CHAPTER VII

CONCLUSIONS AND FUTURE SCOPE OF THE WORK
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The research and development on fiber reinforced ceramic matrix composites has gained significant importance in the last two decades with the aim to use ceramics for high temperature structural applications. Various traditional as well as advanced techniques are being tried to develop these composite materials. Among these techniques, preceramic polymer and organometallic routes have several processing advantages over the traditional melt infiltration, powder metallurgy or CVD routes. However, the processing of these matrix precursors and the science of pyrolysis of these preceramic polymers through sol-gel route as well as their composites need to be studied in detail. The research work carried out on formation of ceramics, resulting composites and their properties compiled in thesis is an attempt in this direction.

The ceramics can be prepared by the hydrolysis of organometallic compounds followed by pyrolysis. Both hydrolysis and pyrolysis conditions are very important for getting the tailor-made properties of the ceramics. By optimizing these conditions, monolithic silica gels can be obtained by hydrolysis of tetraethylorthosilicate. Further the sol-gel method can be used for the incorporation of carbon into silica matrix yielding organic groups in the silica gel through copolymerization of silica sols and a carbon containing monomer, which on polymerization yield C-Si and Si-O group bonding depending on the pyrolysis temperature.

Modified silica gel network could be synthesized by incorporating carbon through copolymerization of organometallic compound using two routes (i) Aqueous route and (ii) Non aqueous route. The products obtained in both cases on pyrolysis at 1000°C yield black glasses or silicon oxycarbide and at higher temperature silicon carbide. An analysis of Infrared studies and thermal analysis of cured samples at 180°C supports the
copolymerization between organometallic and organic compounds. The modified gels on pyrolysis to 1000°C results in the formation of new linkages such as Si-C and Si-O-C. Such multielement ceramics though disordered in structure, exhibits high thermal stability and can be used as matrices for composites.

Gels prepared with Furfuryl alcohol to TEOS i.e. aqueous route in the molar ratio of 0.65 : 1 on sintering to 1000°C gives oxidation resistant Si-O-C carbon/silica glass. Samples show almost no weight loss in air when heated in air at 1000°C. On further heat treatment to 1400°C Infrared spectra and X-ray diffraction pattern showed combined peaks for Si-O and Si-C. On heat treatment to 1600°C it gets converted to material of highly crystalline nature, β SiC. The formation of β SiC has been confirmed by X-ray diffraction.

The gels prepared through non-aqueous route with Butanediol to TEOS in the molar ratio of 4 : 1 on heat treatment to 1000°C give oxidation resistant Si-O-C carbon/silica glass, showing almost no weight loss in air at 950°C. The Si, C, O containing glasses prepared from Butanediol and TEOS system at 1000°C, when heated in the temperature range 1400°C and above show resinterability to dense structures.

The various parameters optimized for the synthesis of gels both by aqueous and non aqueous routes were used for the fabrication of composites. Two types of fibers were used as reinforcement i.e. glass fibers and carbon fibers. For reinforcement staple fibers as well as unidirectional and bi-directional carbon fibers were used. The aim of making composites with glass fibers was to study the infiltration of the matrix precursor into the fibrous structure.

The studies on composites carried out by using staple fibers revealed that, when the length of fiber is more than 1-2 mm, long cracks were observed in the composites, which were attributed to more tension in porous matrix of composite. Due to tension in the matrix, brittle matrix cracks. It was further seen that uniform distribution of fibers in matrix also play a vital role. The composites were prepared by using gel prepared
by non aqueous route and carbon fiber as reinforcement. The composites were found to be highly porous on heat treatment to 1000°C. Heat treatment to 1400°C to reduce porosity resulted in further decrease of density, which may be due to gaseous evolution of the reaction products as a result of thermal instability of silicon oxycarbide matrix. On impregnation of these composites, an increase in density of the order of 10% has been noticed. The composites though structural integrated were still having interconnected and closed porosity.

After heat treatment and impregnation the samples were still having interconnected and closed porosity. These results are further supported by Optical microscopy and Scanning electron microscopy micrographs. These micrographs show that within the composite, the fiber/matrix bonding is of intermediate strength nature. The fibers are well surrounded by the matrix. On heat treatment of the composites at 1000°C, within the matrix, different types of voids, macropores and micropores are seen which account for the porous nature of composites. The bonding between fibers and matrix appears to be good and of intermediate type. Further composites heat treated at higher temperature i.e. 1400°C show dense nature and non catastrophic failure.

Sol-gel derived silica prepared through the hydrolysis of TEOS can be used as an effective precursor material for the development of Silicon oxynitride by high temperature reaction with ammonia. Samples nitried at 950°C in ammonia show almost no weight loss when heated in air at 1000°C.

The studies made and reported in the thesis are results of the first attempt in this direction and show that preceramic polymer synthesized by sol-gel technique are promising materials to be used as matrix precursor for fabricating high temperature oxidation resistant ceramic composites. Though good structurally integrated oxidation resistant fiber reinforced composites could be obtained using sol-gel technique, the matrix exhibits vide pores and voids. The resintering tendency of the matrix (HTT 1000°C) suggests that dense composites can be obtained by hot pressing the composites at a
temperature around 1400°C. Such composites will possess higher density and mechanical properties. Synthesis of preceramic polymer through sol-gel route can be further taken up to study the effect of higher treatment temperatures of 1600°C and more on the matrix structure as well as on the thermophysical behaviour of the composites developed therefrom.

Fiber reinforced nitrogen containing silica matrix (silicon oxynitride) composites can be prepared using sol-gel technique by first making composites with silica sol followed by drying and nitriding the matrix by heating it at a temperature around 900°C in presence of ammonia. These composites show good oxidation resistance at higher temperature and hence can be an promising material at high temperature in oxidizing environment.