The Division of Engineering Materials comprises of three groups, namely, Metals & Alloys, Advanced Carbon Products and High Pressure Technology. The Division is primarily engaged in technology development of materials, components and processes, for strategic, high-performance and general engineering applications. Several developmental projects were undertaken for user organizations/agencies, like, Hindustan Aeronautics Limited, Bangalore; Vikram Sarabhai Space Centre, Thiruvananthapuram; Bhabha Atomic Research Centre, Mumbai; Institute for Plasma Research, Gandhinagar; Indian Oil Corporation, Faridabad; etc., during this year. A few joint collaborative projects with international agencies are also currently underway.
Metals & Alloys
The Metals & Alloys group is mainly engaged in the primary, secondary processing and characterization of monolithic and composite materials. During this year, developmental efforts were concentrated mainly on the projects pertaining to development of process technology for critical components for aerospace agencies. Considerable progress was also made in an on-going in-house project on Spray Forming and process parameters were optimized for the synthesis of Aluminium using the Spray Atomization & Deposition technique. An international joint collaborative project, on Precision Forging, with Mechanical Engineering Laboratory, Japan, was also successfully completed this year.

Precision Forming for Near-Net Parts
This project, which was in joint collaboration with Mechanical Engineering Laboratory, AIST, MITI, Japan, was successfully completed this year. In this project, some prototype Ti-alloy fasteners on reduced scale were developed. The sequence of operation for forging, estimation of forging pressure, design of tooling and actual forging experiments were undertaken for the development of these prototype Ti-fasteners. Several exchange visits from both countries were also made under this project. In the this year, cold/warm forging tooling worth Rs. 10.5 lakh was gifted by Mechanical Engineering Laboratory, AIST, MITI, Japan, to NPL under this project.

Development of Oval Shaped Tube, as Skid Landing Gear for Advanced Light Helicopter (ALH) Phase - I
This is an on-going project which was sponsored by Hindustan Aeronautics Limited, (HAL) Bangalore. This project is under the indigenization and weight reduction programme for Advanced Light Helicopter and envisages a substantial weight reduction. This project is to be undertaken in two phases. The first phase comprises development of actual sized oval shaped tubes from circular mother tubes of Al-alloy, within the dimensional tolerances, as specified by HAL.

In the current year, six numbers of oval shaped Al-alloy tubes were developed after optimizing the process parameters. These tubes had the dimensions: 150 mm X 100 mm (oval cross-section), 3 mm thickness and about 4.5 m in length. These six tubes developed were inspected by the representatives of HAL and CEMILAC, Bangalore and have met all the dimensional tolerances, as specified by HAL.

The first phase of this project has been successfully completed in this year and HAL has issued the test report for these six oval shaped tubes developed at NPL.

Development of Oval Shaped Tube, as Skid Landing Gear for Advanced Light Helicopter (ALH) Phase - II
This is the second phase of the HAL sponsored project to develop oval shaped tube as skid landing gear for ALH, which has commenced in this year. This project involves solutionizing the 4.5 m long oval shaped tubes (developed in the first phase of the project) followed by quenching and finally giving a 2-3% reduction draw on a draw bench to achieve T3 condition. These solutionized and cold-drawn oval shaped tubes are then to be bent from the front end, to achieve a radius of 800R.

The oval tubes were solutionized in a horizontal furnace, (at 495±5°C) which had to be suitably modified to accommodate this 4.5 m long oval shaped tube. The tubes, on water quenching (subsequent to solutionizing at high temperature), were getting distorted in shape due to their large length/diameter ratio and thickness, leading to a banana-type shape. In order to avoid this distortion, 4.5 m long special clamping fixtures had to be developed and installed on the oval shaped tubes, so as to encapsulate its entire length, while solutionizing and quenching. It was found that the shape distortion of these oval shaped tubes could be avoided using these clamping fixtures. These tubes were subsequently cold drawn to 2-3% reduction on a draw bench to achieve final T3 condition. Experimental trials are in progress to bend one end of these tubes to achieve a radius of 800R, on a specially developed tube-bending machine, which has been conceptualized, designed and fabricated in-house. Presently, these tubes are experiencing wrinkles in the inner curvature due to bending. Efforts are presently underway to improve the design of the bending machine and to make it more automated so as to avoid these wrinkles on the inner curvature of these tubes, on bending.
Selection of Sequence of Operation and Design of Tooling for the Development of Prototype Titanium Fasteners (Reduced Scale) using Cold/Warm Forging Technique

This project, sponsored by Hindustan Aeronautics Limited, Bangalore, was also concluded in this year. This project, under the technology indigenization programme of HAL, was undertaken as there is tremendous demand of Ti-fasteners in the aerospace industry, the needs of which are met solely through imports.

