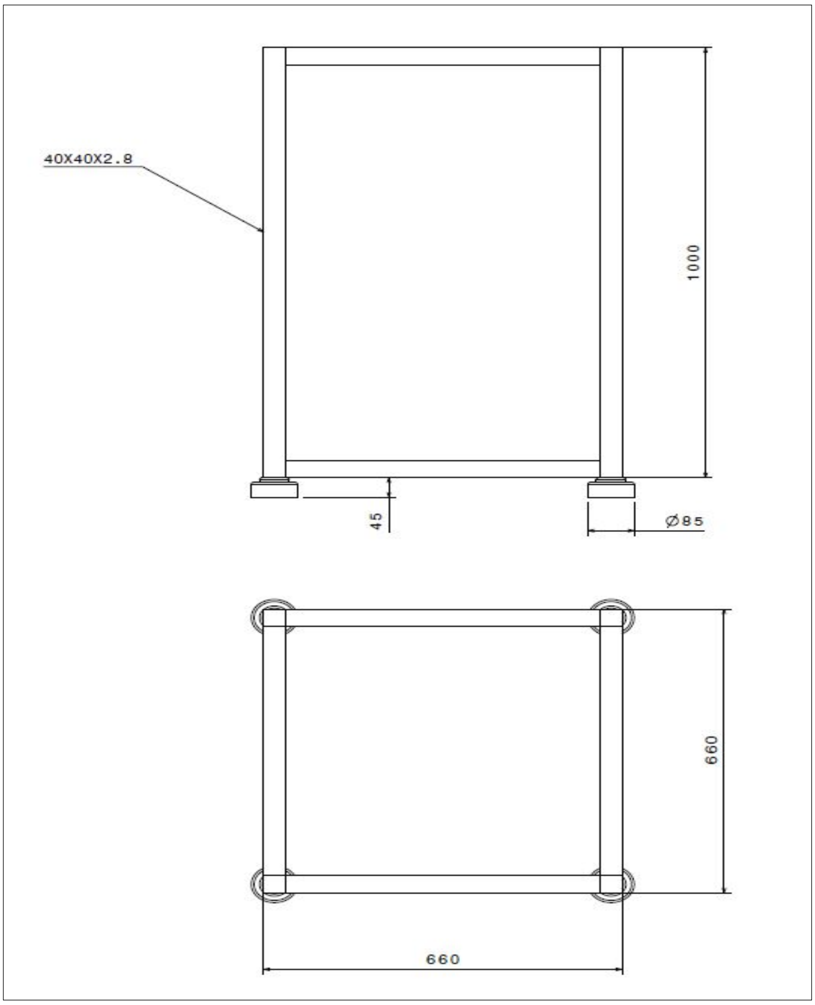
From direct splitter the fibre passes through parallel splitter which is used to direct the split and sprayed sub-tows towards sleeving unit.



Machine Frame

Material used: Structural Steel; Dimensions: 660x660x1100 mm

This machine part is to support whole assembly firm and rigid without vibration while operating.



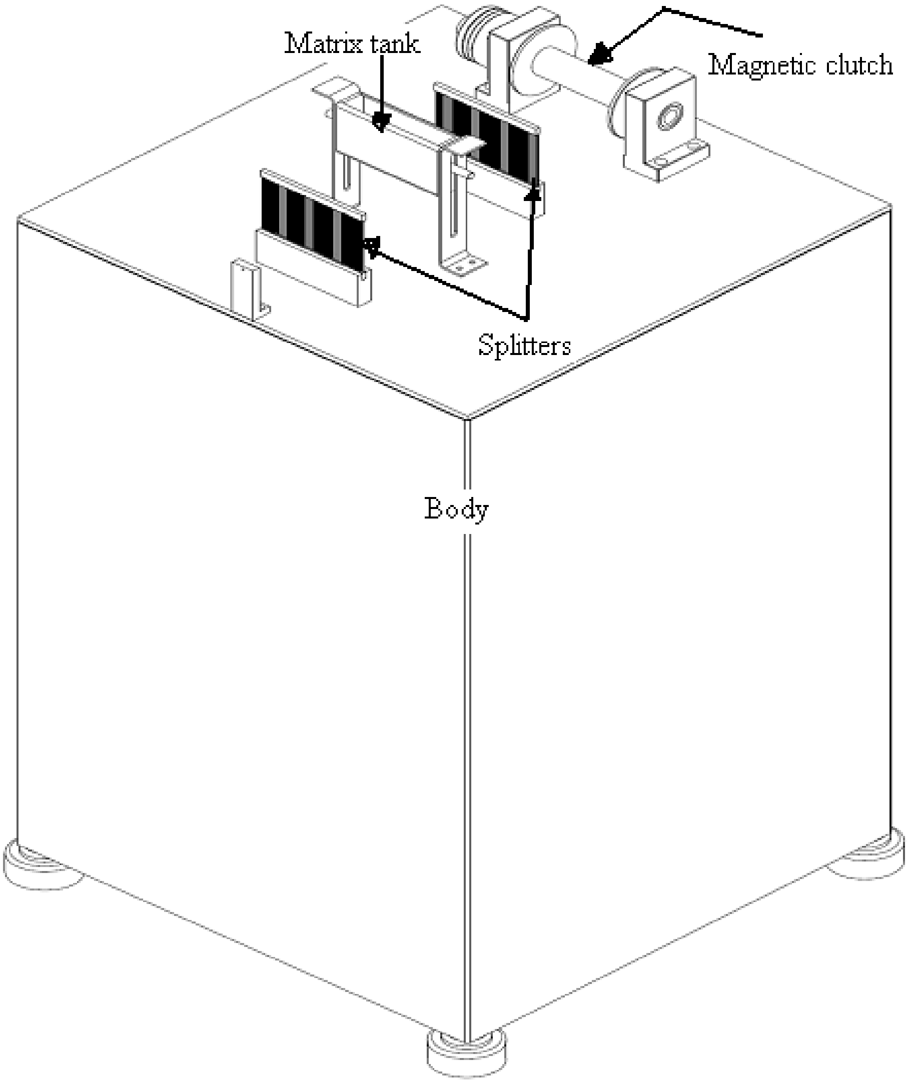


Fig. 3.4 Shows assembled proto type PY making machine model.

3.2 Raw Materials Used For PY Preparation

In order to simplify the manufacturing of C/C composites, it was decided to develop a new production method, using preformed yarns (PY), which contain the carbon fibre bundle as well as the matrix of coke and pitch binder surrounded by Nylon-6, which is the sleeving material. After completion of PY machine, it was planned to prepare at least three types of preformed yarns with three different compositions. Composition I (Sample 1) consisted of 50 wt% carbon fibres, 35 wt% matrix containing 50 wt% coke powders, and 50 wt% binder and 15 wt% thermoplastic sleeve. In composition II (Sample 2) and III (Sample3), the ratio of the binder (pitch) in matrix was increased to 60wt% and 75wt% respectively, fibre content was decreased correspondingly. The preformed yarns were then named as PY- B50, PY- B60 and PY- B75 respectively in accordance with the binder percentage. The materials used in the manufacture of carbon-carbon composites are: 1) Carbon fibre as reinforcement. Table 3.2 gives the properties of carbon fibre which was fabricated by controlled pyrolysis of an organic fibre precursor. Polyacrylonitrile, known as PAN, is the commonly used precursor.

Table 3.2 Properties of carbon fibre used (6k)

Diameter	7 µm
Length of the fibre	5-6 m
Density	1.8g/cc
Tensile strength	3.5GPa
Young’s modulus	230 GPa
Elongation	1.5%

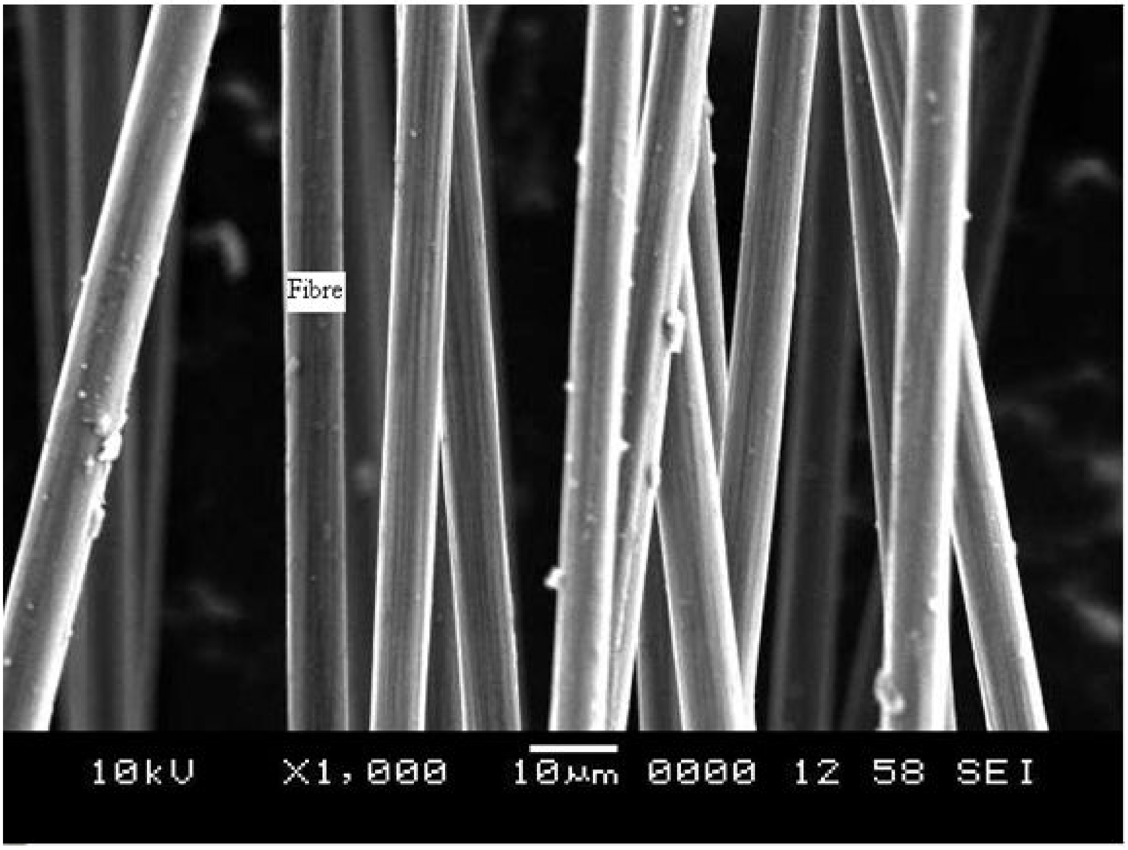


Fig. 3.5 Carbon fibres used for present investigation

Polyacrylonitrile (PAN) carbon fibres with a tow size of 6K were used in the processing of the investigated C/C composite as shown in Fig. 3.5. 2) Pitch and coke as matrix materials, and nylon-6 as a sleeve material, which were supplied by DRDO (Defense Research and Development Organization), Hyderabad, India. Three different samples of C/C composite with different carbon fibre content such as 30wt%, 40wt% and 50wt% weight fractions were prepared using the above-said PY machine. It was decided to prepare three unidirectional C/C composite samples of different weight percent carbon fibre content (30wt%, 40wt% and 50wt %). The compositions of the prepared samples are given in Table 3.3.

Table 3.3 Compositions of preformed yarn

Specimen	Matrix (Wt %)		Fibre (Wt %)	Sleeve (Wt %)
	Pitch and coke together (Wt %)	Pitch fraction in matrix (Wt %)		
Sample1	35	50	50	15
Sample 2	35	60	40	25
Sample 3	35	75	30	35

The chopped yarns were obtained by cutting the endless preformed yarns, which were used to produce carbon fibre reinforced C/C composites. The prepared preformed yarn is shown in Fig. 3.6. These PY enable us easily to fabricate primary work pieces such as unidirectional sheets (UD), cloth sheets, tapes and chopped yarns. Further, it is possible to prepare pipe and tube shaped standby preforms. From these work pieces and preforms, the C/C composites can easily be produced by hot press molding method.

3.2.1 Processing method

The C/C composites were prepared according to the flowchart shown in Fig. 3.7. The materials selected for the present study are UD-reinforced C/C composites. The arrangements of carbon fibres in the investigated composites are shown schematically in Fig. 3.8. The carbon fibres in the investigated C/C composites are unidirectional configuration. In the UD-C/C composite, the carbon fibres in the form of PY are kept in one direction which is perpendicular to hot press direction. The investigated C/C composite was fabricated through hot press molding method at 600^0C subsequently impregnated the carbon fibre preforms by pitch at 250^0C under a pressure of 0.1 MPa for 2-3 hrs. Then the pitch impregnated carbon fibre preforms were subjected to 800°C with hot isostatic pressing at 90-100 MPa pressure for 24 hrs. This was followed by carbonization at 900^0C for 48 hrs and graphitization at 2500^0C for 24 hrs, each under 1 atm. pressure. The process cycle from pitch impregnation to graphitization was repeated 2 to 3 times, until a density of 1.7 g/cc could be obtained in the investigated C/C composites. Die with sample preparation is seen in Fig. 3.9.

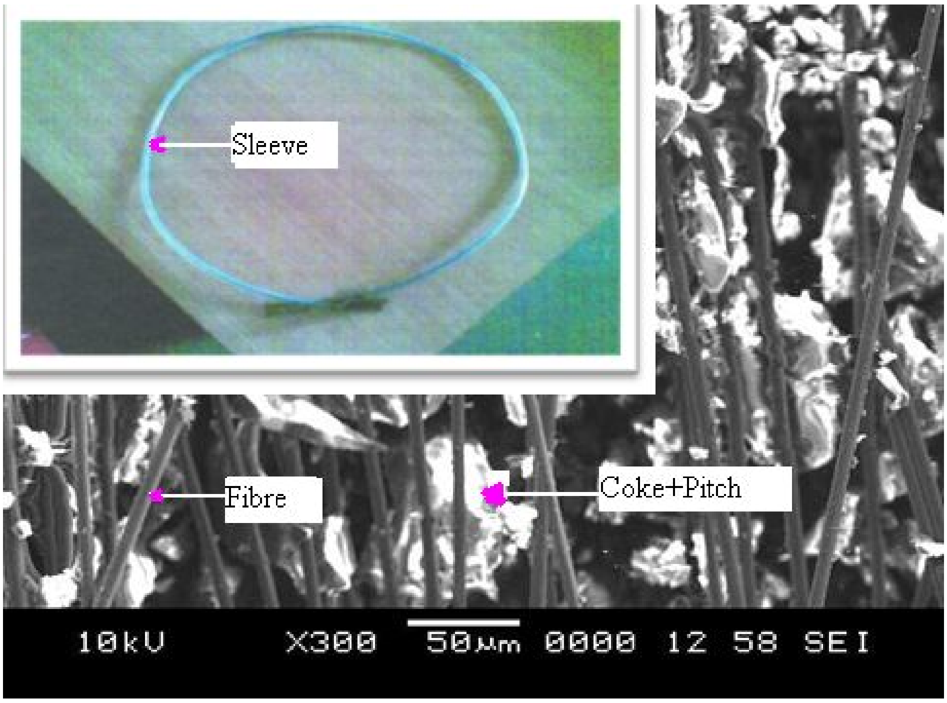


Fig. 3.6 Prepared PY contains carbon fibres, coke, and pitch and nylon sleeve

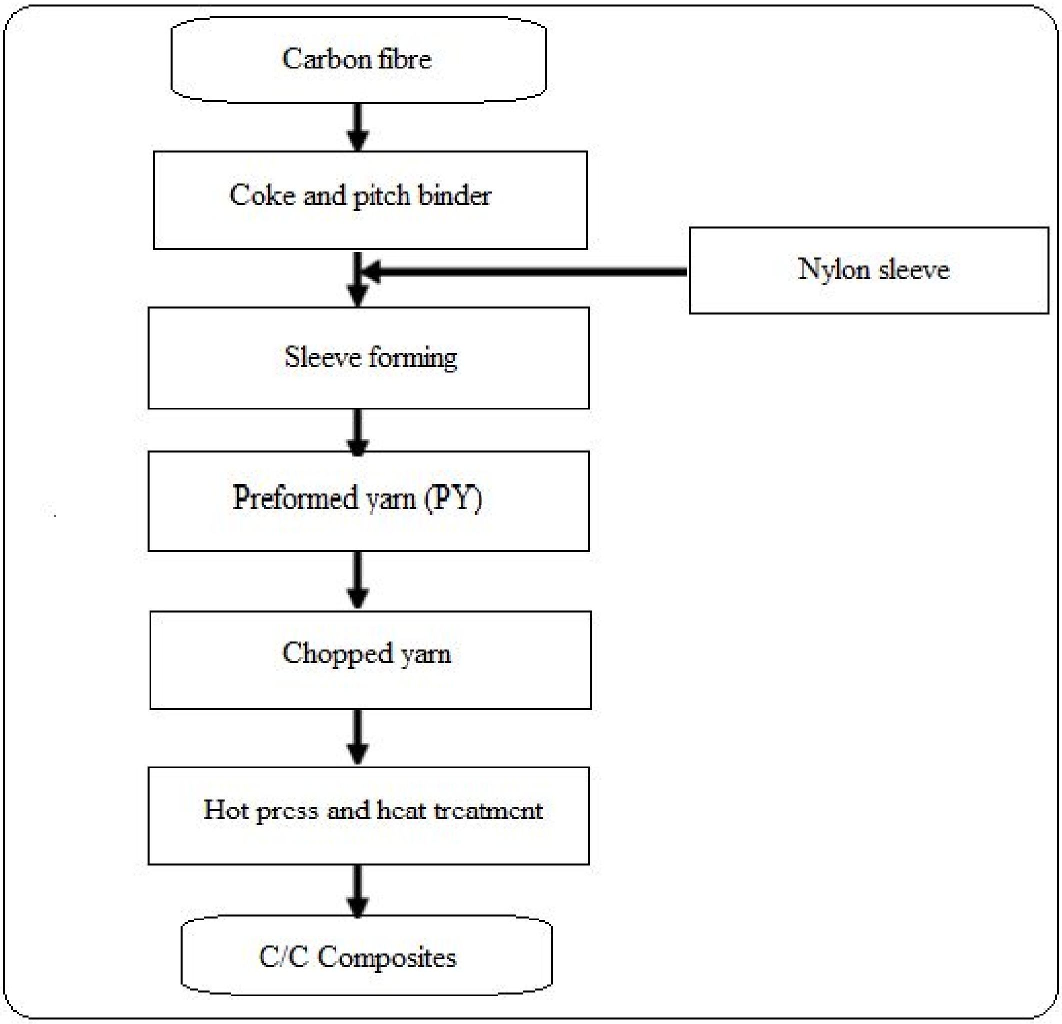


Fig. 3.7 Flow chart for C/C composite preparation

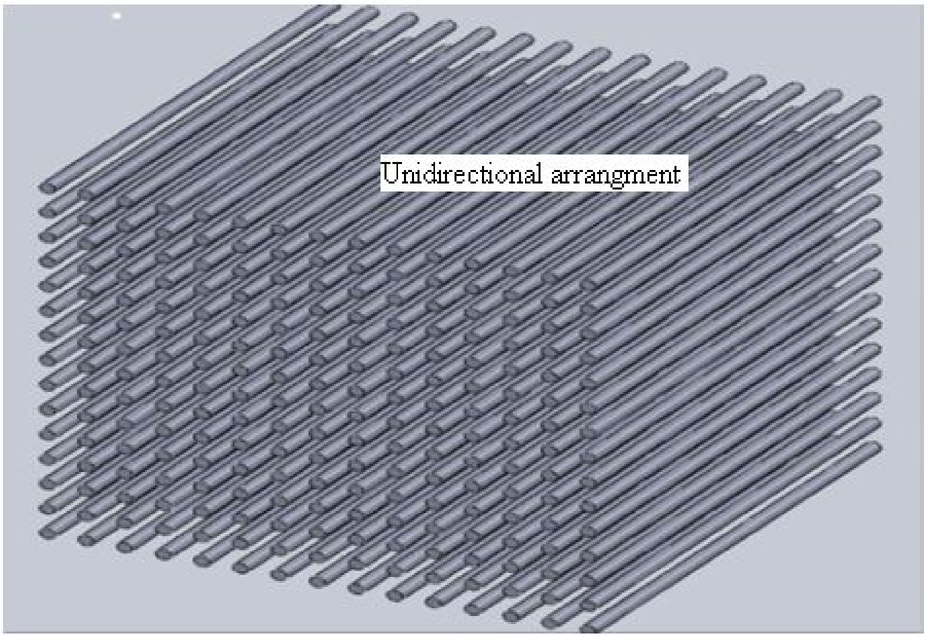


Fig. 3.8 Schematic illustration of the unidirectional C/C composite

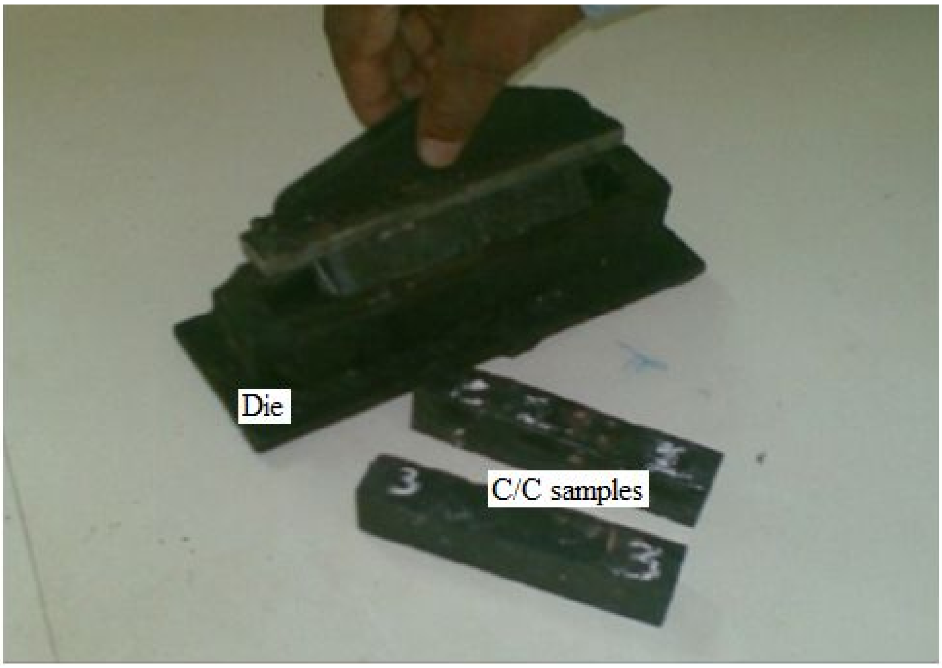


Fig. 3.9 Die with hot pressed samples

3.3 Characterization Of C/C Composites

3.3.1 Scanning electron microscopy (SEM)

The SEM micrographs of C/C composites with 3 different weight fractions of carbon fibers and carbon matrix were obtained using the scanning electron microscope (model-JEOL-JSM-6380). Before observation, the surface of the sample was sputtered with an even thin film of gold powder to improve the conductivity of the surface. The EDAX (energy dispersive analysis of x-ray) was also carried out for the C/C composites.

The images were taken in secondary electron modes. Micrographs were taken at suitable accelerating voltages for the best possible resolution using the secondary electron imaging. The final samples, which were taken for SEM analysis, were heated to 2500^{o}C. For all the samples SEM photographs were taken to study how the fibers were oriented in matrix and to verify the bonding of fibre and matrix at the interface region. The fractured and worn surface morphologies were also observed.

3.3.2 X-RD analyzer

As a non-destructive testing technique, x-ray diffraction is a powerful tool for the analysis of crystalline structure. X-ray has wavelengths comparable to the crystalline lattice constants and thus it can be used for the accurate measurement of lattice parameter, crystallite size etc. X-ray diffraction (X-RD) was utilized to investigate the molecular structure rearrangement in the composites. C/C composites with 3 different weight fractions of carbon fibres and carbon matrix were taken for analysis. X-ray diffraction of the control sample for the required size was prepared and performed using the diffractometer. X-ray diffraction patterns were recorded from 10° to 60° with a computer controlled powder/thin specimen diffractometer (Model: Jeol-Dx-Ge-2p) at a speed of 2.0° per minute using copper radiation, Cu K_{α} (λ=1.542A^{o}), with an accelerating voltage of 40 KV and 30 mA.

3.3.3 Mechanical properties

3.3.3.1 Hardness

Initially the three different weight fractions of C/C composites materials were taken and the test specimens were cut as per the ASTM-E-10 specification and placed in the standard Brinell hardness tester (make: JMT). Brinell hardness test consisted of indenting the composite surface with a 2.5 mm diameter steel ball at a load of 62.5 kg, and the load was applied for a standard time period of 15 seconds and the diameter of the indentation was measured with a low power microscope after removal of the load. The hardness readings were taken for each specimen at different locations to circumvent the possible effects of fibre-segregation. The average of four readings of the diameter of the impression was taken for calculation. The indentation was made on smooth surface free from dirt or scale. The Brinell hardness number (BHN) is expressed as the load P divided by the surface area of the indentation. And the experiment was repeated for all the three samples with different composition.

Rockwell hardness of the samples was also measured using Rockwell hardness tester with ‘F’ scale (1/16`` dia. ball indenter and 60 Kg load). A loading time of 30 seconds (s) was taken for each reading and the average of three readings was recorded.

3.3.3.2 Compression test

Initially the three C/C composite materials, with different weight fractions of the constituents, were taken and the test specimens were cut from the laminates for the required size by using hacksaw frame. Compressive strength was determined at room temperature, for all the three samples according to ASTM-E9-95 standard**,** using a computerized tensile/compression testing machine with a capacity of 20kN. Initially the sample was placed in a compression test fixture and then the machine was made for pre-adjustment procedure and gradually the cross head movement was given with a rate of about 0.05 mm/min. The test was continued until the failure of specimen occurred, and all the data regarding the test were recorded for the further calculation. The procedure was repeated for all the three types of C/C composite samples. Three specimens were tested for each test and the average reading was taken.

3.3.3.3 Flexural properties

Three-point bending tests of C/C composites were carried out using computerized universal testing machine (UNITEK 9450PC, Make: Blue Star) in accordance with ASTM D 790 standard at a deformation rate of 1 mm/min. The specimen for flexural test is shown in Fig. 3.11 (a). The set-up is shown in Fig. 3.10. The flexural strength and flexural modulus values were determined for each composite. The average of three readings was used.

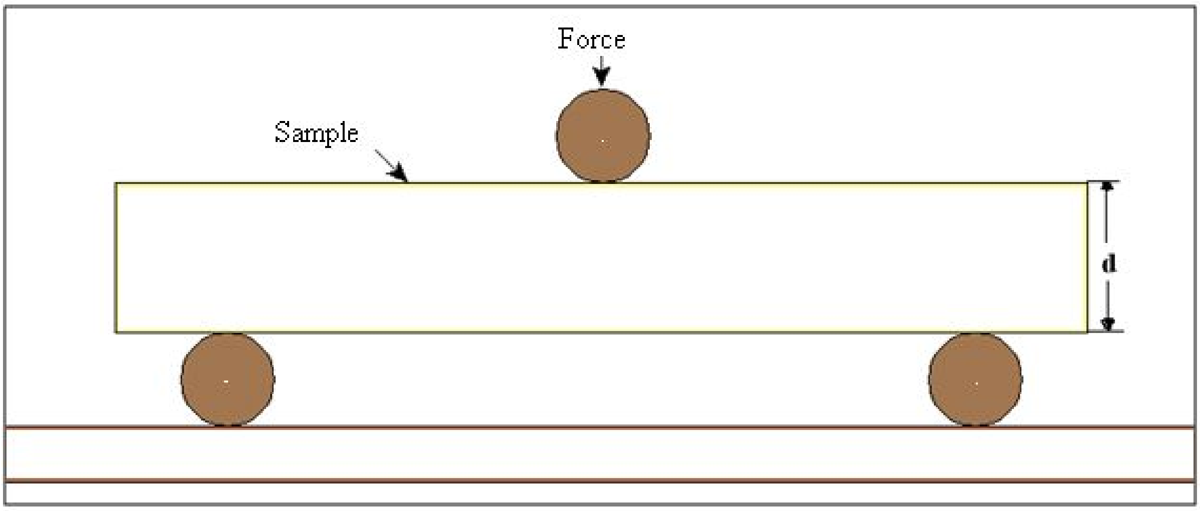


Fig.3.10 Flexural test with three-point loading

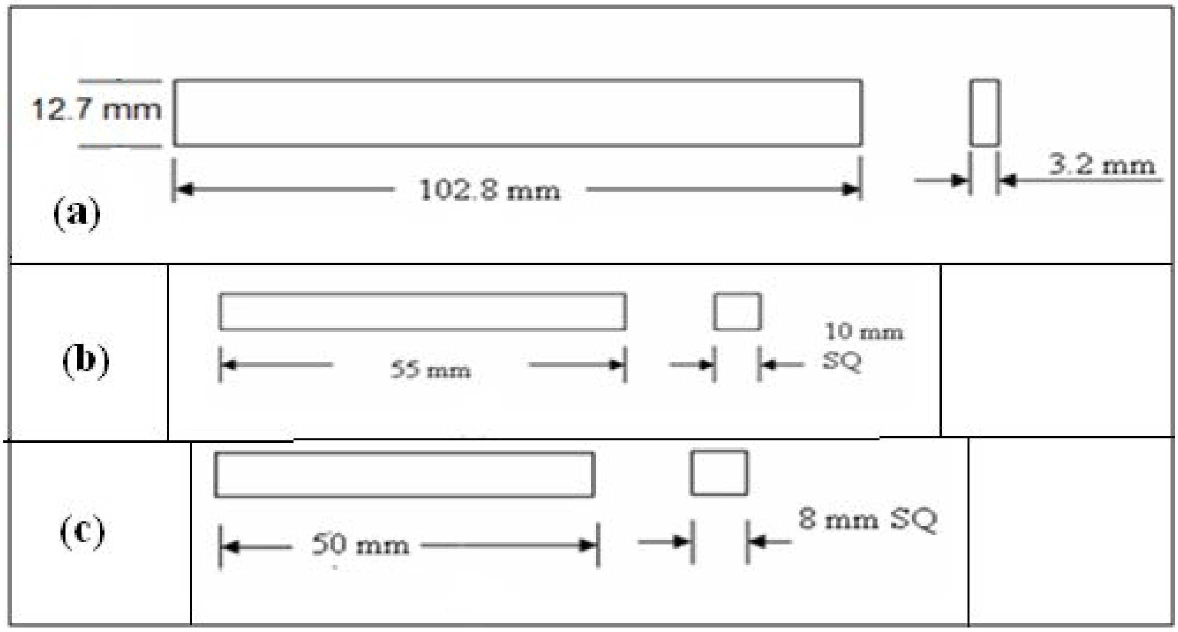


Fig. 3.11 (a) Flexural test specimen, (b) Impact test specimen and (c) Wear test specimen

3.3.3.4 Impact strength

Un-notched Charpy impact tests were conducted on each specimen using impact tester (IT-30, Make: FIE). The average of three readings was reported. The schematic diagram of impact test is shown in Fig. 3.12 and dimensions of specimen are shown in Fig. 3.11 (b). The test consists essentially of a hammer with a given amount of energy striking a notched test piece of fixed dimensions and recording the energy required to fracture the test piece at a specific temperature. The experimenter can also record whether the fracture mode was ductile or brittle.

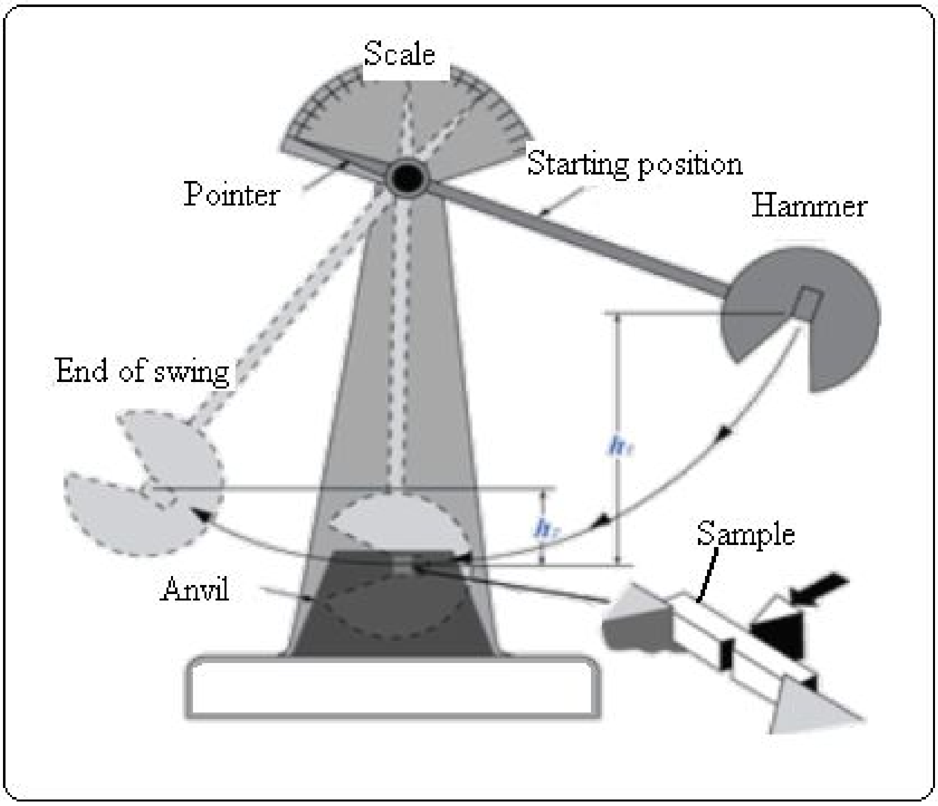
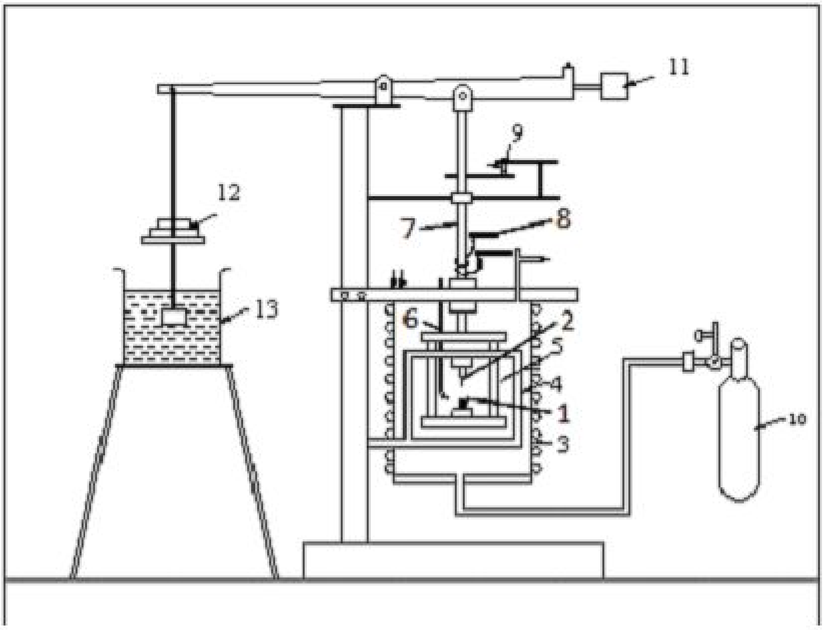


Fig. 3.12 Schematic of basic pendulum function of Charpy test machine

3.3.3.5 Creep test

The creep tests were conducted on the composite samples which were heat treated at 2500^{0}C. These samples were fitted in an impression creep test set up shown schematically in Fig. 3.13.



