

Mechanical Properties of 3D Noninterlaced C_f/SiC Composites Prepared through Hybrid Process (CVI+PIP)

A. Udayakumar, M. Rizvan Basha, M. Stalin, V.V Bhanu Prasad

Abstract—Three dimensional non-Interlaced carbon fibre reinforced silicon carbide (3-D-C_f/SiC) composites with pyrocarbon interphase were fabricated using isothermal chemical vapor infiltration (ICVI) combined with polymer impregnation pyrolysis (PIP) process. Polysilazane (PSZ) is used as a preceramic polymer to obtain silicon carbide matrix. Thermo gravimetric analysis (TGA), Infrared spectroscopic analysis (IR) and X-ray diffraction (XRD) analysis were carried out on PSZ pyrolysed at different temperatures to understand the pyrolysis and obtaining the optimum pyrolysing condition to yield β -SiC phase. The density of the composites was 1.94 g cm⁻³ after the 3-D carbon preform was SiC infiltrated for 280 h with one intermediate polysilazane pre-ceramic PIP process. Mechanical properties of the composite materials were investigated under tensile, flexural, shear and impact loading. The values of tensile strength were 200 MPa at room temperature (RT) and 195 MPa at 500°C in air. The average RT flexural strength was 243 MPa. The lower flexural strength of these composites is because of the porosity. The fracture toughness obtained from single edge notched beam (SENB) technique was 39 MPa.m^{1/2}. The work of fracture obtained from the load-displacement curve of SENB test was 22.8 kJ.m⁻². The composites exhibited excellent impact resistance and the dynamic fracture toughness of 44.8 kJ.m⁻² is achieved as determined from instrumented Charpy impact test. The shear strength of the composite was 93 MPa, which is significantly higher compared 2-D C_f/SiC composites. Microstructure evaluation of fracture surfaces revealed the signatures of fracture processes and showed good support for the higher toughness obtained.

Keywords—3-D-C_f/SiC, charpy impact test, composites, dynamic fracture toughness, polysilazane, pyrocarbon, Interphase.

I. INTRODUCTION

THE continuous carbon fibre reinforced silicon carbide ceramic matrix (C_f/SiC) composites have gained considerable attention for thermo-structural applications, such as the hot-end components of gas turbine engines, thermal protection system of spacecraft, ultra light-weight mirrors, and aircraft brakes etc., because of its superior strength, fracture toughness and abrasive properties [1]-[5].

Three-dimensionally (3D) woven ceramic matrix composite materials are being developed to reduce the anisotropic

behavior in mechanical and thermal properties [6]-[15]. Isothermal isobaric chemical vapor infiltration (ICVI) technique is widely used for the fabrication of SiC ceramic matrix composites, since it yields β -SiC matrix having outstanding mechanical properties [4], [5]. However, it is an expensive process with product thickness limitation, involving hazardous precursors and requires long processing time (several hundred hours). Also, ICVI has slow densification rate and the densification eventually stops when the pores on the surface closed by clogging [6]-[8]. In the recent past, polymer to ceramic transformation process has enabled significant breakthroughs in the development of ceramics in terms of cost, ease of process without much compromise in the properties. Several polymer precursors such as polycarbosilanes, polycarbosiloxanes, polysilazanes etc., have been used as pre-ceramic polymers for the preparation of ceramics. Polymer impregnation pyrolysis (PIP) technique can be used for making SiC matrix in C_f/SiC composite which will enable infiltration of SiC matrix [9]. However, composites prepared using PIP technique alone will have a matrix with poor properties due to poor crystalline nature of the matrix [10].

The present research work involves, the preparation of 3D-C_f/SiC composites by ICVI combined with polysilazane pre-ceramic PIP process and study the mechanical properties. The objectives of the present study is to develop and understand the effect of the fibre architecture in the composite on the mechanical properties, the failure behavior of the 3-D C_f/SiC composites, and to develop an hybrid process (PIP+CVI) to reduce the processing time, and make relatively thicker materials. The scanning electron microscopy study is also carried out on the polished cross section and fracture surfaces of the composite to understand the failure modes.

