CHAPTER 8

CONCLUSIONS
AND
FURTHER SUGGESTIONS
The role of reinforcing fibers in the development of high temperature composites is technologically important aspect as it associates with itself many processing and performance factors. One which has drawn attention of maximum researchers is fiber-matrix interactions, more specifically fiber-matrix bonding. This is of particular interest in carbon-carbon composites wherein both fibers and matrix are of same element and are processed through similar routes. Though this problem has been taken up by many researchers world over but always gets new look with the development of newer types of carbon and alike fibers. Other important aspect for commercial application of these composites is exploring alternative reinforcing fibers for making these composites. Present studies were aimed at understanding fiber-matrix bonding in carbon-carbon composites in general and studying these aspects in commercially available carbon fibers. After studying this, the work was extended to use oxidized PAN fibers as reinforcement for making carbon-carbon composites and to study various processing parameters affecting the fiber-matrix bonding and performance of the composites processed at different temperatures. Various conclusions drawn from present studies are given in this chapter.
Polymer composites on pyrolysis exhibit maximum weight loss and shrinkage up to 1000°C. On subsequent heat treatment to 2000°C and above, both the changes are hardly 10% of those occurring up to 1000°C. The carbonization shrinkage is observed to be anisotropic in nature and greatly dependent on the type of the fibers i.e., on fiber-matrix bonding. Composites having stronger fiber-matrix bonding exhibit higher cross-sectional shrinkage. It is found to vary with reinforcing fibers as follows:

HM (High modulus fibers) < IM (Intermediate modulus fiber) < IPCL (High strength fiber) < T-300 (High strength fiber).

Carbonized composites exhibit low densities which can be increased by subsequent infiltration/carbonization of carbonaceous precursors. The rate of pickup has been found to be dependent on the porosity of the carbonized composites specially the macropores which are found to be maximum for composite made with HM and T-300 carbon fibers. Fiber-matrix bonding in the composites at the polymer level is not only found to effect the processing of composites but also the mechanical properties. Composites having strong fiber-matrix bonding at polymer level result in low strength carbon-carbon composites. On subsequent heat treatment to 2000°C and above results in decrease in fiber-matrix...
interactions and improves the properties of the composites. At 1000°C, the failure of the composite is matrix dependent while at 2000°C and above it is found to be fiber controlled.

When polyacrylonitrile fibers are heated in presence of air at temperature higher than 220°C, chemical transformations take place in the molecular chains of the polymer resulting in flame proof black fiber called oxidized or stabilized PAN fibers. The reaction is found to be diffusion controlled and is therefore time dependent. The rate of reaction also increases with temperature. Present studies have shown that not only the internal cyclization reaction increases with time and temperature of treatment but the oxygen surface groups on the fiber specially the carboxylic groups also increase linearly. These surface chemical complexes make chemical bonding with the free groups available in the polymer matrix. The pyrolysis of the fibers alone to 1000°C result in higher cross-section shrinkage (about twice) than linear shrinkage. The amount of shrinkage being dependent on the oxidation conditions i.e. time and temperatures.

The present studies show that carbon - carbon composites can be fabricated by making fiber reinforced
polymer matrix composites with oxidized PAN fibers and thermosetting resins (phenolic or polyfurfuryl alcohol) followed by pyrolysis to 1000°C. The fiber–matrix has been observed to be much more important in these composites than in the case with carbon fiber composites. This has been controlled by chemical treatment of the fibers with pyrogallol solution. Like oxidized PAN fibers and cured phenolic resin, the polymer composites are also found to exhibit maximum weight loss and linear shrinkage in the temperature range 400–500°C. The weight loss during pyrolysis of oxidized PAN fibers reinforced polymer composites is less than the sum total of the weight loss during pyrolysis of the individual components. This has been attributed to the protection of the fiber surface by polymer layer. The pyrolysis shrinkage of the composites is found to be anisotropic. It is more (22-30%) in width and thickness directions and lower (9-18%) in the fiber direction. The exact amount of shrinkage is dependent on the oxidation conditions of the fibers. It reveals that like in carbon fiber reinforced composites, the pyrolysis shrinkage of OXPAN reinforced composites is also controlled by the reinforcing fiber and hence can be controlled by treatment of the fiber surface.
with retarding agents such as epoxy or pitch. The densities of the composites increase with heat treatment temperature.

Optical microscopic and SEM examination of the composites have revealed good fiber-matrix bonding at all stages, polymer to carbonization and graphitization. At all stages fiber and matrix retain their identities. Polarized light optical microscopic examination of the carbonized composites has revealed that anisotropy in the matrix starts at the fiber-matrix interface and progresses into the matrix. Even the fibers also exhibit development of anisotropic structure. The development of anisotropic structure in the matrix as well as in the fibers is due to shrinkage stresses developed in the composites during pyrolysis. These shrinkage stresses of course depend on the fiber-matrix bonding and are obviously maximum in this composites. On heat treatment of the composites to temperatures higher than 2000°C, the carbon matrix gets highly graphitized. Therefore these studies show that a highly graphitized carbon-carbon composites can be obtained by using oxidized PAN fibers as reinforcement. Not only this, the graphitized composites made with oxidized PAN fibers have been found to possess high flexural strength (150-180 MPa) with pseudoplastic fracture.
Studies made with short fibers (carbon and oxidized PAN fibers) as such or in matrix reveal that the fabrication method; prepregging, isotropic casting with pressure or isotropic casting without pressure have great influence on the macrostructure of the ultimate composites. Taking into consideration the macrostructure, density etc., it is concluded that short fibers of 10 mm length are more suitable than 1 mm length fibers. When short oxidized PAN fibers are used as reinforcement, even the bulk matrix remote from the fibers gets graphitized. Mixed short fiber composites are found to possess lower densities at the carbonized and graphitized level than those made with single type short fibers. These was the initial studies made with short oxidized PAN fibers and mixed fibers. However, future studies may reveal some more interesting scientific results. The results of the initial studies made with short oxidized PAN fibers and mixed fibers have been presented here. The future studies reveal some interesting scientific results which may have wide industrial applications.