1. Specimen, 2.Indenter, 3.Furnace, 4.Fixed Frame,

5. Moving frame, 6. Thermocouple, 7.Vertical Rod,

8. Cooling Coil, 9.LVDT, 10.Argon Gas Cylinder,

11. Balancing Weight, 12.Weights, 13. Dashpot system.

Fig. 3.13 Indentation creep test set-up (Schematic)

The test set up consists of a super alloy cage with two rigid frames- one fixed to the base and the other free to move. The former has an indenter of about 1.08 mm diameter and latter has a seat to hold test specimen exactly below the indenter. The setup also consists of lever arm on which a known weight could be placed in a pan attached to its end, and connected by frames which are free to move. Depth of the indentation which is the displacement of the indenter, sensed through the movement of the pull rod, is measured by LVDT with an accuracy of about (±0.001mm) and a timer is used to measure the time in seconds with respect to the displacement in mm. All the experiments were carried out at room and elevated temperatures.

3.3.4 Tribological properties

Unlubricated sliding wear tests as per ASTM G-99 standard were carried out using pin-on-disc set up (TR-201CL, Make: DUCOM). The schematic diagram of pin-on disc machine is shown in Fig. 3.20 and dimensions of specimen are shown in Fig. 3.11 (c). The disc material is made up of steel (diameter 160 mm and 8 mm thickness) having hardness value of 65 HRC. Before each test, the surfaces were polished with metallographic abrasive paper of 600 grade. Then the samples and the counter surface were cleaned in acetone and thoroughly dried. A constant 80 mm track diameter was used throughout the experimental work. The counter surface slid against the C/C composite sample. Sliding was performed in air with the ambient temperature of around 28^0C over a period of 1 hr. at a sliding velocity of 0.83 m/s and a normal pressure of 0.8 MPa. The samples were then cleaned with brush to remove the wear debris and weighed. The test samples were accurately weighed before and after the wear run using electronic balance to determine the wear loss. A load cell was fixed tangential to the lever arm through which the frictional force was measured. The coefficient of friction was calculated by taking into account the normal load and the frictional force. A characteristic value, which describes the wear performance under the chosen conditions for a tribo-system is the specific wear rate W_s which was calculated from the equation:

$$W_s = \frac{\Delta m}{\rho \times F_N \times L} \ (\text{mm}^3/\ \text{Nm}) \tag{3.1}$$

where Δm is the mass loss in grams (g), ρ is the measured density of the composite in (g mm^{-3}), F_N is the normal load in (N), and L is the sliding distance in meters (m). The results reported were the average of three readings.

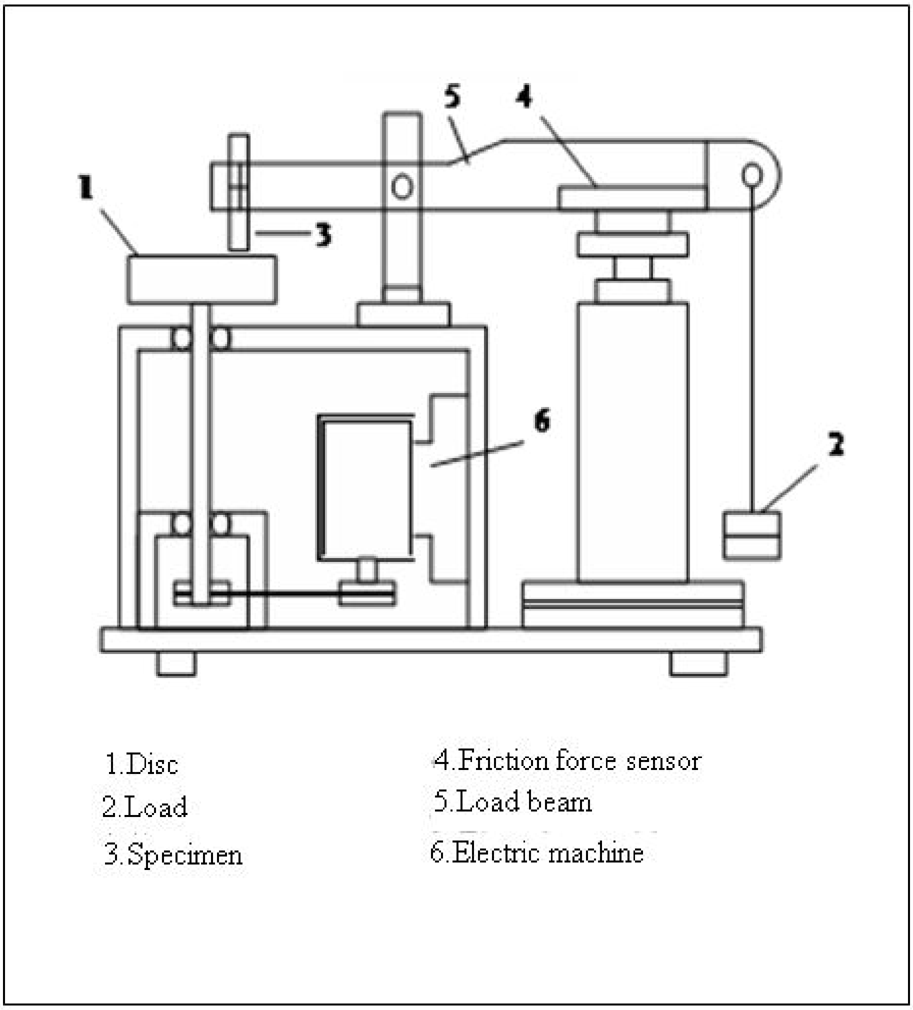


Fig. 3.14 Schematic diagram of pin-on-disc machine

3.3.5 Thermal analysis

Thermogravimetric analysis was conducted in nitrogen gas with a heating rate of 10^0C/min. from ambient to 1000^0C. The TGA samples were cut into small pieces and were machined using a mechanical grinder to maintain sample weights of 20 mg. The experimental setup for oxidation test is shown in Fig. 3.15; it consisted of TGA machine (EXSTAR-6300, Nanotechnology Inc.) with a platinum crucible used for placing the samples and a nitrogen cylinder connected to the furnace inside of which the samples were placed. Nitrogen was used to create an inert atmosphere. The composite samples were placed in the form of powder. The whole system is PID (proposnal integral derivative) controlled and is directly connected to the computer.

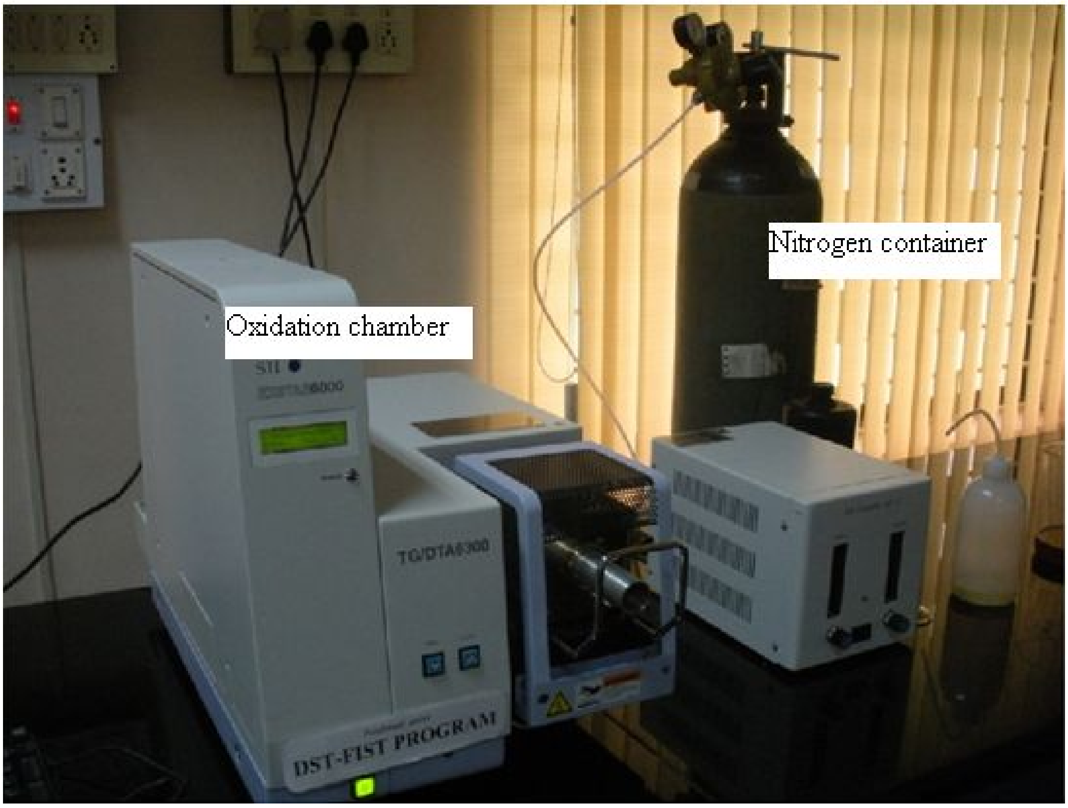


Fig. 3.15 Oxidation experiment set-up

CHAPTER-4

RESULTS AND DISCUSSION

4.1 SEM And EDAX Analysis Of C/C Composites

The SEM photographs were taken to study the orientation and distribution of the fibre in the matrix for all the samples and are shown in micrographs. All the carbon fibres were oriented in unidirectional manner and the diameter of the carbon fibres were measured and it was found to have an average value of about 6.95~7.1 µm as shown in Fig. 4.1. Further, it has been observed that all the carbon fibres were not completely surrounded by the carbonaceous matrix and due to breaking of fibres during hot-pressing some fibres were oriented in other directions.

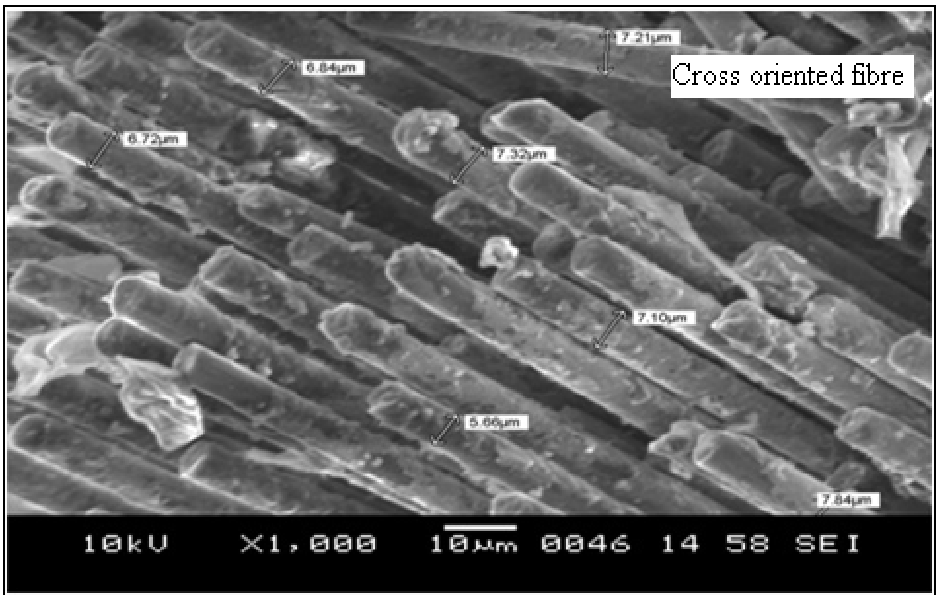


Fig. 4.1 SEM micrographs showing unidirectional orientation-50wt%fibre

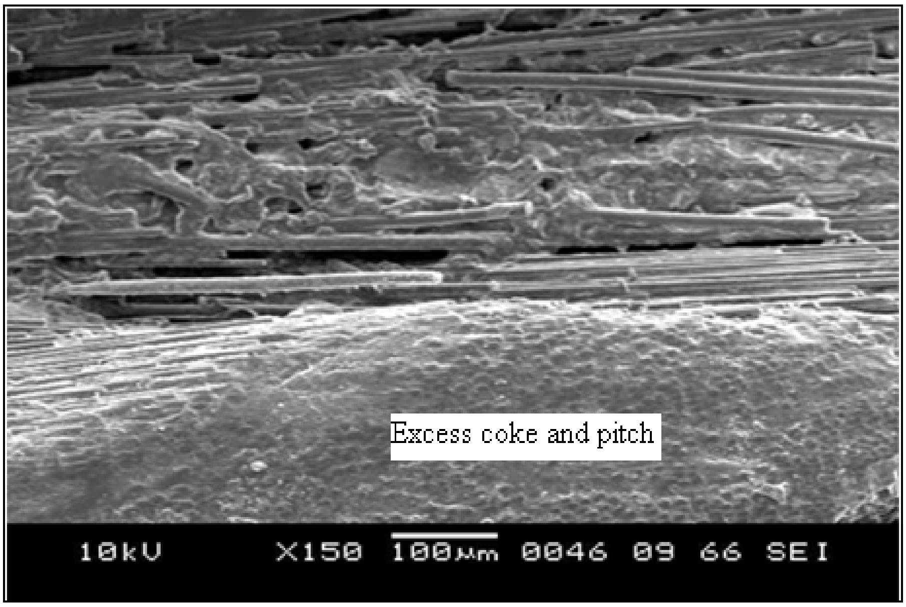


Fig. 4.2 SEM micrographs showing good fibre-matrix bonding-30wt% fibre

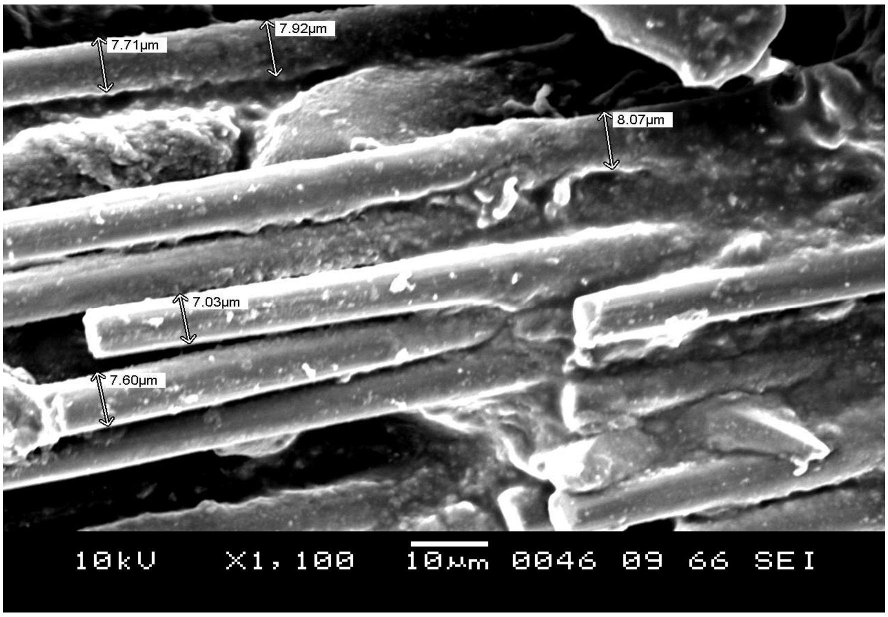


Fig. 4.3 SEM micrographs showing fibre diameter increased due to matrix binder-40wt% fibre

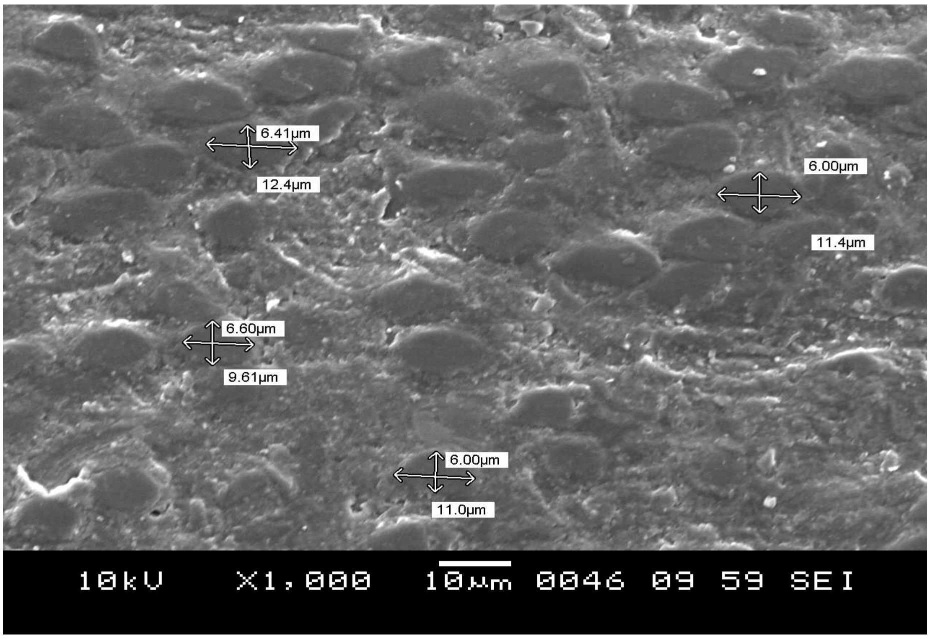
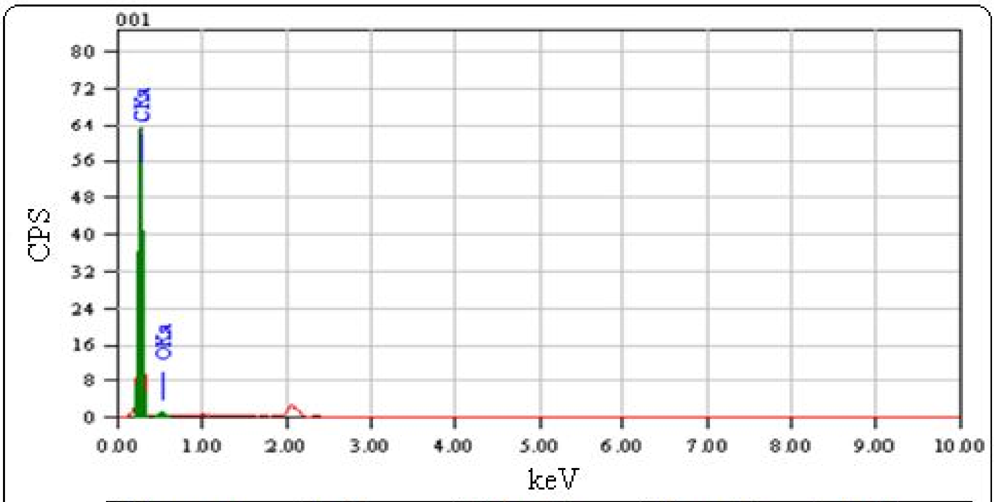


Fig. 4.4 SEM micrographs showing carbon fibres strongly bonded-50wt%fibre

The carbon fibres are oriented in parallel direction in the carbon matrix and binders surround the carbon fibres as shown in Fig. 4.2. The diameter of the carbon fibres was measured and average of 7.76 µm was obtained as shown in Fig. 4.3. The carbon fibres are in perpendicular direction to paper which is closely surrounded by the carbon matrix and the diameter of the carbon fibres were measured and average of 11.1 µm was obtained as shown in Fig. 4.4. Higher fibre content samples got more oval shapes due to hot pressing. Hence, the diameter of carbon fibres in 50 weight% fibre-composite was increased up to 12.4 µm as shown in Fig. 4.4. The SEM analysis also reveals that the interfacial bond between the carbon fibres and carbon matrix was moderate in sample two (40wt% fibre) and three (50%wt fibre). Hence, the values of the properties of the prepared C/C materials had not reached to the expected ones. This is also because of many factors such as insufficient matrix or binder, hot press conditions, number of pitch impregnation cycle and final heat treatment which play an important role in manufacturing C/C composites. Optimization of all these factors would yield better interfacial bond strength and excellent properties.

The energy dispersive X-ray analysis of the C/C composites had been carried out in order to determine the elements present and also it gives the amount of elements presents in C/C composites. All the samples showed same EDAX pattern and percentages of elements present were also the same as shown in Fig. 4.5.

As already explained in the literature, the C/C composites are made up of carbon fibre and carbon matrix. In Fig. 4.5, it is seen that the carbon was the major element constituting 96% of the composites and the remaining 4 % was oxygen. The small amount of oxygen could be because of the oxidation of the surface layer of the composites when the C/C composites were heated above 400°C.



Elements	keV	Mass %	Error %	At %
Carbon	0.277	95.15	0.04	96.31
Oxygen	0.525	4.85	0.40	3.69
Total		100		100

Fig. 4.5 EDAX of C/C composites with elements present

4.2 X-RD Analysis

The XRD patterns of raw materials are shown in Figs. 4.6, 4.7 and 4.8 and comparison with 30wt%, 40wt% and 50wt% of carbon fibre samples are shown in Figs. 4.9, 4.10 and 4.11 respectively. The amorphous carbon present in the raw materials was converted into crystalline graphite in the prepared samples.

The "d" spacing, d (0 0 2) was calculated using Bragg's equation:

$$n\times\lambda = 2\times d\times\sin\theta \text{ -----------(4.1)}$$

And the average crystallite dimension in the c-direction (stacking height of crystallite, L_c) was calculated according to the Scherer equation [L. Dobiaova et. al.2002]

$$t = 0.9\times \lambda / B\times\cos\theta \text{ --------- (4.2)}$$

Where 'n' is an integer, the 'λ' is X-ray wavelength, the 'θ' Bragg angle of the (0 0 2) reflection peak and 'B' the width at half-height of the same diffraction peak (measured in radians). The obtained curve was smoothened. From this, the $2\theta_1$ and $2\theta_2$ values and the 'B' the width at half-height of the diffraction intensity were obtained, and the't' was calculated, which relates the thickness of the crystal to width of the curve. The following results for all the three samples were obtained, and these were found to be almost the same as those shown in Figs. 4.9, 4.10 and 4.11.

- 2θ (deg) = 26.63^o
- Peak intensity (counts) = ranges from 360 to 700
- Half width (deg) = 0.285
- d_{002} spacing (nm) = 0.3346
- t in (nm) = 2.86

By comparing with JCPDF it is concluded that carbon is present in graphite form for all the samples at 26.63^o with a 'd' spacing of 3.3346 A^o. With this we can also conclude that graphitization has been initiated and almost completed in sample 1 but the extent of graphitization is not completed in samples 2 and 3. The glassy phases reveal the presence of some amount of amorphous phase in samples 2 and 3, which could be understood from XRD peak width and serration.

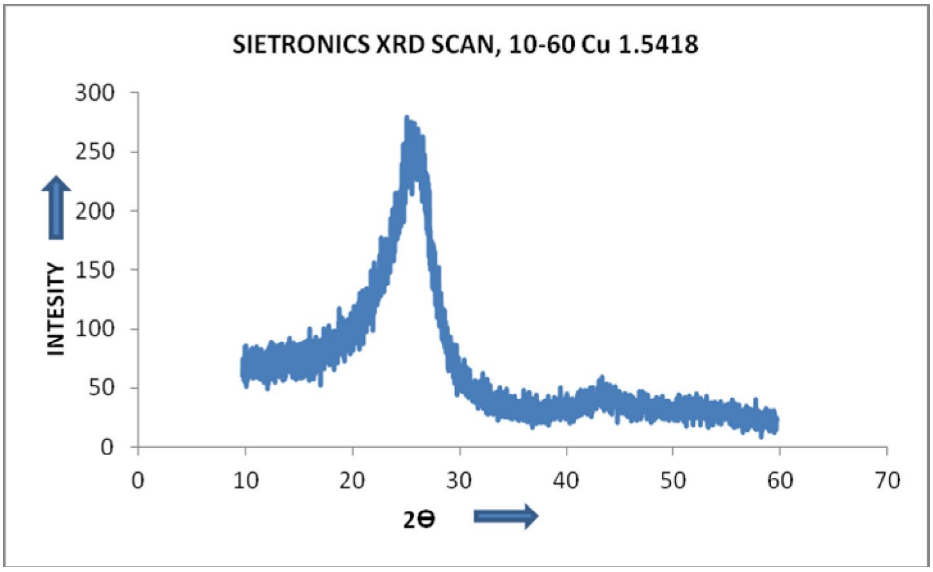


Fig. 4.6 XRD pattern of raw petroleum coke

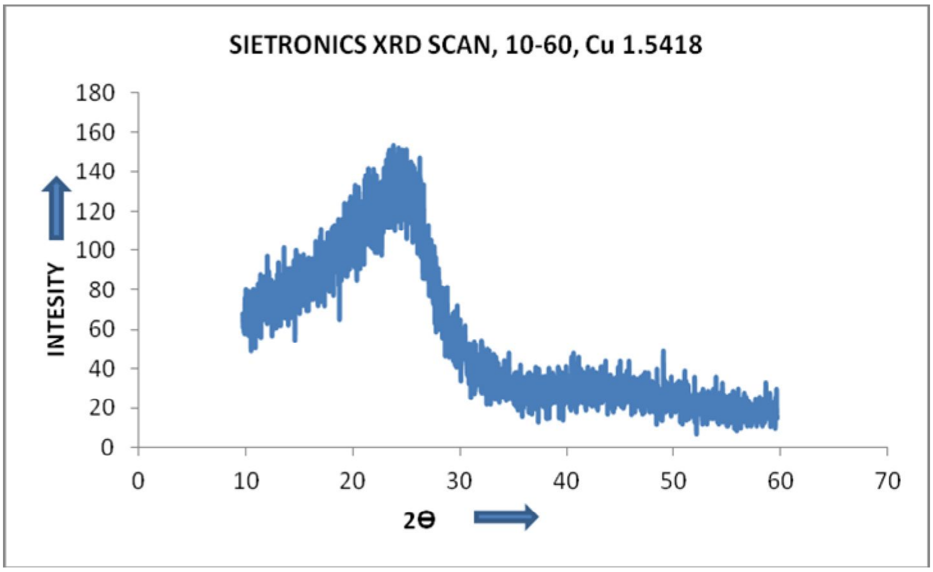


Fig. 4.7 XRD pattern of raw coal tar pitch

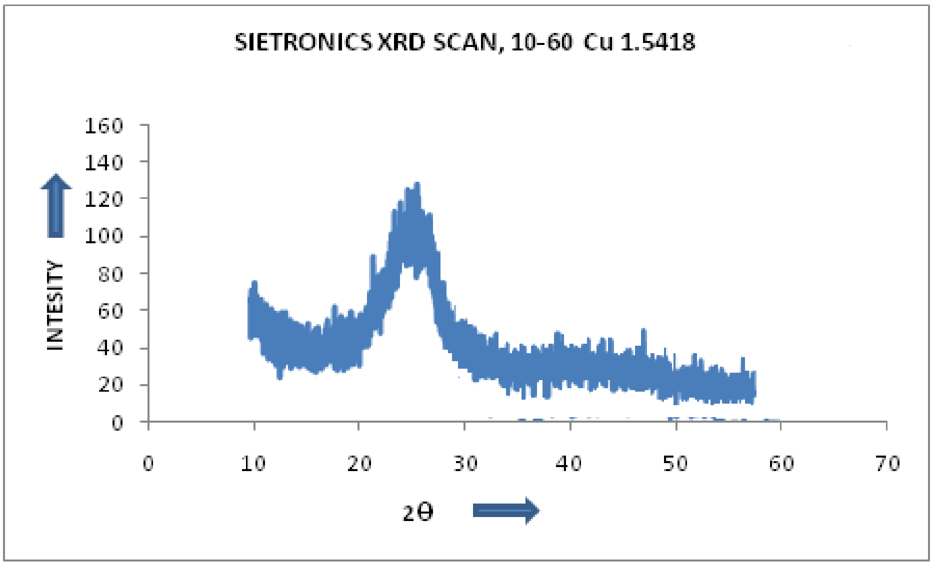


Fig. 4.8 XRD pattern of as received fibre (6K) before processing

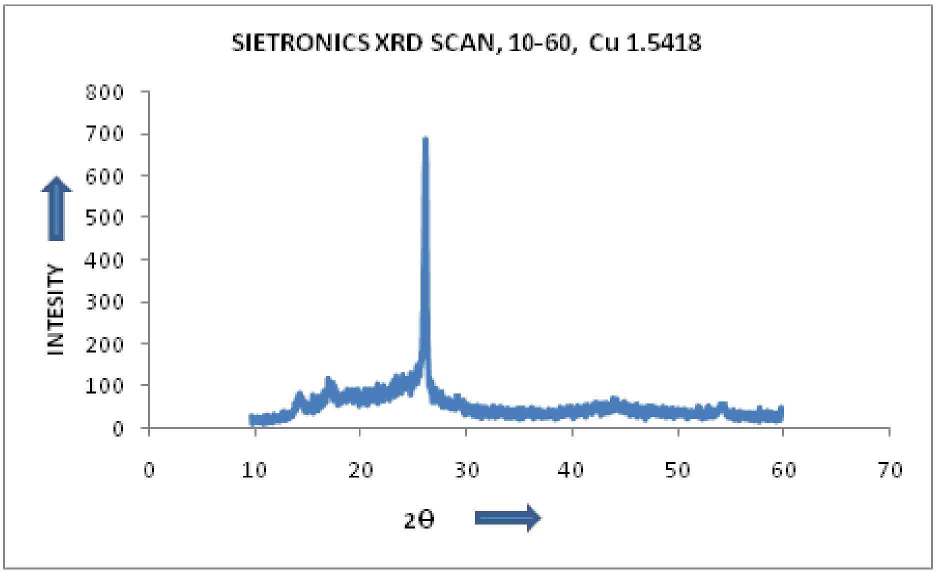


Fig. 4.9 XRD pattern of the sample with 30wt% carbon fibre

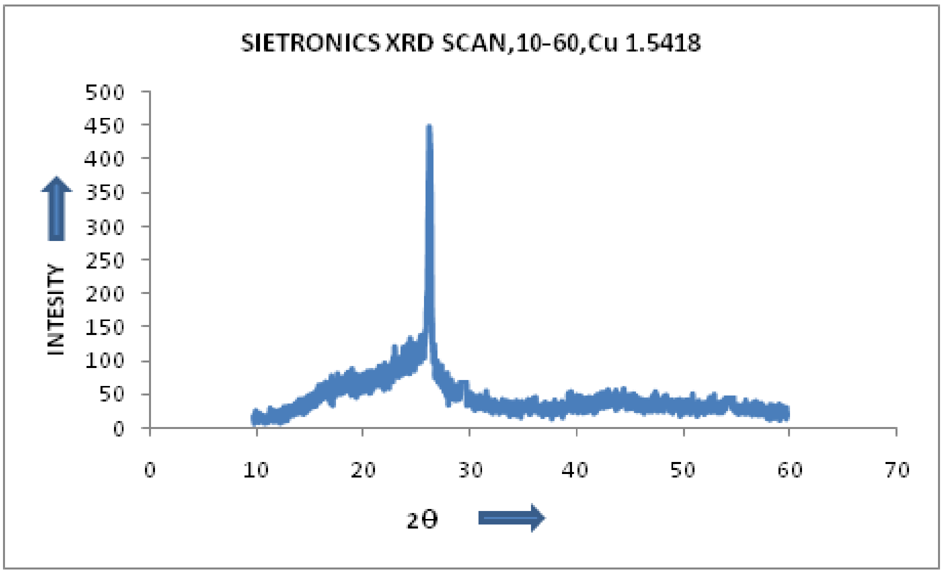


Fig. 4.10 XRD pattern of the sample with 40wt% carbon fibre

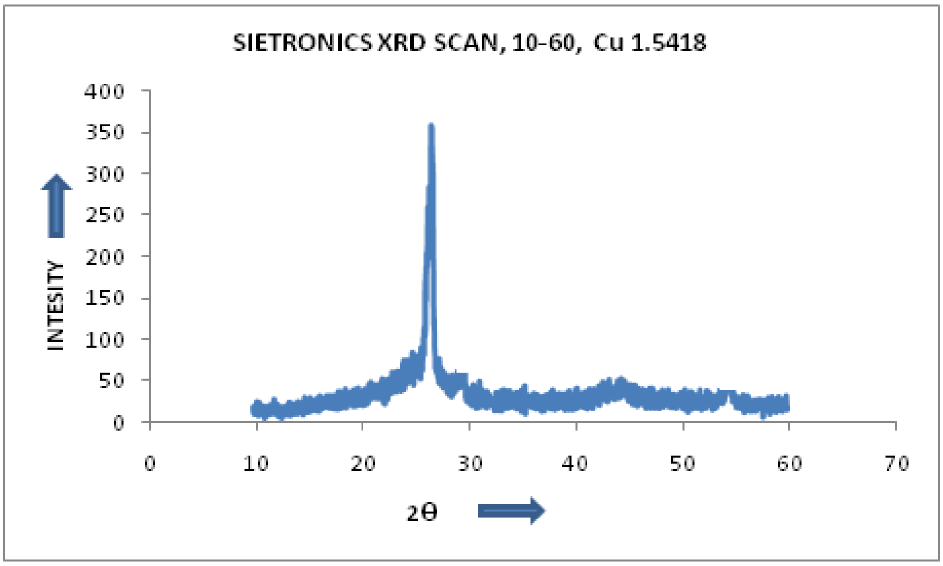


Fig. 4.11 XRD pattern of the sample with 50wt% carbon fibre

4.3 Analysis Of Mechanical Properties

4.3.1 Hardness

The hardness tests were carried out on all the three samples. Both BHN and Rockwell Hardness tests were observed; the following results have been found.