II. MATERIAL AND METHODS

A. PSZ Curing and Pyrolysis

Polysilazane 20 (PSZ) (KION corporation, USA) is mixed with 0.1 wt% of 98% pure dicumyl peroxide initiator (Sigma Aldrich, Japan), cured at temperature 413 K for 5 h. and a pale yellow transparent PSZ cured solid is obtained. Cured solid is then heated at a heating rate 2°C min⁻¹ with argon flow (1-2 standard litres per minute (SLM)), and pyrolyzed at different temperatures between 850-1500°C for 3 h. X-ray diffraction analysis is carried out for all the pyrolyzed samples to identify the temperature at which the β -SiC is formed. TGA and FTIR analysis are also carried out to understand the curing and pyrolysis of PSZ.

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B. Fabrication of the Composites

T-300 carbon 12k fibre is employed for the fabrication of 3-D preforms (~320 mm in length, 320 mm in breadth and 8 mm in thickness) by knobbing technique (Thulasi Industries, Coimbatore, India). The fibre volume fraction maintained in warp, weft and Z directions are 0.4, 0.4 and 0.2. The basic unit-cell geometry of the preform is illustrated in Fig. 1.

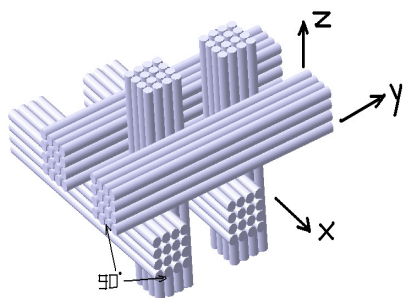


Fig. 1 Basic unit-cell geometry of the preform

Pyrolytic carbon (PyC) interphase layer and SiC matrix are obtained via ICVI process, which has been described previously in detail [16]-[18]. PyC interphase is deposited on the surface of the carbon fibres by cracking methane (CH_4) with nitrogen as dilution gas prior to matrix densification. Reaction condition used for the deposition of PyC interphase is namely: temperature 1473 K, pressure 0.1-0.2 kPa, and coating period 5 h. Methyltrichlorosilane (MTS, CH_3SiCl_3) vapor and hydrogen as carrier/catalyst gas are used for SiC matrix infiltration. Typical conditions used for the densification of the preforms with SiC matrix are: temperature 1233 K, hydrogen: MTS molar flow rate ratio 16:1, and a pressure 0.1-0.2 kPa. After 160 h of SiC matrix infiltration, the partially densified laminate is obtained and it is subsequently vacuum impregnated with PSZ pre-ceramic polymer and cured at 150°C. The cured laminate is then pyrolysed at 1350°C for 5 h in argon flowing atmosphere for getting SiC matrix. The laminate is continued to densify with CVI SiC matrix for further period of 120 h and final 3-D C_f/SiC composite laminate (Fig. 2) is obtained.

C. Mechanical Properties Evaluation

The density of the specimens is determined using Archimedes method (EN 1388-2004) [19]. Mechanical properties of the composite materials are studied under tensile, flexural, shear, and impact loading. Dog bone type specimens measuring 200 mm in length and 10 mm in width prepared using water jet cutting machine (HWM-6025-1/2D, Waricut Cutting Systems, Germany) with a finished gauge section measuring 40 mm long, 8 mm wide, and 4.5 mm thick are used for tensile testing. Tensile strength is measured at RT, 500°C, and 1000°C. Flexural strength is measured using the three-point-bending method at RT. Inter-laminar shear stress (ILSS) of 3-D C_f/SiC composites is measured by loading a double-notched specimen of uniform width as per ASTM C 1292-10 (2005) standard in compression [20]. The V-notch depth and width are 5.72 mm and 0.5 mm, respectively. The

Impact tests are performed using instrumented Charpy equipment. The specimen having the dimensions: 56 mm (L), 6.7 mm (B), and 10 mm (T) is used for the test. Impact velocity 3.35 m.s^{-1} is chosen for the test. The microstructure of the polished cross section and fracture surfaces is observed using scanning electron microscope.



Fig. 2 Photograph of 3-D C_f/SiC composite laminate

III. RESULTS AND DISCUSSION

A. Polymer Curing/Pyrolysis Study

The thermo-gravimetric (TG) curve obtained for the PSZ is shown in Fig. 3. It reveals three distinct regions based on the weight loss with increasing temperature namely: region-1 (150-300°C), region-2 (300-550°C), and region-3 (550-750°C).