In this project, exploratory work for the prototype development of Hexagonal and Allen-headed fasteners in two grades of Ti-alloys, viz, (a) Ti-6Al-4V and (b) Ti-16V-4Sn-3Al-3Nb, on a reduced scale, was taken up. The design of the sequence of operation for cold/warm forging and design and fabrication of cold/warm forging tooling, for development of Hexagonal and Allen-headed

Fig. 3.1 (a) : Pictorial view of the Ti-6Al-4V fasteners (allen headed)

Fig. 3.1 (b) : Pictorial view of the Ti-6Al-4V fasteners (hexagonal headed)
Ti-fasteners, was completed. Several experiments were conducted in order to understand the deformation characteristics of these two types of Ti-alloys at different temperatures. After optimizing all the process parameters it was concluded that, it was not feasible to cold forge Ti-6Al-4V alloy and this alloy could only be warm or hot forged. However, it was found that Ti-16V-4Sn-3Al-3Nb alloy was cold forgeable. It has thus been suggested to the user agency that these Ti-fasteners would be made using Ti-6Al-4V alloy by hot forging followed by cold working on the shank. Figure 3.1 shows a pictorial view of the Ti-6Al-4V fasteners, made in three sequence of operation of forging.

**Rapid Solidification of Aluminium Alloys**

This is an on-going in-house project to develop aluminium/aluminium-alloys using Spray Atomization and Deposition (SAAD) unit, which was developed in-house in an earlier AR&DB funded project.

In this project, commercial grade aluminium has been successfully spray deposited by disintegrating the liquid melt by a stream of nitrogen gas jets and depositing the atomized droplets on a rotating water-cooled substrate, which also has a provision of fine vertical/rotary movement. Several experiments were carried out on the Spray Atomization & Deposition Unit and the process parameters (such as, melt temperature, flight distance, gas pressure, melt flow rate, substrate rotation/vertical movement speed, atomizer design, etc.) were optimized to spray deposit pure aluminium in flat and conical shapes. A gas atomizer, used in the SAAD experiments, was also fabricated in-house in this project. Typical yields of the as-sprayed conical deposits are in the range 60-70% of the weight of the melt with a density of about 90% of the theoretical density in the core region. The microstructure of the as-sprayed deposits indicated uniform microstructure with equiaxed grains and no indication of dendritic features, normally observed with conventional cast alloys. A few cylindrical billets, machined out of conical as-sprayed aluminium deposits (typical dimensions of 73 mmØ and 190 mm length), were secondary processed on a vertical hydraulic press, using the extrusion technique. Different extrusion process parameters were optimized to obtain a cylindrical rod with 36:1 extrusion ratio at 400°C extrusion temperature. The as-extruded rod exhibited a density of more than 99.9 % of the theoretical density with UTS of about 350 MPa.

**Advanced Carbon Products**

The advanced carbon products group is engaged in both basic and applied research, covering various aspects of carbon. The emphasis is on developing new carbon products as import substitutes or for strategic applications. In the current year, work was continued on several projects sponsored from national and international agencies. A few new projects were also initiated in this year.

**Development of Carbon-Ceramic Composites**

Carbon-Ceramic composites were developed by incorporating SiC (particulates or through sol-gel) and B₄C (particulates) in the NPL-developed green coke based carbon matrix. The composites were heat-treated to 1400°C and characterized for their physical, mechanical and oxidation resistance properties. It was observed from X-ray diffraction studies that silicon alkoxide alone could not yield SiC in the composites and B₄C helped in the formation of SiC in the sol-gel technique. By changing the carbon to ceramic and ceramic to ceramic ratio, the carbon-ceramic composites with improved oxidation resistance were obtained using both the techniques. However, composites developed through the particulate route were found to exhibit better oxidation resistance as revealed by a lower weight loss of 6.6% at 1000°C as compared to 14.3% in the case of sol-gel route. Further work is in progress to obtain carbon-ceramic composites exhibiting a weight loss of ≤1% at 800-1000°C.

