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INTERLAMINAR SHEAR STRENGTH OF TEXTILE REINFORCED CARBON-CARBON COMPOSITE

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Interlaminar fracture of carbon fabric reinforced carbon composites with different volume fractions of reinforcement was investigated by tensile loading of double-notched specimens. They were carbonised in nitrogen at 1000 °C and heat treated in argon at 2200 °C, or twice impregnated by the resin and finally graphitised. The elastic properties of unnotched specimens were measured by the resonant frequency technique. Dynamic tensile and in-plane shear moduli of all materials increased with increasing volume fraction of reinforcement. However, double impregnation and recarbonisation process led to more prominent growth of these elastic parameters, which should be attributed to the role of matrix in transfer of stresses between fibres. After graphitisation, the in-plane shear modulus fell remarkably which is due to "stress" graphitisation of the matrix resulting in its softening in shear. The interlaminar shear strength yielded maximum values for specimens made up of 8 reinforcing layers (volume fraction $V_f \approx 48$ *%). Both the graphitisation and repeated impregnation improved the shear strength, the former being much more effective at lower volume fractions of reinforcement (i.e., higher content of matrix) than under inverse conditions. The large impact of heat treatment upon properties of matrix-rich composites can be understood, as the matrix is more susceptible to changes induced by heat treatment than the fibres.*

Keywords: carbon-carbon composite, fabric reinforcement, interlaminar shear strength, dynamic tensile and shear modulus, fracture surface.

Laminated composites are prone to interlaminar failure even when loaded by a general stress. The interlaminar mode of failure is potentially the major life-limiting failure process in these composites. Under flexural load their interlaminar fracture is by far more probable than tensile or compressive failure - unless the composite plate is sufficiently thin. Interlaminar failures occur in the plane of reinforcement and their fracture mechanisms are governed by matrix fracture and fibre-matrix separation while fibre breaking is sporadic. On a microscale the interlaminar fracture can occur under mode I tension or mode II in-plane shear. In both cases the separation occurs by brittle tension the only difference being the orientation of tension stress with respect to the plane of failure.

It is interesting to study these phenomena in a composite system where the matrix is gradually developed, e.g., in composites with ceramic matrix made by impregnation and pyrolysis of

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polymer precursors. By measuring appropriate mechanical properties (interlaminar shear strength or in-plane shear modulus) after completion of subsequent technological steps the impact of matrix densification and structural transformation can be monitored. In the present study, interlaminar fracture of carbon fabric reinforced carbon (or, C-C) composites was pursued during investigation of their suitability as substrates for biocompatible thin films.

Experimental

The material was manufactured in laboratory using a plain weave fabric reinforcement woven from PAN-based Toray T800-6K carbon tow. In order to optimise the construction of reinforcement a series of specimens were made with different volume fractions of reinforcement. This was achieved by stacking 6 to 10 layers of fabric soaked with matrix precursor (diluted phenolic resin) and curing them at ≈ 125°C during pressing to the same thickness (approximately 2.5 mm). The reinforcement volume fraction V_f was calculated using fibre density 1.81×10^6 g.m^{-3} and fabric density 270 g.m^{-2} to range 36 – 60 %.

The cured plates were cut to specimens 50 × 10 × 2.5 mm oriented along the weft tows. They were carbonised in nitrogen at 1000°C (*C*), "graphitised" (*CG*) i.e. heat treated in argon at 2200°C, or twice impregnated (*I*) by the resin, re-carbonised (*C-I-C-I-C*) and finally graphitised (*C-I-C-I-C-G*). The carbonised only material was rather porous due to mass-loss on pyrolysis. During further processing its open porosity decreased by 9 - 14 % on average. 5 specimens of each construction (6 – 10 layers) and treatments *C, CG,* and *C-I-C-I-C-G* were tested to interlaminar shear strength.

Prior to strength testing of the composites the elastic properties of the unnotched specimens were measured by a resonant frequency technique. The dynamic tensile modulus E_{11} was determined from the basic longitudinal resonant frequency of a beam with free ends. The in-plane dynamic shear modulus G_{12} was measured by a resonant frequency method described in [1]. The method relies on direct numerical solution of the complete frequency equation for flexural vibrations of a beam with free ends. The resonant frequencies were measured using the electrodynamic resonant frequency tester Erudite (CNS Electronics Ltd., London, UK) at frequencies up to 100 kHz. The vibrations were excited and detected without mechanical loading so that the boundary condition of free-end beam was fulfilled.