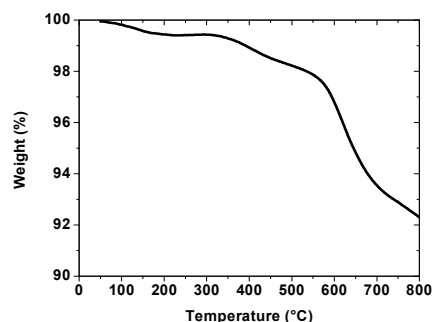


Fig. 3 TGA curve of PSZ precursor in nitrogen atmosphere

In region-1, a minimal weight loss of about 0.2% is observed. In the region-2, a gradual loss in weight of about 1.8% is noted. A steep reduction in weight of about 7.4% has been observed in region-3. Small fractional loss below 350°C corresponds to an optimized degree of cross-linking [21]. In region-1, the loss of low molecular weight oligomers occurs by volatilization. This may be attributed to inadequate cross-linking, depolymerisation reactions such as dehydrogenation, vinyl polymerization, and hydrosilylation. The transamination reaction leading to the formation of Si_3N_4 may be attributed to the reduction in weight in the region-2 [22]. In region-3, the N-H, Si-H and C-H bonds are broken and small molecules such as hydrocarbons and hydrogen are evolved accounting for the large weight loss observed. However, the weight loss is restricted by the presence of the methyl group in the repeating

unit which offers steric hindrance to the reaction which helps enabling a higher yield [23].

The Infrared (IR) spectra obtained for cured neat PSZ, and cured PSZ pyrolyzed at different temperatures (850°C, 900°C, 950°C, 1000°C, 1100°C, 1200°C, 1350°C and 1500°C) is shown in Fig. 4. The pyrolysis heating rate is maintained at 2°C min⁻¹ for all the samples as the heating rate inversely affects the ceramic yield.

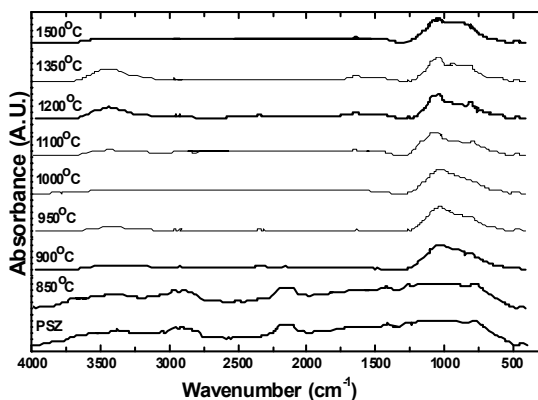


Fig. 4 Infrared spectra of polysilazane pyrolysed at different temperatures in argon atmosphere

When the pyrolysis temperature is increased (Fig. 4), a decrease in the intensity and broadening of the absorption bands are observed. The spectrum of the as cured sample reveals the band intensities corresponding to vinyl group (1404 cm⁻¹). At 850°C, the absorption bands arising from Si-H (2158 cm⁻¹), C-H (2916 cm⁻¹), and N-H (3383 cm⁻¹) are observed [24], [25]. With further increase in pyrolyzing temperature, the organic to inorganic transformation occurs. In the temperature range 900-1100°C, the absorption bands corresponding to Si-H, C-H, and N-H are completely eliminated. The spectra show broad overlapping absorption bands arising from Si-C and Si-N in the 600-1200 cm⁻¹ range. This marks the formation of amorphous SiC. In the temperature range 1200-1350°C, in addition to the absorption bands corresponding to Si-C, the presence of absorption bands corresponding to N-H (3434 cm⁻¹) are also noticed. Beyond 1350°C, the stability and the crystallisation of the Si-C phases depends on the nature of the pyrolysis atmosphere. The decomposition under argon yields a pyrolytic residue almost totally free of nitrogen but enriched by a crystalline SiC phase.

The XRD patterns of the precursors pyrolysed at different temperatures (850-1500°C) are shown in the Fig. 5. It is evident that SiC is not formed till 1000°C. The prominent peak of β -SiC at $2\theta=35.5^\circ$ emerges at 1350°C marking the onset of crystallization.

