

THE SIGNIFICANCE OF SOL GEL DERIVED INTERPHASE ON THE MECHANICAL PROPERTIES OF C/C COMPOSITES.

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Introduction

Fibre/matrix interactions are the very essence of a composite material and have been recognized as useful parameter for controlling the mechanical properties of C/C composites, the detailed mechanism is not fully understood, despite extensive studies. Also, there are no published studies of the effect on mechanical properties of hybrid C/C composites, that is, composite containing an interphase material other than carbon in between the fibre and matrix. This paper presents the results of a study that addresses this subject.

Experimental

Tetraethoxysilane (TEOS), water, acetonitrile and hydrochloric acid in the molar ratio of 1:8:4:0.05 and dimethyldiethoxysilane (DMDEOS), water, ethanol and hydrochloric acid in the molar ratio of 1:8:2:0.05 are used for sol gel processing of polydimethylsiloxane (1). Final mole percentage of DMDEOS : TEOS is kept 90:10. Coatings of the processed sol were applied on carbon fibres with uptake values of 4.6×10^{-4} g/cm (sample S-4), 7.8×10^{-4} g/cm (sample S-8) and 11.2×10^{-4} g/cm (sample S-12).

PAN based carbon fibres (having T.S. 2.5 GPa and Y.M. 195 GPa) are used in the fabrication of composites ($V_f = 45 \pm 3\%$).

RESULTS AND DISCUSSION

Two basic requirements of an interphase (to hinder the crack propagation and to enhance overall mechanical properties of the ultimate C/C composites) are :

- (i) Interphase should reduce the adhesion between the cured phenolic resin and the carbon fibre surface, so that the resin shrinkage during precarbonization treatment (HTT 600 °C) should not lead to damage of either matrix or fibre.
- (ii) Interphase should improve the interactions after carbonization. This may be achieved by converting the interphase into a material that develops an optimum degree of bonds with carbon fibre and carbon matrix.

An extensive study on the sol gel derived polydimethylsiloxane revealed that it is not an active species because of limited number of active groups such

as Si-OH. Apart from this, it is polar in nature and thermally stable upto a temperature of ~ 600 °C. At this temperature the transformation of the resin is nearly complete. Therefore there is a possibility that after the application of polydimethylsiloxane interphase, composites would not get destroyed because of the stresses generated during shrinkage. At higher temperatures, sol gel derived polydimethylsiloxane gets converted into an intimate mixture of carbon and silica via the formation of Si-OH, Si-CH₂-Si, Si-CH₂-CH₂-Si and linked to carbon by Si-C or Si-O-C bonds (1). Thus there is a large tendency of formation of strong interactions with carbon matrix. Sol gel derived polydimethylsiloxanes, therefore, seemed to exhibit these desired properties required for an appropriate interphase.

Figure - 1 shows the load deflection curves of the carbonized composites. Curve of S-0 composites shows a brittle fracture. This type of curve reflects strong fibre /matrix interactions which makes debond energy very high. No debonding takes place under load and thus composite fails catastrophically. Curve of S-4 composites reflects that there is debonding in the this system. Stress decreases steadily at strain higher than the matrix failure strain. The failure is not catastrophic, fibre pull out contributes to the work of fracture. Curves of S-8 and S-12 composites show the retention of applied load in initial stages and debonding of interphase thereafter. This may indicates the formation of strong interphase in these composites. Scanning electron micrograph, as shown in figure - 2, supports the above results. Development of a strong interface between carbon matrix and interphase after carbonization, because of the formation of Si-C or Si-O-C linkages, makes the interfacial sliding stress low enough to allow relative movement of the two phases as load is transferred to the fibres. Since there is debonding in the system, fibre did not fail in the matrix crack plane, allowing some fibre pull out to contribute to the work of fracture.

Figures 3 and 4 show the flexural strength and interlaminar shear strength of polymer and carbonized composites. The low strength values are unusual but clearly reproduced. The figures show variation of flexural and interlaminar shear strength of the composites with the extent of interphase. The polymer composites show a sharp decrease in the values. However, the carbonized composite show a strong increase of the flexural and

ILSS at low interphase content, but also a remarkable decrease in the properties is noticed with further increase in the interphase content. The maximum values are nearly twice as high as that found for the composites produced without the application of interphase.

