INTERFACIAL SHEAR STRENGTH OF C/C COMPOSITES AND DEBOND DETECTION USING WAVELET ANALYSIS

Soydan Ozcan[a,c], Jale Tezcan[c], Bijay Gurung,[a,c], Peter Filip[a,c]

a. Mechanical Engineering and Energy Processes, Southern Illinois University, Carbondale, IL, USA
b. Civil and Environmental Engineering, Southern Illinois University, Carbondale, IL, USA
c. Center for Advanced Friction Studies, Southern Illinois University, Carbondale, IL, USA

Introduction

The use of C/C composite has exhibited significant advantages in high temperature braking and structural aerospace applications due to excellent mechanical and thermal properties coupled with light weight of C/Cs [1-4]. Extensive theoretical and experimental studies have been dedicated to investigate the properties of fiber/matrix interface of the polymer and ceramic matrix composites [5-9] compared to only a few related to that of C/C composite [10, 11]. In this paper, a single fiber push-out technique (pushing on a single fiber until it separates from the matrix) using a nanoindenter is combined with a mathematical approach that will provide additional insight into the process of debonding in C/C composites.

Experimental Procedures

Commercial C/C composites, consisting of three directional needled felt PAN fibers and rough laminar CVI matrix, were used. The C/C composites were kindly provided by Honeywell Aircraft Landing Systems. And then heat treated in graphitization furnace The provided C/C samples were heat-treated using a graphitization furnace at 1800° C, 2100°C and 2400°C and named CC-D18, CC-D21, CCD-24, respectively

Microstructure characterization techniques, namely optical microscopy (Nikon Eclipse LV 150), and scanning electron microscopy (SEM, Hitachi S570) The terminology used for the description of optical textures is in accord with the classification used in the literature [13-18]. Correspondence between extinction angle (Ae) and the classification of the texture of pyrocarbon varies as: isotropic (Ae<4°), dark laminar (4°≤Ae<12°), smooth laminar (12°≤Ae<18°), and rough laminar (Ae≥18°) [13-18].

For push-out tests, 200 µm samples were prepared and mounted on a 3mm transmission electron microscopy copper grid using a crystal bond wax to form a beam and placed on a sample holder. Schematic of testing set-up and deformation mechanism is given in Fig. 1.

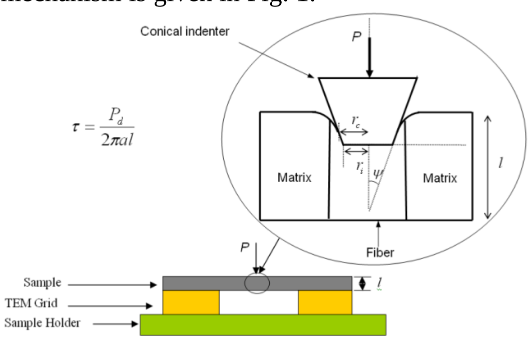
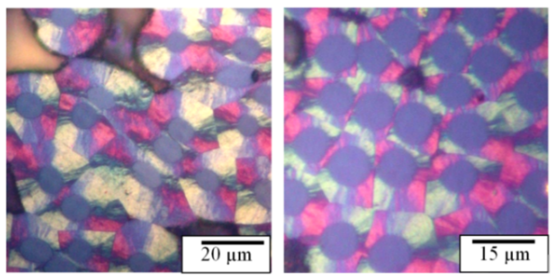


Fig. 1. Schematic of sample set-up and deformation during the push-out test in the nanoindenter

IFSS was determined by push-out tests conducted at room temperature using a Nano Indenter® (XP system, MTS Nanoinstruments, Knoxville, TN). A sixty degree cone with a 5µm diameter flat-end indenter tip was employed for the single fiber push-out test (Fig. 1). A constant load rate of 0.66 mN/sec was applied until the preset maximum load of 108 mN was reached.

Results and Discussion

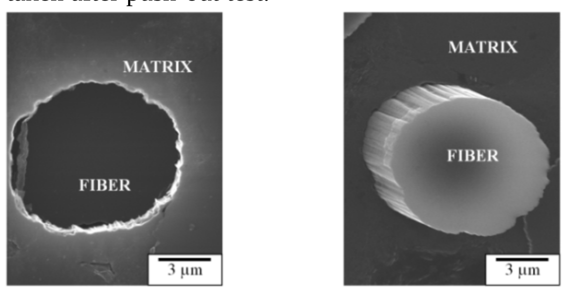
Characteristic light microscopy images of C/C composite samples used for pushed out tests are shown in Fig. 2. The C/C composite consists of PAN-based carbon fiber and chemical vapor infiltrated (CVI) carbon matrix. The CVI carbon matrix exhibited 20° extinction angle (Ae). This indicates highly anisotropic rough laminar pyrocarbon.



(a) (b)

Fig. 2. Light microscopy image of polished surface showing carbon fiber cross sections at different magnifications. (a) CCD-18, and (b) CCD-24.

Typical scanning electron microscopy images shown in Fig. 3 were taken after push-out test.



(a) (b)

Fig. 3. SEM images taken after single-fiber push-out tests. View from top (location of applied force) (a) and bottom (pushed-out fiber from the matrix) (b).