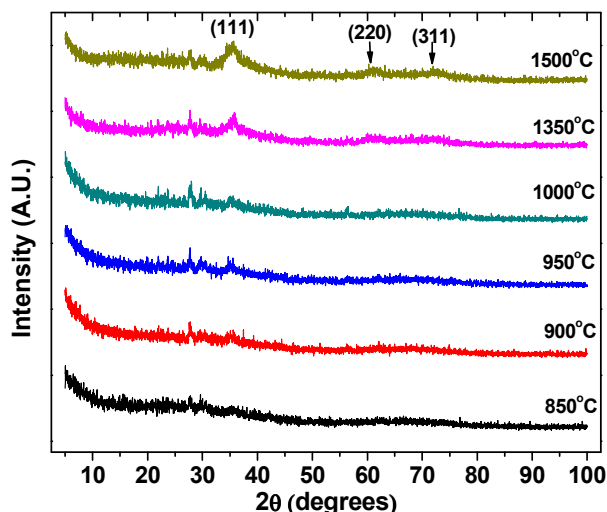


Fig. 5 X-ray diffraction patterns of polysilazane pyrolysed at different temperatures

The XRD pattern of the PSZ pyrolysed at 1500°C show three diffraction peaks at $2\theta=35.5, 60$ and 72° which are due to (111), (220) and (311) planes of β -SiC as marked in Fig. 5 [26]. No clear diffraction peak has been observed corresponding to Si-N or Si-N-C ceramics. This indicates that at a temperature of 1500°C in the presence of argon, the crystallization of SiC is favoured. This also indicates that a minimum temperature of about 1350°C is required to obtain crystalline SiC. The ceramic yield of the precursor is found to be 74 %.

B. Mechanical Characterisation: Tensile Loading

The density of the composite is 1.94 g.cm⁻³ after the three dimensional non-interlaced carbon fibre preform was SiC infiltrated by CVI for 280 h with one intermediate cycle of polysilazane pre-ceramic polymer impregnation pyrolysis. Figs. 6 and 7 show the typical tensile fracture behavior of 3-D C_f/SiC composite at RT, and at elevated temperatures. The mechanical behavior is found initially elastic followed by non-linear region reflecting the matrix cracking (region of stable crack growth). It is also observed that the transition from linear region to non-linear region was very smooth. The composites failed catastrophically after reaching the ultimate tensile strength. The fibre architecture in the composites played a major role in the observed tensile behavior. Few fibre bundles (12K) are available in the cross section of gauge section and they pulled out. From the tensile stress-strain curve, it is evidenced that the interphase coating has controlled the interfacial bond strength (weak enough) between fibre and matrix, which in-turn facilitated the de-bonding, fibre pullout etc.

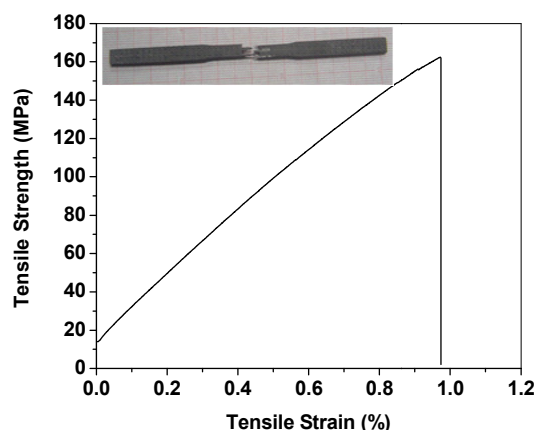


Fig. 6 Tensile stress vs strain curve obtained for 3-D C_f/SiC composites at RT

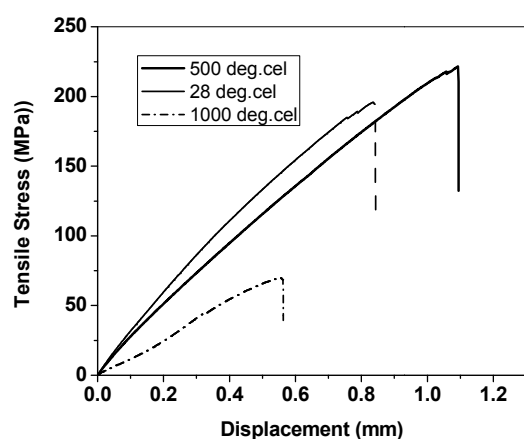


Fig. 7 Tensile stress vs displacement plot obtained for 3-D C_f/SiC composite at a) RT b) 500°C, and c) 1000°C

From the SEM micrograph of the polished cross section of 3-D C_f/SiC composite shown in Fig. 8, it is observed that the PyC interface is uniform and thin enough (~188 nm) to facilitate de-bonding. Fig. 9 shows the SEM micrographs of the fractured surface of tensile specimens tested at various temperatures. The energy absorption mechanisms such as fibre/matrix de-bonding, fibre pullout are observed. The tensile data obtained for the 3-D C_f/SiC composites are summarised in Table I.

