C_f/CERAMIC COMPOSITES OBTAINED BY PIP METHOD

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Non-scientific abstract

The development of advanced technologies in the field of materials science requires new materials, which could work under specific conditions and could be more reliable. Ceramic matrix composites reinforced with carbon fibres (C_f/ceramic composites) are potential candidates for construction working at elevated temperatures and in an oxidative atmosphere. They retain their mechanical properties at high temperatures and perform high oxidation resistance.

The area of ceramic matrix composites (CMCs) application concerns mainly space industry, nevertheless over the last years such composites are more and more often found in the motor, aircraft and railway industry and as frictional parts in disc brakes. Moreover, CMCs are major candidates for high temperature applications, such as heating elements, construction elements working at elevated temperature in an oxidative atmosphere or multifunctional materials, which can work both as construction and heating element.

Current technological achievements related to using of ceramic matrix composites reinforced with continuous fibres are far from industrial application because of high costs associated with their manufacturing.

The application of silicon-containing polymers as the precursors of composite ceramic matrices reinforced with carbon fibres is a new economical way and may be an alternative for conventional fabrication of ceramic matrix composites. Starting with our study became feasible due to the presence of low cost commercially available silicon-containing polysiloxane resins. Moreover, the prognoses relevant to carbon fibres in the near future show their prices decreasing.

Technical abstract

PIP method bases on preceramic polymers that are transformed into ceramic phases during heat treatment. The use of preceramic polymers offers perspectives for broadening of the application of continuous fibres reinforced CMCs. This method is the most economical from the point of view of matrix precursor prices and energy saving (lower processing temperatures), do not damage fibres and allows to obtain pure homogenous composites with tailored matrix structure and microstructure.

Many data concerning organosilicon polymers applied as a source of ceramic compounds and CMCs, namely polycarbosilanes, are available in the literature. However, there are limited data on the use of polysiloxanes as ceramic precursors and no data for their application as precursors for CMCs reinforced with carbon fibres.

The aim of this work was to elaborate new CMCs reinforced with continuous carbon fibres, which matrices were obtained from polysiloxane polymer precursors, by PIP technique. Four types of cheap, commercially available polysiloxane resins, differing in carbon to silicon molar ratio and in oxygen concentration, were used in experiments. Conversion mechanism from pure polysiloxane resins to carbide phase and conversion mechanism of these resins to ceramic phase in the presence of carbon fibres were investigated. Selected physical and mechanical properties of composite samples were examined.

Objective

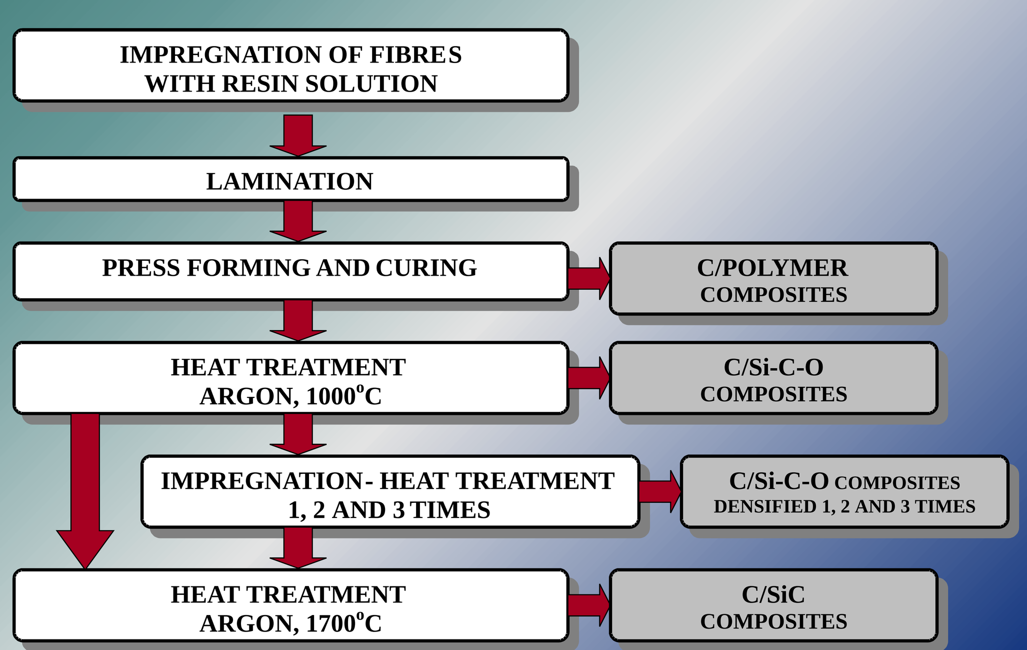
- preparation of ceramic matrix composites reinforced with carbon fibres via PIP technique
- investigation of the mechanism of thermal conversion of polysiloxane resins to carbide phase in the presence of carbon fibres
- determination of selected properties of composite samples