In-plane shear strength was measured by tensile loading of double-notched specimens (Fig. 1). Although an ASTM (D 3846) standard, the specimen shape is often criticized

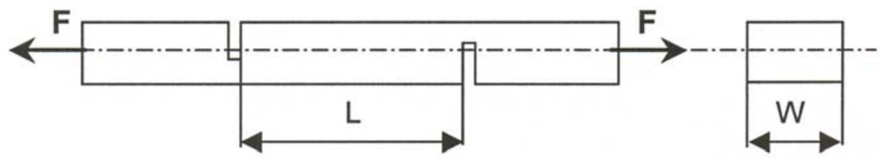


Fig. 1 Specimen shape and arrangement of in-plane shear strength measurement.

because of normal and shear stress concentrations induced at the bottoms of the notches. They can lead to premature local failures propagating immediately across the whole region between the notches. Furthermore, the ILSS values strongly depend on the relative notch distance [2].

Similarly to the short beam shear test (ASTM D 2344) and to the DN compression test (ASTM D3846), the applied test can yield only "apparent" interlaminar shear strength, which at best should be used only for comparison of material performance. On the other hand, the specimen is easy to fabricate and the test procedure can be accepted if no design data are required.

In our case the specimens were mounted into pneumatic grips of an Instron 6025 testing machine and loaded in tension at a crosshead speed 1 mm.min^{-1}. The interlaminar shear strength was calculated from the maximum load as

$$ILSS = \tau_{\max} = \frac{F_{\max}}{L \times W}$$

where F_{max} is the maximum load, L = 20 mm is the notch distance, and W is the specimen width.

After test completion the fracture surfaces were inspected under S.E.M. (Tesla BM 301).

Results and discussion

Dynamic tensile and in-plane shear moduli of all materials generally increased with increasing number of fabric layers (and, therefore, volume fraction of reinforcement) in the composite (Fig. 2 and 3). The double impregnation and recarbonisation process led to more prominent growth of these elastic parameters, which should be attributed to the role of matrix in transfer of stresses between fibres constituting thus composite's elastic properties. Another aspect of the matrix role manifested itself in the influence of high-temperature treatment on the moduli: while a minor rise of the tensile modulus after graphitisation was detected (Fig. 2) the in-plane shear modulus fell remarkably (Fig. 3). The latter is due to "stress" graphitisation of the resin-derived matrix with lamellar-type microstructure oriented parallel to the fibres resulting in its shear-softening [3].

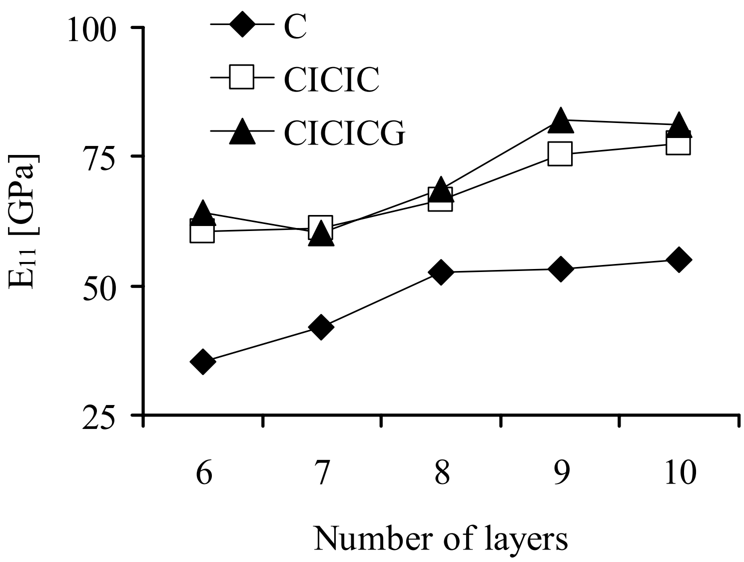


Fig. 2 Dynamic tensile modulus E_{11} plotted against number of fabric layers.