TABLE I
SUMMARY OF MECHANICAL PROPERTIES OF 3-D C_f/SiC COMPOSITE

PROPERTY	Value
Fracture toughness (K _{IC}) @28°C	39±7.9 MPa m ^{1/2}
Young's modulus	35 ±1.7 GPa
Tensile Strength at 28°C	178 ±25.7 MPa
Tensile Strength at 500°C	194 ±15 MPa
Strain to failure	0.75±0.1
work of fracture	2.8 kJ.m ⁻²
Dynamic fracture toughness through Charpy impact test	44.8 kJ.m ⁻²
Interlaminar shear stress	93.0 MPa

MPa = Mega Pascal; m = metre; GPa = Geiga Pascal; kJ = kilo joule

Lower tensile strength observed at 1000°C is due to the oxidation of PyC interphase and C fibre because of oxidising atmosphere. The oxidation of carbon constituents is clearly observed from SEM micrograph (Fig. 10) of respective fracture surfaces.

It has been reported that the thermal expansion co-efficient of the SiC matrix and the PAN based carbon fibre are $2.2 \times 10^{-6} \text{ K}^{-1}$ and $7-12 \times 10^{-6} \text{ K}^{-1}$ (in the direction parallel to fibre), respectively [26], [27]. After the composites are cooled from the infiltration temperature (960°C) to RT, a tensile stress is generated across the interphase layer. Hence, it is quite easy for the fibre to be pulled out from the SiC matrix. When the temperature was increased above the SiC infiltration temperature (960°C), a compressive stress is generated across the interphase layer and resulted in the strong interfacial bond between fibre and matrix. This makes fibre difficult to be pulled out of matrix (cleavage fracture).

The fibre pull out length for the sample tested at 1000°C (Fig. 9 (c)) is shorter than samples tested at RT and 500°C (Figs. 9 (a) and (b)). This observation is similar to the observation made for the three dimensional textile Hi-Nicalon SiC_f/SiC composites prepared by chemical vapor infiltration [28]. Fibre bundle pullout was always observed at RT and high temperature (Fig. 9).

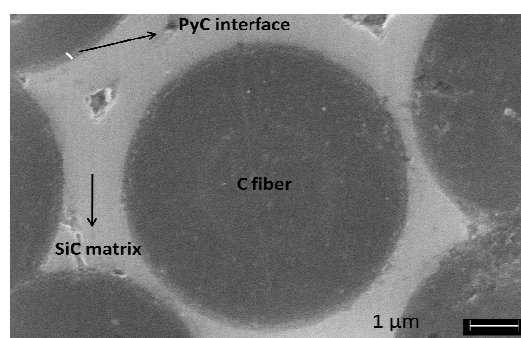


Fig. 8 The SEM microstructure of the polished cross section of 3-D C_f/SiC composite