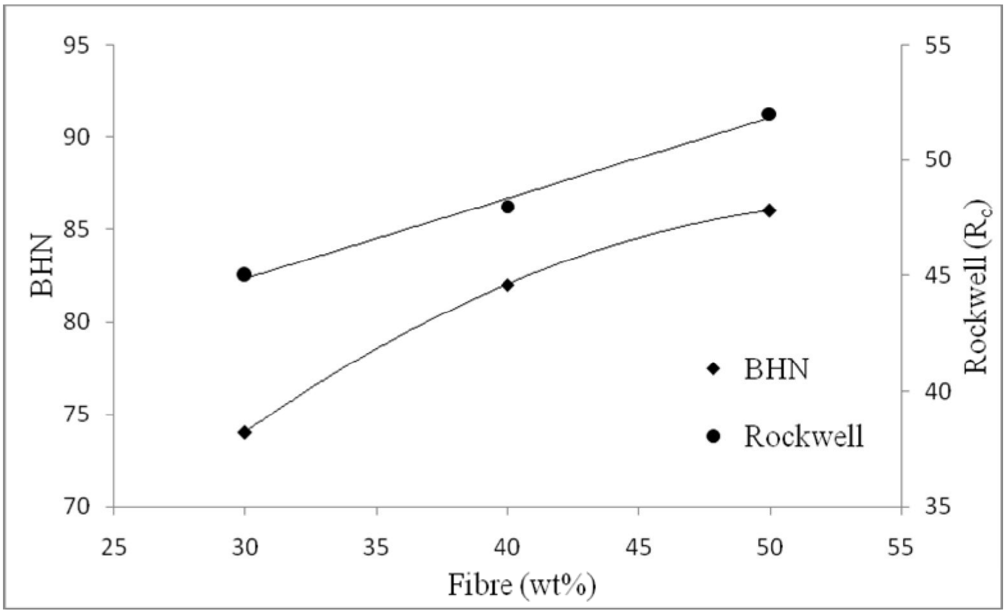


Fig. 4.12 Hardness values of C/C composites as a function of fibre content

The test results reveal that the hardness of the material gradually increased from the value of the sample of 30wt% carbon fibre reinforced composite by 16% in 50wt% of carbon fibre reinforced one. Fibres were uniformly distributed throughout the matrix. The more the fibre content per unit area, the more will be the hardness. Hence higher wt% of carbon fibre reinforcement yields higher hardness. Hardness is resistance to penetration, wear, a measure of flow stress and resistance to cutting and scratching. It is generally known that, when fibres or other types of reinforcement are incorporated into a binder, the presence of the reinforcement can affect the curing process; this can affect the properties of the cured binder [S. Charles et. al.2003]. Fig. 4.12 shows that increasing the carbon fibre content has also improved the hardness value. The hardness values for 30wt%, 40wt%, and 50wt% carbon fibre reinforcement were BHN 74, BHN 82 and BHN 86 respectively, the same trend was observed in Rockwell also.

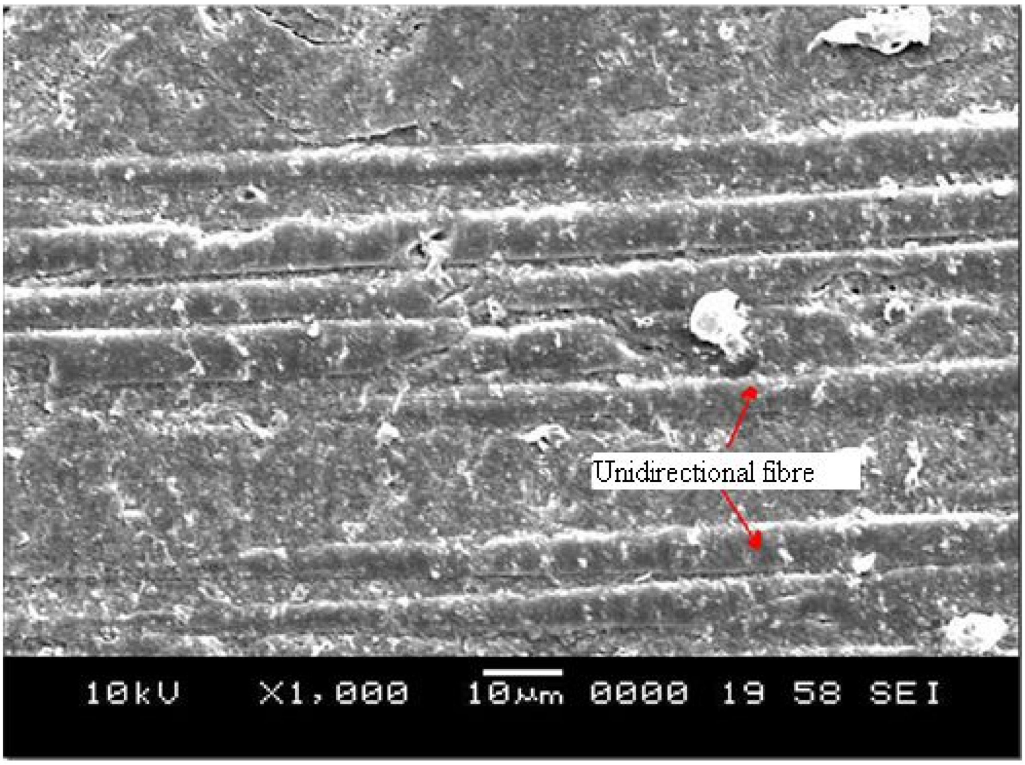


Fig. 4.13 SEM micrographs showing uniform distribution of carbon fibres – 30wt%fibre

This shows that the mixture of carbon fibre and the binder were highly homogeneous and fiber per unit area is more as the weight percentage of fibre increases; this could be observed from SEM micrographs as shown in Fig. 4.13. Increasing the carbon fibre content restricted the percentage of binder available for the cross linking and resulted in a rigid interface, this improves the hardness. By these results we can also conclude that for all the three samples carbonization process have been completed and the graphitization process have been initiated and almost completed as revealed from X-RD data. This shows that increase in the fibre content resulted in increased load carrying capacity of C/C composites. Since fibre is stronger than matrix this behavior is expected. The fibre strengthening effect is expected to be retained even at elevated temperatures and for expected time period, because the fibres are not reactive with the matrix phase [K.H.W.Seah.et al.1997].

4.3.2 Compression strength

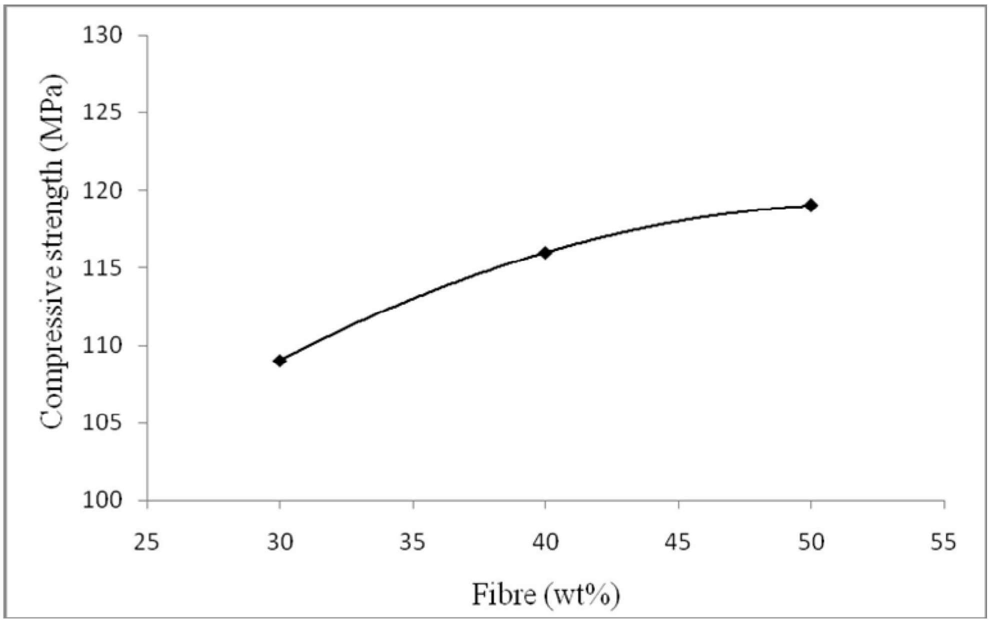


Fig. 4.14 Compressive strength of C/C composites as a function of fibre content

The test results suggest that the increase in the addition of carbon fibre increases the compressive strength, which obviously increases the load bearing capacity. This is because initially the force is taken up by the matrix and later it is transferred to the fibres in fibre-matrix interface region. It is observed that the compressive strength of the composites is increased by about 7 percent as the reinforcement content increases from

30wt% to 40wt%. As the reinforcement content increases to 50wt%, the compression strength of composite is increased from 7 to 10 percent.

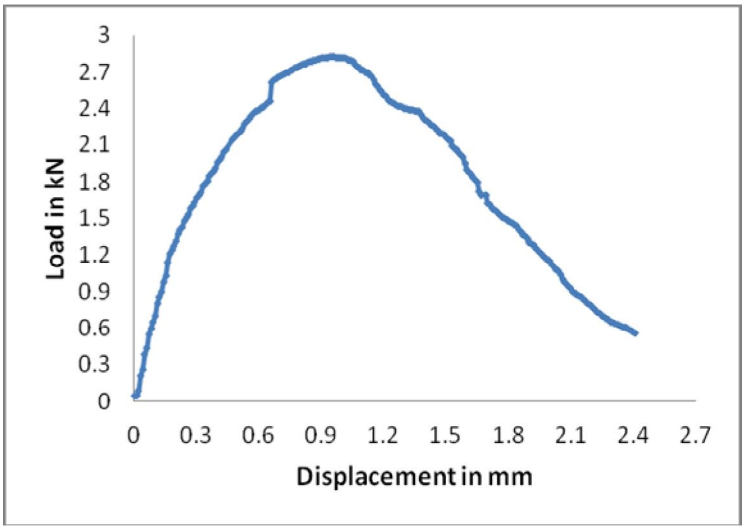


Fig. 4.15 Load versus displacement diagram of 30wt% fibre C/C composite sample

Earlier researchers concluded that increasing the fibre concentration through compaction during processing was the most effective method of improving the strength of the composites. As we go on compressing, the load increases in the C/C composites until a peak value called ultimate load is reached; afterwards load decreases due to the fracture caused by the crack initiation and propagation in the sample. Many papers suggest that the fibre and matrix adhesion or bonding increases as the percentage of carbon fibre increases [Hiroshi Hatta et al. 2005]. The failure mode in the carbon fibres in the composite depends strongly on the adhesion between fibre and matrix. With the low adhesion the filament delaminates from the matrix and the fibres undergo columnar buckling; at 30wt% of carbon fibre intermediate adhesion occurs and the fibres undergo micro buckling along the line of maximum shear stress; and at 40wt% and 50wt% of carbon fibre it was seen that there is a high adhesion contributing to fibre compressive failure occurs in several planes owing to the strong lateral support to the fibre columns.

The composite with higher percentage of fibres, therefore, can be compressively loaded to their maximum capacity without much buckling.

The Fig. 4.14 shows an increase in compressive strength; that is due to the increase in the percentage of carbon fibre which has more compressive strength than matrix. Load vs. displacement diagram for 30wt% fibre reinforced sample is shown in Fig. 4.15.

4.3.3 Flexural strength

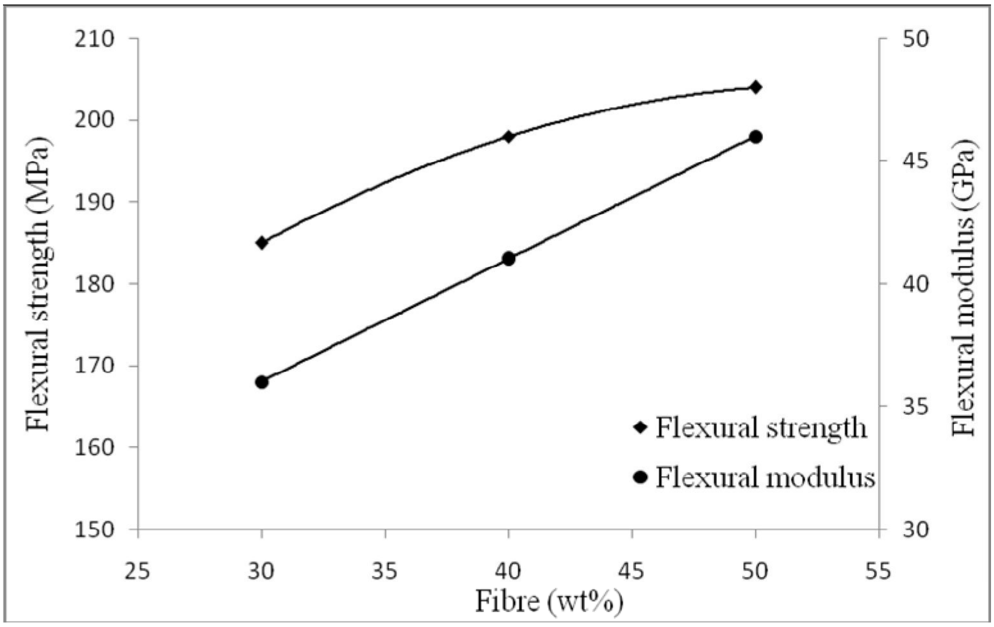


Fig. 4.16 Flexural strength and modulus of C/C composites as a function of fibre content

The effect of fibre on the strength of composite was investigated by flexural tests as a measure of the bonding of fibre to the matrix as well as mechanical strengthening. Fig. 4.16 shows the flexural strength and modulus of C/C composites for 30wt%, 40wt% and 50wt% carbon fibre contents. The flexural strength increased from 184 MPa in the case of 30wt% fibre to 203 MPa for 50wt% fibre composite. The flexural modulus increased from 35GPa in the case of 30wt% fiber to 46GPa for 50wt% fibre composite. For fibre contents between 30wt% and 50wt%, the modulus is increased steadily. It is observed that the flexural strength and flexural modulus of the composites is increased by about 10 and 30 percent respectively as the reinforcement content increases from 30wt% to 50wt%. The increasing of flexural modulus shows that a strong fibre-matrix bond exists for all fibre content samples. This shows that the flexural strength of C/C composites increased sharply to 203 MPa and the modulus increased to 46GPa for 50wt% fibre content samples whereas the values decreased gradually for lower fiber content. It is clear that the flexural properties of C/C composites were enhanced due to the addition carbon fibres. The PY C/C composites exhibited higher flexural strength compared to conventional C/C composite.

Some important characteristics of composites have to be considered to correlate the results obtained. The quality of the interface in composites i.e. the fibre-matrix bonding and the interface stiffness play a very important role in the material's capability to transfer stresses and elastic deformation from the matrix to the fibres [H.H. Kuo. et al. 2005, Lin G. et al. 1993]. This is especially true for composites because, they impart a high proportion of interface. If the fibre-matrix interaction is poor, the fibres are unable to carry any part of the external load. The yield strength of fibre composite can be higher than that of the matrix alone; when there is a good fibre-matrix bonding [Wu C. L. et al. 2002]. A high interfacial stiffness corresponds to a high composite modulus. Hence, the increase in flexural strength and modulus as observed for the C/C composites suggests that stresses are efficiently transferred via the interface. Comparing our results with Across Company results; the modulus value obtained by us is less than that of the Company's i.e. Across C/C had a maximum modulus of 80GPa whereas ours is 46GPa. Hence more number of pitch impregnation cycles and higher synthesizing temperature need to be done with our composite.

Fracture surface of the C/C composites as shown in Fig. 4.17 (a) reveals a brittle behavior characterized by smooth areas. In addition to the smooth surfaces it also shows a micro-rough structure characterized by flow patterns aligned perpendicular to the direction of the main crack propagation, along with hackle like features. Such matrix deformation may occur by an energy-consuming mechanism in C/C composites, and this mechanism has been reported by several authors [Roulin-Moloney A. C. 1986, Yee A. F. et al. 2001]. Other energy consuming mechanisms are fibre de-bonding, and fibre pull-out as shown in Fig. 4.17 (b) and the initiation of secondary cracks at local in homogeneities are indicated by curved region.

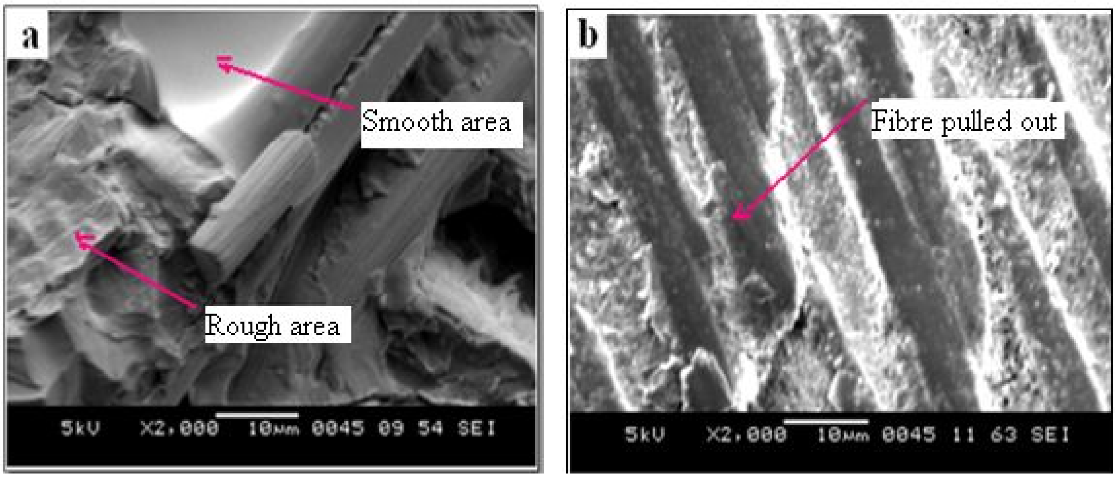


Fig. 4.17 SEM micrographs of (a) fracture under three point bending (b) fibre pull-out region

4.3.4 Impact strength

Charpy impact tests are high speed fracture tests measuring the energy to break a specimen under bending conditions. The specimens are deformed within a short time and therefore exposed to high strain rates. In the present work, the un-notched specimens were selected for impact testing, keeping in mind that the notches may induce stress concentration at their vicinity and the impact strength of the C/C composites may be further reduced.

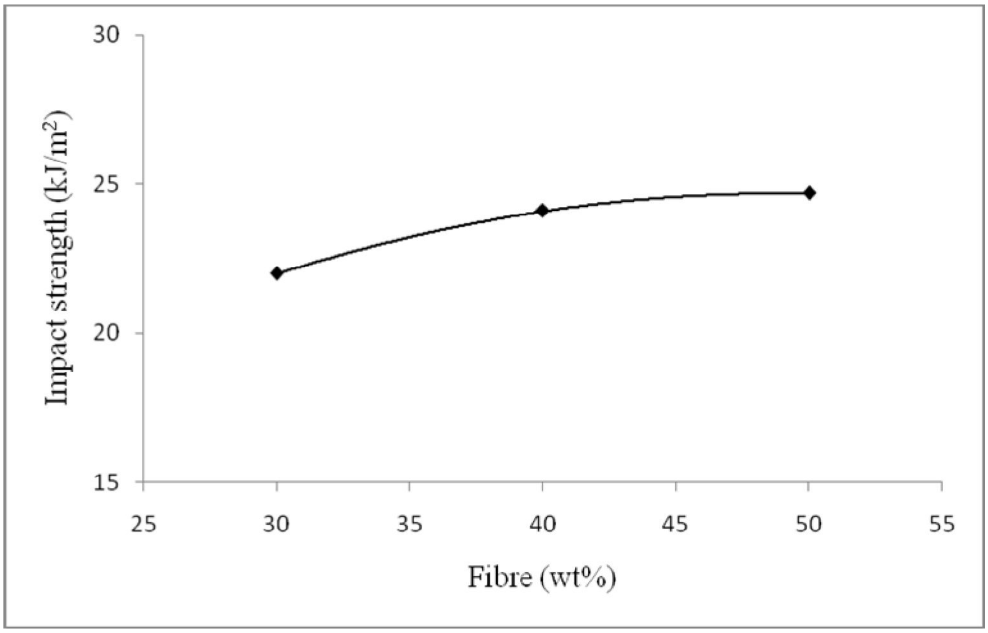


Fig. 4.18 Charpy impact strength of C/C composites as a function of fibre content

The Fig. 4.18 shows the impact behavior C/C composites. The impact strengths of 30 wt%, 40 wt% and 50 wt% C/C composites are reported as 22.00 kJ/m^2, 24.10 kJ/m^2 and 24.69 kJ/m^{2}respectively. It is clear that the amount of fibre increase resulted in increased impact strength of C/C composites. It is observed that the impact strength of the composites is increased by about 9 percent as the reinforcement content increases from 30wt% to 40wt%. As the reinforcement content increases further to 50wt% the impact strength of composite increases from 9 to 12 percent.

In general, the fibre enhances the impact properties of composites [Riley A. M. et al. 1990] while, fibres with high percentages induced more strength and hardness as it was seen. It was also confirmed that C/C composites do not become so brittle under high strain-rate conditions. The main mechanisms here is fiber de-bonding and fibre pull-out as shown in Fig. 4.19.

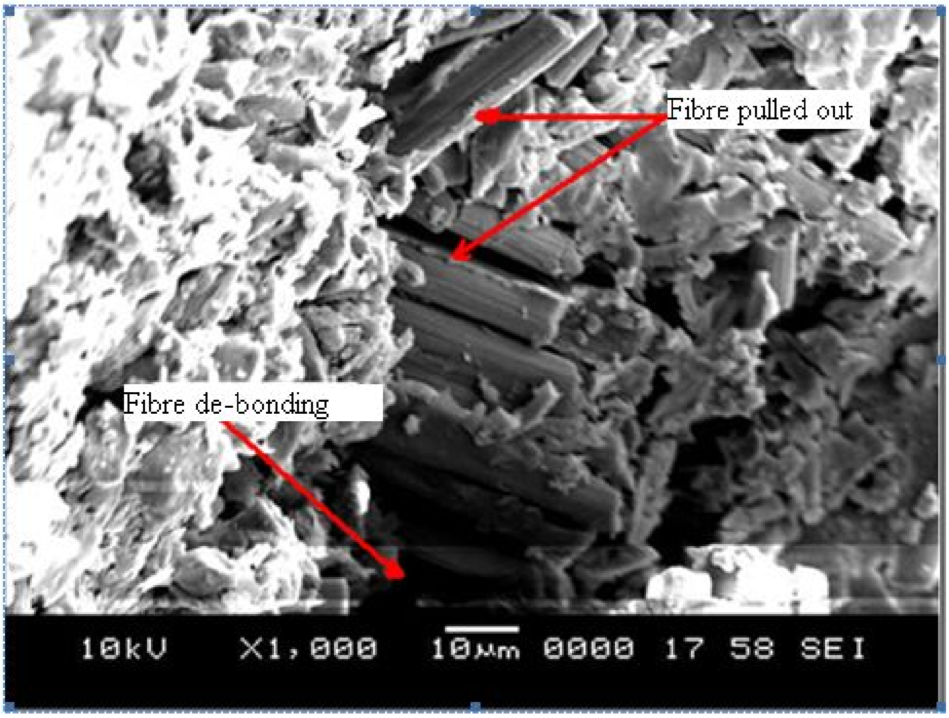


Fig. 4.19 SEM micrographs showing fibre de-bonding and pull-out

4.3.5 Creep test

Impression creep tests were carried out for all the three types of samples by using impression creep test setup at both room and elevated temperatures. The test results are presented graphically as the profiles of depth of indentation against time. Tests were conducted on the composites of different composition and the following observations were recorded. The Figs. 4.20, 4.21 and 4.22 show the results of creep test conducted on samples with variable load and temperature for 30wt%, 40wt% and 50wt% carbon fibre respectively and they are the plots of strain versus time.

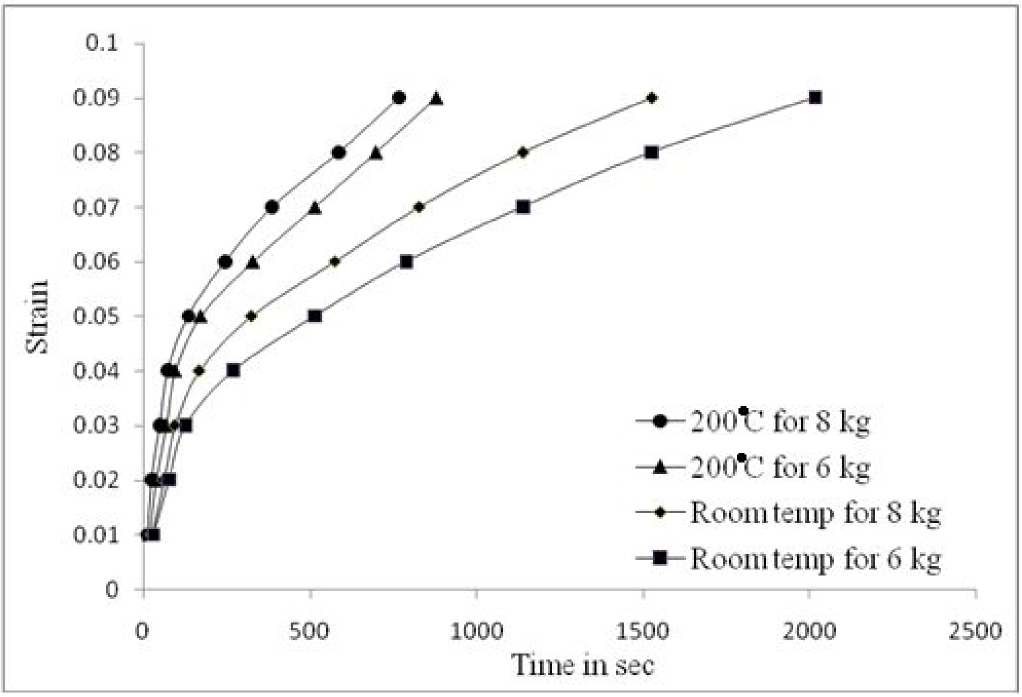


Fig. 4.20 Strain v/s Time plot of creep test for 30wt% fibre

Impression creep test was carried out on all the three weight fraction samples which were heated to 2500ºC. Creep characteristics were compared with each other at both room and elevated temperatures. Assessment of impression creep properties of the fiber matrix in the interface region of the aerospace material is important (R. G. Bathgate et al 1997) on the following counts.

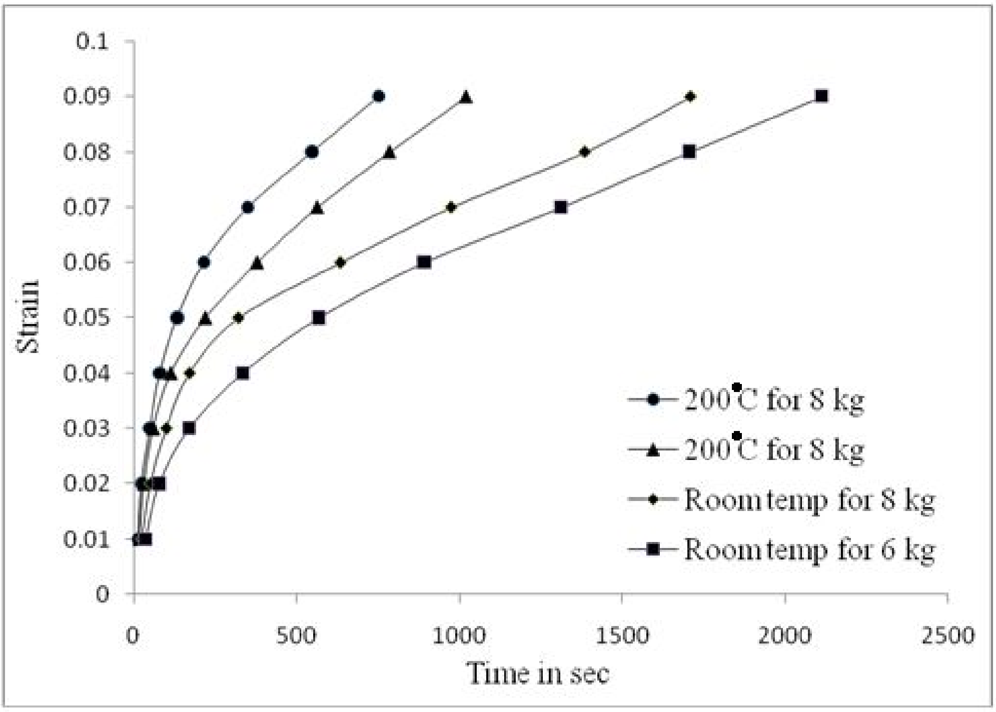


Fig. 4.21 Strain v/s Time plot of creep test for 40 wt% fibre

These properties can be taken as characteristic properties to evaluate and to compare the integrity of the material, and to understand the behavior of C/C composites under the condition of creep in critical components used in aerospace applications. Samples at elevated temperature register lower creep resistance compared to samples at room temperatures at all the loads, because difference in coefficient of thermal expansion of matrix and carbon fiber leads to weakening of matrix-fibre interfacial bond, which leads to lower creep resistance.

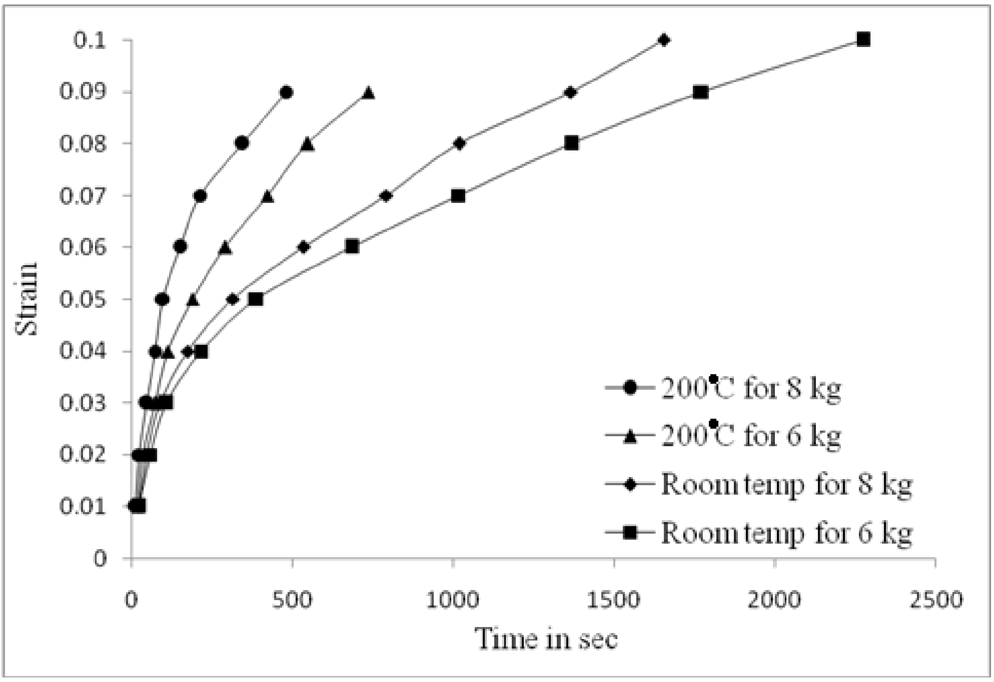


Fig. 4.22 Strain v/s Time plot of creep test for 50 wt% fibre

Creep resistance depends on both load and temperatures; as the load increases the time taken to deform decreases and vice versa and also as the temperature increases the creep resistance decreases. This is due to the fact that above room temperature softening of the matrix will take place at fibre-matrix interface region and dislocation movement is easier [J. Schjùdt-Thomsen et al 2000]. Hence, the fibre-matrix interface region will become weaker. By observing the creep data, we can conclude that as the percentage of fibre increases the creep resistance of the composites also increases for samples one and two but for sample three it slightly decreases: this is due to the fact that extent of graphitization is not completed in 50wt% fibre sample. The energy dispersive x-ray analysis (EDAX) shows that the element present is only carbon in the form of graphite but glassy phases (amorphous form) could be seen from X-RD analysis, this may lead to reduce interfacial bond between fibre and matrix. The lack of binders in higher fiber weight fraction content specimen creates many voids. The indenter diameter was almost

1mm; therefore indenter directly may pass through these voids. As the percentage of carbon fibre increased from sample one to two creep resistance also increased, but for sample three fibre amount is more (only 50wt% of binder when compared to 75wt% and 60wt% for sample one and two respectively). From this we can conclude that sufficient binder is also required to fill the entire fibre-matrix interfacial region to give good bond strength and to enhance creep resistance of C/C composites.

