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THE WORK-OF-FRACTURE OF CARBON-CARBON COMPOSITES

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ABSTRACT

The work-of-fracture was measured for three different types of carbon-carbon composites including structures with substrates of a three dimensional woven preform and a fiber reinforced carbon felt. A semi-random chopped fiber structure was also measured. The fracture surfaces were examined by scanning electron microscopy. The work-of-fracture values are considerably larger than conventional carbon and graphite materials and can be partially explained in terms of the crack substrate interactions derived from the basic nature of the composite structure.

INTRODUCTION

Carbon-carbon composites are advanced composite materials noted for their high specific strengths and elastic moduli as well as their wear resistance, thermal shock damage resistance, and excellent ablation resistance at very high temperatures. Their mode of synthesis, one of first constructing a carbon substrate which may then be infiltrated or impregnated to contain a carbon matrix, permits the structure of carbon-carbon composites to be adapted to precisely tailorable properties. Because of these unique processing characteristics and their excellent thermochemical and thermomechanical properties, carbon-carbon composites have become leading candidates for the thermal protection materials on re-entry vehicles and in the nozzle systems of rocket motors. These composites are also

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finding application as the wear resistant components of high performance brake linings.

Considerable research has been conducted on the physical and mechanical properties of some of these composites[1-9], however, relatively little of it has been focused on their fracture characteristics[10,11]. Consequently, there does not exist a clear fundamental understanding of the fracture process of these types of materials. In order to gain further improvements of the properties of carbon-carbon composites and to guarantee their reliability in advanced applications, it is important to have a better understanding of their fracture processes. Since many of the aforementioned applications represent thermal stress situations, the crack propagation resistance or the work-of-fracture, γ_{wof}, is of prime consideration as it relates to the crack propagation under thermal shock conditions. It is the objective of this paper to report measurements of the work-of-fracture of several advanced carbon-carbon composites in an attempt to identify some of the fracture mechanisms. One goal of this study is to identify some of the fracture surface features and to correlate them with experimental fracture measurements and with the intrinsic characteristics of the substrate and matrix carbons, thus increasing the understanding of the fracture process and ultimately providing a basis for continued improvement of these most interesting composite materials.

EXPERIMENTAL

Three different types of carbon-carbon composites were studied in this investigation. Their descriptive classifications are as follows:

(i) (CC-T) a three dimensionally woven carbon-carbon composite,

(ii) (CC-F) a carbon felt substrate, fiber reinforced carbon-carbon composite, and

(iii) (CC-B) a semi-random, chopped fiber reinforced carbon-carbon composite.

Although the processing details of these composites are proprietary, a number of selected room temperature properties are listed in Table 1.

For the work-of-fracture measurements made in this study, two types of notched specimens were used: (a) a straight sawed notch specimen and (b) a Chevron type of notched specimen with a reduced triangular cross section. The notches were introduced by sawing with a 0.025 cm thick diamond blade to a specimen depth of approximately one half the sample thickness. Specimens 5x5x40 mm

Table 1. Selected Room Temperature Properties of the Composites

Composite:	CC-T	CC-F	CC-B
Density (gm/cm^3)	1.90-1.95	1.73-1.77	1.55-1.57
Young's Modulus (GPa)	48	32	31
Bend strength (MPa)	140	97	120
CTE $(25\text{-}1000)\times 10^{-6}/C$	1.5	2.6	--

were tested in three point bending at room temperature on a commercial testing machine using a 30 mm span and a 5.08×10^{-3} cm/min crosshead speed. The work-of-fracture was determined by integrating the area under the load displacement curve, $\int Fds$, and dividing it by twice the projected fracture area of one specimen half, A_f. The work-of-fracture is then defined as:

$$\gamma_{wof} = \frac{\int Fds}{2A_f}$$

To accurately measure the work of fracture, a completely stable fracture is necessary, however, this was no problem with these composites as catastrophic failure does not occur even for unnotched specimens(12-14). To identify specific fracture characteristics of these composites, their fracture surfaces were examined in a scanning electron microscope after coating with gold.