Materials

- carbon fibres HTS 5131 (Tenax-J)
- polysiloxane resins - Lucebni zavody, Kolin (Czech Republic)

Labelling	Type of polymer	C/Si molar ratio
P5	*polymethylsiloxane*	*4.87*
P3.1	*polymethylphenylsiloxane*	*3.11*
P3.2	*polymethylphenylsiloxane*	*3.14*
P1	*polymethylphenylsiloxane*	*1.14*

Sample preparation - liquid impregnation technique



Methods

- the porosity was measured by mercury-porosimeter CARLO ERBA 2000 with MACROPORES accessory
- the microstructure of composite samples was studied by scanning electron microscopy (SEM) and EDS analysis
- the oxidation resistance was determined by mass losses of samples heated in air atmosphere at 600°C, duration 2h
- to determine mechanical parameters, the composite samples were investigated in three points bending test on Zwick machine model 1435. The samples in the form of bars were loaded during the test with the constant rate of 2 mm/min.

Composite characteristic

- fiber volume fraction in C_f/polymer composites 0.50 % +/- 0.03 %
- open porosity of C_f/Si-C-O composites - about 20 %
- open porosity of C_f/SiC composites - about 35 %

Results

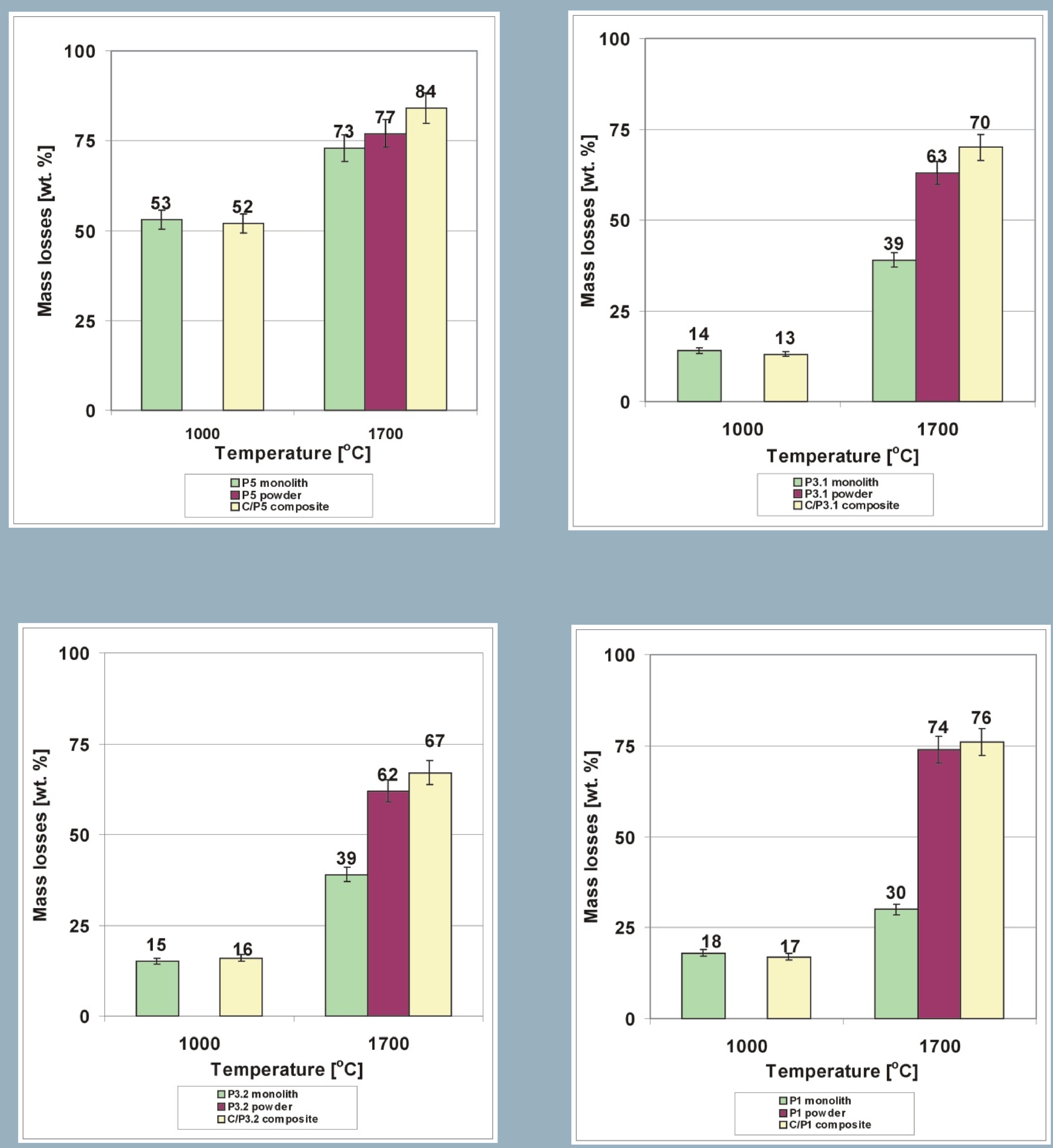


Fig. 1. Mass losses of polymers heat treated at 1000°C and 1700°C: Polymers in form of monolith, powdered polymers, polymers in the presence of carbon fibres