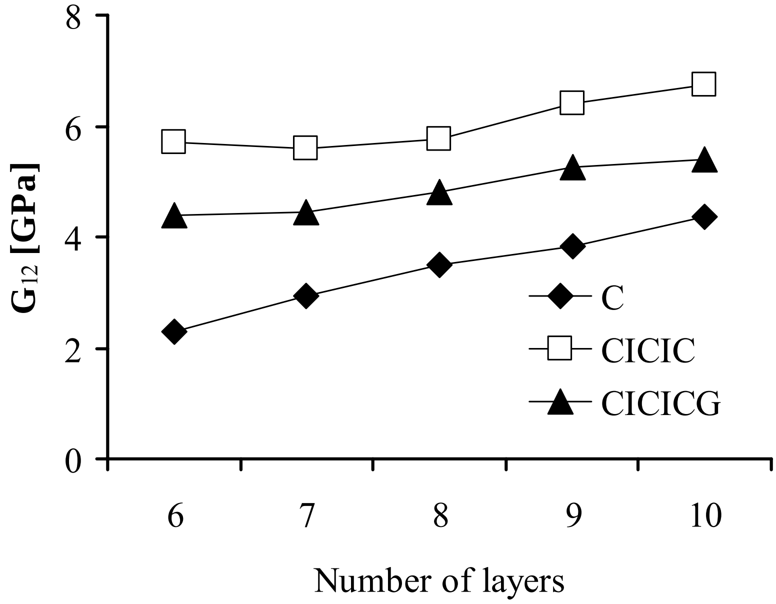


Fig. 3 Dynamic in-plane shear modulus G_{12} plotted against number of fabric layers.

While the elastic moduli increased monotonously with V_f. the interlaminar shear strength yielded maximum values for specimens made up of 8 reinforcing layers (Fig. 4). The corresponding reinforcement volume fraction V_f (approx. 48 %) is optimal from the viewpoint of shear strength for all the investigated processing levels. Both the graphitisation and repeated impregnation improved the shear strength, the former being much more effective at lower volume fractions of reinforcement (and – consequently – higher content of matrix) than under inverse conditions (Fig. 4). Having in mind that the matrix is more susceptible to changes induced by heat treatment than the (already heat treated during manufacture) fibres the large impact of heat treatment upon properties of matrix-rich composites can be understood.

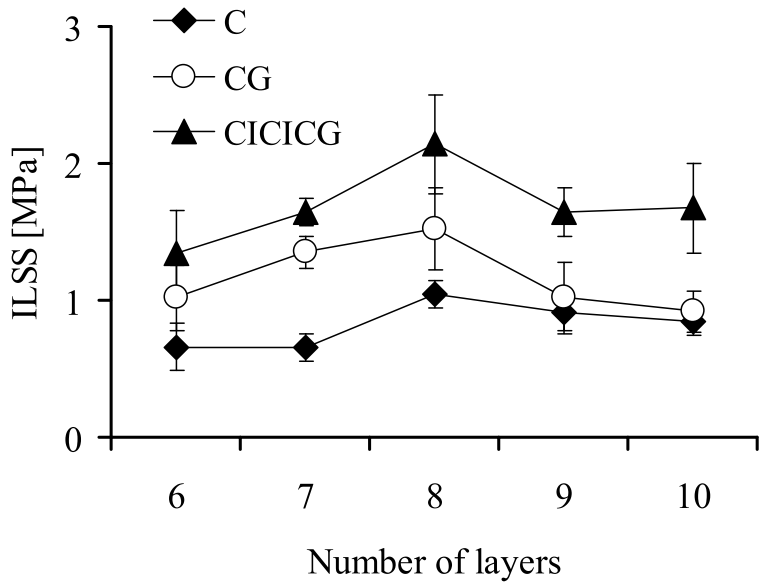
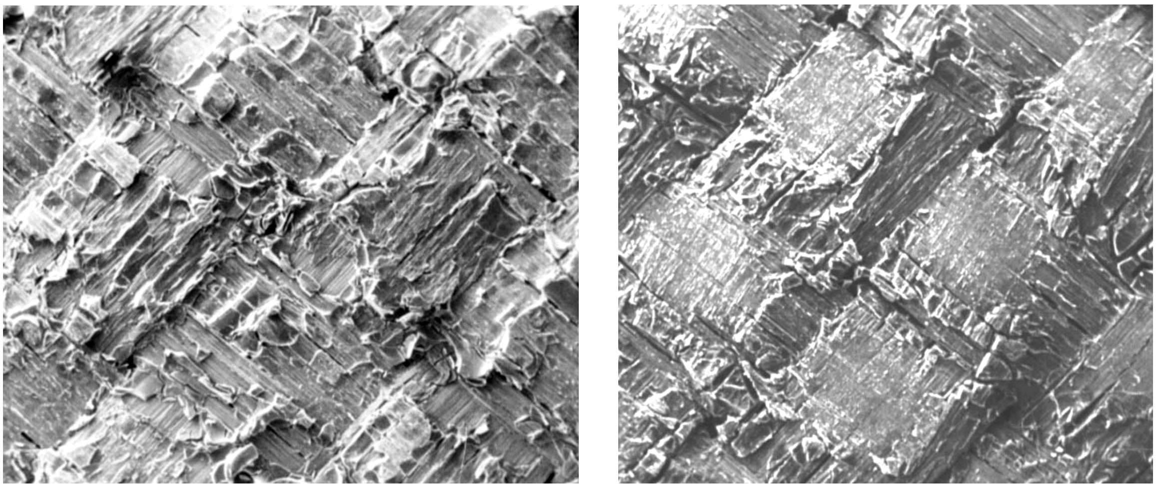