RESULTS AND DISCUSSION

The work-of-fracture values for the two specimen types of these different carbon-carbon composites are reported in Table 2. There is good agreement between the two types of notches, but there exists a wide difference in the magnitudes of the work-of-fracture values of these three carbon-carbon composites. The three dimensionally woven composite CC-T is clearly the most difficult structure in which to propagate a crack, while the chopped fiber composite CC-B is nearly as energy consuming; however, even though the fiber reinforced felt substrate CC-F was the lowest, it has a considerably larger work-of-fracture value than most other carbon and graphite materials. The work-of-fracture values of a number of other carbon and graphite materials are listed in Table 3 for comparison purposes(15-18).

Table 2. Work-of-Fracture of the Composites (J/m^2)

Sample Types	CC-T	CC-F	CC-B
Straight-Notch	5,430	360	1,750
Chevron-Notch	5,100	340	1,630

To a first approximation the relative ranking of these three carbon-carbon composites may be quite simply related to the crack propagation. For the fiber reinforced felt composite, CC-F, the developing cracks travelled parallel to the major plane of the felt, or in the direction of preferred crystalline alignment. This suggests that CC-F should probably exhibit the lowest work-of-fracture of these three carbon-carbon composites, and in fact it does. The interlocking structure of the three dimensionally woven substrate of the CC-T material promises very difficult crack propagation as fibers must be broken or pulled-out for all aspects of the crack propagation process in this composite. This indicates that very high work-of-fracture values are to be expected and indeed large work-of-fractures do result. The work-of-fracture values for CC-T of over 5,000 J/m^2 are some of the highest work-of-fractures ever reported for any brittle materials. For the carbon-carbon composite CC-B, the developing crack travelled essentially parallel to the direction of molding, which is perpendicular to the partial alignment of the chopped fibers. Because this results in considerable crack interaction with the chopped fibers, the work-of-fracture of CC-B is also expected to be high. It lies between that of CC-F and CC-T and indeed is quite high relative to the other carbon and graphite materials summarized in Table 3. This rather qualitative approach to the fracture of these composites is further justified from the macroscopic appearance of the fracture paths in the composites. Figure 1 illustrates this for the three composites. It is evident that relatively little crack branching occurs in CC-F, while there is considerable overlapping of cracked regions and auxiliary microcracking of fiber containing regions in the CC-B material and also strong evidence of a tortuous crack path in CC-T, the three dimensional woven substrate composite.

Although the previously discussed structural features and the manufacturing processes explain the order of the work-of-fracture values for these carbon-carbon composites, it is appropriate to further examine the fracture surfaces of these materials in greater detail to gain additional understanding of the structural effects