4.4 Analysis Of Tribological Properties

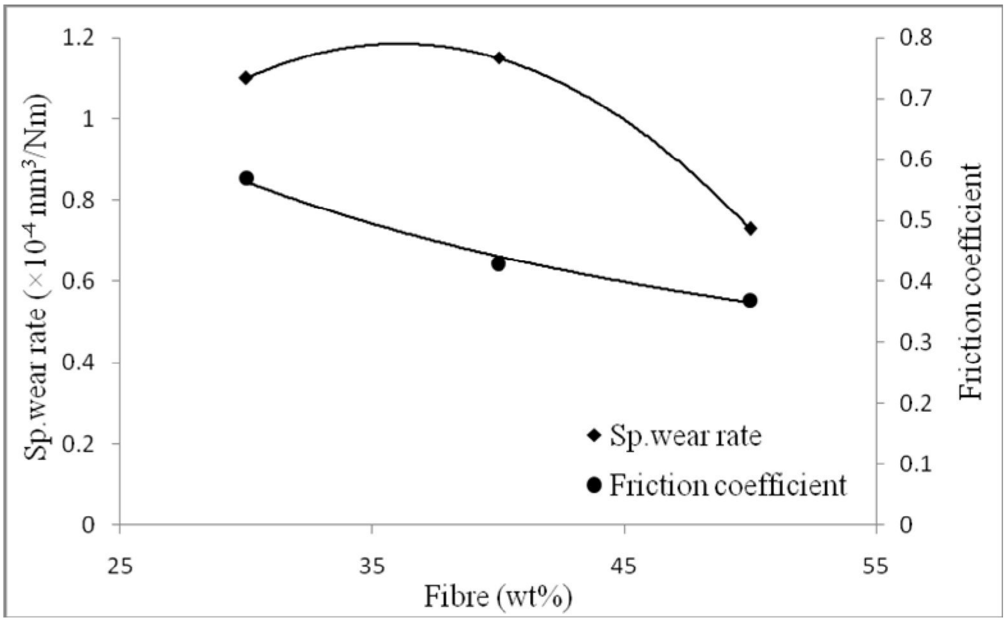


Fig. 4.23 Specific wear rates and friction coefficient of C/C composites as a function of fibre content for 4kg load at 200rpm for 1hr

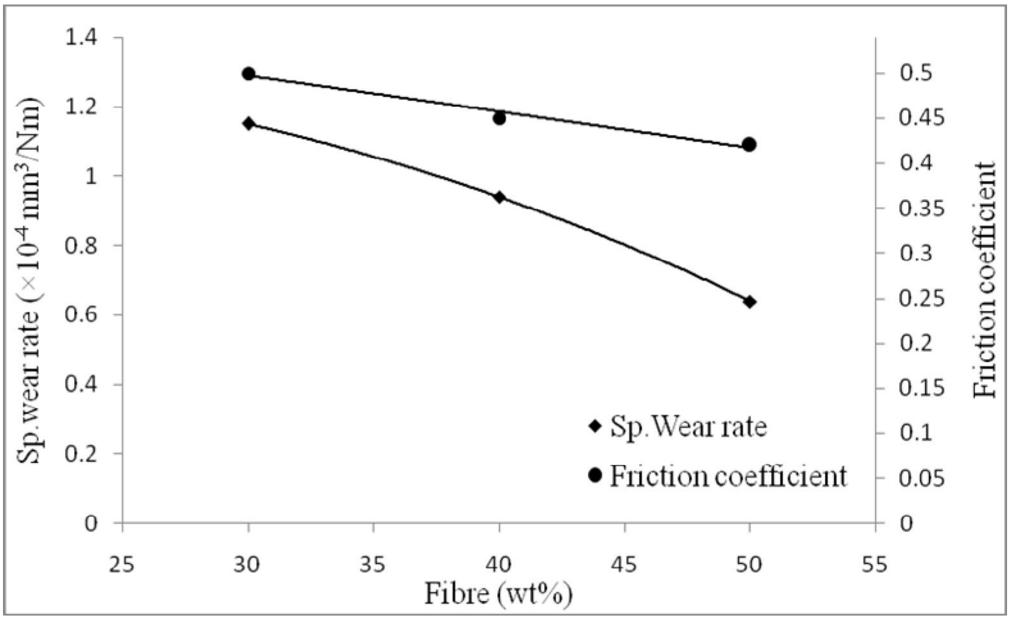


Fig. 4.24 Specific wear rates and friction coefficients of C/C composites as a function of fibre content for 5kg load at 200rpm for 1hr

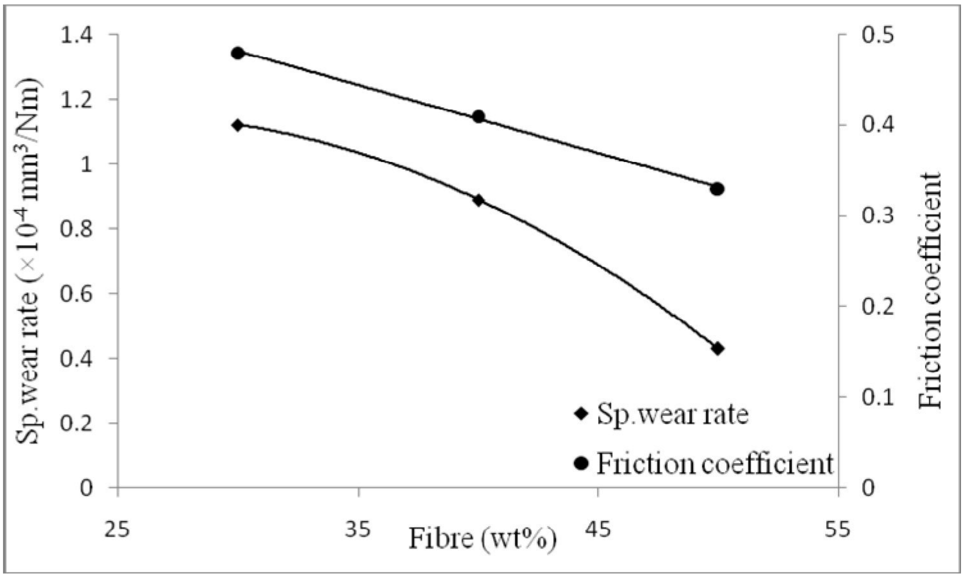


Fig. 4.25 Specific wear rates and friction coefficients of C/C composites as a function of fibre content for 6kg load at 200rpm for 1hr

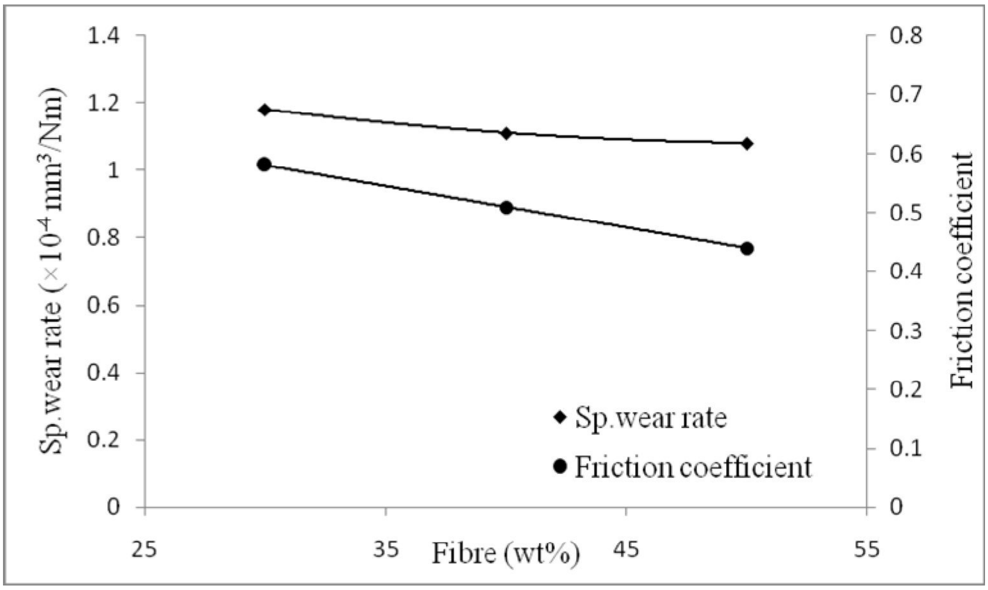


Fig. 4.26 Specific wear rates and friction coefficients of C/C composites as a function of fibre content for 4kg load at 400rpm for 1hr

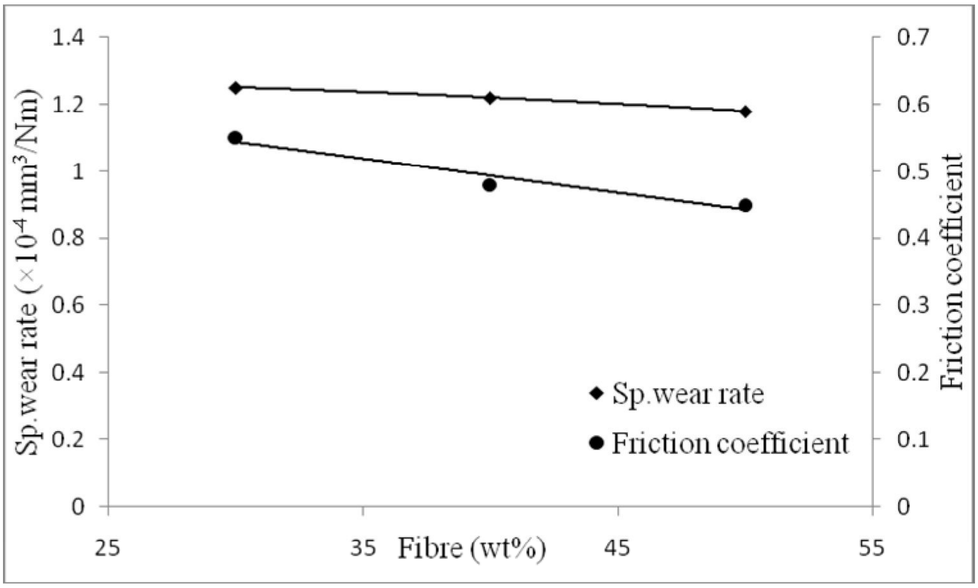


Fig. 4.27 Specific wear rates and friction coefficients of C/C composites as a function of fibre content for 5kg load at 400rpm for 1hr

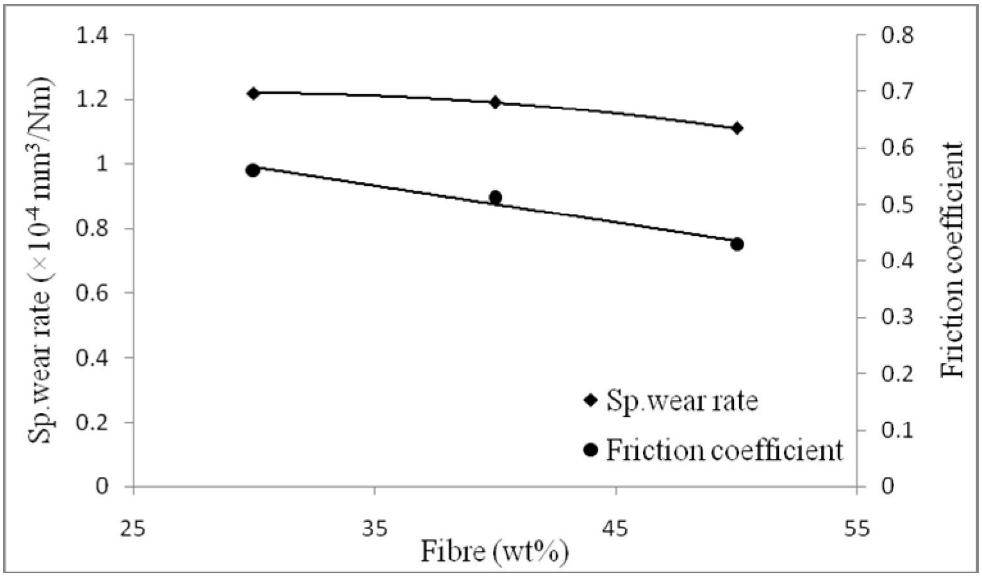


Fig. 4.28 Specific wear rates and friction coefficients of C/C composites as a function of fibre content for 6kg load at 400rpm for 1hr

Wear test was carried out for all the three fibre weight percentages of samples by using dry sliding wear testing machine at room temperature. The test results are presented graphically with the profiles of wear rate against weight fraction of carbon fibre. The test was conducted on the composite at loads of 4kg, 5kg and 6kg with the corresponding speeds of 200 and 400rpm. The readings of specific wear rate and friction coefficient values are recorded for three weight percentage of C/C composites. Wear characteristics were compared with each other for tests conducted at room temperature and is presented in Figs. from 4.23 to 4.28. Assessment of wear properties of the carbon fibre as well as matrix in the interface region of the aerospace breaking material is important on the following counts. These properties can be taken to evaluate the tribological behavior, to compare the integrity of the material, and to understand the behavior of C/C composites under the condition of wear in critical components used in aerospace braking applications. All the samples were applied with a load of 4kg, 5kg and 6kg with a speed of 200 and 400 rpm, and they registered a gradual decrease in their wear rate as the percentage of carbon fibre increased. This is due to the presence of graphite particulates from matrix acting as a solid lubricant. This study clearly says that the percentage of carbon fibres mainly decides the wear behavior of C/C composites. By observing the wear rate data, we can conclude that as the percentage of fiber increases the wear resistance of the composites also increases, that is wear rate decreases from sample 1(30wt%), through 2 to 3; this is due to their high temperature resistance and their perfect bond between fibre and carbon matrix. In the larger fibre-weight fraction composite, more carbon fibre will be in contact with the disc, as compared to less weight fraction one. These fibres will be having higher hardness as compared to the matrix resulting in lower wear rate and this also shows that a good fibre-matrix interface is established in the C/C composites. During the Pin-on Disc type wear process thermal stress gradient may be generated within the sample and this gradient may produce cracks. Reinforcements in the form of fibre can stop these cracks hence higher weight fraction of carbon fibre will lead to higher probability of stopping of cracks compared to lower weight fraction of carbon fibre. Therefore, higher weight fraction of carbon fibre shows low wear rate compared to lower weight fraction of carbon fibre. During any friction and wear process a significant heat is generated between any two sliding surfaces; if the surface contains more weight fraction of carbon fiber the amount of heat generated will be less compared to surface with low weight fraction of carbon fiber and more of matrix, which is easy to soften. In the present study, it is concluded that matrix has worn out faster than fibre. Hence, wear loss is caused mainly by matrix wear but fibers only got twisted and partially worn out; this could be revealed from SEM micrograph, shown in Fig. 4.29.

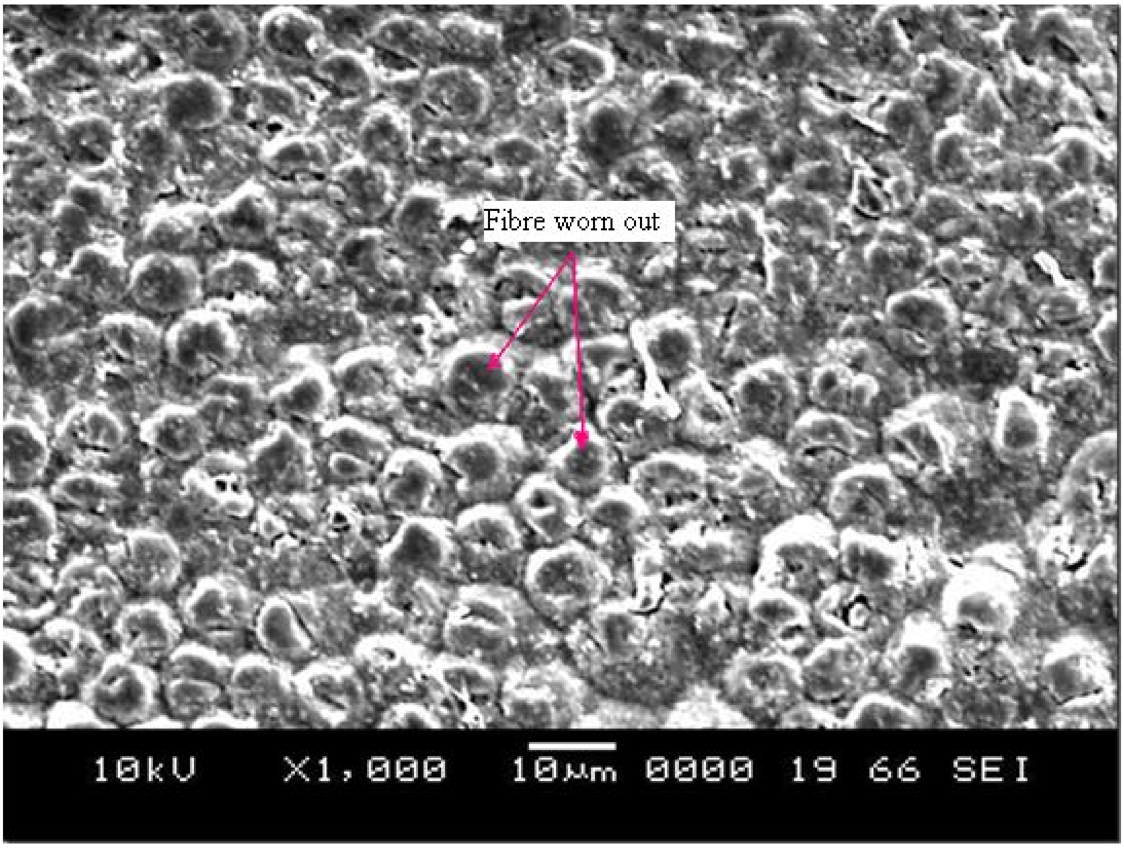


Fig. 4.29 SEM photomicrograph of worn surface of a C/C composite / 50wt % fibre /6kg/ 400rpm

Carbon fibres are being produced for the development of advanced new materials especially for aerospace applications. Composites can easily make use of the carbon fibres because of their micron or nano size diameter. With the large surface area of fibres very small quantities are needed to observe significant friction altering effects as observed in the current investigation as well as noted by other numerous researchers. Specifically, when the fibres become part of the friction layer they will increase the nominal area of contact thereby also the mechanical properties and typically increase or stabilize the coefficient of friction and reduce wear when percentage of fibre increases in the sample. It was seen that the wear occurring was due to severe mechanical abrasion whereby worn and scratched surfaces were observed, with the presence of fibre-matrix un-bonding; broken and torn-out fibres and the formation of high amounts of third body, demonstrating the fragile behavior of the C/C composite at room temperature. If the fibre reinforcement size is reduced to the nano-scale level (carbon nanotubes), the wear resistance of the nano-composites is significantly increased compared to that of micron-sized fibre-reinforced composites. The same results could be observed in nanoparticles-reinforced composites which were conducted by Wang Q. et al. (1996) and Wetzel B. et al. (2006). The results showed that, the smaller the particle or fibre diameter was, the better the wear and fracture resistance of the composites. Increase in impact fracture strength correlating with a decrease in specific wear rate was found out by G. Shi et al. (2004).

More number of experiments varying various processes parameters can throw some more light on mechanisms involved other than what has been already presented, which need to be performed in future by researchers. The wear results have been compared with Across Company results. The results were similar i.e. friction coefficient is 0.3 in both the cases.

4.5 Analysis Of Thermal Properties

Figures 4.30, 4.31 and 4.32 represent the thermo gravimetric analysis (TGA) and differential thermo gravimetric (DTG) curves in nitrogen atmosphere for C/C Composites of 30wt%, 40wt% and 50wt% carbon fibre, respectively. A sharper drop in a nitrogen atmosphere indicates that a more drastic thermal decomposition reaction takes place and that C/C composites decompose more rapidly in air than in nitrogen. The more rapid the pyrolysis, the greater the amount of volatiles generated. Dynamic TG experiments were conducted in a nitrogen flow at heating rate from 0.5 to 15°C /min. Mass was lost as temperature increased which caused the rate of yielding going on reducing as degradation was taking place. Mass lost in an oxidative atmosphere during yielding of the char in nitrogen at 1000°C is dependent on heating rate.

In 30wt% of carbon fibre composite, it was observed that a sharp drop occurred in the temperature range of 275ºC to 415ºC and maximum rate of degradation was found at 313.9°C with a major weight-loss of about 33.9% in the first step in Fig. 4.30. Similarly thermal degradation started from 415°C to 690ºC and maximum rate of degradation was found at 629° C with a major weight-loss of about 28.5% in second step. For the degradation from 690ºC to 1000°C and the maximum rate of degradation was started at 863º C with a major weight-loss of about 27.5% in third step. After completion of the third step, about 89.9% of C/C Composites was degraded and about 10.1% of sample remained.

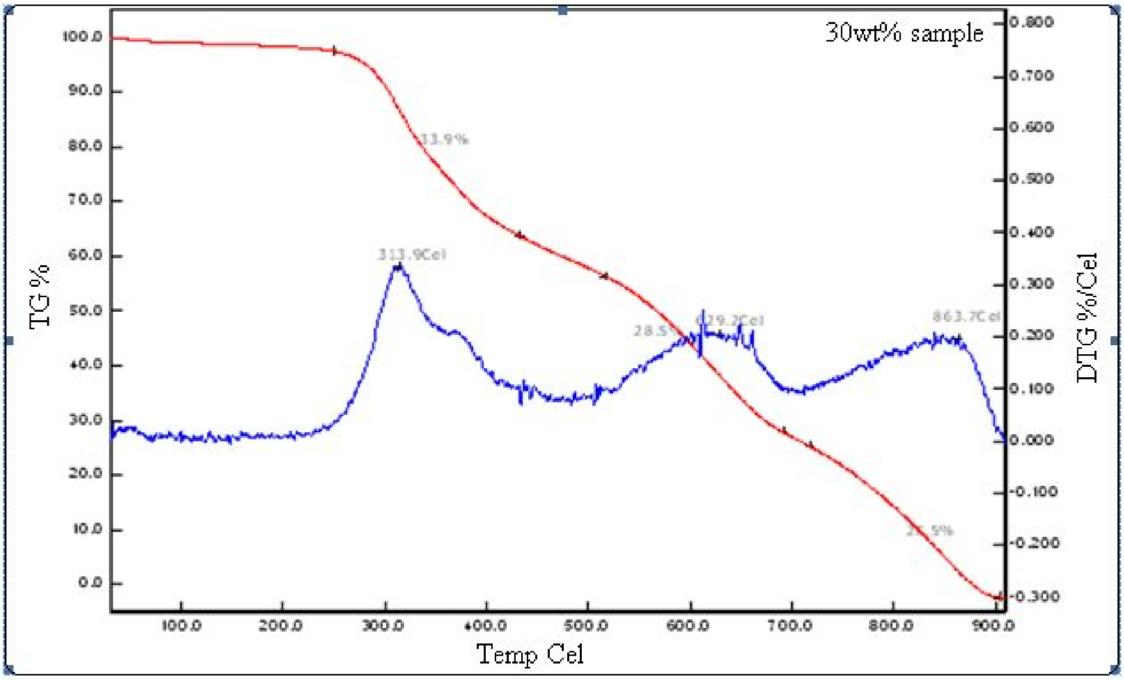


Fig. 4.30 TG/DTG v/s. temperature for sample with 30wt% carbon fibre

In 40wt% carbon fibre composite, it was observed that a sharp drop occurred in a range of temperature from 220ºC to 450ºC and maximum rate of degradation was found at 356° C with a major weight-loss of about 26.4 % in this range. Similarly, in the second range of 450°C to 620ºC, degradation rate was maximum at 543°C with a total weight-loss of about 25.7%. In the third range of 620º C to 1000°C, maximum rate of degradation was found at 794ºC with a total weight-loss of about 33.6%. After 1000^{0}C, about 85.7% of C/C composite degraded and about 14.3% of sample remained.

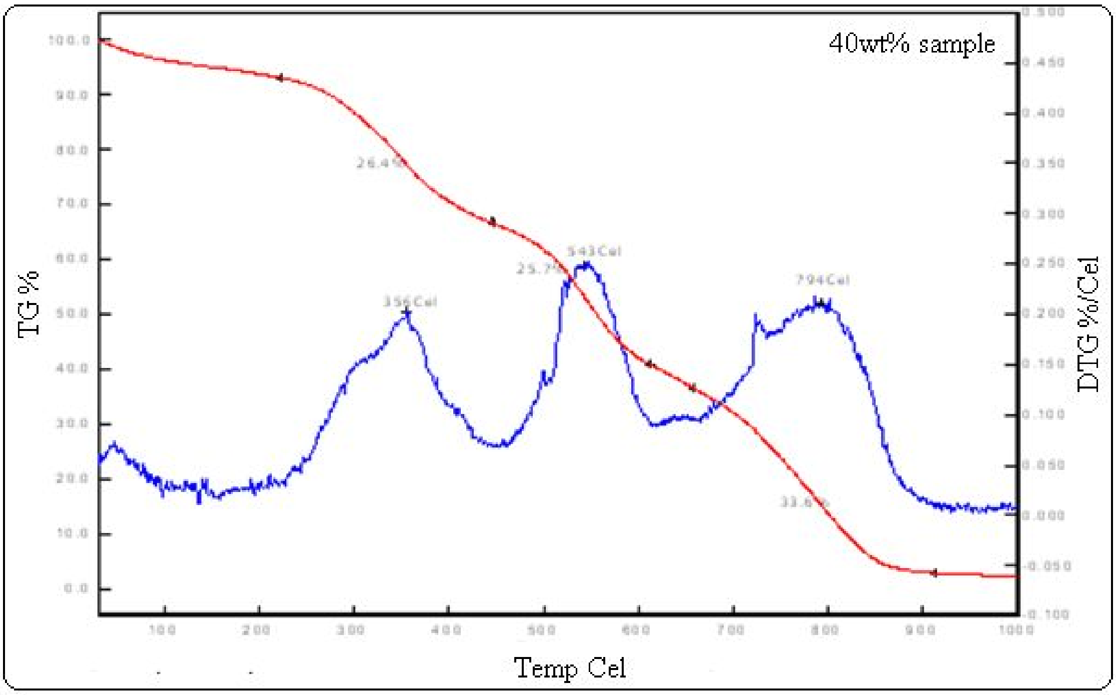


Fig. 4.31 TG/DTG vs. temperature for the sample with 40wt% carbon fibre

In 50 wt% carbon fibre composite, it was observed that a sharp drop occurred in a range of temperature from 220º C to 465ºC and maximum rate of degradation was found at 322°C with a total weight-loss of about 26.2 % in this range. Similarly, degradation was observed from 465°C to 745ºC and maximum rate of degradation was found at 644°C with a total weight-loss of about 27.3% in this range. Degradation was observed from 745ºC to 1000°C and maximum rate of degradation was not found. In some experiments the derivative of dynamic TG curve is not well-defined in nitrogen gas particularly in the third range. After completion of the third range about 53.5% of C/C composites were degraded and about 46.5% of sample remained.

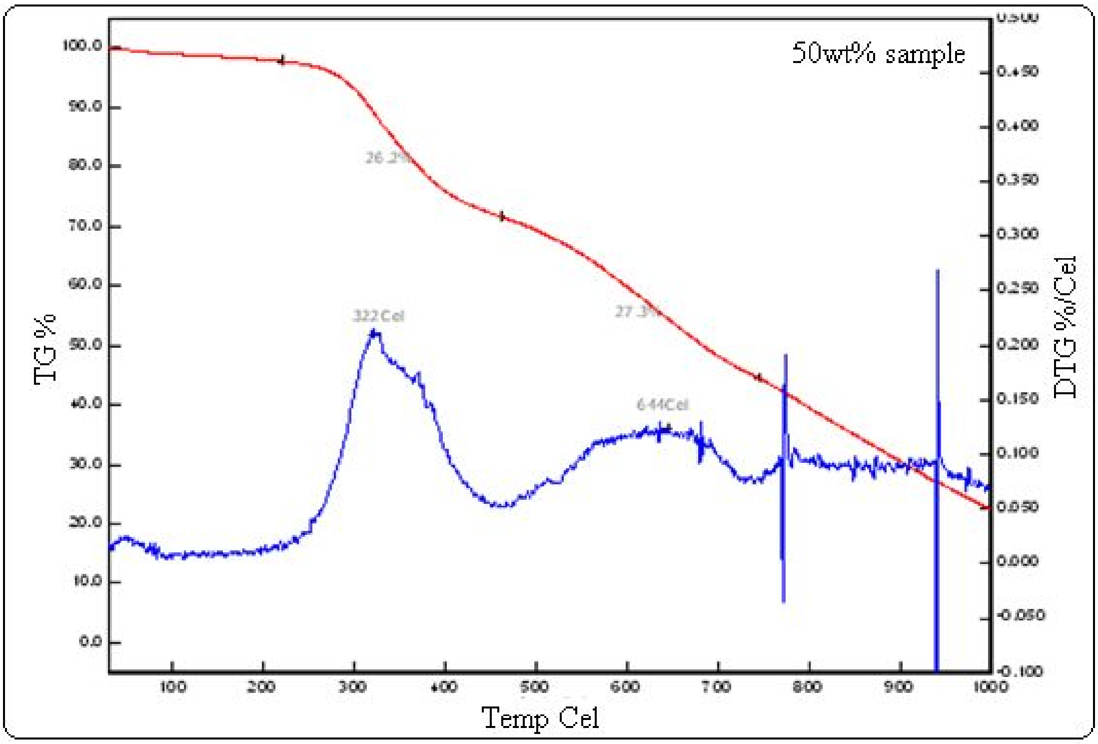


Fig. 4.32 TG/DTG vs. temperature for a sample with 50wt% carbon fibre

The results show a decrease of about 52 to 89% in weight up to 1000ºC in nitrogen gas. This weight-loss is most likely due to release of hydrogen from the matrix and also from the evolution of CO or CO_2 gases. Oxidation reaction was severe leaving about 11% residual materials after one hour for sample number one i.e. 30wt% carbon fibre-reinforced composite shown in Fig. 4.30. For sample number two i.e. 40wt% carbon fibre composite, about 14.3%, and for sample number three i.e. 50wt% carbon fibre composite, about 46.5% respectively were the remaining weights (Figs. 4.31 and 4.32). The major cause of weight-loss is due to the chemical reaction occurring at low temperatures, carbon fibre-matrix diffusion at medium temperatures and boundary layer diffusion at elevated temperatures and finally micro-cracks existed in fibre. The total damages of composites occurred at higher temperatures.

4.6 Constructional Feature And Drawings Of Automatic PY Production Machine

After completion of design and development of making manual PY making machine model, PY has been successfully prepared using this machine and C/C composites have been synthesized by hot-press-mould method. The superiority of the synthesized the new class of materials has been proved by testing for various mechanical, tribological and thermal properties. As per the experiment 50 wt% carbon fibre composites showed the best properties among the three different samples. Hence, it was decided to develop semi-automatic PY machine for the composition of 50 wt% carbon fibre, 45 wt% matrix and 5 wt% sleeve in order to minimize the processing steps of C/C composites for various applications. The part names, material and quantities required for automatic PY machine are listed in Table 4.1.

Figures 4.33 (a) and 4.33 (b) show the isometric view and side view respectively of the automatic yarn making machine describing the three stages of operation in which, stage A shows the carbon fiber splitting and spraying arrangements, stage B shows the fixing of yarn diameter and stage three shows the sleeving arrangement. In the first stage, fiber from roll is passed through a pivot and is pulled up by the rollers and widening the fiber is done to a specified width. Further it is mixed with coke and pitch powder, then preheated and passed to the next stage for rolling it into a required diameter using number of rollers. Then fibre passes through second stage where it is cooled to -20˚C to be ready for the third stage. After cooling to a constant temperature in the cooling chamber it is passed further through the third stage where it comes in contact with a spherical groove roller. The groove roller has molten polypropylene (PP) adhered to the surface which is maintained at a temperature in between 165˚C and 176˚C. By passing the fibre with coke and pitch through this roller, fibre is coated with PP and is further rolled by another roller to maintain the final required diameter. Further, the preformed yarn is collected by

Table 4.1 List of Main Parts and Materials for automatic PY machine

Part number	Part name	Quantity	Material
1	Carbon fibre spool	1	Carbon
2	Fiber guiding bush	1	MS
3	Driven roller and driver roller with rubber layer	2	MS
4	Circular groove roller with rubber layer	2	MS
5	Hopper for pitch	1	SS
6	Hopper for coke	1	SS
7	Sprinkling bed	1	Al
8	Heating coil	1	SS
9	Plain driver and driven rollers	2	MS
10	Metal die or lofted die	2	MS
11	Circular groove metal rollers	6	MS
12	Adjacent supports for rollers	1	MS
13	Chassis mounting	1	MS
14	Spur drive gear	9	MS
15	Idler gear	14	MS
16	Mounting for hoppers	4	SS
17	Tension bar for bearing housing	30	MS
18	M6x25mm slotted bolt	30	MS
19	Bearing mountings	60	MS
20	Chassis	6	MS
21	Chassis spacer pins	12	MS
22	Guide bushes	6	Brass
23	Timing Belt	1	Urethane
24	Fiber guiding block	1	MS
25	Timing pulley	1	Al
26	Plain driver rollers	2	MS
27	Fibre with coke and pitch coating	1	Carbon
28	Cooling unit	1	SS
29	Timing belt	1	Urethane
30	Timing pulley	2	Al
31	Idler timing pulley	1	Al
32	Circular groove metal rollers Groove width 2.2	2	MS
33	Circular groove metal rollers Groove width 2	2	MS
34	PP bath	1	SS
35	Molten PP catch roller	1	MS
36	Timing Belt	1	Urethane
37	Circular groove roller for sleeving PP	1	MS
38	Pivoted fiber guide	1	MS
39	Fibre Collection Spool	1	Bobbin
40	Adjacent supports for collection spool	1	MS
41	Timing belt	1	Urethane
42	Timing pulley	1	Al

spinning it on a spool, which is run at a constant rpm. All the controls are maintained by a programmable logic controller (PLC). The rollers are powered by 0.5 HP motor.