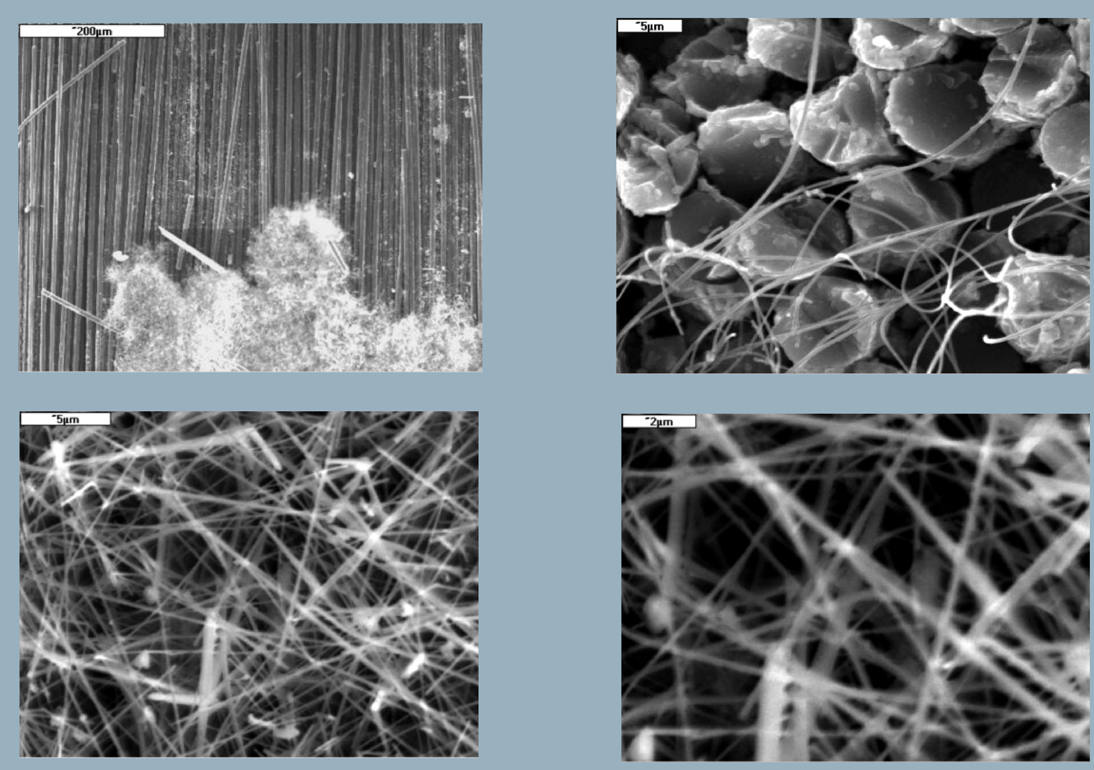


Fig.2. SEM images of C_f/SiC composites

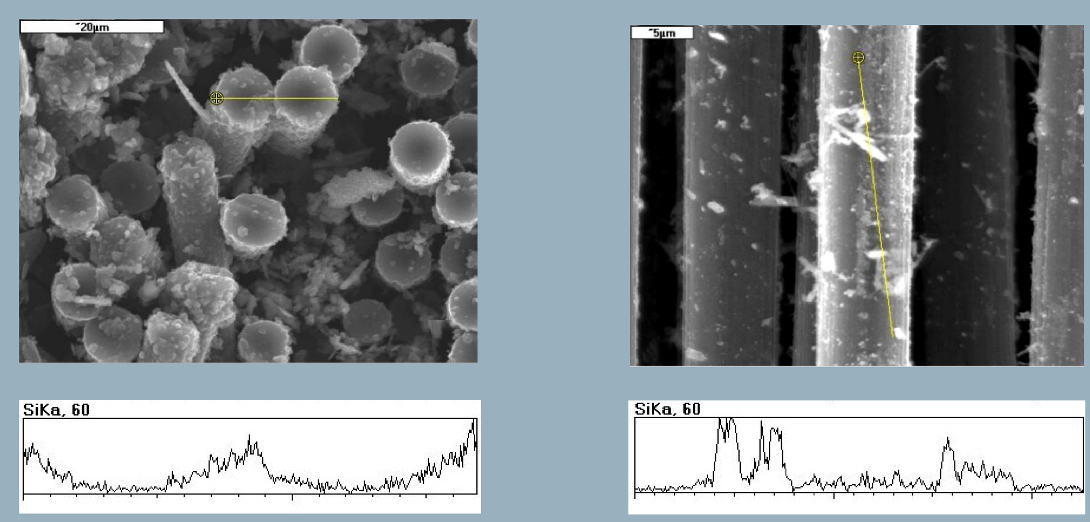


Fig. 3. SEM images and EDS analyses of C_f/SiC composites

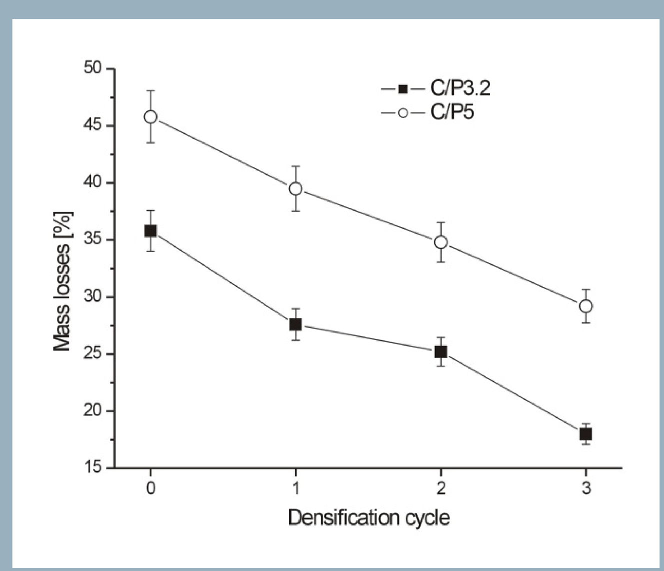