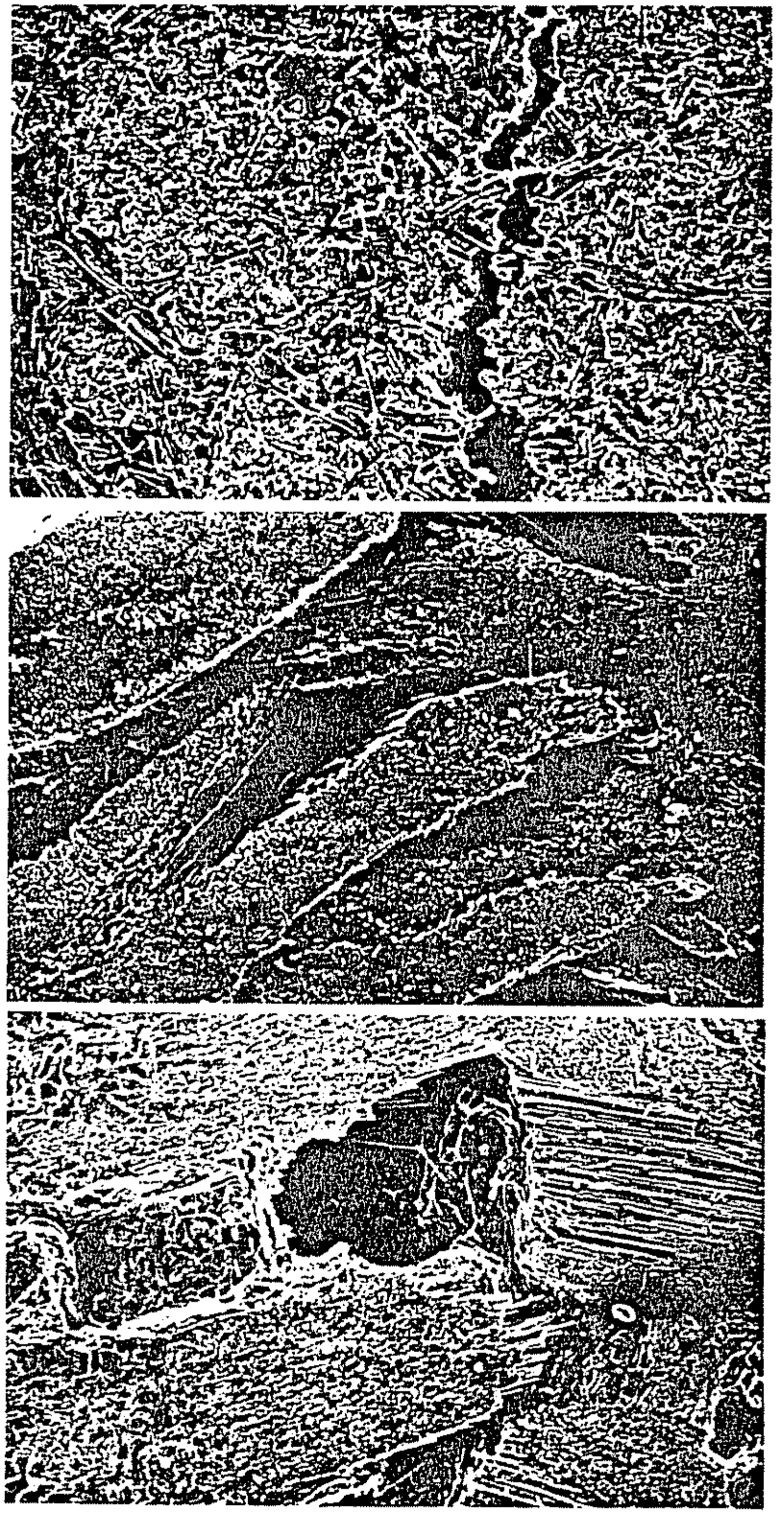

Figure 1. The macrofracture modes of the three carbon-carbon composites at 25X: CC-F (top); CC-B (center), and CC-T (bottom).

Table 3. Typical Work-of-Fractures of Carbon Materials

Material	γ_{wof} (J/m^2)
Glassy Carbons	
GC-10	15
GC-20	21.5
GC-30	31.0
Polycrystalline Graphites	
ATJ-S	42.4
PILE - "A"	49.4
AFXQ 1	69.1
Steelmelting Electrode Graphites	
transverse	210
longitudinal	150

and the role of the substrate. These will be addressed in the order of increasing work-of-fracture values, CC-F, CC-B, and CC-T, respectively.

The carbon-carbon composite CC-F consists of a fiber reinforced carbon felt substrate surrounded by CVD and pyrolyzed matrix carbons. Numerous gaps exist between the matrix carbon sheaths and the carbon fibers. The laminar structure of the matrix carbons is mostly aligned parallel to the longitudinal orientation of the fibers, but some of the graphitic basal planes of the matrix carbons are aligned over a range of different angles, intertwined and interwound like ribbons. Gaps and cracks are always apparent, for the bonding is not perfect. There are also cylindrical matrix carbon layers surrounding the carbon fibers. These cylinders are composed of numerous cylindrical matrix layers. Those that are closer to the fibers are CVD matrix carbon while progressing away from the fiber is the pyrolyzed matrix carbon. Debonding and gaps always seemed to occur at the interfaces of the different matrix carbons.

Fracture surfaces of the carbon-carbon composite CC-F were always quite rough with numerous pits on the fracture surface. These pits indicate spalling of the matrix carbon from the composite substrate and the breakage and pulling out of some of the reinforcing carbon fibers. Occasionally, small bundles of carbon fibers are apparent on the fracture surface, as can be seen in Figure 2. Fracture usually occurred within the laminar structure of the matrix carbons, often at the interface of the CVD matrix carbon and the pyrolyzed matrix carbon, as can be seen in Figure 3. The bonding between the CVD matrix carbon and the resin pyrolyzed carbon was weak as the pyrolyzed matrix carbon often broke into pieces during fracture but the CVD matrix carbon remained on the fiber surfaces in quite good condition. This indicates that the strength of the CVD matrix carbon exceeds that of pyrolyzed matrix carbon. This carbon felt fiber reinforced carbon-carbon composite failed in a matrix-matrix mode along well aligned graphitic laminar layers, especially at the interfaces of CVD matrix carbon and the pyrolyzed matrix carbon. Improvements in this particular carbon-carbon composite could readily be achieved through development of a stronger bond between the two matrix carbons.