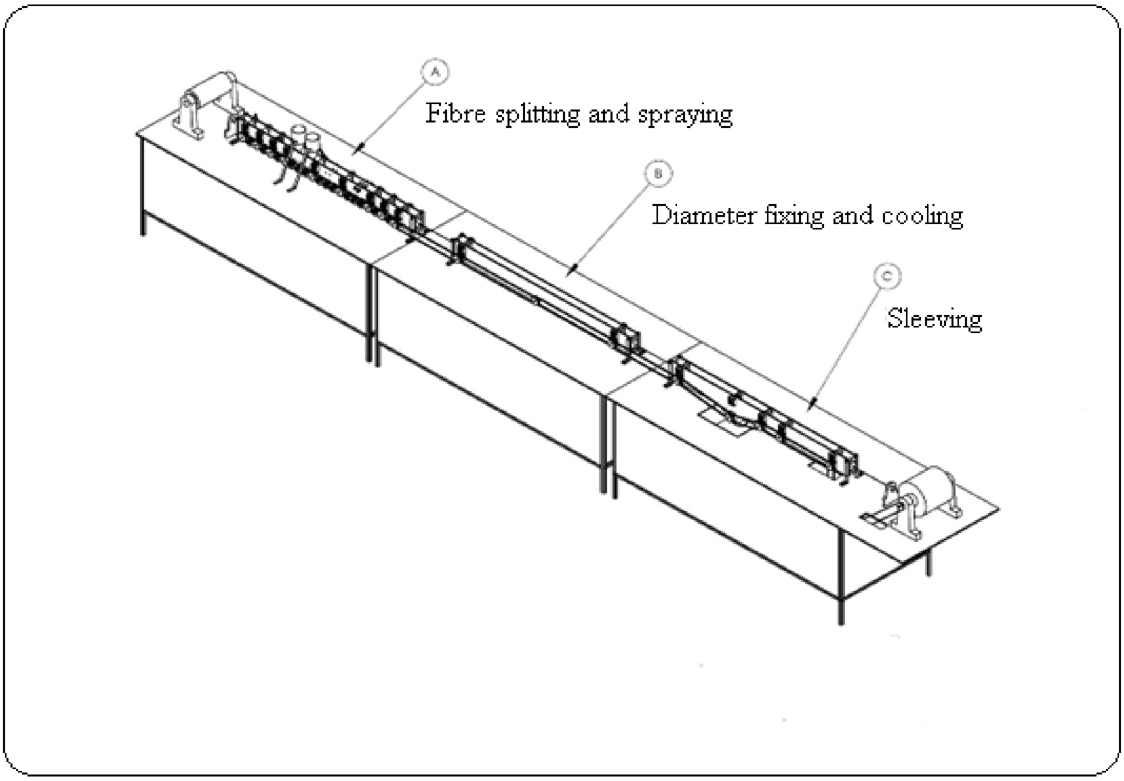


Fig. 4.33 (a) Isometric view of three stages of automatic PY producing machine

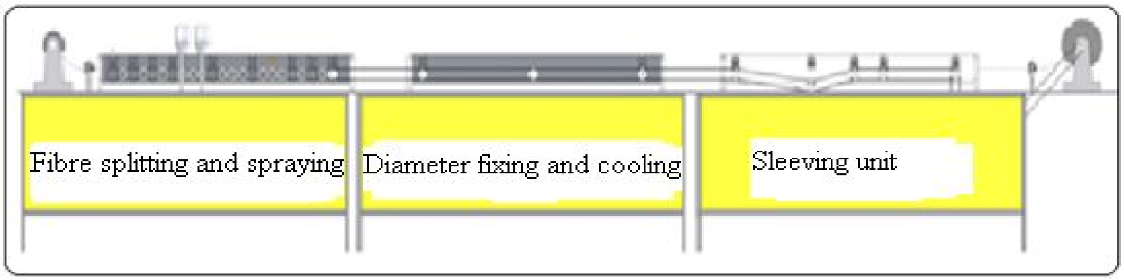


Fig. 4.33 (b) Side view of three stages of automatic PY producing machine

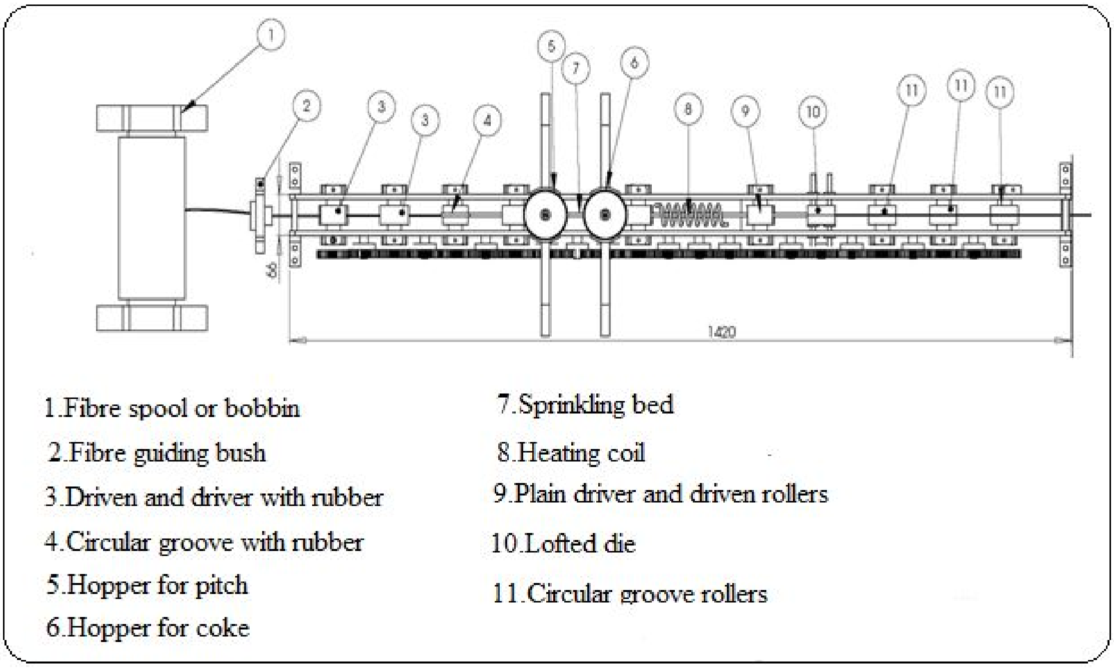


Fig. 4.34 (a) Top view describing the first stage of operation of the machine

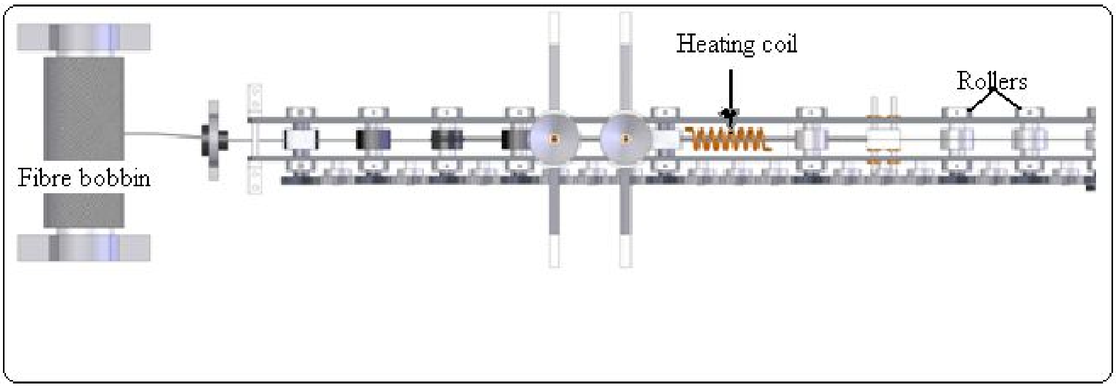


Fig. 4.34 (b) Top view showing the first stage of operation of the machine

Figures 4.34 (a) and 4.34 (b) show the details of the first stage of the machine in top side view. Part-1 represents the carbon fibre spool supported on a vertical pillow type arrangements, where in the fibre is passed on to a hollow pivot-2 with a five mm hole, then it passes through the driven roller-3 which pulls the fiber and pushes it to the next stage. Roller-3 which is made of MS material with hardened surface further applies pressure on the fibre and makes it flat for maximum widening with a width of around four mm, which can be achieved. Further the widened fibre is passed through roller-4 which has unique surface structure with a two mm rubber layer that enables the fibre to be further flattened, there by widening it to a width of six mm. Fibre is then passed through the next roller where it is perfectly aligned to the centre of the whole chassis before it is passed to the sprinkling bed-7. Hoppers -5 and 6 are filled with pitch and coke respectively. The amount of coke and pitch which is to be sprinkled is dependent on the frequency of vibrator at the end of the hopper nozzle. Right amount of spraying coke and pitch is controlled by varying the frequency of the vibrator. Once the coke and pitch deposit on the flattened fibre moving on the sprinkling bed, it will remain on the fibre. The fibre with the coke and pitch mixture is passed through a heating coil-8 where it is heated to a temperature around 41˚C, which is just below the melting point of pitch, where the pitch softens and gets adhered to the surface of the fibre. Further, the fibre is passed through a set of rollers-9 where it is compressed and passed further.

Further, fibre with pitch and coke mixture travels through a metal die-10 which has round loft shape feature with seven mm diameter at the entrance, which narrows to the required diameter 2.5 mm at the other end. The metal die-10 is a split die which opens and makes provision for fibre to be fed through it. As the flattened fiber enters the metal die-10 it gradually narrows and becomes round in shape to a diameter of 2.5 mm, when it comes out of the metal die-10. Further the rounded fibre travels through the rollers -11, which has a hemispherical groove in its center with an approximate radius of one to two mm. When the two rollers meet, they make a perfect round shape. When the fibre passes through rollers, it will be further compressed to a diameter of 2 to 2.2 mm. Similarly, when the fibre that comes out after passing through the set of rollers-11, it will have a final diameter of 1.8 to 2.2 mm.

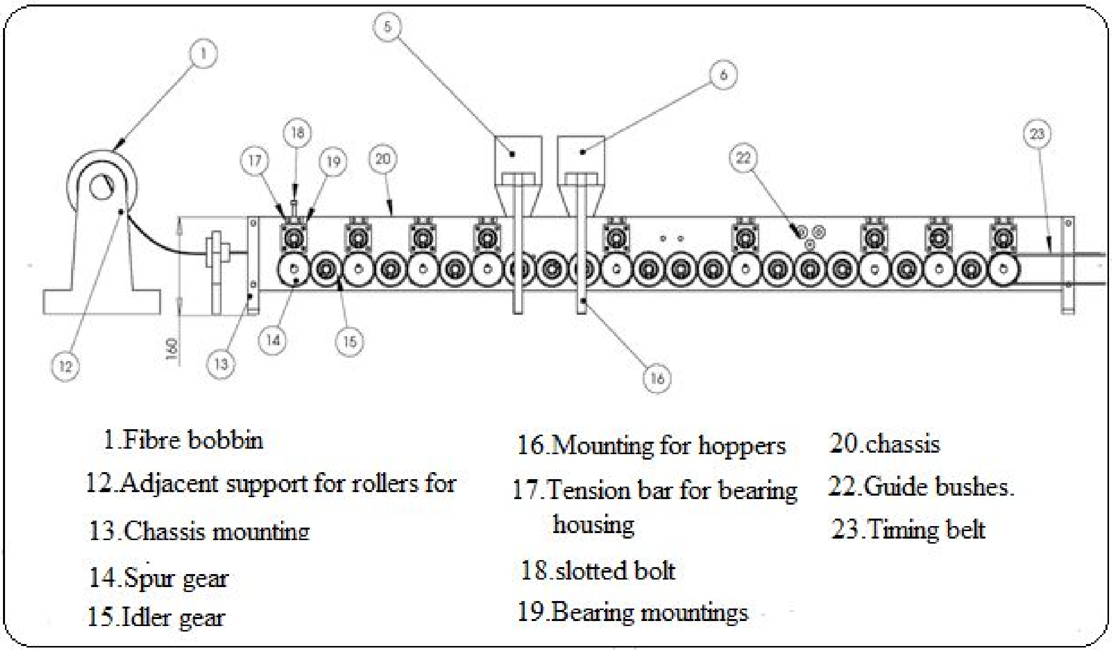


Fig. 4.35 (a) Side view describing the first stage of operation of the machine

Figures 4.35 (a) and 4.35 (b) describe the stage one of PY machine in a side view and isometric view respectively. It is clearly showing the gears, bearing mounting and timing belt coupled to timing pulley. Part-1 as indicated is the carbon fibre spool which rests on the adjacent supports-12, which is free to rotate on its axis. The part-19 represents the bearing mounting along with bearing.

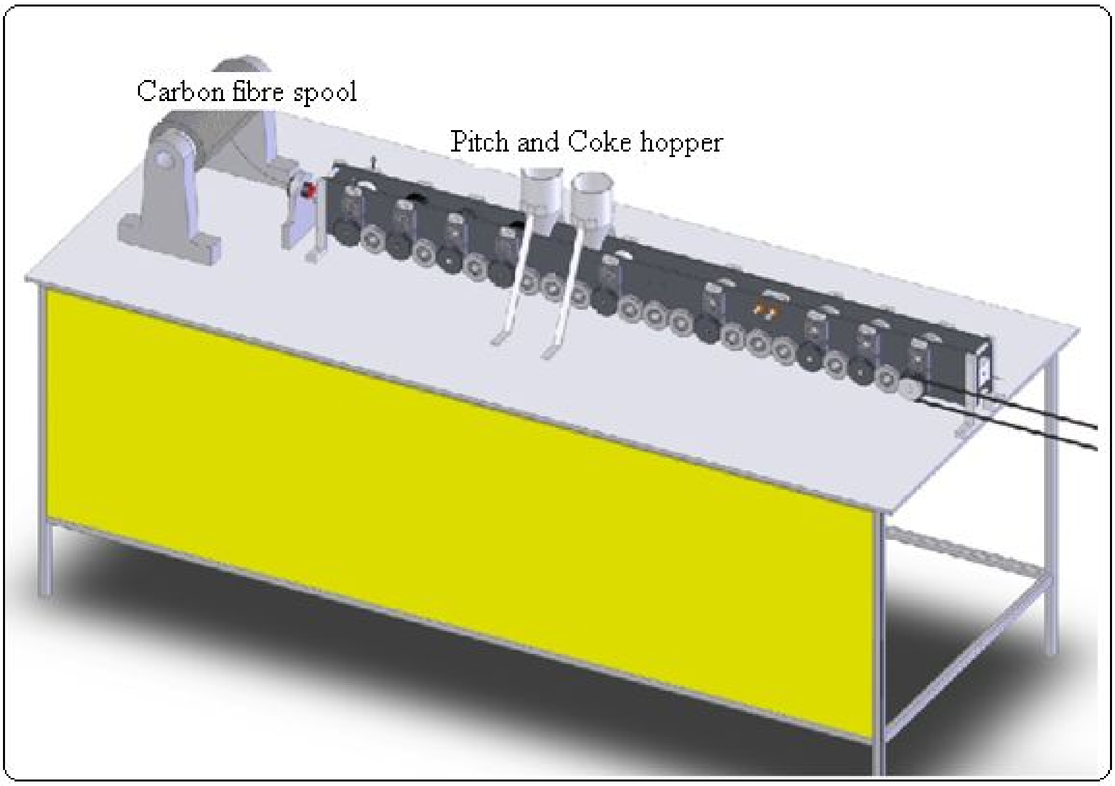


Fig. 4.35 (b) Isometric view describing the first stage of operation of the machine

There are totally eighteen bearing mountings used for each set of rollers. Bearing mounting which supports the main driver roller is firmly fixed to the face of the chassis-20 using bolts and nuts. Bearing mounting supports the driven puller which is free to slide to around three mm in vertical axis; it is mainly done to increase the compressive pressure on passing fibre between driven rollers. Tensioned mountings-17 is attached to the chassis-20 using bolts and nuts. Tensioned mountings-17 has a threaded hole which uses a bolt-18. When the bolt-18 is tightened, it moves downward and applies compressive pressure on the bearing mountings-19, which is free to slide; as such the driven roller supported by the bearing mounting-19 is compressed against another driver roller. Spur gear-14 is mounted on the shaft of the driven roller. Power is transmitted from the roller to roller using the spur gear-14 and the idler gear-15. All driver rollers will be rotating in clockwise direction with a speed of 12 rpm, producing around one meter of preformed yarn per minute. Hoppers-5 and 6 are mounted using mounting-16, which is firmly attached to the base of the machine. Part-22 represents the guide bushes that hold the metal die-10 which guides metal die to move in specified direction. Drive power for the rollers comes from the timing pulley and belt-23, which comes from the next stage two.

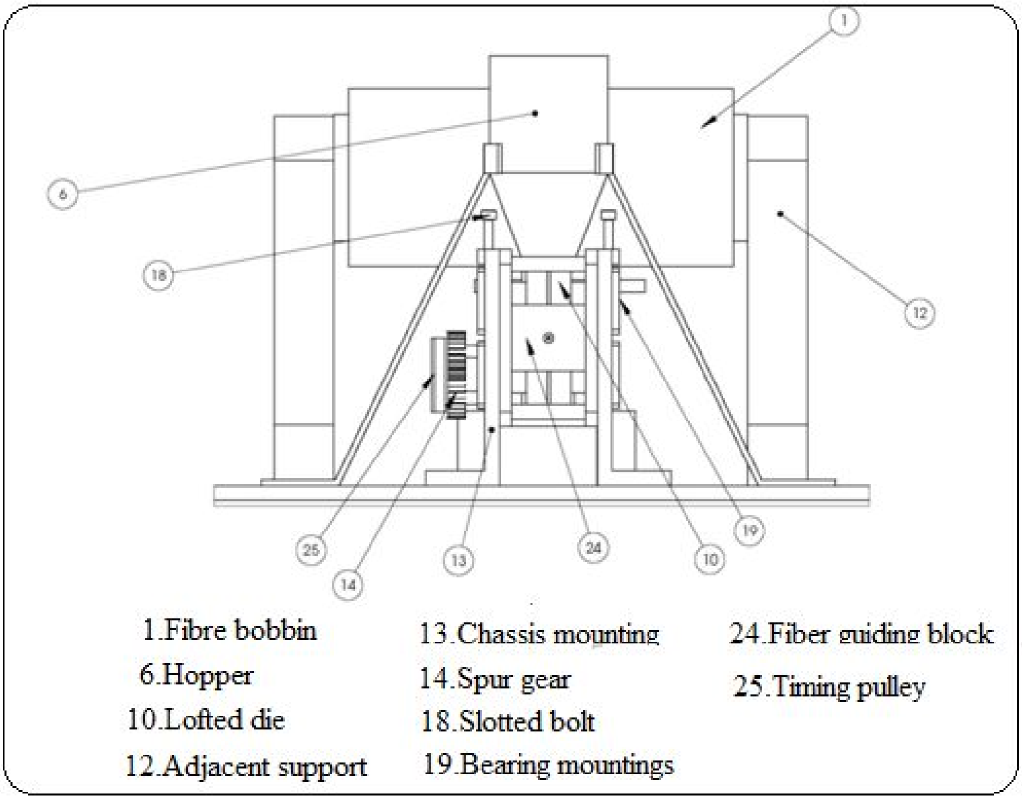


Fig. 4.36 (a) Front view describing the first stage of operation of the machine

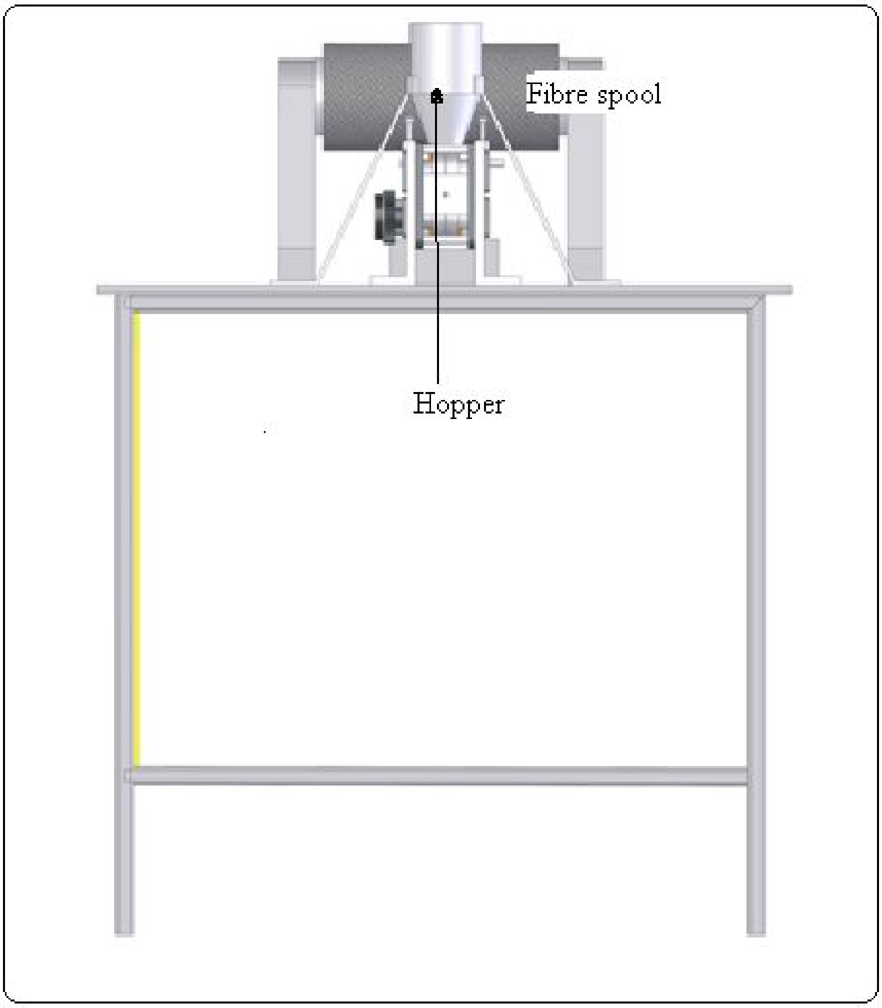


Fig. 4.36 (b) Front view showing the first stage of operation of the machine

Figures 4.36 (a) and 4.36 (b) show the first stage in its front side. Fibre after coating with coke and pitch comes out of the hollow pivot block-24. Part-13 represents the mounting for the chassis-20 to the bench-25, which shows the timing pulley firmly coupled to the driver gear-14. Power from the next stage is being transferred using this timing pulley and belt-23.

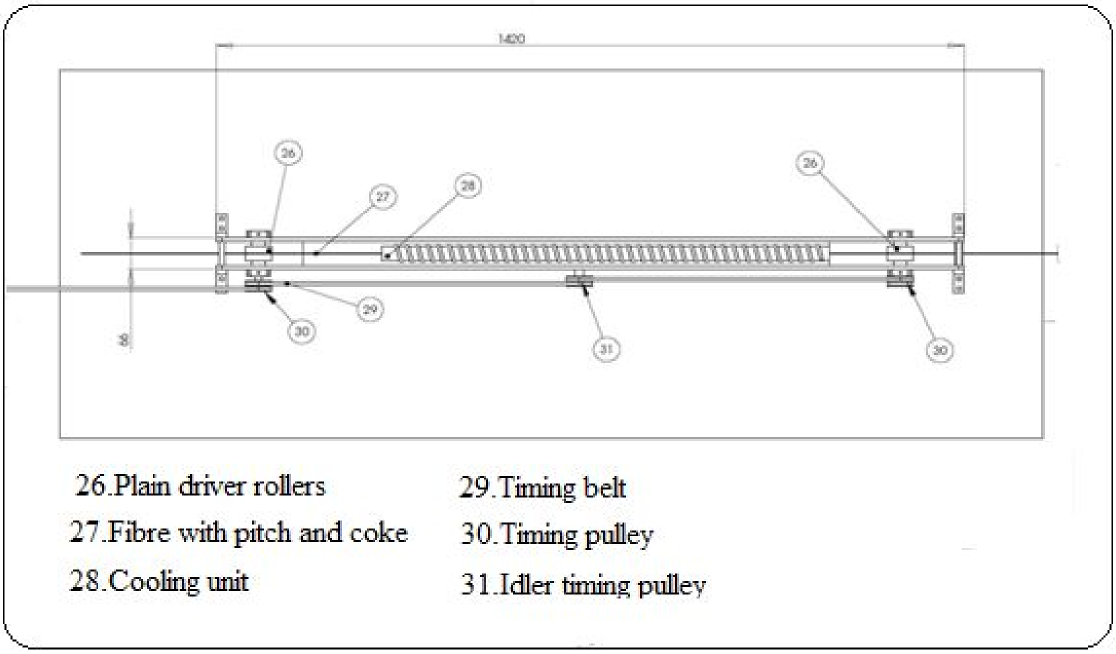


Fig. 4.37 (a) Top view describing the second stage of operation of the machine

Figures 4.37 (a) and 4.37 (b) describe the second stage of the machine. In this stage fibre is cooled to a temperature of -20˚C in one second before passing through the next stage of sleeving with PP (polypropylene). As in the figure, the pair of rollers-26 with a spherical grove of diameter 2.2 mm pulls the fibre, pitch and coke blend (27) and passes it through the cooling unit-28. The total length of cooling chamber is 850 mm where the fiber is instantly cooled; and maintains the same temperature throughout the length of the cooling channel. The cooled fibre comes out of the other end of the cooling channel and is pulled up by the pair of rollers-26. The component-30 represents the 2 rows of timing pulley which are coupled to the driver roller-26. The component-32 represents the two row idler pullers which are used for maintaining the direction of the rotation of the rollers.

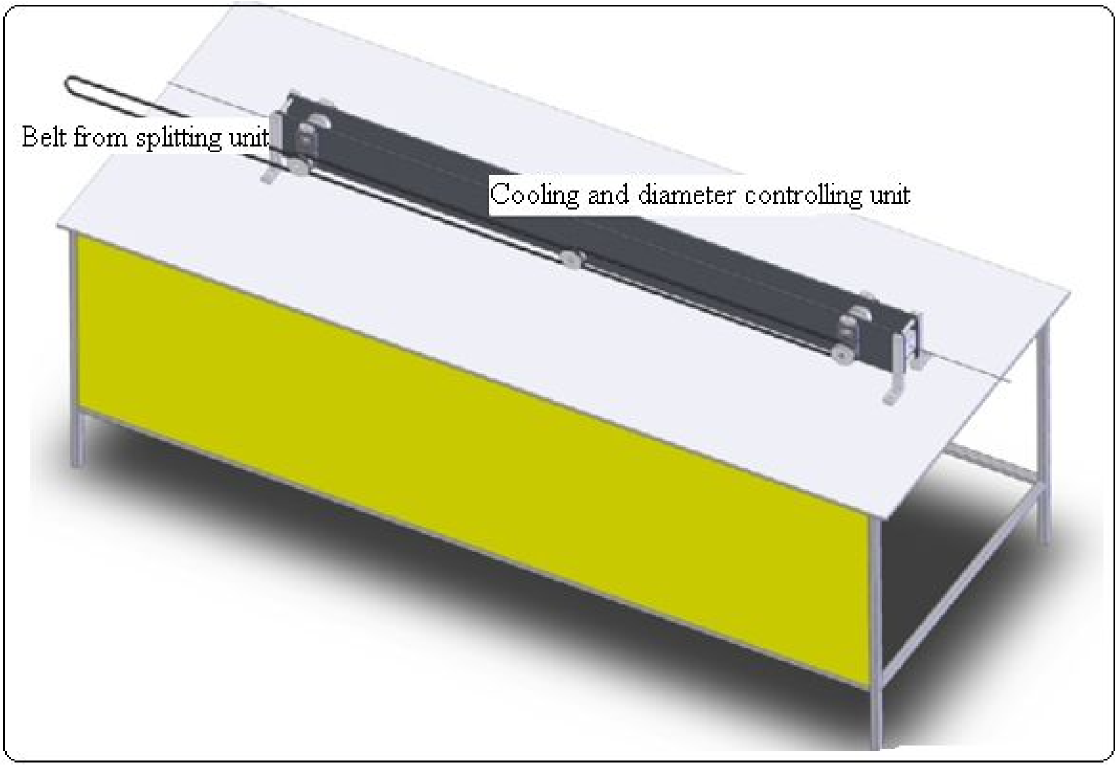


Fig. 4.37 (b) Isometric view describing the second stage of the machine

Cooling is mainly done to counteract the heating at the stage-3. If it is (fibre mixed with coke and pitch) not cooled to -20˚C the pitch will melt, when the temperature crosses above 50˚C. Once cooled and passed to the next stage where the pitch will meet a temperature of 165˚C to 170˚C it will take nearly five seconds to reach from -20˚C to +40˚C. By that time PP will be cooled and will surround the fibre and the coke pitch blend.

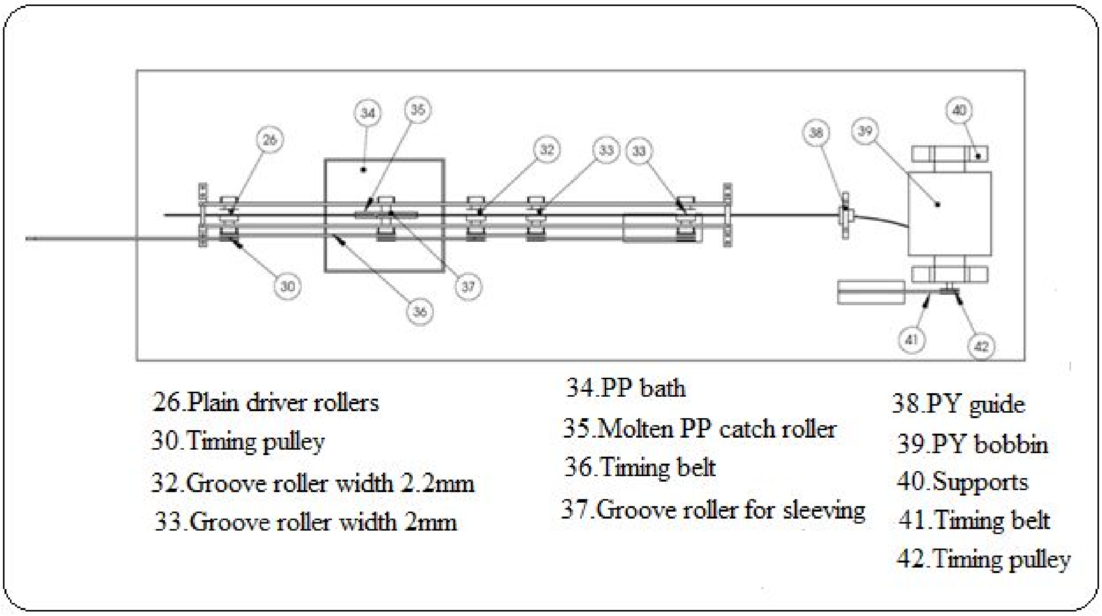


Fig. 4.38 (a) Top view describing the third stage of operation of the machine

Figs. 4.38 (a) and 4.38 (b) describe the third stage of the machine where sleeving is to be done using polypropylene (PP). Roller-26 pulls the fibres and passes it through rollers-35 and 37 with a spherical groove of radius 1-1.2 mm. The roller-35 with a slightly larger diameter is used to dip fibre in the hot PP. The PP bath-34 is maintained at a temperature of 165˚C to 170˚C. When the roller rotates fibre carries along with it the molten PP in between the roller-35 and 37. Further it is passed through the pair of rollers-32 and 33 where it is further compressed to a diameter of 2-2.4 mm as per the specification given. Fibre with the PP sleeve is passed through the last pair of roller-33 and finally comes out with the required diameter of 2.2 mm. Spool-39 is mounted on mounting stand-40 which rotates in its axis. Spool is coupled to a timing pulley-42 and also coupled to the motor within the bench using the timing belt-41. Other end of the timing belt is connected to the pulley of the motor using a slip clutch mechanism.