**Development of Pitch-based Carbon Monofilament**

Work was continued under the on-going project (sponsored by AR&DB) on the "Development of carbon monofilament suitable for CVD-based SiC fibres". Having developed a 'Pitch Spinning Assembly' and a suitable precursor pitch for the carbon monofilament, vigorous efforts were made to improve the strength of the green pitch filament by way of modifying the precursor pitch by adding different types of polymers, namely, high density polyethylene (HDPE), polypropylene (PP), polyvinyl chloride (PVC), polystyrene (PS), high impact polystyrene (HIPS) and polymethyl methacrylate (PMMA). The
resultant pitches were subjected to spinning to obtain the green pitch filaments, which were tested for tensile strength, tensile modulus and strain-to-failure ratio and also subjected to scanning electron microscopy. The additions of PMMA and PS have been found to lead to significant improvement in the handleability and flexibility of the resulting green pitch filaments with respective green tensile strength of 220 and 70 MPa and tensile modulus of 10 and 12 GPa, respectively. However, the PMMA modified pitch led to a highly porous carbon filament with poor mechanical properties. The PS, on the other hand resulted in a carbon filament of tensile strength of 880 MPa and tensile modulus of 68 GPa. Further work is continuing to optimize the process parameters and to study the microstructure.

Development of High Thermal Conductivity Graphite

Extensive work was carried out under the on-going project (sponsored by IPR, Gandhinagar) entitled, "Development of high thermal conductivity special graphite for first wall component of SST-1 tokamak". The special graphite to be developed under this project is required to possess a thermal conductivity of 90 W/mK, bulk density of 1.7 g/cm³, bending strength of 40 MPa, electrical resistivity of 2mΩcm and an ash content of ≤ 0.2%. To accomplish the development of such a graphite, the 'green coke' method of producing the high density graphite was made the basis wherein the green coke was modified by way of addition of small amounts of synthetic graphite or natural graphite into the precursor pitch. The resultant graphite, made by both conventional pressing as well as isostatic pressing, was found to meet all the required characteristics of the special graphite except the thermal conductivity where a value of 77 W/mK was obtained with synthetic graphite addition as against the threshold value of 90 W/mK. Efforts are continuing to obtain this special graphite with a thermal conductivity value of 90 W/mK by the above said modification of green coke.

Feasibility Studies of Petroleum Streams as Precursor for High Performance Carbon Fibres

Under the on-going project (sponsored by IOC) entitled, "Feasibility studies on various petroleum refinery streams as precursor for high performance carbon fibres", work was carried out on the development of standard pitches (by techniques of condensation, polymerisation and distillation) with softening points of 125 ± 10 °C from all the five streams, namely, Short Residue (SR), Residual Fuel Oil (RFO), Blue Oil Extract (BOE), Clarified Oil (CLO) and Coker Fuel Oil (CFO), supplied by IOC R&D Centre, Faridabad. The resultant pitches were studied for pyrolysis behaviour and mesophase formation at different temperatures and periods of time. With respect to the yield of pitch obtained from the different streams, the streams are found to be in order of SR (35.5%), RFO (25.0%), BOE (11.5%), CLO (5.8%) and CFO (3.5%), whereas w.r.t. the coking yield obtained from the different standard pitches, the streams are found to be in order of CLO (55.9%), CFO (53.5%), RFO (47.0%), BOE (41.3%) and SR (33.1%). Thus, w.r.t. the overall coking yields, the streams were found to be in order of RFO (11.8%), SR (11.7%), BOE (4.7%), CLO (3.2%) and CFO (1.9%). Besides this, pitches from all the streams were found to form liquid crystalline phase (mesophase) on heat treatment, which is a pre-requisite for any material to be a precursor for high performance carbon fibres. Thus, from techno-economical considerations, the RFO could be regarded as the best stream for the development of precursor for high performance carbon fibres. Fig.3.2 shows the optical micrograph of bulk mesophase formed in RFO-based pitch on heat-treatment at 490°C for 1h. Further work to develop general-purpose carbon fibres from this stream is in progress.

Synthesis of Carbon Nanotubes

Using the dc arc discharge technique, it has been possible to obtain carbon nanotubes, both, as cathode deposit, as well as in the carbon deposits, inside the chamber. This has been possible by optimizing various processing parameters as well as using special graphite electrodes. The study has given useful insight into the formation of carbon nanotubes and will be quite helpful in increasing the yield of carbon nanotubes in future and their applications in composites.