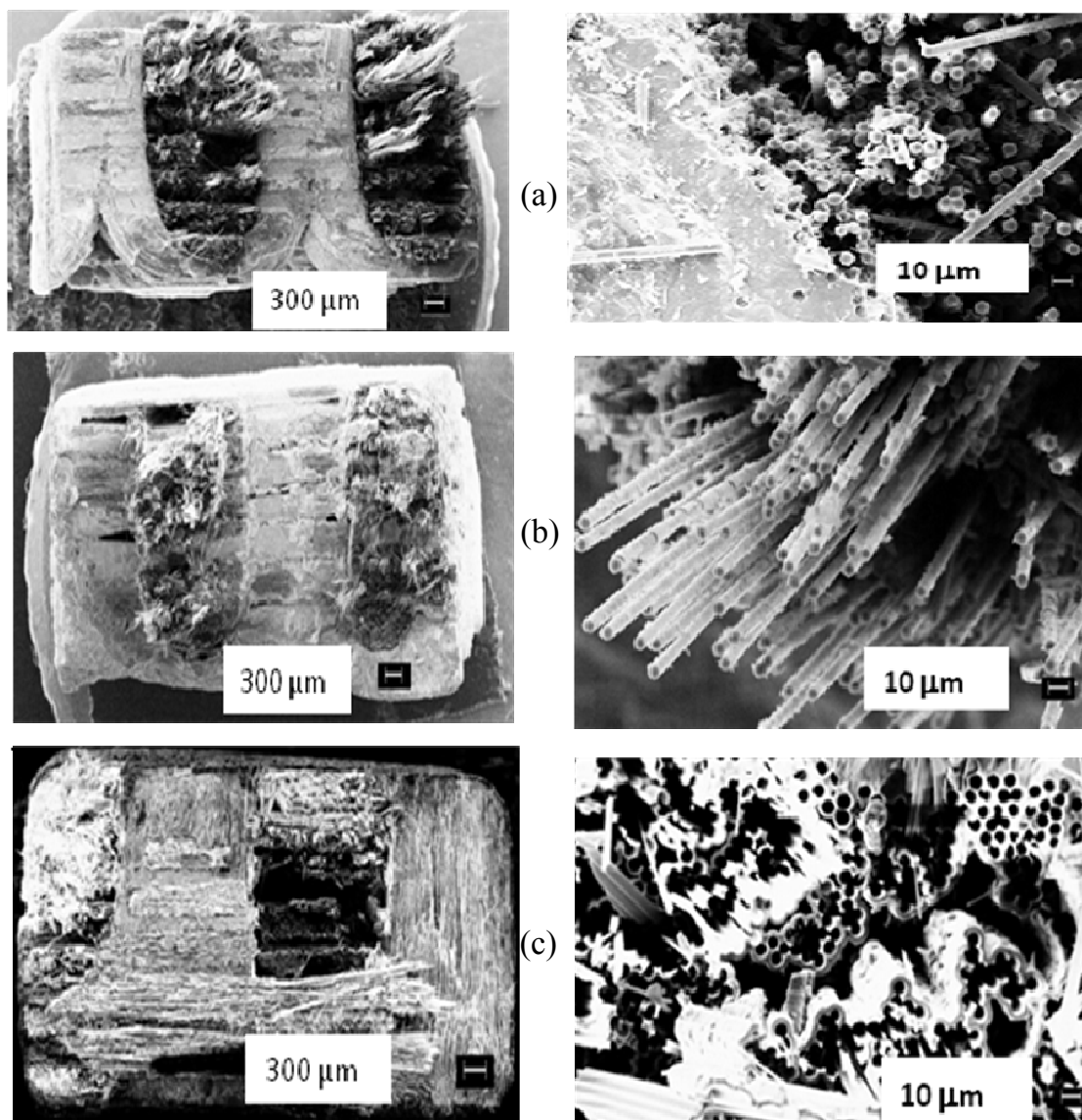


Fig. 9 SEM microstructure of tensile fractured surfaces of 3-D C_f/SiC composites tested at (a) RT, (b) tested at 500°C, and (c) tested at 1000°C

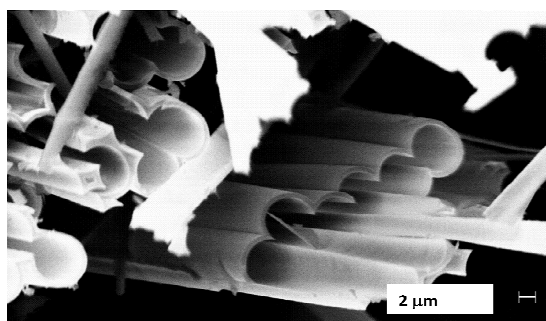


Fig. 10 SEM micrograph of fracture surface of tensile fractured 3-D C_f/SiC composite tested at 1000°C

C. Mechanical Characterisation: Flexural Loading

Typical failure behavior of 3-D C_f/SiC composites under flexural loading at RT is shown in Fig. 11. Flexural stress Vs displacement curve is initially showing linear behavior, then non linear region is observed reflecting matrix cracking and residual strain.

Finally fibre failure occurs causing the fracture of composites. The average flexural strength of 243 ± 25 MPa is obtained. The lower strength is attributed to the lower density and phase impure SiC matrix obtained from pyrolysis of polysilazane. The lower flexural strength is also due to inter-bundle porosity (Fig. 12).