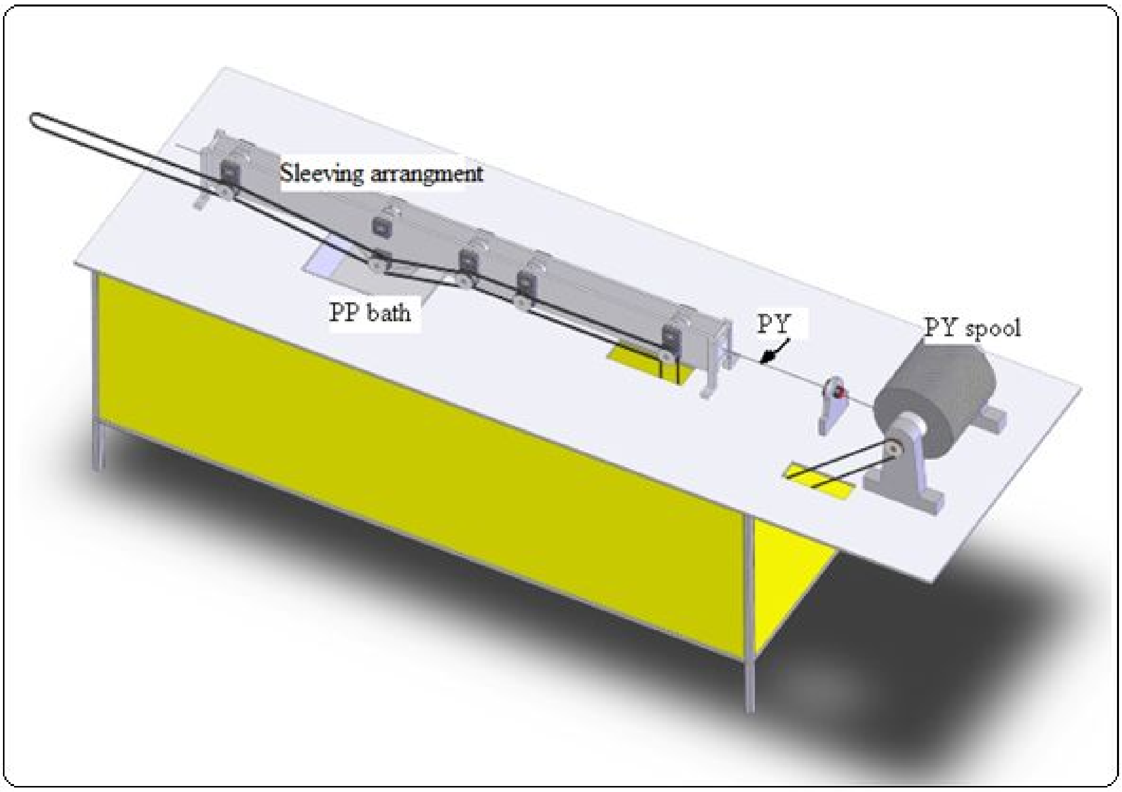
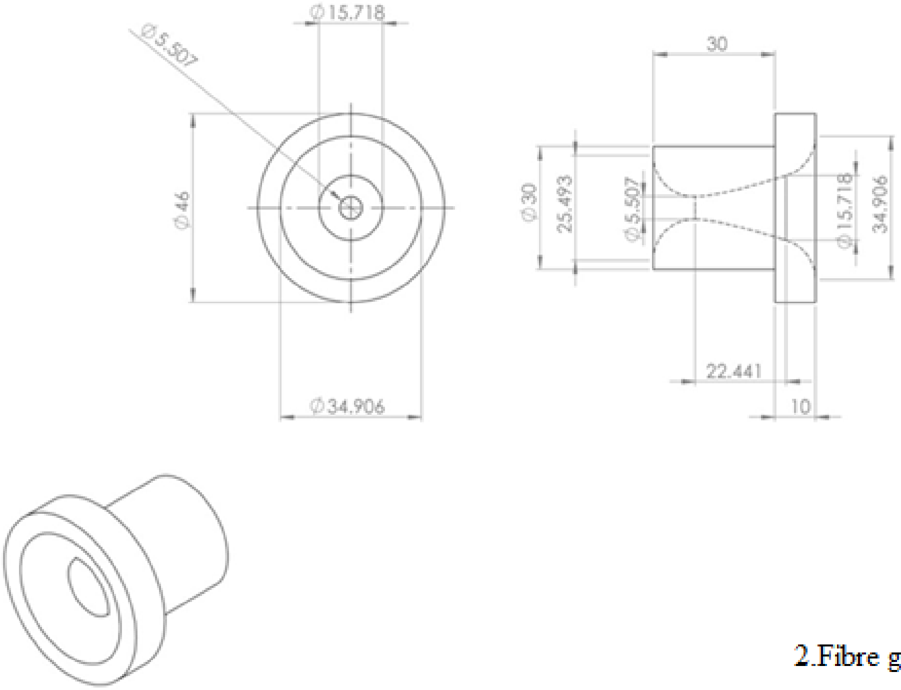
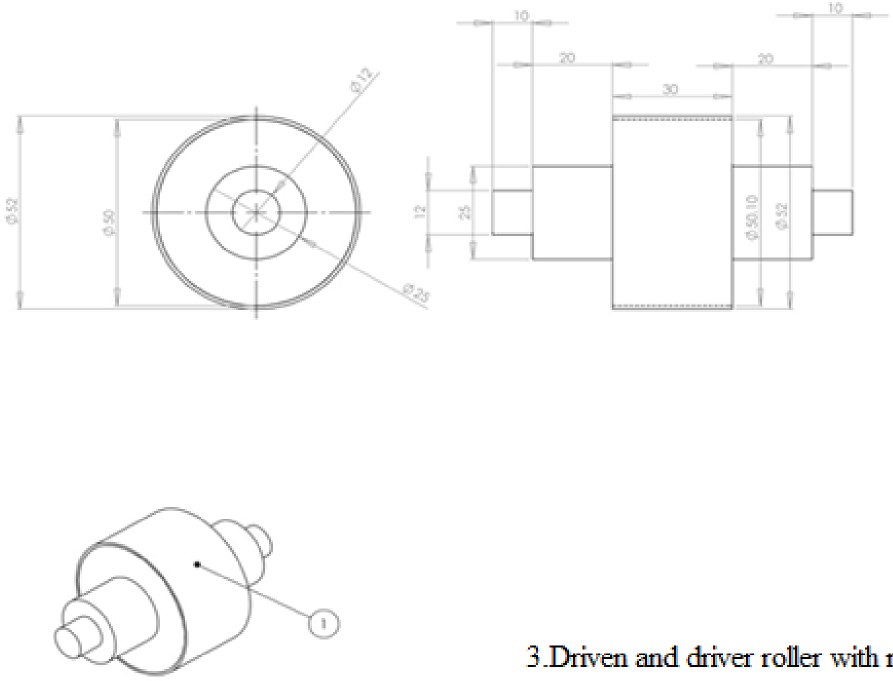


Fig. 4.38 (b) Isometric view describing the third stage of operation of the machine

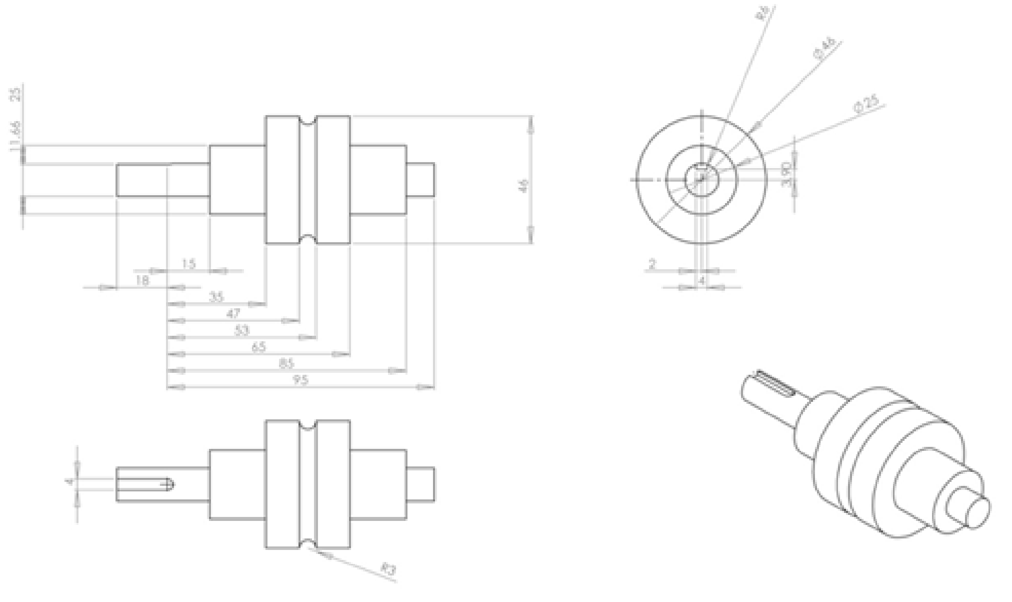
Slip clutch is used to avoid over tightening of the fibre, which may break the fibre. When the pulling torque cross the specified limit of the slip clutch, slipping takes place and the spool-39 looses power and stops till the pull torque reduces. This completes the design and development of automatic preformed yarn machine to produce the preformed yarn. In order to reduce the cost of the machine, readymade sleeve could be used instead of sleeving unit. The same has been incorporated in this work. The final machine, which has been installed, is using readily available sleeve. The detailed drawings of all major parts are given in Figs. 4.39 (All part drawings included, pages 127-144) and photograph of PY machine with splitting and spraying arrangements is shown in Fig. 4.40. The final PY machine with sleeving arrangement is shown in Fig. 4.41.



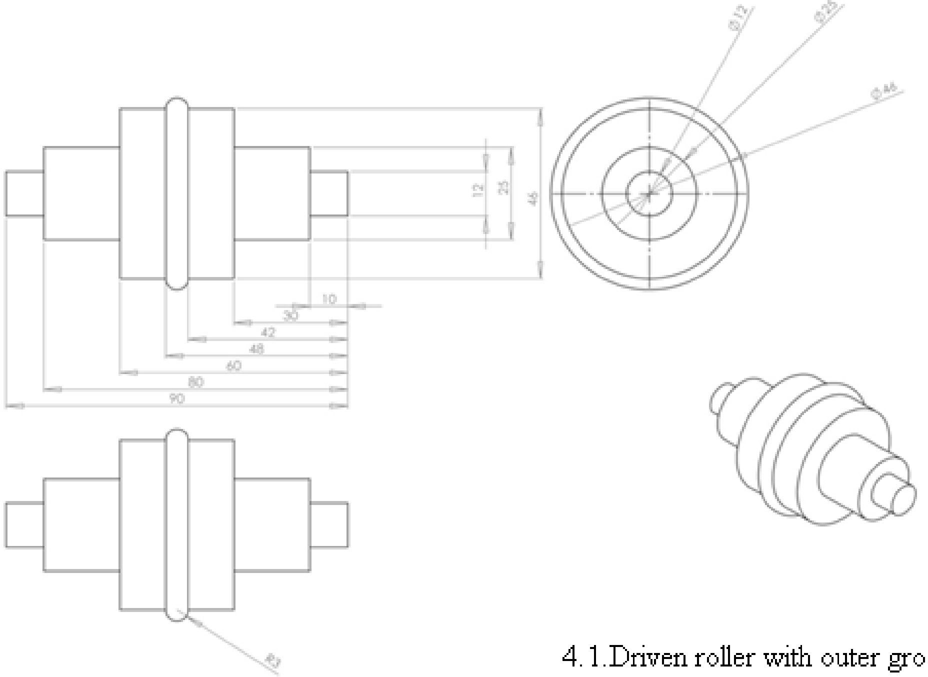
2.Fibre guiding bush



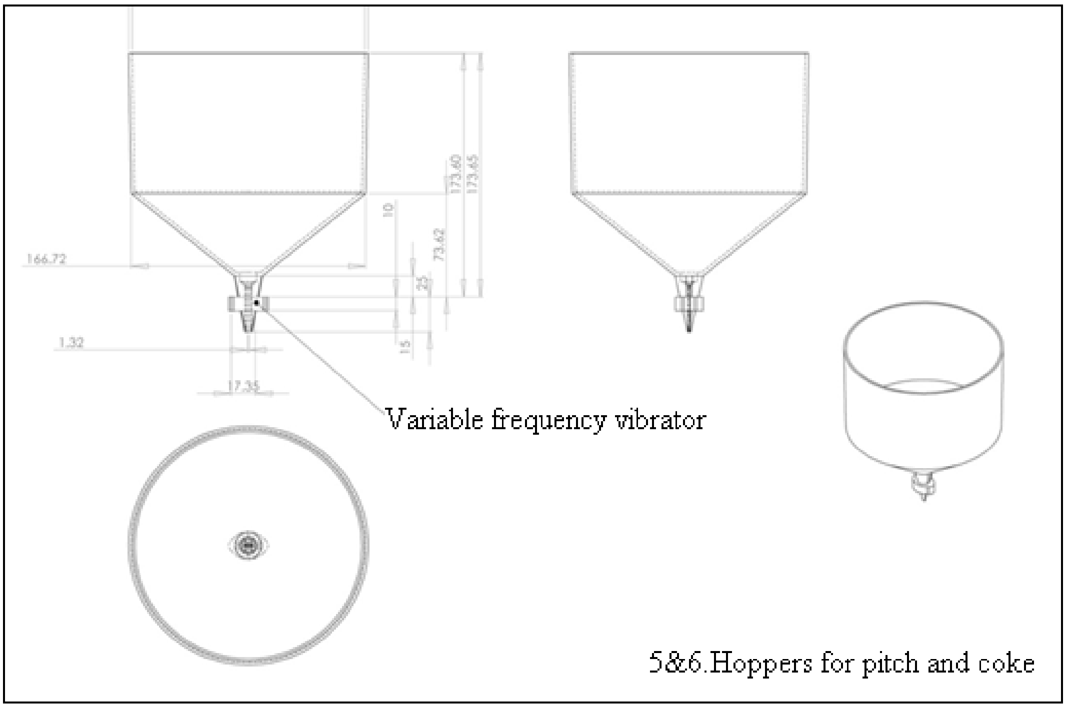
3.Driven and driver roller with rubber layer