From a chemical point of view it may be assumed that the silanol groups of interphase can undergo polycondensation reactions with the carboxylic and hydroxyl groups present on the carbon fibre as well as in the phenolic resin matrix system. Polycondensation reactions occurring among hydroxyl, carboxylic and silanol groups present in the interphase, carbon fibre and phenolic resin matrix may govern the interfacial strength between carbon fibre and interphase as well as between interphase and phenolic resin. At low interphase content, it is obvious that the amount of polycondensation reactions would be less. This led to the low interfacial strength and consequently the lower flexural strength and interlaminar shear strength (ILSS) values of S-4 composites at polymer stage. The extent of polycondensation reactions would be more with the increase of interphase content. Subsequently a strong interface developed which led to the higher flexural and ILSS of S-8 and S-12 composites at polymer stage, figures 3 and 4.

During carbonization upto a temperature of 1000 °C, interphase undergoes numerous reactions. These reactions convert it into an intimate mixture of carbon

and silica via the formation of Si-OH, Si-CH₂-Si, Si-CH₂-CH₂-Si and linked silicon to carbon by Si-C or Si-O-C bonds. Carboxylic and hydroxyl functional groups at the carbon fibre decompose at a lower temperature which limits the interactions between interphase and carbon fibre during carbonization. Thus, there is a greater possibility of formation of strong interactions with carbon matrix. Development of an ideal interface between an optimum interphase and carbon matrix results in the increase in flexural strength as well as ILSS, as observed in S-4 composites (figure 3 and 4). However, development of strong interface between carbon fibres and interphase in S-8 and S-12 composites resulted in the decrease in F.S. and ILSS as well as dual nature of fracture behavior.

This investigation concludes that application of a suitable interphase improves the mechanical properties of C/C composites to a great extent.

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Reference

1. V.K. Parashar, Ph.D. Thesis, Indian Institute of Technology - Delhi, 1996

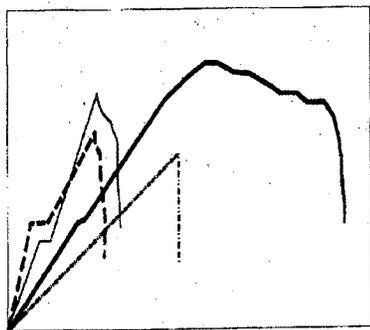


Figure 1. Stress-strain curve of carbonized composite. S-0(•••); S-4(—); S-8(—); S-12(—).



S-0 (X100)

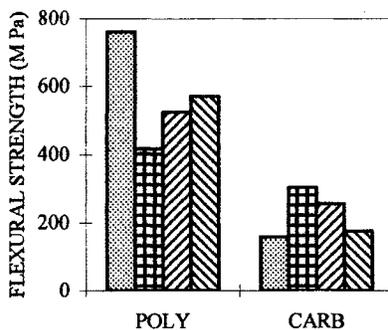
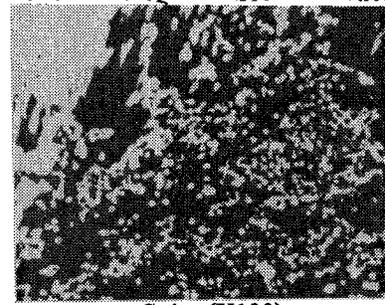


Figure 3. Flexural strength of composites. S-0(•••); S-4(•••); S-8(•••); S-12(•••).



S-4 (X100)

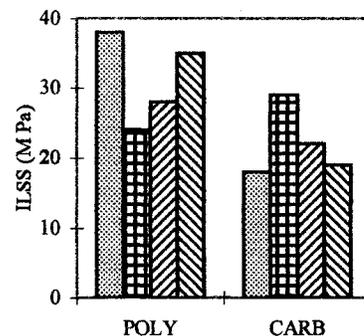
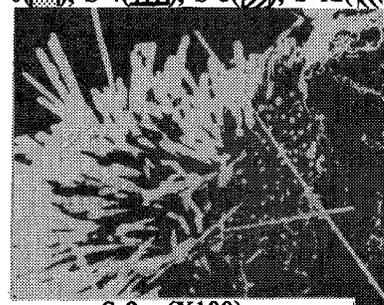


Figure 4. ILSS of composites.

S-0(•••); S-4(•••); S-8(•••); S-12(•••).



S-8 (X100)

Figure 2. Fracture surface of C/C composites