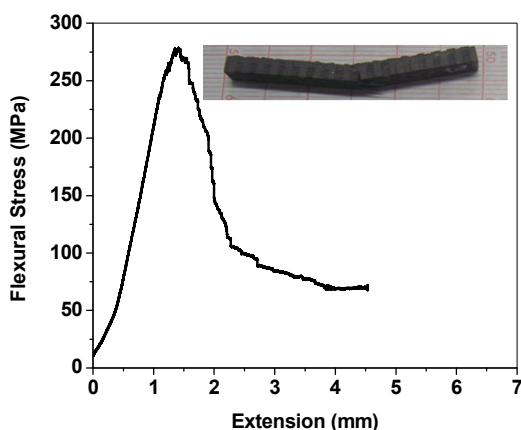


Fig. 11 Stress vs extension trend obtained for 3-D C_f/SiC composites under flexural loading

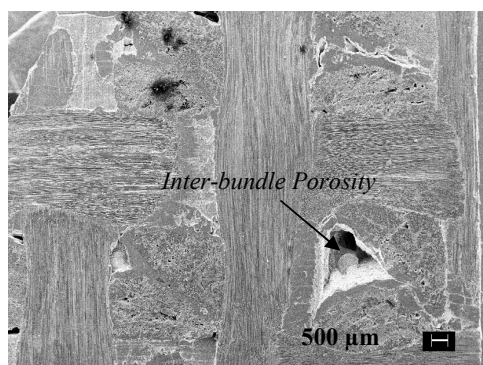


Fig. 12 SEM micrograph polished cross section of 3- D C_f/SiC composite showing the residual Pores

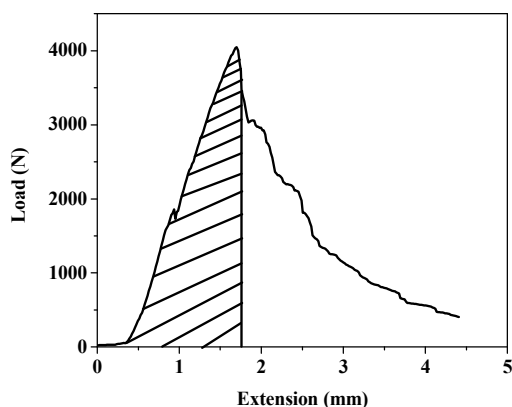


Fig. 13 Load vs displacement trend obtained from the SENB test

D. Failure Behavior of Notched Specimen under Flexural Loading

To determine the fracture toughness of the composites, SENB specimens are generated and tested in three point bending configuration. The span length: 40 mm and loading rate: 0.17 mm.min⁻¹ are used for the testing. Load vs displacement trend obtained from the SENB test is shown in Fig. 13. The fracture toughness derived from SENB test is 39

±1.9 MPa.m^{1/2}, which is nearly 10 times that of typical monolithic ceramic materials (3-5 MPa.m^{1/2}), and two times that of 2D C_f/SiC composites (~20 MPa.m^{1/2}). The work of fracture is obtained from the characteristic area (area under the curve starting from the initial point to the 10% decrease of the curve: Fig. 13) under the load-displacement curve divided by the cross sectional area of the specimen. The average work of fracture value of 22.8 kJ.m⁻² is obtained.

E. Mechanical Characterisation: Impact Loading

Instrumented Charpy Impact tests on un-notched specimens are carried out to obtain the energy absorbing capability and dynamic fracture behavior of the composite materials. Equation (1) is used to calculate the dynamic fracture toughness (α_k).

$$\alpha_k = \frac{\Delta w}{bt} \quad (1)$$

where, Δw is the absorbed energy by composite during impact, b is the breadth and t is the thickness of the specimens. The value of α_k obtained for 3-D- C_f/SiC composite materials is 44.8 kJ.m⁻². It is lower than that of super alloy ($\alpha_k = 80$ -160 kJ.m⁻²). The impact fracture surface is like brush bristles as shown in Fig. 14.