The general structure of the chopped fiber containing carbon-carbon composite CC-B exhibited a number of voids, consistent with its low density. The fracture surface exhibited an overall random distribution of the chopped fibers, many of which were torn away, while others were pulled out generally rough and ragged. This composite exhibited irregular propagation of the cracks, probably due to the random nature of the chopped fibers' orientations. Fracture paths were quite complicated. In contrast to the fiber reinforced carbon felt carbon-carbon composite CC-F, this semi-random chopped fiber carbon-carbon composite CC-B failed in a matrix-fiber mode. Figure 5 illustrates fracture at the interface of the matrix carbon and the carbon fibers. Carbon fibers are actually pulled out of the matrix carbon with the matrix carbon sheaths remaining after the carbon fibers had already torn away. It is evident that the bonding between the matrix carbon and the chopped carbon fibers in CC-B is not very strong, so that any efforts toward improving or increasing the fiber-matrix bond strength might improve the properties of this semi-random chopped fiber carbon-carbon composite.

Microstructural analysis revealed that the structure of the matrix carbon in the three dimensional woven substrate carbon-carbon composite CC-T is a laminar structure with a high degree of basal plane orientation. The well aligned graphitic layers are not extended planes but curved surfaces with considerable waviness and changeable curvature. It contains lots of cracks. Figure 6 shows cracks present in the matrix carbon, but cracks also exist within the fiber bundles. These cracks are very intricate and complicated, sometimes linking together to form elaborate microcrack networks.

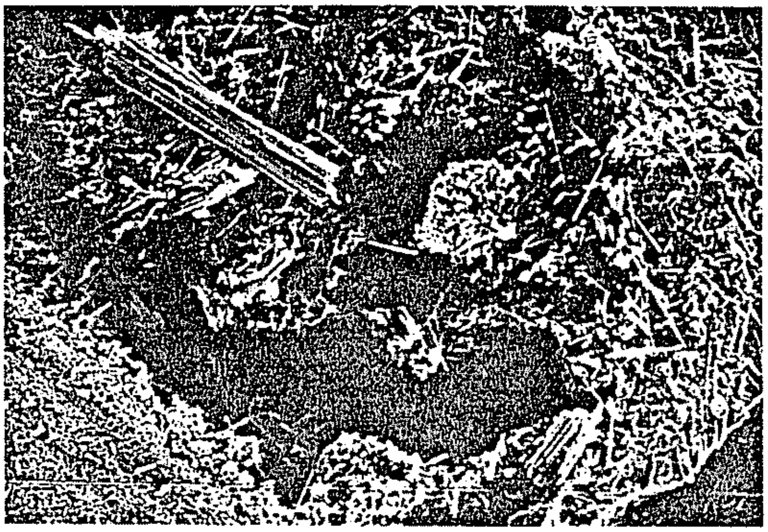

Figure 2. Fracture surface of the carbon-carbon composite CC-F, illustrating fibers and fiber bundles on the fracture surface (50X).

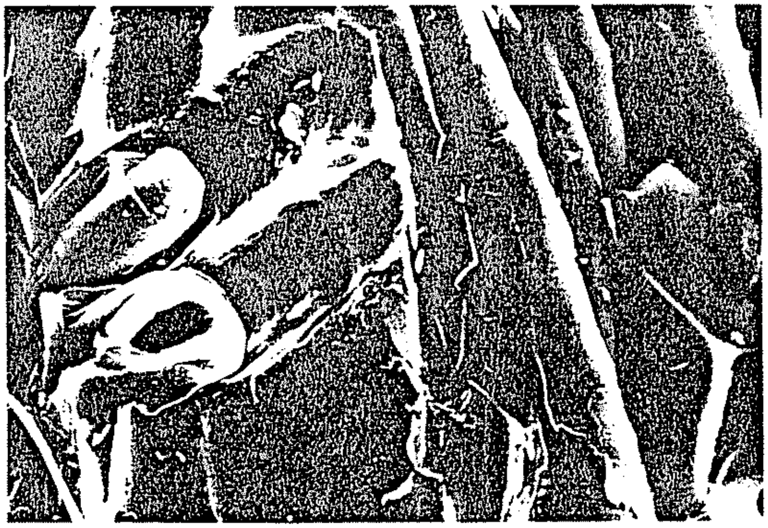

Figure 3. Fracture surface of the carbon-carbon composite CC-F (500X).