Fig.4. Mass losses of C_f/Si-C-O composites after oxidation

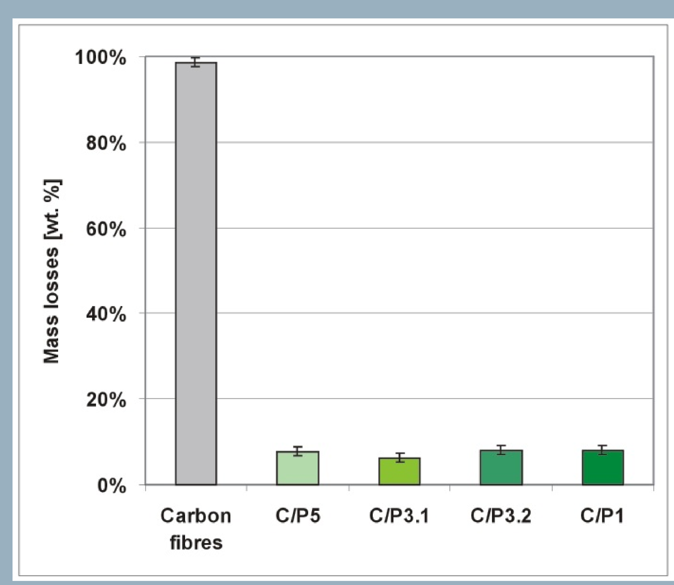


Fig. 5. Mass losses of C_f/SiC composites after oxidation

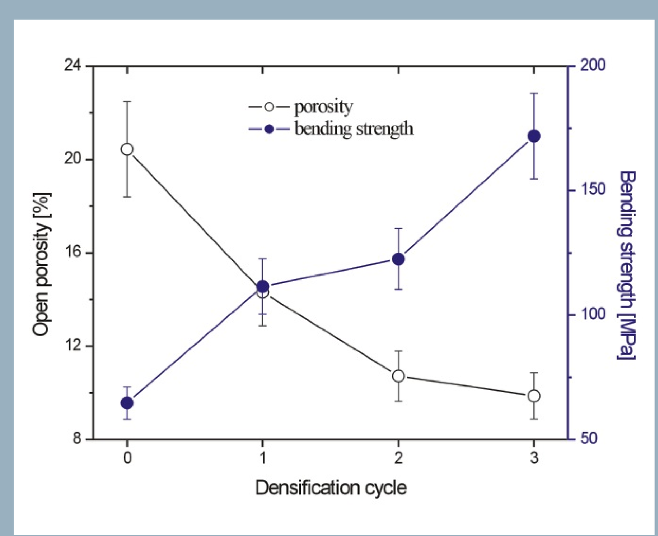


Fig. 6. Effect of cyclic impregnation