Development of Carbon Composite Femoral Arch for Polio Patients

Under this project, carbon fibre reinforced composite 'Femural Archs', also known as 'Italian Arch', were developed as an extension of the earlier project, which was on the development of these rings for Ilizarov Fixator. These have been successfully tried on the polio patients at Agrawal Orthopaedic Hospital, Gorakhpur.
High Thermal Conductivity Carbon/Carbon Composites

Under this sponsored project "Development of High Thermal Conductivity Carbon-Carbon Composites" funded by (UISTRF, INDO-UK), carbon-carbon composites were developed using different reinforcements (chopped PAN and pitch based fibres) and matrices (Coal Tar Pitch, modified Coal Tar Pitch and mesophase Pitch) with the main objective of developing composites having high thermal conductivity (150-200W/mK). Composites were characterized for physical, electrical and thermal properties. X-ray diffractometry showed the crystallite dimensions in ab plane to be 31 nm and d_{202} to be 0.3392 nm. Electrical resistivity of the graphitized sample was found to be 0.5-0.8 m\(\Omega\)cm. A new technique, micro Thermal Analyzer AFM, was used for measurement of Thermal Conductivity which combines the visualization power of Atomic Force Microscopy (AFM) with the characterization capabilities of thermal analysis. Fig. 3.3 shows the topographic and thermal images of F3M1 sample which clearly shows that carbon fibres possess more thermal conductivity compared to the matrix.

Development of Porous Conducting Carbon Paper

The objective of this project is to develop porous conducting carbon paper, which will be used in Fuel cells as a gas diffusion type electrode and also as a catalyst. The specifications of the carbon paper,(sponsored by Naval Materials Research Laboratory) which were targeted and achieved so far, are reproduced in the table:

<table>
<thead>
<tr>
<th>Property</th>
<th>Specification Targeted</th>
<th>Specifications Achieved</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>70 %</td>
<td>&gt;70 %</td>
</tr>
<tr>
<td>Gas Permeability</td>
<td>3.59 cm(^3)/sec</td>
<td>3.5 cm(^3)/sec</td>
</tr>
<tr>
<td>Electrical Resistivity in plane</td>
<td>0.005 (\Omega)cm</td>
<td>0.003-0.008 (\Omega)cm</td>
</tr>
<tr>
<td>Flexural Strength</td>
<td>40 MPa</td>
<td>40-45 MPa</td>
</tr>
</tbody>
</table>
Asbestos - Free Brake Materials for Automobiles - Tailoring, Characterization and Evaluation
This activity was continued and the samples of size 6 cm x 6 cm x 0.5 cm were prepared and characterized for density, hardness, flexural strength & flexural modulus. The studies were mainly confined to control the scleroplastic hardness of the samples which was brought down from a value of 60 reported last year to a desired value of 40-45. Few samples were also given to ITTMEC, IIT Delhi for their characterization of friction and wear properties.

Development of Radiationally Stable Carbons
The studies were continued in the project (sponsored by BARC) to develop carbon composite samples which are isotropic as well as amorphous in nature. Carbon composite samples were prepared by changing fibre and matrix volume fractions. These composites were characterized through XRD for their amorphous nature. The isotropic nature was established by measuring their coefficient of thermal expansion and by optical microscopy. Few samples of size 18 mm dia and 20 mm thickness were supplied to BARC for keeping them inside the nuclear reactor for the evaluation of their thermal stability.

High Pressure Technology
The group has been engaged in the synthesis of cubic boron nitride at pressures lower than those used by other researchers employing the conventional techniques which uses pressure (50kb) and temperatures (>1500°C). We had earlier synthesized cubic boron nitride under a DST funded project at pressures as low as 25 kb. As an extension of this activity, a project entitled "Low pressure synthesis of cubic boron nitride by means of supercritical fluid" in collaboration with Mineralogical Institute, University of Bonn, Bonn, Germany, under DST - DAAD Project Based Personnel Exchange Programme has been approved. In this programme, experiments on cBN synthesis using liquid ammonia, as a supercritical fluid, were carried out. However, the initial experimentation did not show encouraging results with respect to cBN conversion using hBN-Li2N-NH3 system. This work is under progress.

Trial runs were also made to synthesize cBN compacts on 1000 ton press using imported cBN powder (6-9 micron) and TiC/TiN as the binding material. cBN compacts with a microhardness of about 3000 kg/mm2 were obtained.