Figure 4. Fracture surface of CC-B showing fiber pull out, break-off, and fiber tearing from the matrix carbon (200X).

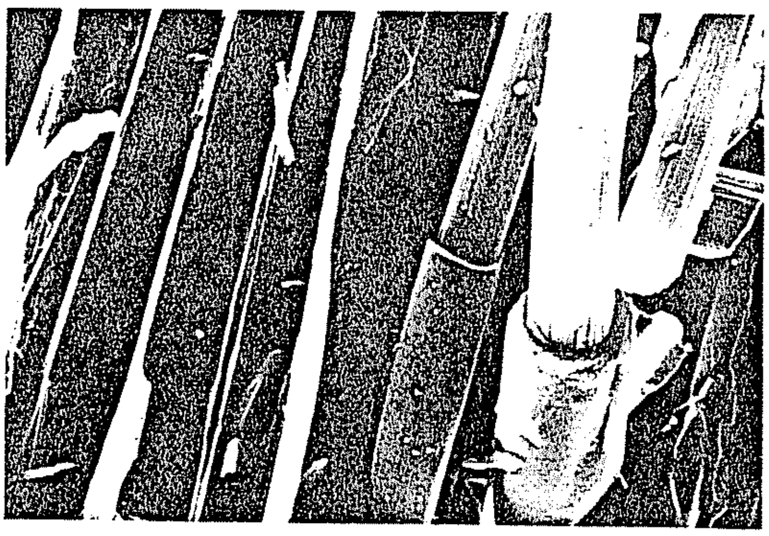

Figure 5. Fracture surface of the chopped-fiber carbon-carbon composite CC-B, showing fiber pull-out and the debonding of the fibers and matrix carbon (1000X).

It is reasonable to assume that some of these cracks might have formed during the repeated graphitization heat treatment during processing, as a consequence of the high transverse thermal expansion of the carbon fibers. A matrix sheath usually surrounds the carbon fibers and is characterized by well aligned graphitic layer planes parallel to the longitudinal direction of the carbon fibers. The fiber surface and the stresses during graphitization may have contributed to this alignment of the graphite basal planes.

In this three dimensionally woven substrate carbon-carbon composite the matrix-rich carbon seems to be the weakest link, for cracking often occurred at the intersection of the matrix pockets and the fiber bundles, and many circumferential cracks occurred in the surrounding matrix carbon. These cracks readily proceeded through the matrix carbon adjacent to the fiber bundles, but the propagation and enlargement of these cracks was not straightforward. In matrix-rich areas the crack propagation was coincident with the orientation of highly aligned laminae of the graphitic layer planes, but due to the wavy nature of these laminae, the propagation of cracks often deviated. If there were voids present in the adjacent matrix carbon, crack propagation sometimes tended to progress through these voids, even though the voids were not in the direct path of the propagating crack. When a propagating crack approached longitudinal fiber bundles, it frequently changed direction and proceeded along the interface region. The fracture of fiber bundles appeared to depend on weaknesses of the individual bundles rather than being an inherent characteristic of the structures. Figure 6 shows the propagation of cracks in the three dimensional carbon-carbon composite illustrating fiber fracture and crack propagation parallel to fibers.

Examination of the fracture surfaces of this three dimensional carbon-carbon composite revealed that the fracture occurred primarily in the matrix-matrix mode, predominantly in matrix carbon areas, sometime through the matrix-rich regions at, or adjacent to carbon fibers, or bundles of fibers. During the fracture, portions of the matrix carbons and sometime even whole pieces might spall off. Due to differences in loading directions, some of the carbon fiber bundles were also torn apart and others pulled out of the matrix carbon, as shown in Figure 7. In all cases there was matrix carbon adhered to and remaining on the surfaces of the fibers.