Fig. 4 Interlaminar shear strength plotted against number of fabric layers.

Morphology of the failure surfaces was quite variable in dependence on the composite construction and processing. In macroscale, a remarkable difference exists between the failure surface patterns of the twice impregnated and graphitised specimens with 6 or 8 fabric layers (Fig. 5 a, b).

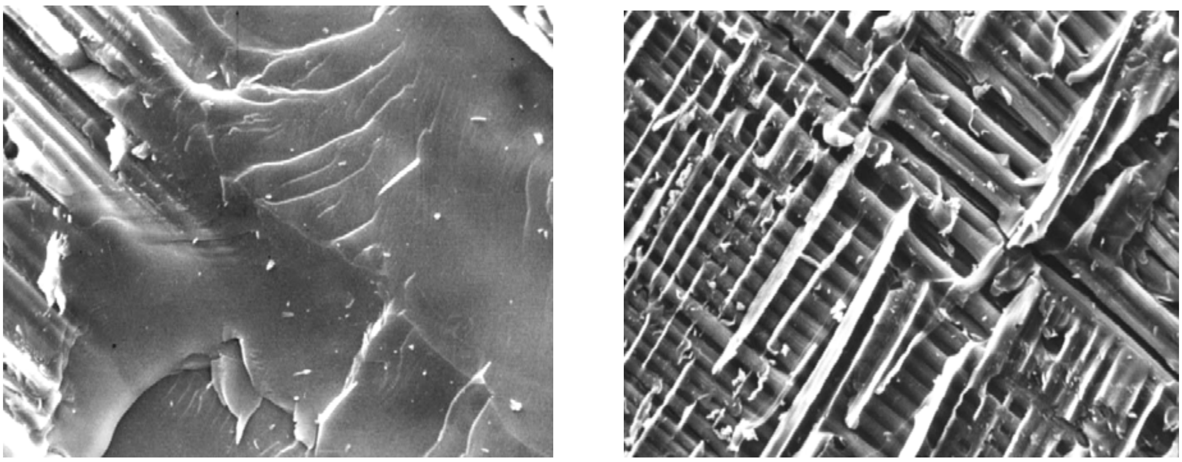


a 1 mm b

Fig. 5a, b. Failure surfaces of the twice impregnated and graphitised specimens (a – 6, b – 8 layers, load direction from the top-left towards the bottom-right corner).

The abundant brittle matrix (Fig. 5a) enables the crack to deviate from the plane of lamina and to propagate easily leaving large matrix fragments stuck at the surface. With an appropriate amount of matrix (Fig. 5b) its fragments are concentrated into the "pockets" near the tow crossing.