on porosity and bending strength of P5 resin - based C_f/Si-C-O composites

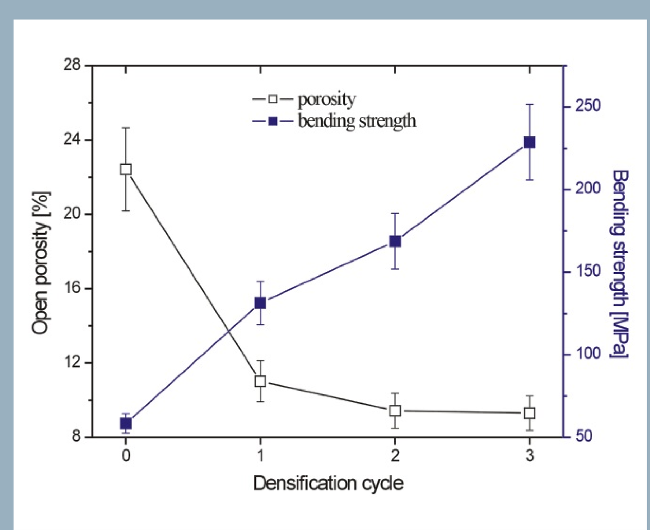


Fig.7. Effect of cyclic impregnation on porosity and bending strength of P3.2 resin - based C_f/Si-C-O composites