It is clear that fiber-matrix debonding did not play a paramount role in the failure of this three dimensional carbon-carbon composite. Thus, efforts to improve the properties of this particular carbon-carbon composite by increasing the fiber-matrix bond strength would not be successful. A more important approach would be to try to improve the matrix system and the related processing cycles which could potentially reduce the microcrack networks in the matrix-rich

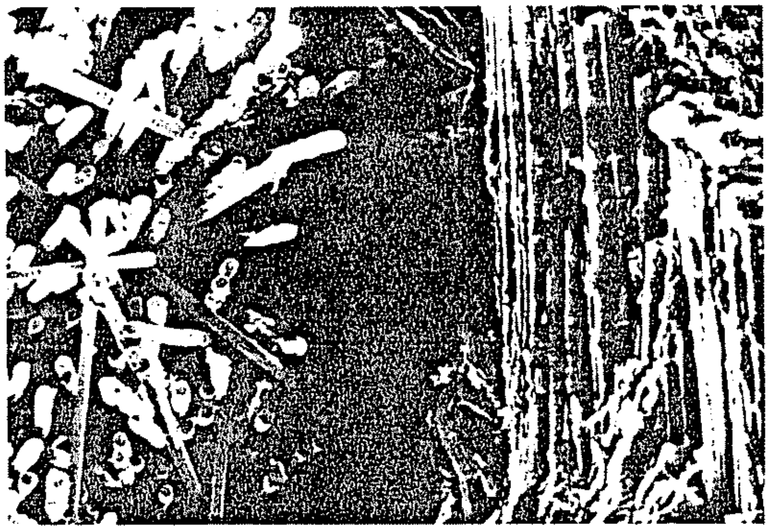

Figure 6. Fracture surface of the carbon-carbon composite CC-T showing fiber fracture and crack propagation parallel to the fibers (200X).

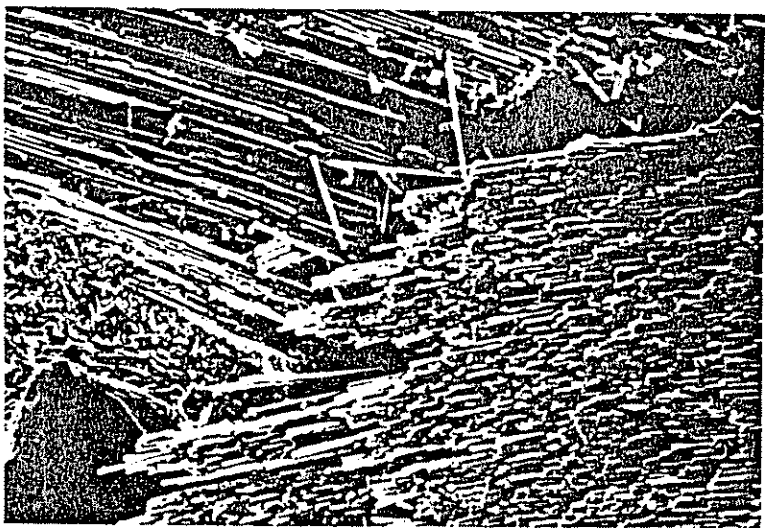

Figure 7. Fracture surface of the carbon-carbon composite CC-T (100X).

carbon areas and thus improve the matrix carbon integrity.

SUMMARY AND CONCLUSIONS

The work-of-fracture values of three different carbon-carbon composites were measured and their fracture surfaces were examined by scanning electron microscopy. Compared to other carbon and graphite materials, it was observed that these composites possessed very high energy requirements for crack propagation and that the failure processes could be ascertained through microscopic examination of the fracture surfaces.

These different carbon-carbon composites failed in different modes. Crack propagation characteristics, as well as the weakest links in these composites are closely related to both the substrates and the matrix carbons of these composites. The highest work-of-fracture composite, the three dimensionally woven carbon-carbon composite failed in the matrix-matrix mode. Matrix-rich carbon areas were the weakest link in this particular composite as fracture often occurred through matrix-rich areas or in matrix regions adjacent to carbon fiber bundles. The crack propagation proceeded in a ragged fashion. The composite with a substrate of carbon felt reinforced by carbon fibers also failed in the matrix-matrix mode. The interface between the CVD matrix carbon and the pyrolyzed matrix carbon was the weakest link of this composite. In contrast to the others, the semi-random chopped fiber reinforced material failed in the matrix-fiber mode. The interfaces of the carbon fibers and the matrix carbon were the weakest link of that carbon-carbon composite as fracture readily occurred at those interfaces.

ACKNOWLEDGEMENTS

The authors wish to express their thanks to Dr. J. L. Wood for assistance in supply of the carbon-carbon composite CC-B. Thanks are also extended to Dr. P. A. Thrower and Mr. W. W. Corbett for their assistance with the scanning electron microscopy.

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