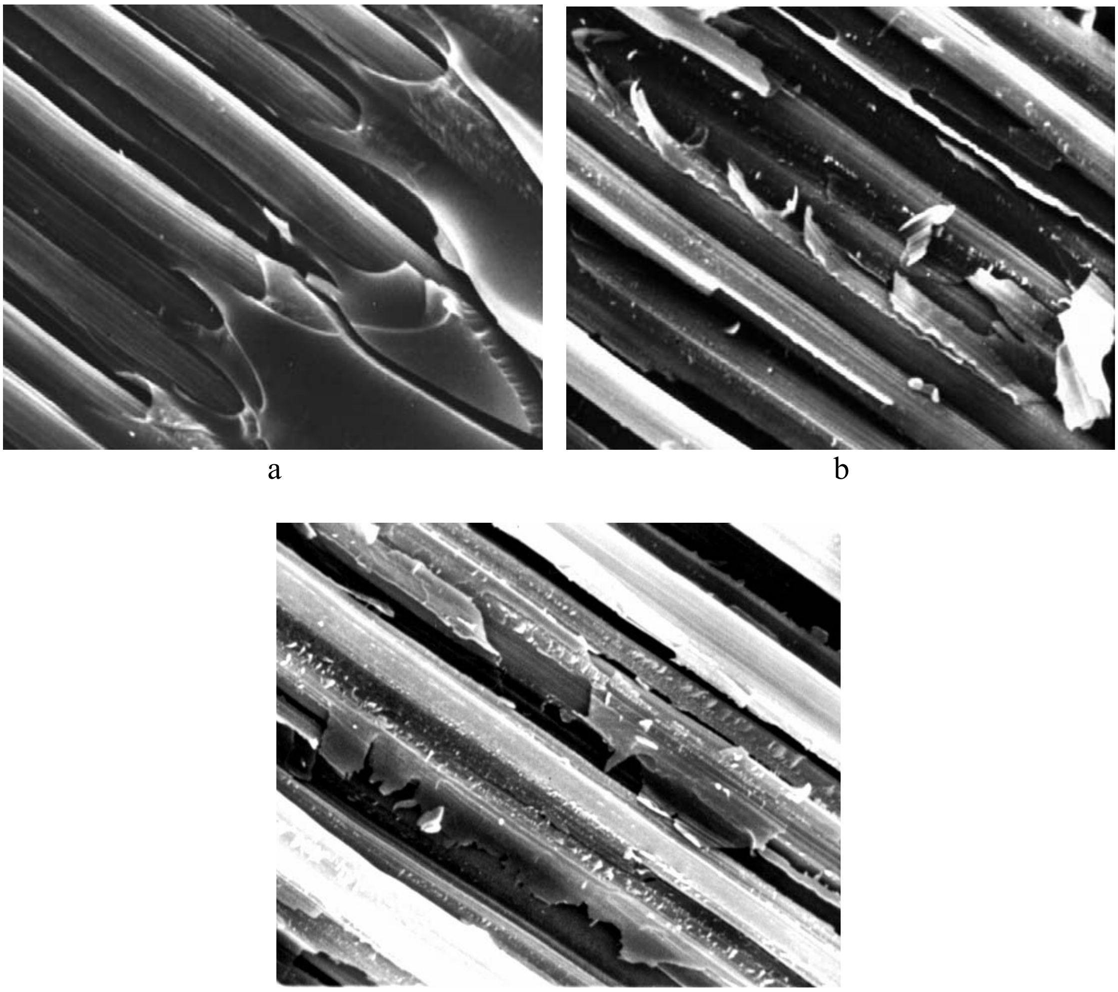
In microscale the fracture mechanisms can be distinguished (Fig. 6a, b). Mode I tension fractures with flat areas and river marks occur more frequently in the matrix-rich areas (Fig. 6a) while separate matrix fragments replicating the adjacent lamina orientation are found in the less abundant matrix (Fig. 6b).



a 40 μm b

Fig. 6a, b. Failure surfaces of the twice impregnated and graphitised specimens (a – 6, b – 8 layers, load direction from the top-left towards the bottom-right corner).

In a close detail (Fig. 7 a, b, c) the appearance of matrix fragments in the graphitised materials containing large, medium, or small amount of matrix is compared.



a b

c 15 μm

Fig. 7 a, b, c. Failure surfaces of the graphitised specimens (a – 6, b – 8, c - 10 layers, load direction from the top-left towards the bottom-right corner).

It can be concluded that in-plane shear behavior of the investigated composite materials is very sensitive to the proper choice of the fabric reinforcement volume fraction and to the parameters of heat treatment processing.

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