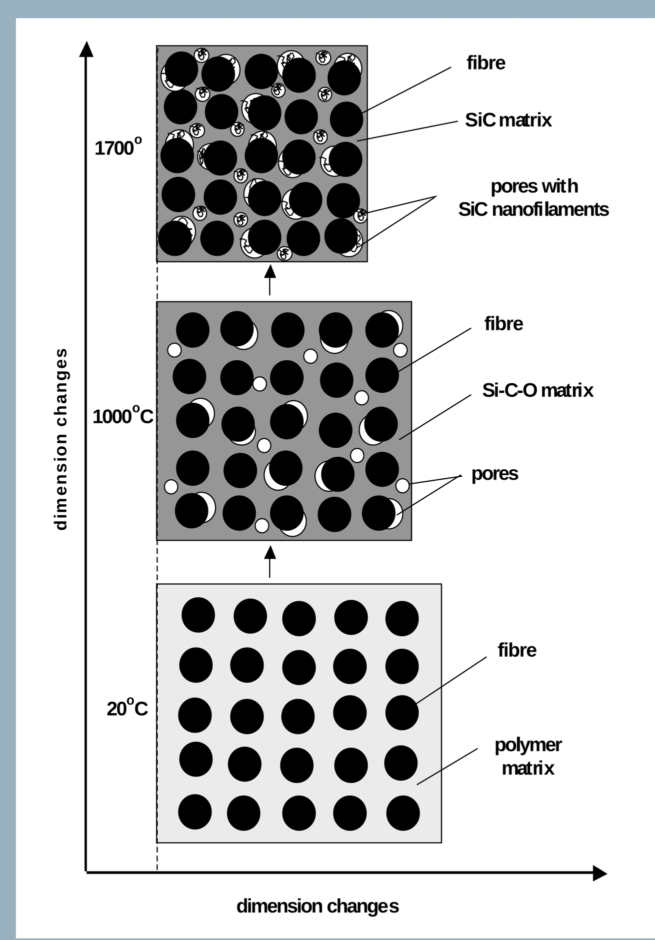


Fig. 8. Scheme of thermal conversion of C_f/polymer composites to C_f/SiC composites

Conclusions

- Thermal decomposition mechanism of pure resins in the presence of carbon fibres up to 1000°C is similar as without fibres
- Above 1000°C thermal decomposition of the matrices in the presence of fibres is more intense. The process occurs in gas and solid phase - the presence of carbon fibres results in developing of matrix surface area and produces higher mass losses and higher porosity of composite
- Above 1000°C, as a consequence of silicon oxycarbide decomposition, nanosized silicon carbide fibres crystallize in composite pores and silicon carbide protection layer forms onto the fibre-matrix interface
- Owing to the presence of protective silicon carbide layer in the fibre-matrix interface, highly porous C_f/SiC composites represent a significantly high oxidation resistance
- The process of repeated densification of porous matrix with polysiloxane polymer and additional heat treatment lead to further improvement of mechanical properties and oxidation resistance of C_f/Si-C-O composites

Glossary

C_f - carbon fibres

C_f/ceramic composites - ceramic matrix composites reinforced with carbon fibres

PIP method (Polymer Impregnation and Pyrolysis method) - in this method the fibres are embedded in a polymeric precursor of the matrix, such as thermosetting resin or silicon-containing polymer (e.g. polycarbosilane for SiC) or pitch for carbon. Then, as received green composite is pyrolyzed.

polysiloxanes - polymers backbones of which consist of alternating atoms of silicon and oxygen. They can exist as elastomers, greases, resins, liquids and adhesives. Their great inertness, resistance to water and oxidation, and stability at high and low temperatures have led to a wide range of commercial applications

preceramic polymers - polymers transformed into ceramic by heat treatment

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