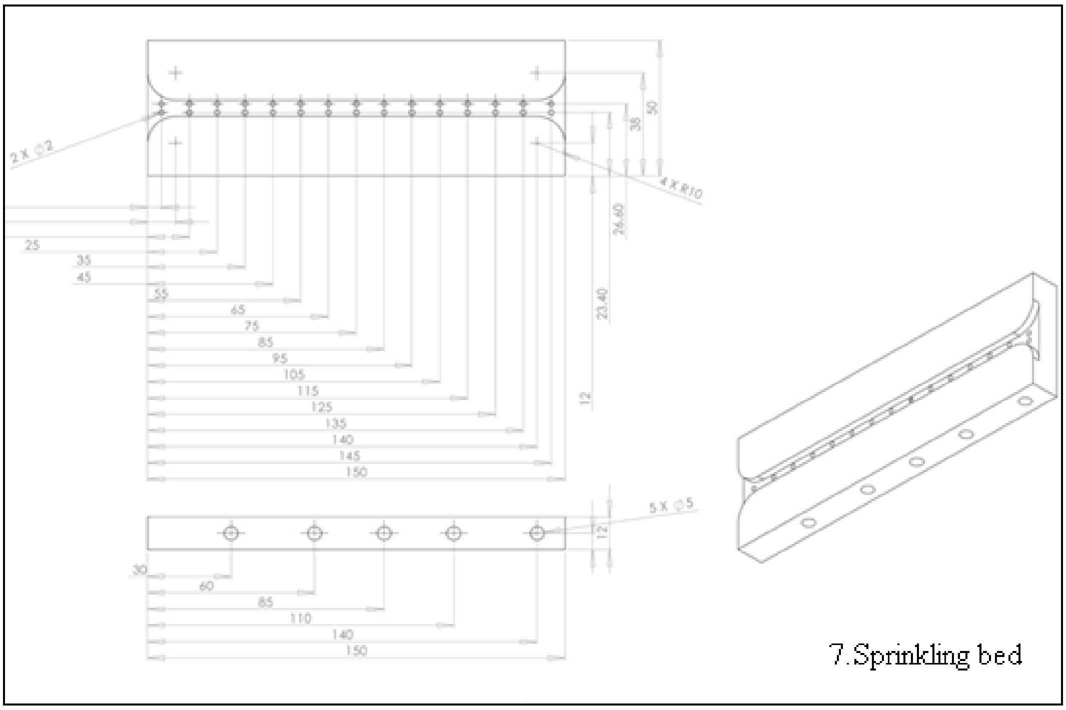
4.Driver roller with spherical groove



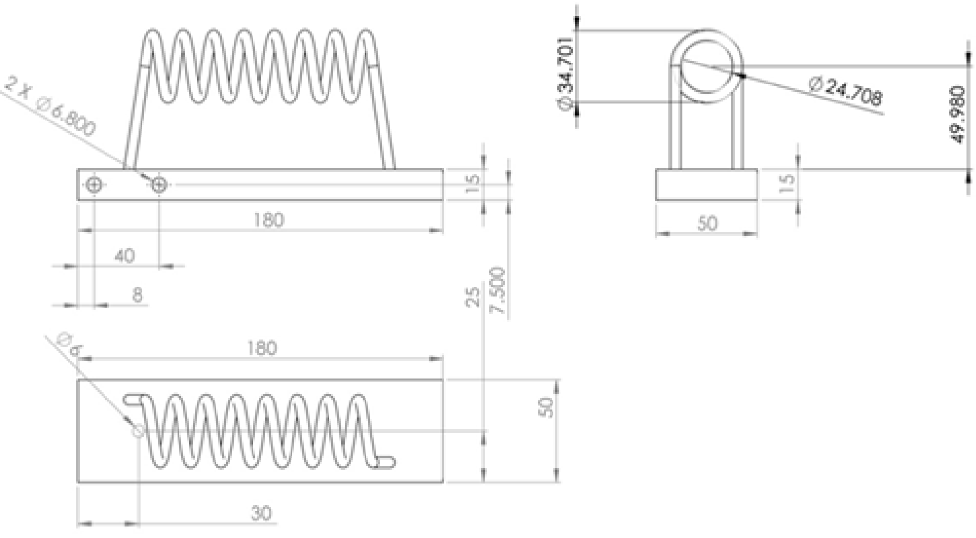
4.1.Driven roller with outer groove



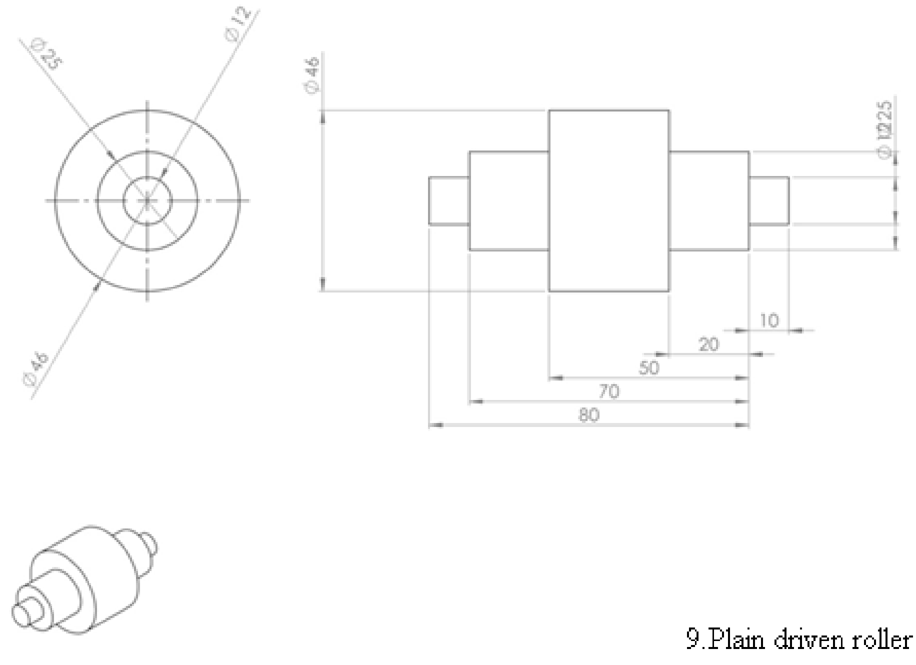
5&6.Hoppers for pitch and coke



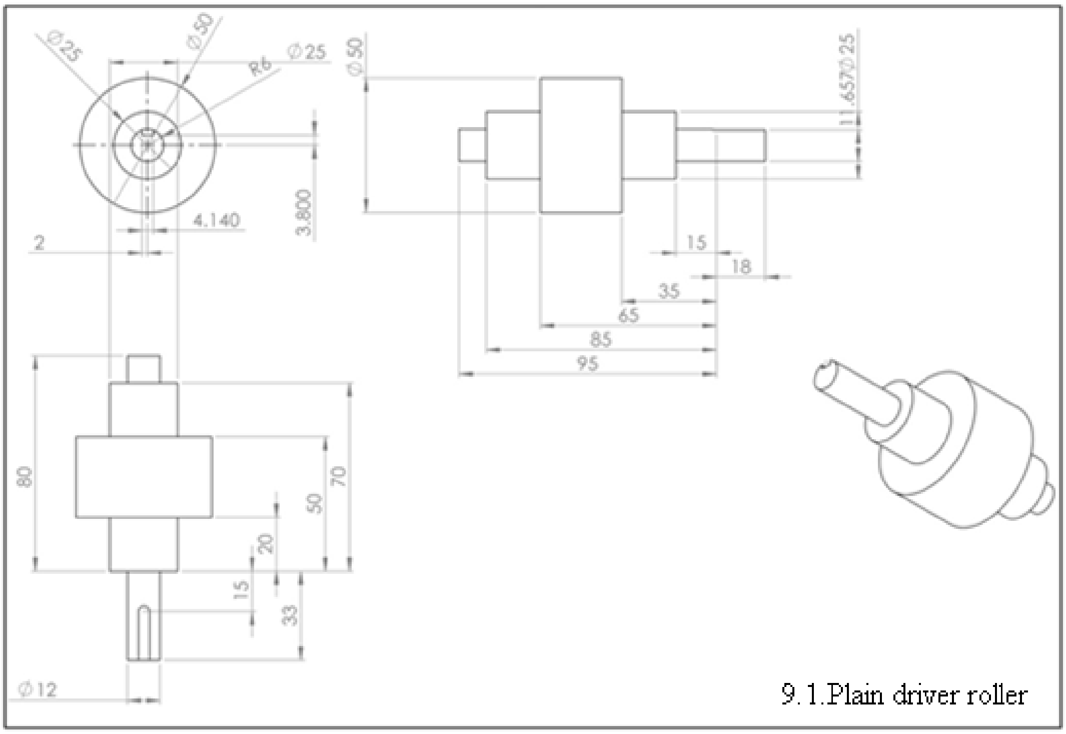
7.Sprinkling bed



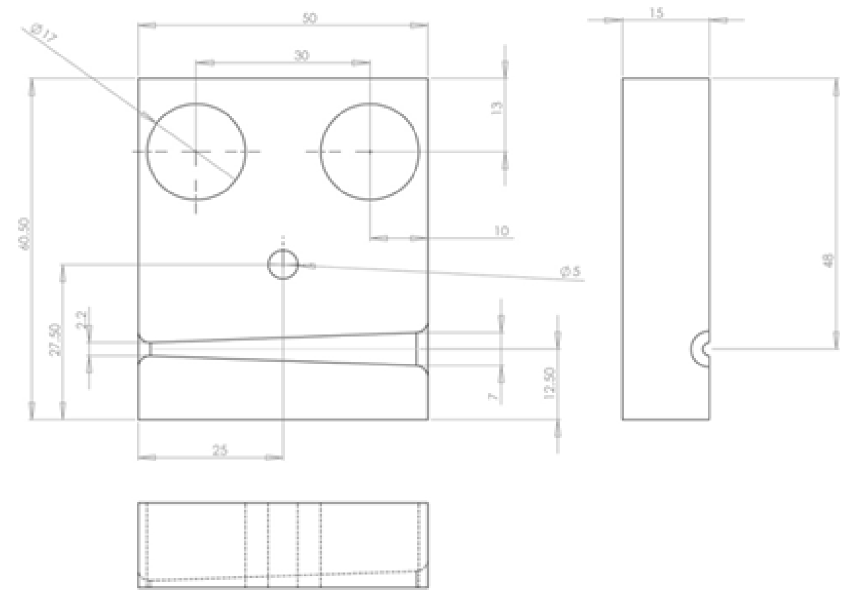
8.Heating coil



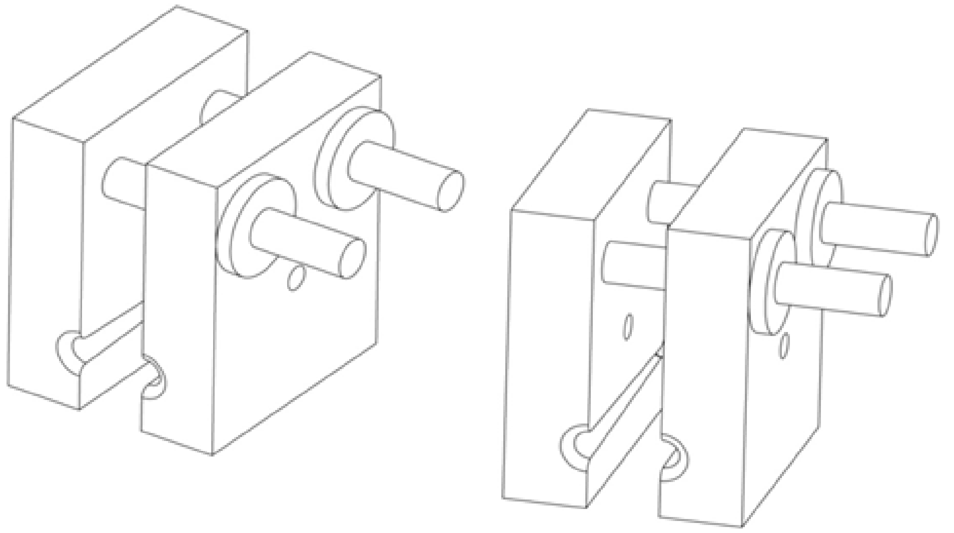
9.Plain driven roller



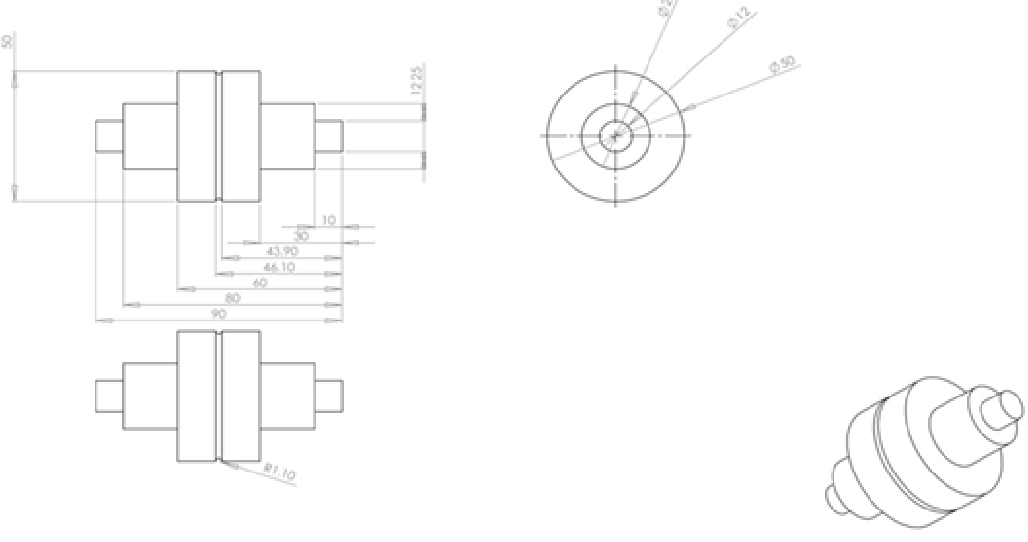
9.1.Plain driver roller



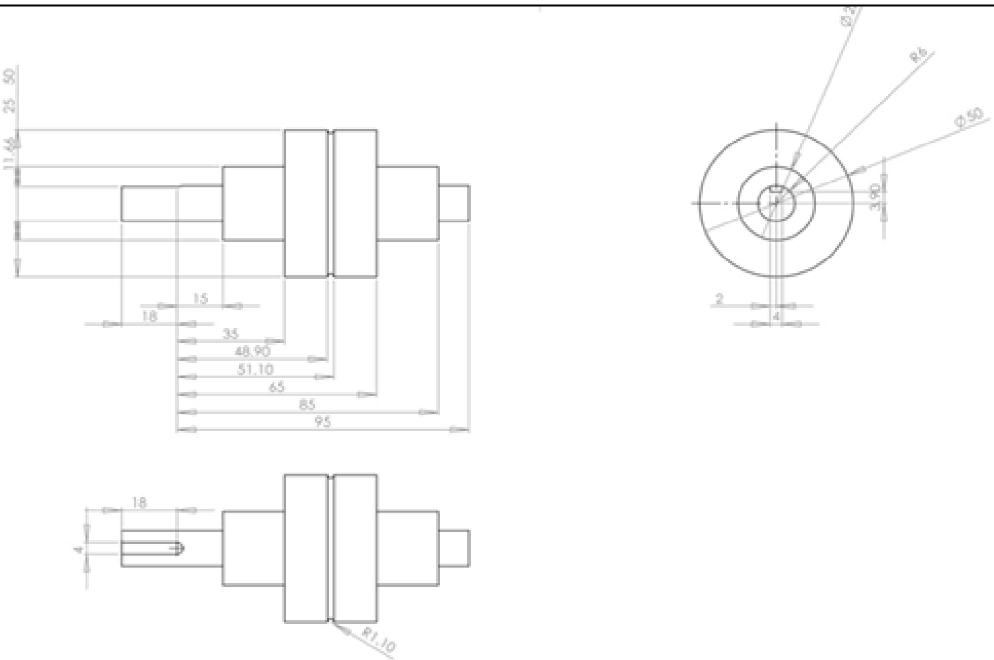
10.Lofted die



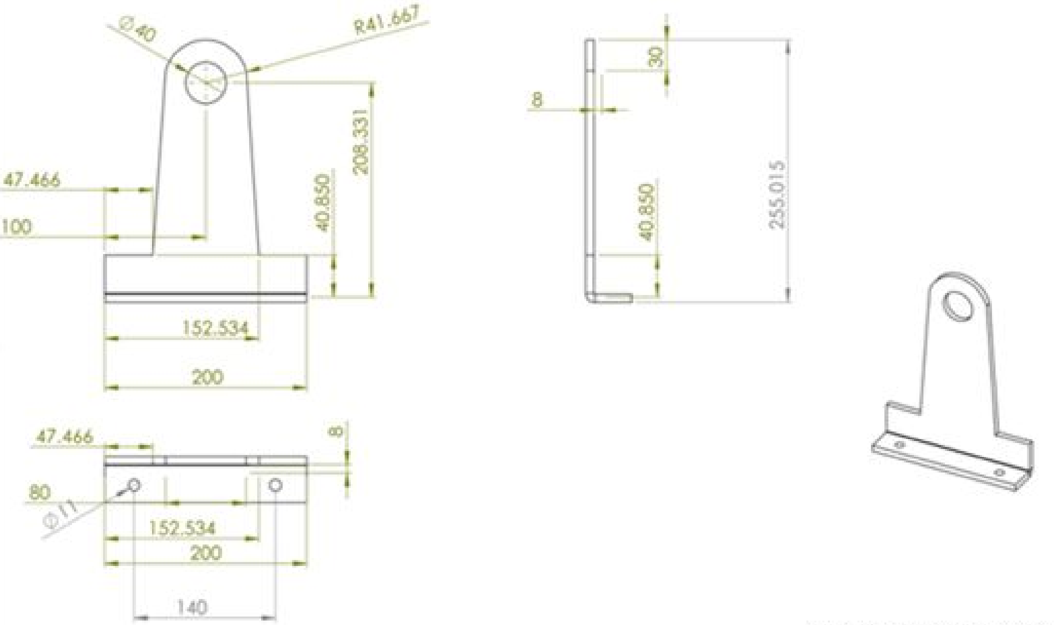
10.1.Pair of lofted die



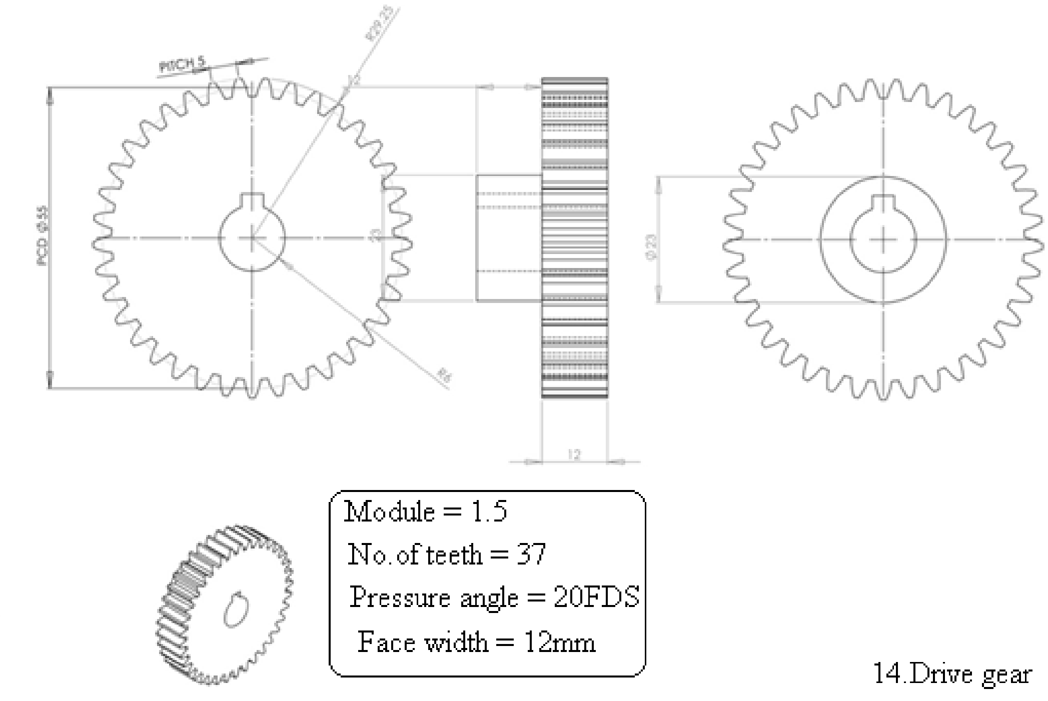
11.Circular groove driven rollers



11.1.Circular groove driver roller

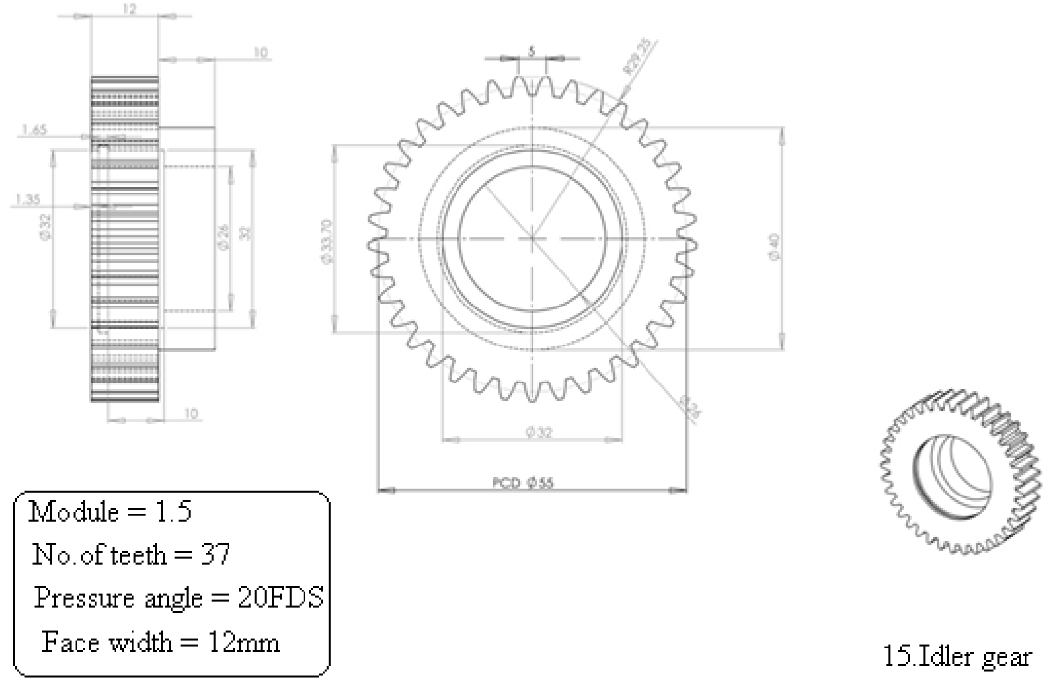


12.Adjacent support



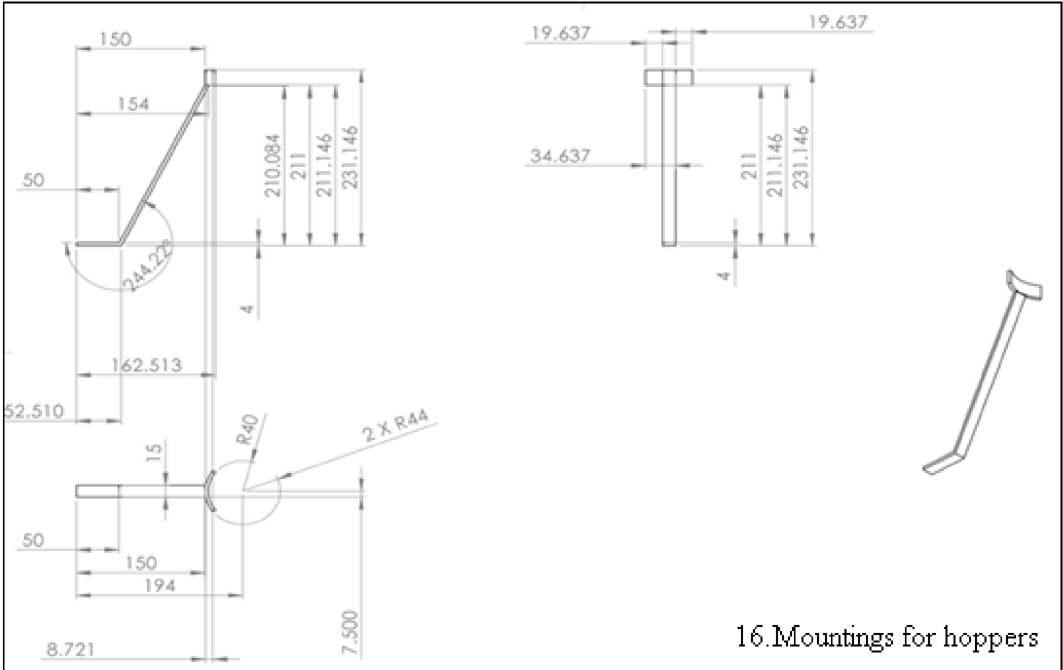
Module = 1.5
No.of teeth = 37
Pressure angle = 20FDS
Face width = 12mm