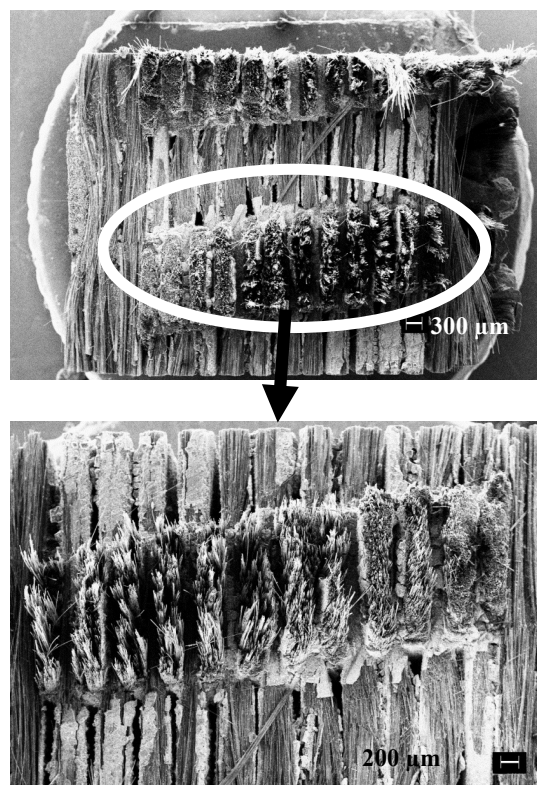


Fig. 14 The SEM micrographs of impact fracture surface of 3D- C_f/SiC composites

The results revealed that the 3D C_f/SiC composites exhibited excellent resistance against dynamic impact.

F. Failure Behaviour under Shear Loading

It is observed that shear failure occurred between two centrally located notches generated half way through the thickness and faced a fixed distance apart on opposing faces. Equation (2) is used for calculating ILSS.

$$ILSS = \frac{P_{max}}{A} \quad (2)$$

where P_{max} is the applied maximum load and A is shear stressed area. Equation (3) is used to compute A .

$$A = wh \quad (3)$$

where, w is the average specimen width and h is the distance between the V-notches. Shear strength of 93 MPa is obtained, and it is much higher than that of 2-D C_f/SiC and 2D SiC/SiC composites. Interlayer de-bonding is not occurred in the present composites, which are favored to get low or nil rejections due to de-lamination during fabrication.

IV. CONCLUSION

High toughness, non-interlaced, three dimensional T-300 carbon fibre reinforced silicon carbide matrix composites (3-D C_f/SiC) were fabricated by ICVI process with one intermediate polysilazane preceramic polymer impregnation pyrolysis cycle. The density of the composite was $\sim 1.94 \text{ g.cm}^{-3}$ after the preform was silicon carbide infiltrated for 280 h and densification by one intermediate polysilazane preceramic polymer impregnation pyrolysis. The values of tensile strength were $178 \pm 25.7 \text{ MPa}$ at RT, $194 \pm 15 \text{ MPa}$ at 500°C and $70 \pm 8 \text{ MPa}$ at 1000°C . The RT flexural strength was 280 MPa. At 1000°C , the failure behavior of the composite became brittle due to compressive stress generated cross the interphase layer caused by the thermal expansion coefficient mismatch between fibre and matrix. The fracture toughness and work of fracture were as high as $39 \text{ MPa.m}^{1/2}$ and 22.8 kJ.m^{-2} . Significantly higher shear strength (93.3 MPa) was achieved as required to have nearly isotropic properties. The composite also exhibited excellent resistance against dynamic impact. The dynamic fracture toughness was 44.8 kJ.m^{-2} .

ACKNOWLEDGMENT

The authors would like to thank Mrs. Kalavati for the SEM studies, Mr. P. Kumara, Mr. S. Ganesh, Mr. M. Suresh and Ms. M.V. Geetha for their help in carrying out the experiments, and Mr. A. Sri Ganesh for mechanical testing. The authors record sincere thanks to Dr. S K Bhaumik, Head, Materials Science Division of CSIR NAL Bangalore for his continuous support and encouragement. Thanks are due to Dr. Nick J. Archer and Mr. John Yeatman from M/s. Archer Technicoat Limited, UK for their support and suggestions. The authors thank Dr. Debalay Chakraborty and Dr. Rahul Mitra for instrumented Charpy test. The authors also thank the Director, CSIR-NAL for his active support and constant encouragement. Financial support from DMRL and CSIR is also acknowledged.

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