14.Drive gear

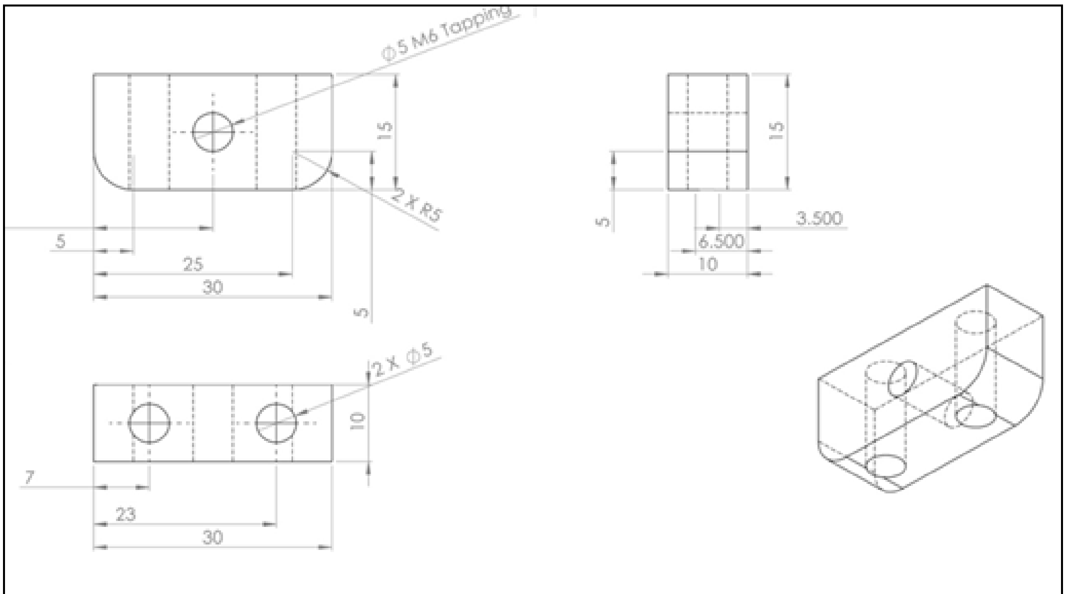


Module = 1.5
No.of teeth = 37
Pressure angle = 20FDS
Face width = 12mm

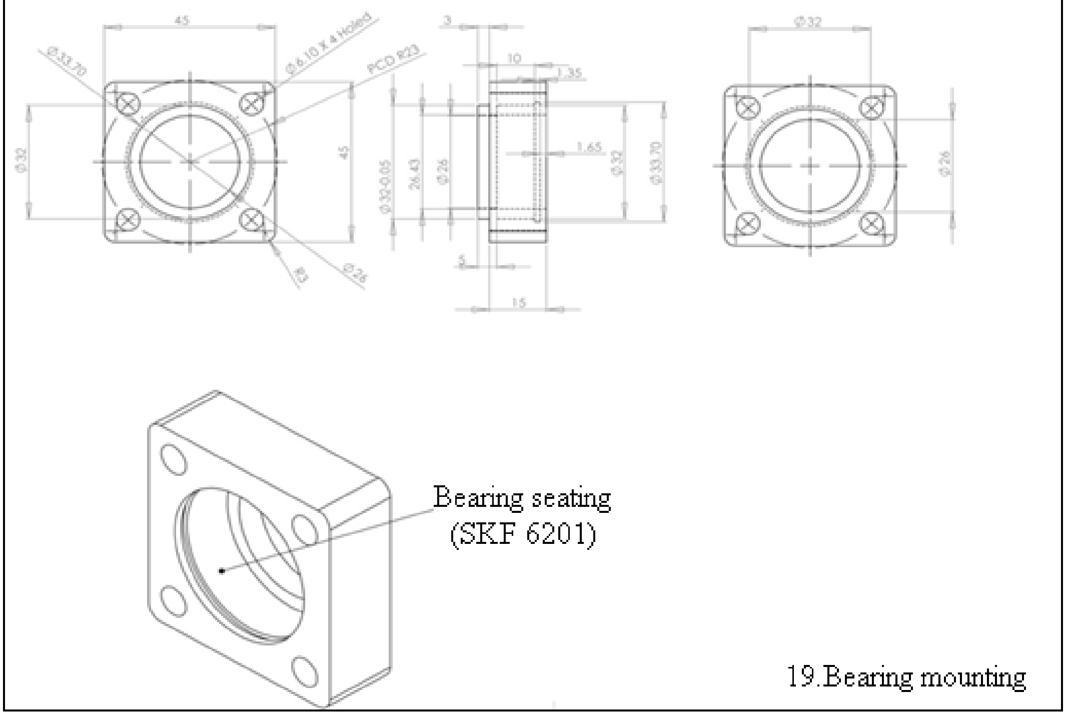
15.Idler gear



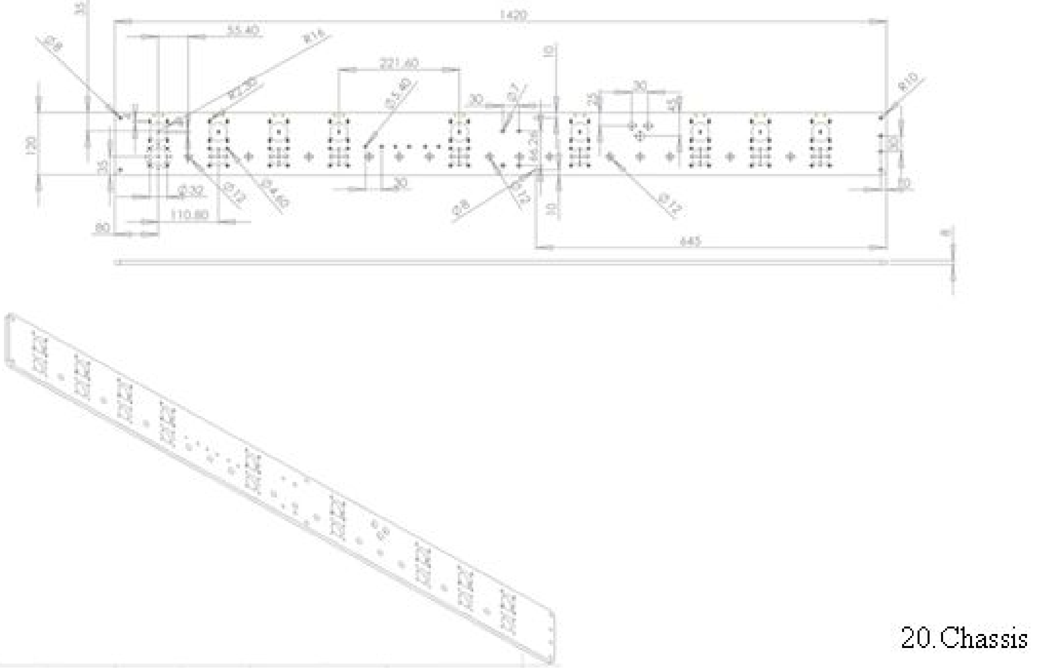
16.Mountings for hoppers



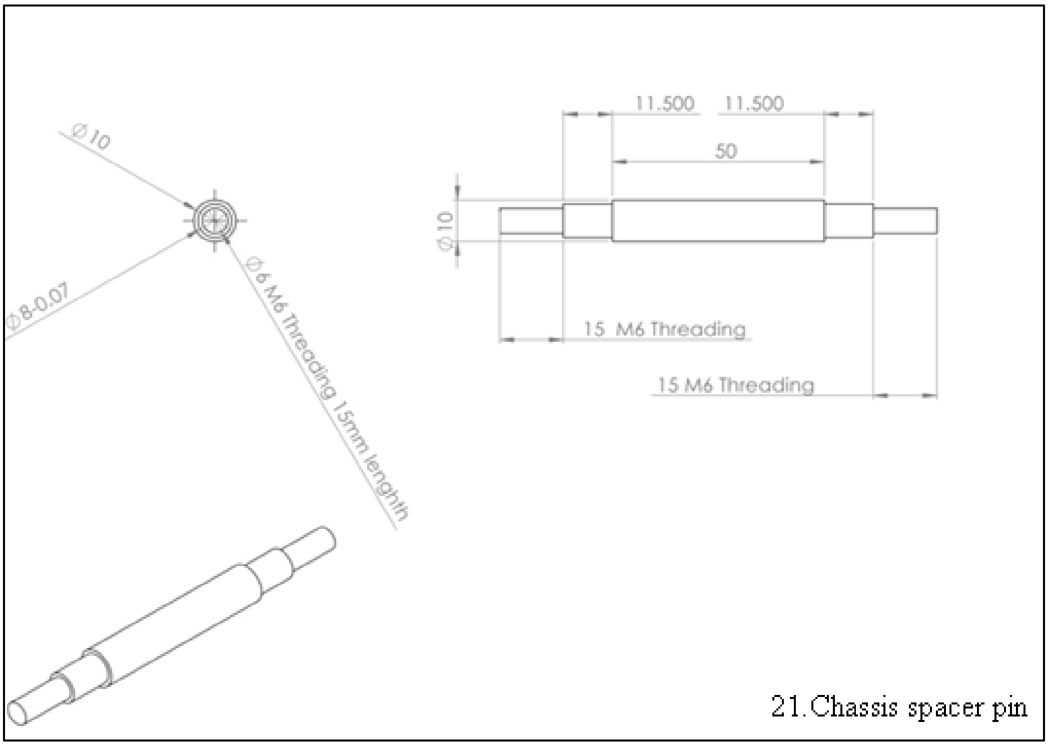
17.Tenser for bearing housing



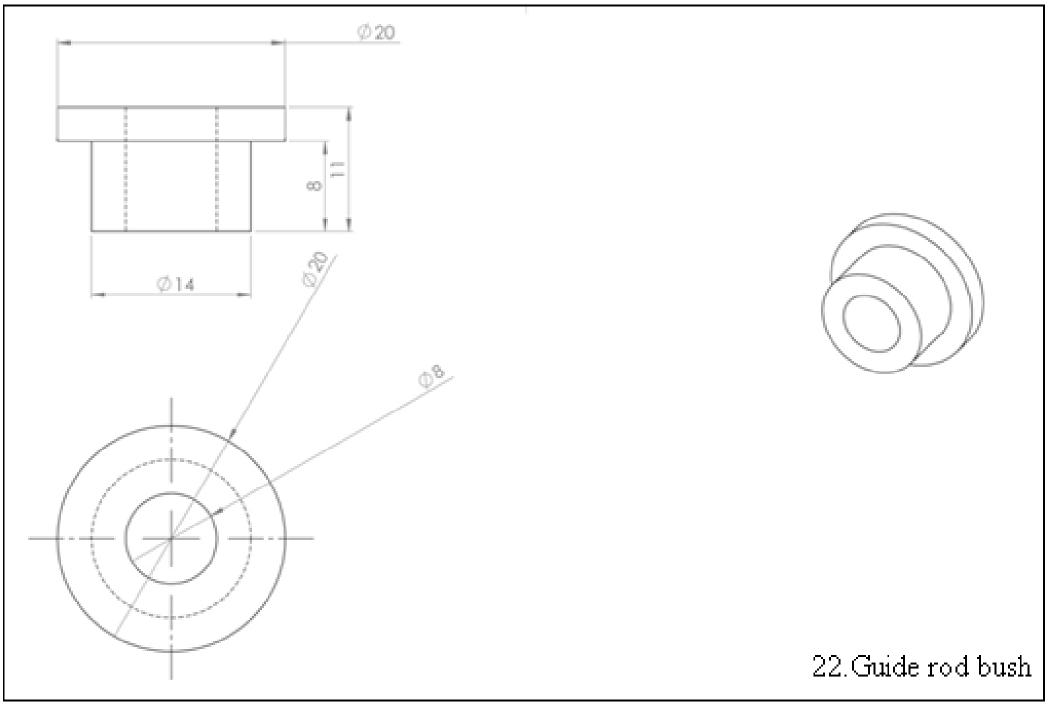
19.Bearing mounting



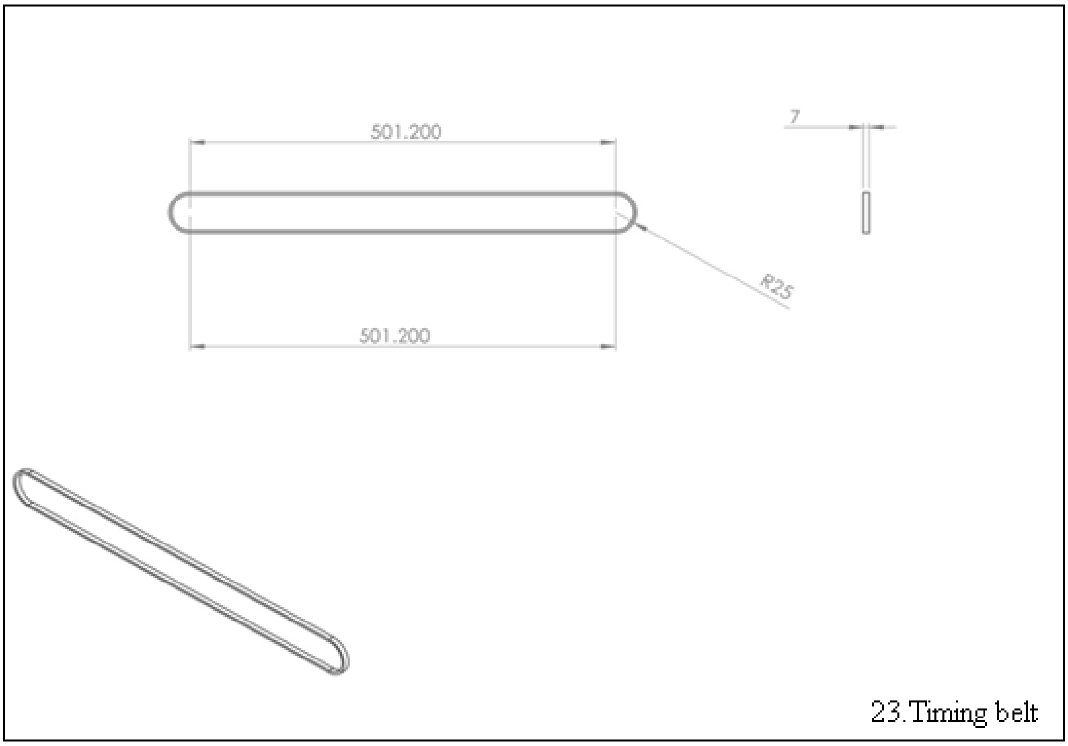
20.Chassis



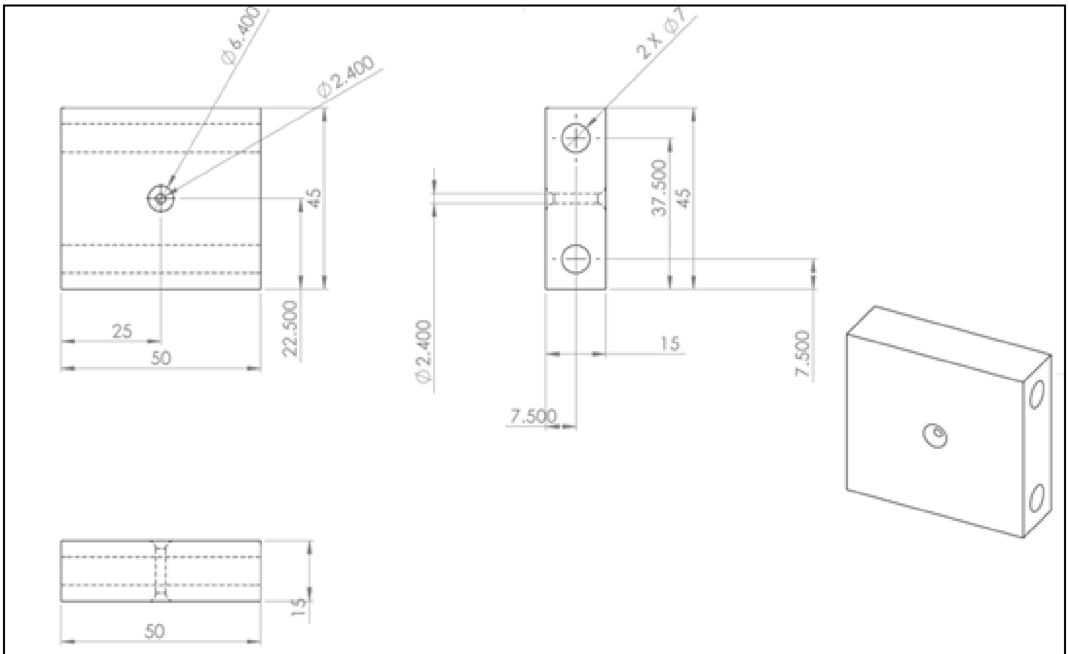
21.Chassis spacer pin



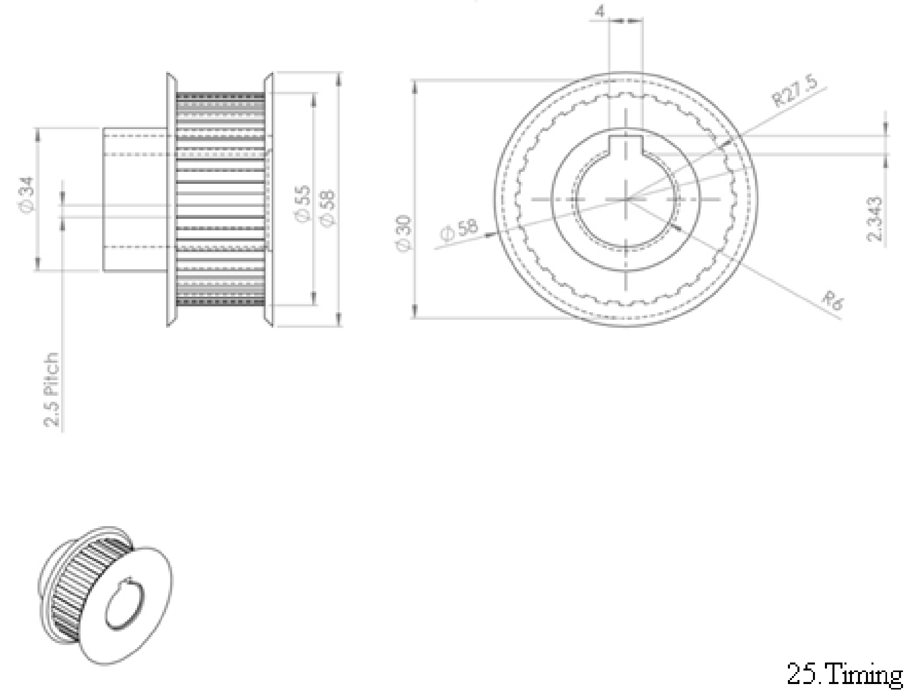
22.Guide rod bush



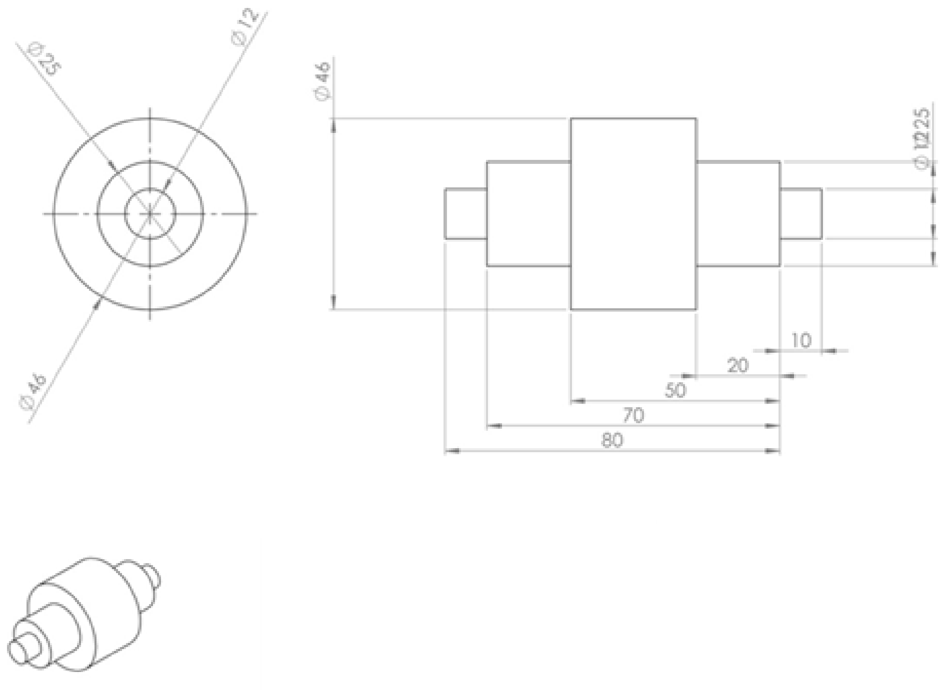
23.Timing belt



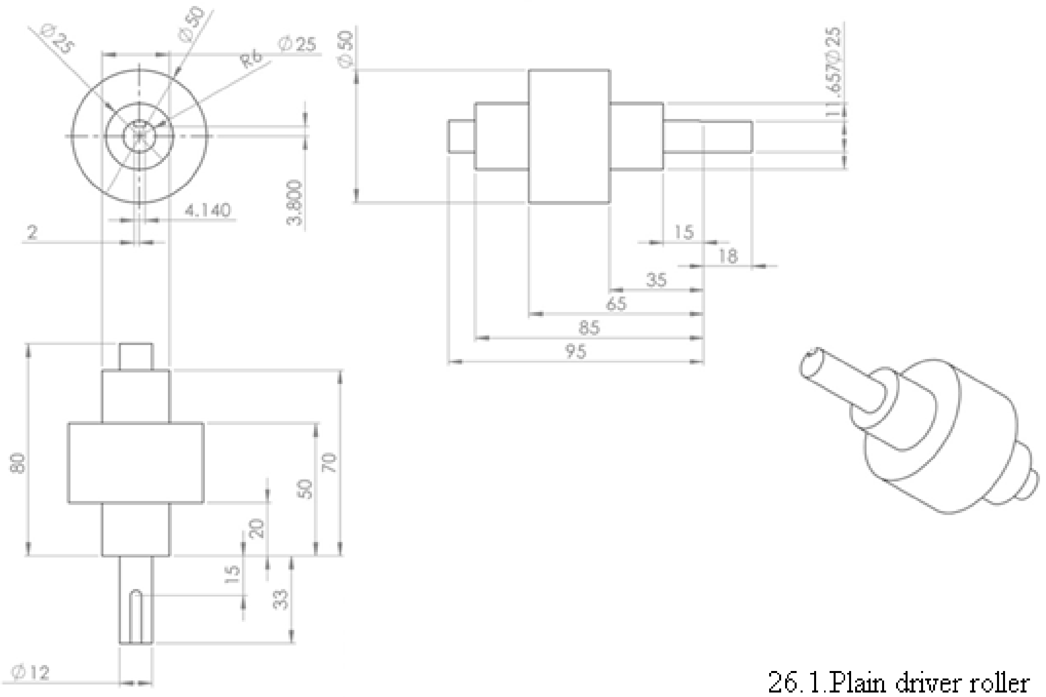
24.Fibre guiding block



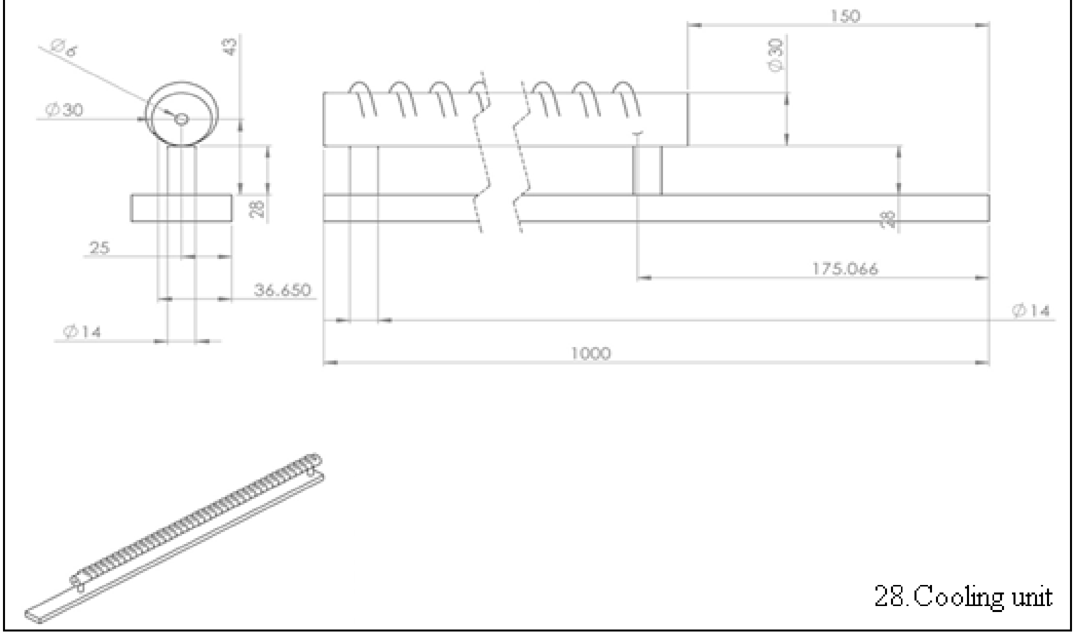
25.Timing pulley



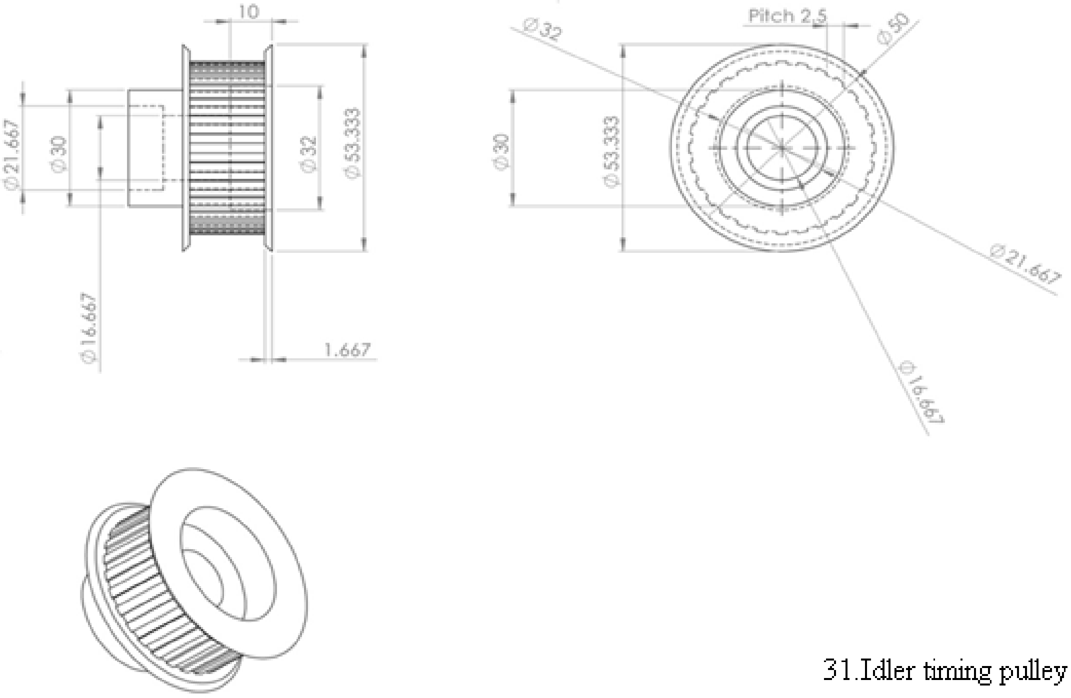
26.Plain driven roller



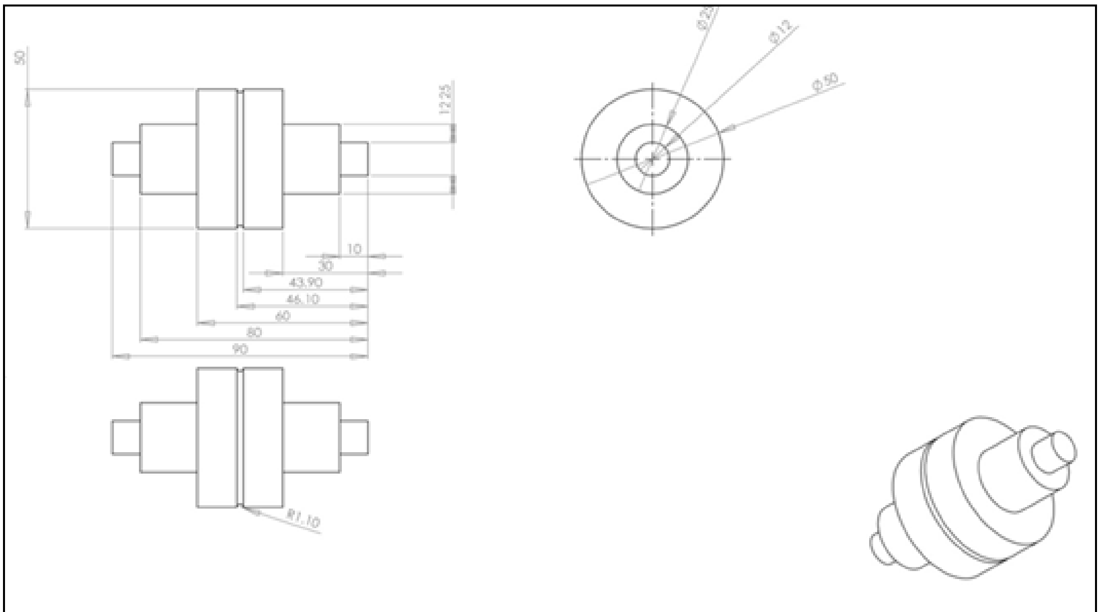
26.1.Plain driver roller



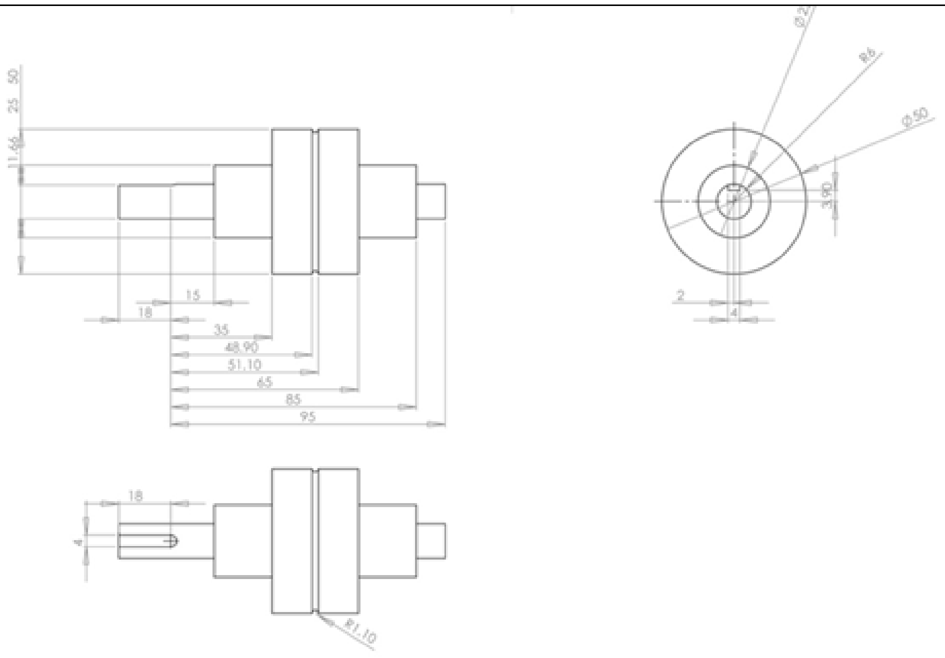
28.Cooling unit



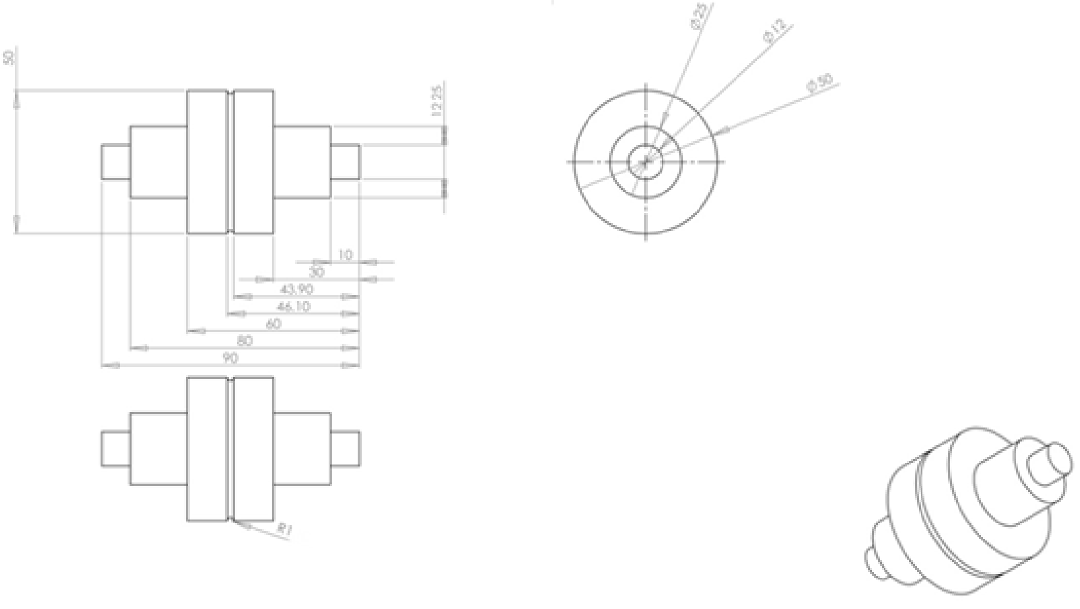
31.Idler timing pulley



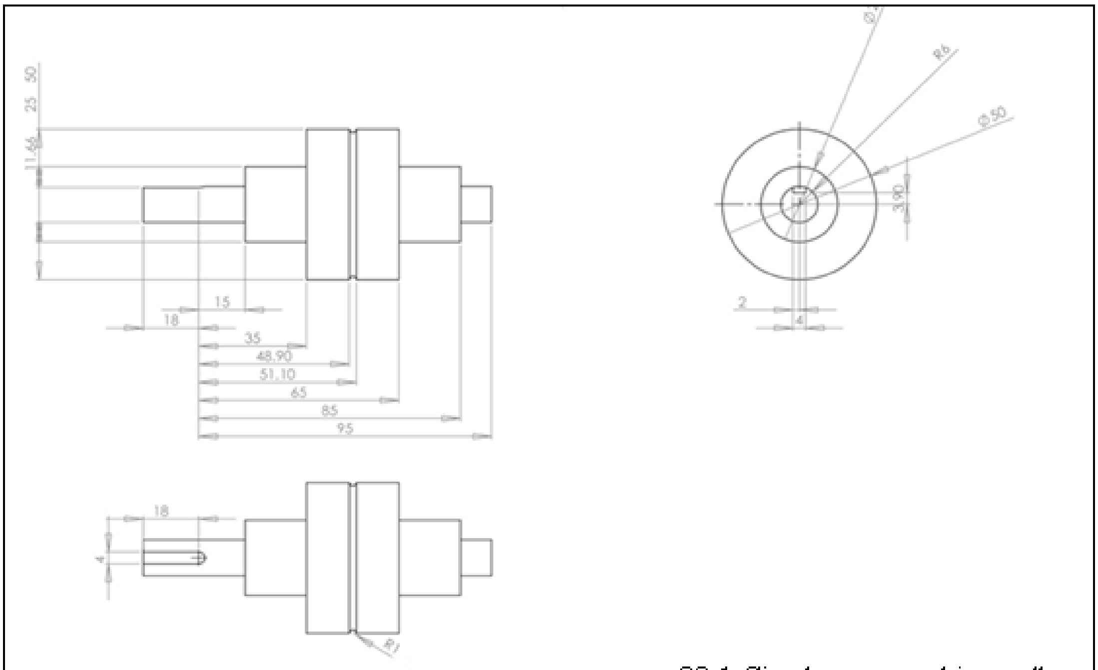
32.Circular groove driven roller



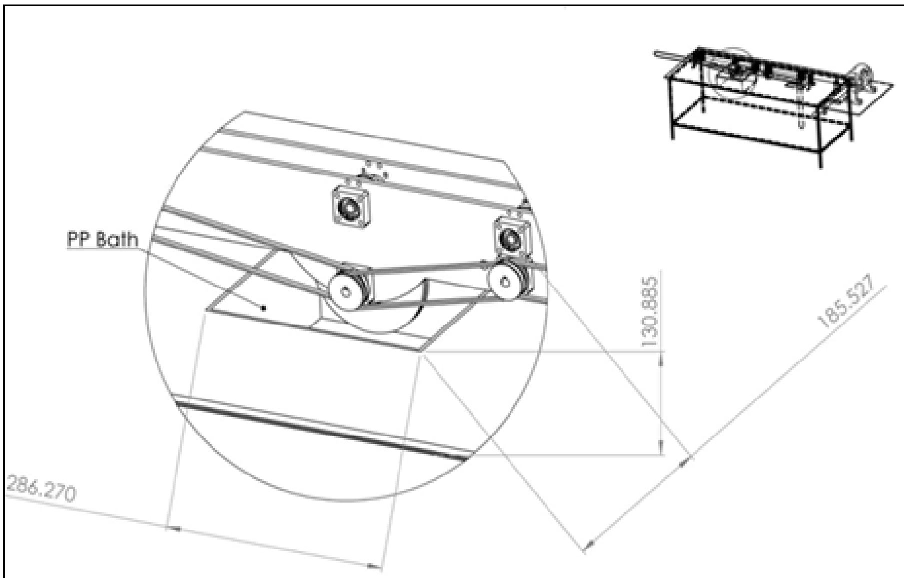
32.1.Circular groove driver roller



33.Circular groove driven roller



33.1.Circular groove driver roller



34.Sleeve bath

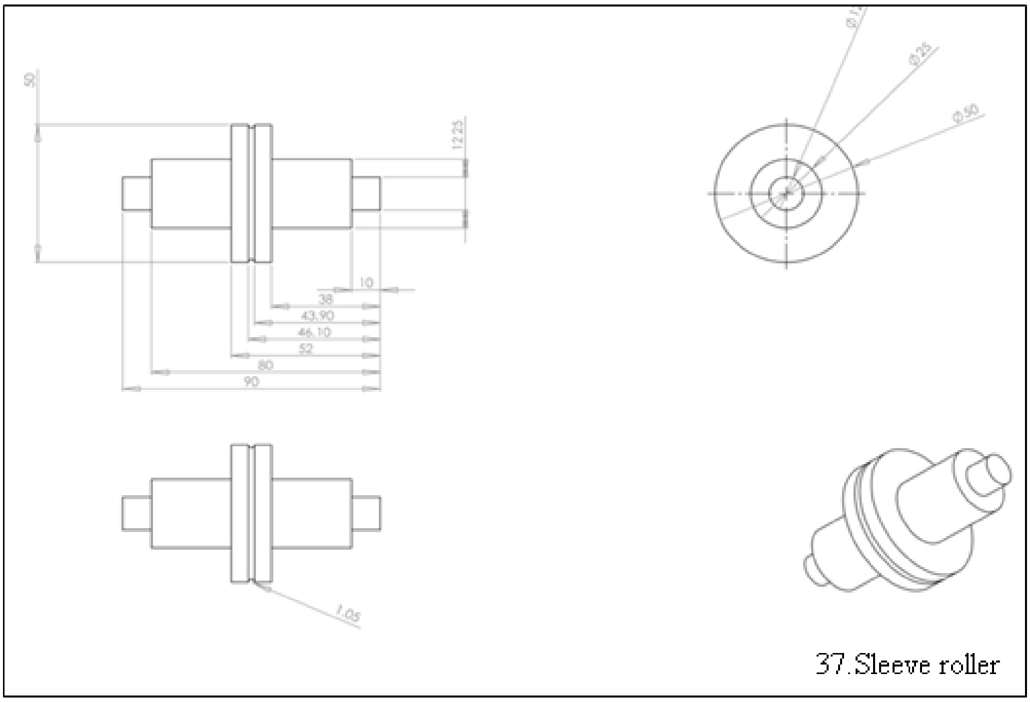


Fig. 4.39 Detailed drawings of automatic PY machine

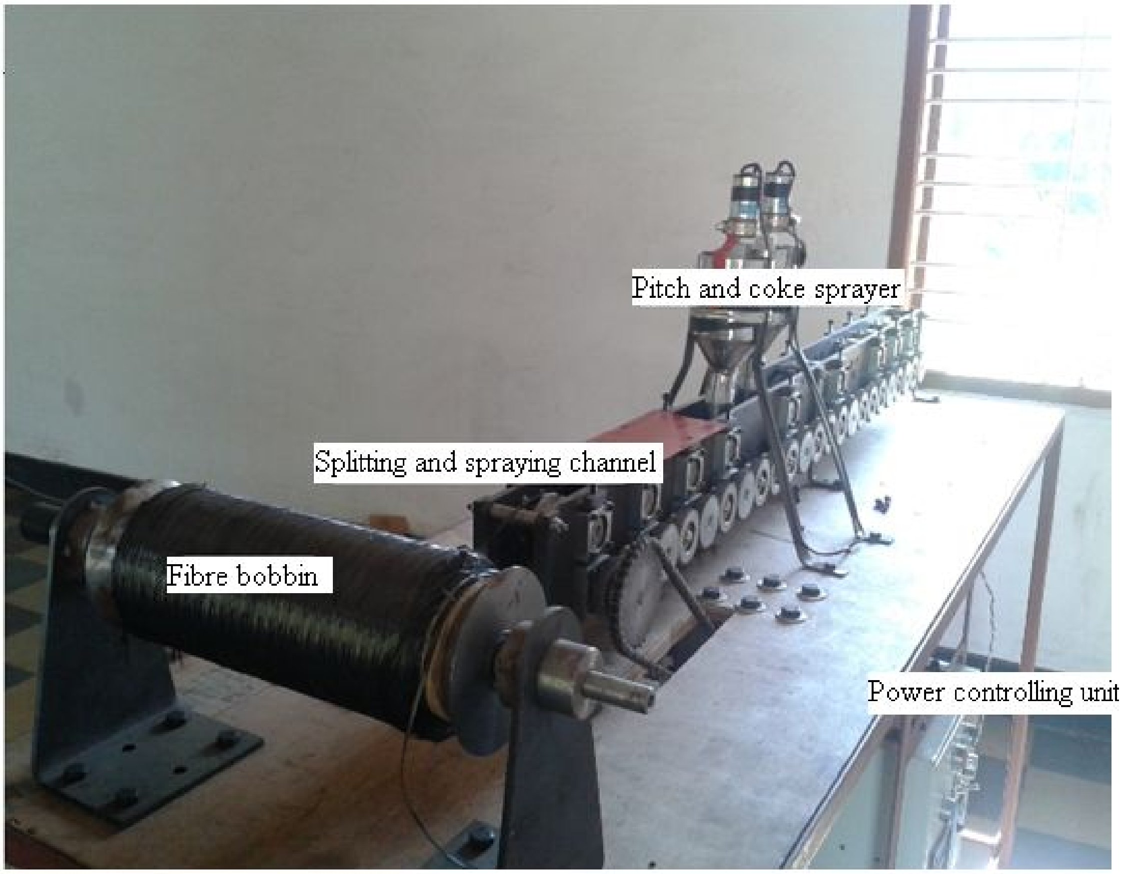


Fig. 4.40 Photograph of PY machine with splitting and spraying arrangement

Fig. 4.41 Photograph of final PY machine with splitting, spraying and sleeving arrangement

4.7 Summary

This thesis comprises of 5 chapters. The conclusions drawn on the experimental results are listed in chapter-5. Referred articles are listed separately. The contents of chapter-1 to chapter-4 are summarized hereunder.

Chapter 1 is of introductory nature about the basics of carbon and forms of carbon, brief information about the importance as well as influence of different precursor material on the mechanical and tribological properties of C/C composites with specific scope and objectives of the present work.

Chapter 2 is a thorough review of the published literature on influence of carbon fibres on the mechanical and tribological properties of C/C composites. This chapter reviews the methods of preparation of conventional C/C composites, characterization techniques for C/C composites, influence of weight fraction on the mechanical properties, wear behavior and work carried out on tribological behavior of C/C composites. After going through the literature, a new manufacturing method of C/C composites i.e. PY method was identified and methodology of conducting the experiments were decided with the specific objectives.

Many have worked on the experimental production of carbon fibre-carbon matrix composites by hot press molding method. They developed a simplified molding method allowing forming of lamination of unidirectional or cloth type carbon fibres with a mixture of coke powder and a carbonaceous binder such as pitch. The latter two materials are used to form the matrix of the C/C composites. The binder which is used for the above simplified manufacturing process of C/C composites has the following characteristic, namely, the binder in matrix softens with rising of temperature in the hot press process and penetrates into the fibre bundles. In this simplified process, the binder is prevented from the complete penetration because of its high viscosity, from which certain voids are formed in the matrix and therefore, a much stronger C/C composites will not be obtained. Elemental carbon oxidizes readily in air at temperatures exceeding 400°C, forming gaseous oxides which offer very mild, if any, inhibition to further oxidation. So, the subject of oxidation protection is a subject in itself and considerable amount of work is being done currently. In order to have a clear understanding of the performance and also the merits and demerits of the various methods of protection, the kinetics of decomposition of uninhibited carbon is essential. Further, for exceedingly short time applications such as nose-cones or stabilizing fins of missiles, wherein the life-time is of the order of a minute, protection is not essential and uninhibited carbon-carbon is the material of choice.

This chapter deals with both conventional and PY method to produce C/C composites and their mechanical and tribological properties are included. In order to avoid aforementioned problem, a new method was developed which is called composites preparation with preformed yarn; a preformed yarn (PY) already contains the components of the matrix within the carbon fibre bundles. The purpose of this research is to explain the newly developed manufacturing process of preformed yarns with the machine and also proving the better mechanical properties of the new C/C composites thus manufactured from the preformed yarns. In the present study, an attempt is made to construct preformed yarn machine model and to introduce the PY method especially in India – the said machine has not been available so far to synthesize C/C composites for many applications.

In chapter 3, the present work deals with some aspects of fabrication and characterization of C/C composites with special emphasis on the PY production model and mechanical properties as well as on the C/C composite’s microstructure. Manufacturing of PY to synthesize C/C composites by varying the weight percentage of fibre was mainly considered. Developed PY machines are both manual type and automatic types. Properties of manual type PY C/C composites are compared with conventional C/C composites; then superiority of the new class composites has been demonstrated. The PY developed in the current study was carbon fibre bundle surrounded by coke and pitch which were enclosed in Nylon-6; three types of samples with fibre percentages of 30wt%, 40wt% and 50wt% respectively, were fabricated. The PY was subjected to hot pressing at 600^0C followed by final heat treatment at 2500^0C. Subsequently, in order to eliminate the voids, sufficient number of pitch impregnation cycle was performed on the samples, by hot isostatic pressing until density of 1.7g/cc was reached. PAN based PY C/C composite was fabricated using the hot press impregnation-carbonization route. The matrix precursor was petroleum coke and coal tar pitch was used for densification. Characterization of the specimens was then carried out in terms of scanning electron microscopy (SEM) to identify the matrix morphologies formed at various stages of processing. The degree of graphitized composites depends on the matrix precursors. The extent of graphitization was investigated from the X-ray diffraction data.

The dimensions of samples for the determination of hardness, impact energy, flexural properties, thermal analysis, dry sliding wear behavior and worn surface study are clearly explained. The behavior of carbon-carbons at temperatures in the range of 400°C to 1000°C has been studied using thermogravimetry. The experiments were conducted and are explained to show the effect of fibre content on the mechanical properties, and thermal properties of PY C/C composites. The method of using computerized universal testing machine and pin-on-disc wear testing machine are also discussed. Observation of worn surface morphology before and after the wear test was carried out using SEM. Constructional feature of PY making machine model and experimental procedure for the evaluation of mechanical properties is clearly mentioned in this chapter. The detail of part drawing of manual type PY model is given in this chapter.

Chapter 4 addresses the results of the tests done to determine the mechanical, and thermal properties of PY C/C composites. Carbon-carbons find increasing use as primary load bearing structures and material properties such as hardness, compressive strength, impact, flexural, creep and wear properties are considered for designing these composites. As a part of the present investigation tests were performed to determine these and also to study the failure mode. An attempt to study the effect of configuration of the loading with respect to the axis of the load was made. The failure patterns have been studied using SEM to understand the fracture features. Keeping in view the high temperature applicability of the carbon-carbons, oxidation strengths were evaluated up to a temperature nearing 1000°C.

The variation of mechanical properties, thermal, specific wear rate and friction coefficient as a function of fibre content is shown using graphs. SEM photographs of fracture surfaces and worn surface morphology after the various tests are given and effort has been made to correlate the obtained results with the SEM images. The chapter also presents the effects of normal pressure, and sliding velocity on the dry sliding wear behavior of the samples. The effects of increase in fibre content and functionalization of carbon fibre on mechanical, thermal and dry slidng wear behaviour of PY C/C composites are discussed. The identification of type of wear mechanism has been explained by worn surfaces-study. As per the experiment 50 wt% carbon fibre composites showed the best properties among the three different samples. It was decided to develop automatic PY machine for the composition of 50 wt% carbon fibre, 45 wt% matrix and 5 wt% sleeve in order to minimize the processing steps of C/C composites for various applications. The Chapter 5 gives the conclusions drawn from the present work.

CHAPTER-5

CONCLUSIONS

Preformed yarn producing machine has been successfully developed. Addition of fibre on mechanical, thermal and tribological properties of PY C/C composites has been investigated. Having discussed the results of the experiments the following conclusions are recorded:

i. The design and development of manual PY model were carried out. Preformed yarns containing three different weight percentage of carbon fibre were successfully drawn from the PY model to produce the PY C/C composites.

ii. Increasing in the weight fractions of carbon fibre in preformed yarn C/C composites have shown remarkable increase in various mechanical, tribological and thermal properties of composites over reported conventional C/C composites.

iii. From the hardness, compression, flexural and impact studies, it was observed that as the weight fraction of carbon fibre increased the material properties also increased. But, these properties are still less than the expected values because the extent of graphitization was not completed. This could be observed from glassy phases present in the XRD pattern.

iv. As the percentage of fibre increased, the creep resistance of the composites increased for sample one and two containing30wt% and40wt% fibre respectively but for sample containing 50wt% fibre, three was slight decrease. This was due to incomplete graphitization and lack of binders in higher fibre-content specimens, which created many voids in these specimens, thereby weakening interfacial bond between fibre and matrix.

v. By observing the wear data, we can conclude that as the percentage of fibres increases the wear resistance of the composites also increased for all the three weight-percentages of C/C composites. This is because of the ability of the C/C composites to operate in high temperature and various wear conditions. Increasing the weight percentages of carbon fibre contributed significantly in reducing friction and exhibited better wear resistance. The resistance to wear is maximum in 50wt% carbon fibre composite as compared to 40wt% and 30wt% carbon fibre composites.

vi. It has been observed in the TGA curves of all three PY C/C composites samples that as the percentage of carbon fibres increased the residual of PY C/C composites increased slightly from sample one and two but a large amount of residual of PY C/C composites remained in sample three, resulting in an increase in the thermal degradation resistance of C/C composites. As this is a high temperature material used for aerospace applications, this is the most important property.

vii. The properties of PY C/C composites are dependent on fibre weight fraction, fibre distribution, binder ratios and various processing parameters. Unlike polymer matrices, carbon matrices contribute significantly to the ultimate properties of the composites, especially in the case of pitch and coke-derived carbon matrices.

viii. The automatic PY production machine has been successfully designed and drawings are presented for one composition of 50wt% fibre, 45wt%matix and 5wt% sleeve, which gave higher properties among the three C/C samples. The matrix contained 50 wt% coke and 50 wt% pitch.

SCOPE FOR FUTURE WORK

Following suggestions are made for carrying out the investigative work in future in this field:

i. By listing out the draw backs of the existing PY model, and then modifying it accordingly, the PY production rate could be increased that would enhance the performance of the machine.
ii. The diameter of yarn and C/C compositions can be varied with a slight modification in the existing model by a properly designing the rollers.
iii. Optimization of diameter and composition need to be done using suitable simulation software.
iv. Fibres used in the present study are untreated ones. Chemical treatment of the fibre surface could improve the oxidation resistance in the matrix as well as bonding between matrix and fibres. There is a scope for further enhancement of the mechanical, wear and thermal behavior of C/C composites.
v. Wear is a complex phenomenon in which fracture also plays a very important role. Growth of the crack in sub-surface area, which is controlled by fracture toughness of the material, is an important aspect to be studied. It is necessary to find out the relation between the wear properties and fracture toughness of the material so that resistance can be improved by increasing the fracture toughness of the material. The method of increasing the fracture toughness in C/C composites is an important area for future studies and
vi. Compared to unidirectional fibres, such as short fibres, the improvement in wear resistance obtained by the use of unidirectional is modest. It is a traditional route to integrate various functional fibres. To date, however, little effort has been directed towards the development of hybrid composites by integrating nano-fibres with micro fibres. There is scope for developing wear and creep resistant C/C composites by using a combination of nano- and micro- fibres.

APPENDIX I

Research Work Planned/Accomplished

YEAR	**WORK PLANNED/ACCOMPLISHED**
06-07-2007	Registered for PhD.
July 2007-July 2008	Course work of PhD curriculum completed and obtained **CGPA of 9.25**.
18-06-2009	Research proposal given
30-12-2010	First progress seminar given
17-05-2012	Second progress seminar given
27-12-2012	Pre-synopsis seminar given
04-04-2013	Thesis submission

Specimen Calculation

(i) Calculation of Flexural Strength (S) Of C/C Composites

$$S = \frac{3PL'}{2bd^2}$$

S= flexural strength in MPa

P = Maximum load in kN = 1.5 kN

L`= Support span in mm = 90 mm

b = Width of the beam in mm = 12.4 mm

d = Depth of the beam in mm = 9.4 mm

$S = \frac{3 \times 1.5 \times 90}{2 \times 12.4 \times (9.4)^2}$ = 0.18487 kN/mm^2 = 184.87MPa

(ii) Flexural Modulus (E)

$$E = \frac{L^3 m}{4bd^3}$$

L= Support span in mm = 90 mm

m = slope of load-deflection curve = $\frac{\Delta P}{\Delta \delta}$ = $\frac{0.8 - 0.2}{0.45 - 0.15}$ = 2

b = Width of the beam in mm = 12.4 mm

d = Depth of the beam in mm = 9.4 mm

$E = \frac{90^3 \times 2}{4 \times 12.4 \times 9.4^3}$ = 35.39 kN/mm^2 = 35390 MPa=35.39GPa

(iii) Charpy Impact Strength Of C/C Composites

(Energy absorbed in Joules (J))/ (Cross-sectional area of specimen in mm^2)

Energy absorbed = 2.2 J

Cross-sectional area = (10 x10) = 100 (mm^2)

Impact strength = (2.2) / (100) =0.022 J/mm^2 =22 kJ/m^2

(iv) Calculation For Wear Properties Of C/C Composites

a) Specific wear rate (W_s)

$$W_s = \frac{\Delta m}{\rho \times F_N \times L} \qquad (mm^3/N\text{-}m)$$

Δm = worn specimen mass (g),

= Initial weight (W_1) –Final weight (W_2)

= 3.259- 3.2368

= 0.0222 g

ρ = measured density of specimen (g/mm^3)

= W_1 (g) /volume of specimen (mm^3)

= 3.259 / 1917.48

= 0.0017 g/mm^3

F_N = Normal force in Newton (N) = 4 Kg = 39.24 N

L = Sliding distance (m) = $\pi \times D \times N \times T$

D = Track diameter in m = 0.08 m

N = Speed in rpm = 200 rpm

T = Time in min. = 60 min.

L = 3.142 x 0.08 x 200 x 60

= 3016.32 m

$$W_s = \frac{0.0222}{0.0017 \times 39.24 \times 3016.32}$$

= 1.1 x 10^{-4} mm^3/N-m

b) Coefficient of friction (µ)

µ = Frictional force (N)/ Normal force (N)

= 22.5 / 39.24

= 0.573

c) Normal pressure (p)

p = Normal force (N)/ Cross-sectional area of specimen (mm^2)

= 39.24/ (7 x 7) = 0.8 N/mm^2

=0.8 MPa

d) Sliding velocity (v)

v = Sliding distance (m) /time (s)

$= \pi \times D \times N \times T$

D = Track diameter in m = 0.08 m

N = Speed in rpm = 200 rpm

T = Time in min. = 60 min.

L = 3.142 x 0.08 x 200 x 60

= 3016.32 m (Sliding distance for 1 h.)

v = 3016.32/3600

= 0.83 m/s

(v) Design Of Materials

a) Design of driven and driver roller. [Data hand book used- K. Mahadevan and Balaveera Reddy, 1987]

$P = 2\pi NT/60 \times 1000$

$$T = \frac{60 \times 1000P}{2\pi N}$$

T = 60 x 1000 x 373/$2\pi \times 60$

T = 59364.79 N-mm

The torque equation is given by

$T/J = \tau /R$

T = Torque in N-mm

J = Polar movement of inertia

J = $\pi d^4/32$ in mm^4

R=Distance from neutral axis = d/2 in mm

τ = Shear stress developed, normally shear stress = half of the normal stress

Then $\tau = \sigma/2$ in N/mm^2 or MPa

Then equation (1) becomes

$T/\ \pi d^4/32 = \tau\ /d/2$

$\tau = 16T/\ \pi d^3$

$d = \sqrt[3]{16 \text{ x } 2 \text{ x } T/}\ \pi\ \sigma$

d=16 x 2 x (59364.79/3.14) x 416

d =11.32mm.

From the table 3.4 chosen slandered diameter of shaft = d =12mm

First step of the roller is = d x 2 + 1 = 12 x 2 + 1 = 25mm

Second step of the roller is = First step of the roller x 2 = 25 x 2 = 50mm

Then 2mm rubber layer on 50mm diameter roller, hence outermost diameter or Final diameter of roller = 50+2 = 52mm.

b) Design of key

From table 4.1

For shaft with d = 12mm selected parallel key.

Used width = b = 4mm

Height = h = 4mm

Standard length, minimum = 8mm, maximum = 45mm.

Here chosen length = 18mm

c) Design of ball bearing

For shaft with d = 12mm

From table 16.9a

Chosen 12BC02- SKF 6201 series with dimensions as follows

D = 32mm, d = 12mm, B = 10mm.

d) Design of spur gear

Torque transmitted

T = 59364.79N-mm

$m^2 = [2Mt / \sigma_d C_v (b/m) \pi y z]$ ----------------- (12.16)

$m = [2Mt / \sigma_d C_v K \pi y z]^{1/2}$

$m = [2Mt / \sigma_d C_v K Y z]^{1/2}$

m = module in mm

M_t = Transmitted torque in N-mm

σ_d = Allowable static stress = 549 MPa, from Table 12.7

C_v = velocity factor = 0.5

$k = b/m$

Y = Form factor = πy

Then y = Lewis form factor for 20^0 FDI (Full Depth Involutes) system = 0.144, from Table 12.5

Z = No of teeth on gear = 37

$m = 2 \times 59264.79/549 \text{ x } 0.5 \text{ x } 12 \text{ x } \pi \text{ x } 0.144 \text{ x } 37]^{1/2}$

$m = 1.46$

From standard table 12.2 chosen 1.5mm

$m = 1.46 \equiv 1.5$

e) Dynamic strength of gear (F_d)

$F_d = F_t + Fi$

F_d = Dynamic load in N

F_t = Tangential load at pitch line in N

F_i = Incremental load in N

$F_d = F_t + K_3 v\, (cb + F_t) / K_3 v + \sqrt{}\, (cb + F_t)$ -------------------------------- 12.33

C = Dynamic factor = 237.3, taken from Table 12.12

$K_3 = 20.67$

$$V = \frac{\pi d1N1}{60000}$$

$$V = \frac{\pi \text{ x } 55 \text{ x } 60}{60000}$$

v = 0.1727m/s

$M_t = F_t \times r$

M_t = Transmitted torque in N-mm

r = Pitch circle radius of gear in mm

$F_t = M_t / r$

$F_t = 59365/ (55/2)$

$F_t = 2158N$

$F_d = F_t + F_i$

$$Fi = \frac{20.67 \text{ x} 0.1728\ (237.3 \text{ x } 12 + 59365)}{20.67 \text{ x } 0.1728 + \sqrt{237.3 \text{ x } 12 + 59365}}$$

$F_i = 878.31$

$F_d = (2158+878)$ N

$F_d = 3036.72N$

f) Beam strength or dynamic strength of teeth

$F_{en} = Fs = \sigma_{en}\ b\ \pi ym$ ------------------------- (12.34)

$Fs = 741.15 \text{ x } 12 \text{ x } \pi \text{ x } 0.144 \text{ x } 1.5$

Fs = Dynamic strength of the gear in N

F_{en} = Endurance strength of gear in N

$\sigma_{en} = 1.35 \text{ x } \sigma_d = 1.35 \text{ x } 549 = 741.15$

Fs = 6034N

Fs $\geq$ Fd Hence, design is safe against dynamic loading.

g) Design against wear load

$F_w = d_1bQK$ -- (12.36a)

$F_w =$ Limiting load for wear

$d_1 =$ Pitch diameter of gear = 55 mm

K = load stress factor = 5.913, from Table 12.17

Q = ratio factor = 1

$F_w = 55 \times 12 \times 1 \times 5.913$

$F_w = 3902.58N$

Fw > Fd Hence, design is safe against wear.

---------∞----------

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4. Padmayya Naik and A.O. Surendranathan (2011), "New Manufacturing Process of Carbon-Carbon Composites and their Mechanical Properties", International Journal of Earth science and Engineering (IJEE), Vol. 04, No.05, pp. 916-921.

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