The 5 µm diameter flat-end nanoindenter tip was pushing on a single fiber until it separated from the matrix. SEM images represented in Fig. 4 clearly indicate the carbon fiber movement through the matrix without any visible damage (cracks) on fiber or matrix. This demonstrated that the debonding only occurs at the fiber/matrix interface after the push-out. For cases of sudden and complete debonding, the IFSS can be approximated as the average shear strength of the entire interface [20]:

$$\tau = \frac{P_d}{2\pi\, a\, l}$$

where P_d, a and l are the debonding load, radius of fiber and thickness of the specimen, respectively.

In this study, a mathematical analysis tool called the wavelet transform was used to examine the initiation and progression of fiber/matrix debonding. Wavelet transform decomposes a function into its multiscale components using basis functions which are the scaled and shifted versions of a wavelet [12].

The wavelet coefficients of a function $y(x)$ are calculated from

$$W_\psi(x', s) = \frac{1}{\sqrt{s}} \int_{-\infty}^{\infty} y(x)\psi^*\left(\frac{x - x'}{s}\right)dx$$

where $\psi(x)$ is the analyzing wavelet, and s and x' are the scaling and shifting parameters, respectively. Wavelet transform allows detection of critical points that are hidden in the signal itself, but apparent in the higher derivatives. When applied to the push-out data, stiffness changes can be located more accurately than using the classical curve fitting approaches.

Figure 4 demonstrates 3-level wavelet analysis of the measured indentation displacement data for a sample treated at 1800 °C which exhibits sudden and complete debonding. The wavelet coefficients for three scales (D1, D2, D3) are concentrated around the load (~80mN) where the debonding occurs.

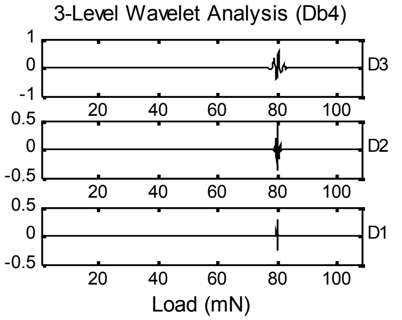


Fig. 4. Example application of wavelet analysis to detect debonding

Figure 5 shows the average measured IFSS values for samples subjected to heat treatment temperatures at 1800 °C, 2100 °C and 2400 °C.

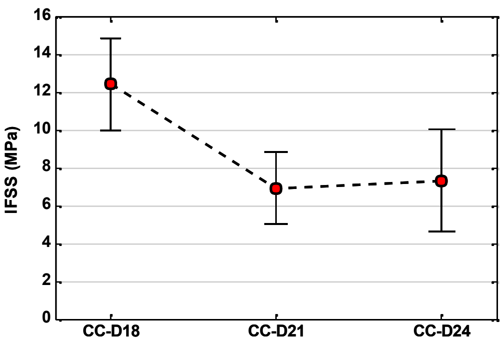


Fig. 5. IFSS of C/C samples subjected to different HTT.

Debonding of the fiber was confirmed using SEM analysis, and the samples with partial debonding were excluded from the analysis.

Conclusions

The interfacial shear strength decreased drastically after heat treatment of C/C composites at 2100 and 2400°C. The decrease of IFSS is attributed to:

1- The thermal mismatch between carbon fiber and pyrocarbon matrix which deteriorates interphase (introduces internal stresses) and weakens the IFSS. This may also cause defects in the interphase.

2- Reorganization of carbon atoms and formation of extended graphene sheets parallel to fiber/matrix interphase in the pyrocarbon matrix with a low shear resistance leads a weaker IFSS,

3- The heat treatment has not provided additional strengthening in the fiber-matrix interface by forming chemical bonds between fiber and matrix.

Acknowledgement

This research was sponsored by the National Science Foundation (Grant EEC 3369523372), State of Illinois and a consortium of 11 industrial partners of Center for Advanced Friction Studies (http://frictioncenter.engr.siu.edu)

References

[1] Fitzer E, Manocha L. Carbon Reinforcements and Carbon/Carbon Composites: Springer 1998.
[2] Savage G. Carbon-Carbon Composites: Kluwer Academic Publishers 1993.
[3] Schmidt D, Davidson K, Theibert L. Unique Applications Of Carbon-Carbon Composite Materials(Part Three). SAMPE. 1999;35(5):47-55.
[4] Ozcan S, Filip P. Microstructure and wear mechanisms in C/C composites. Wear. 2005;259(1-6F):642-50.
[5] Faber K. Ceramic composite interfaces: Properties and Design. Annual Reviews in Materials Science. 1997;27(1):499-524.
[6] Herrera-Franco P, Drzal L. Comparison of methods for the measurement of fibre/matrix adhesion in composites. Composites. 1992;23(1):2-27.
[7] Kerans R, Parthasarathy T. Theoretical Analysis of the Fiber Pullout and Pushout Tests. Journal of the American Ceramic Society. 1991;74(7):1585-96.
[8] Mandell J, Grande D, Tsaing T, McGarry F. Modified Microdebonding Test for Direct In Situ Fiber/Matrix Bond Strength Determination in Fiber Composites. 1986: ASTM International; 1986.
[9] Marshall D, Oliver W. Measurement of Interfacial Mechanical Properties in Fiber-Reinforced Ceramic Composites. Journal of the American Ceramic Society. 1987;70(8):542-8.
[10] Furukawa Y, Hatta H, Kogo Y. Interfacial shear strength of C/C composites. Carbon. 2003;41(9):1819-26.
[11] Sakai M, Matsuyama R, Miyajima T. The pull-out and failure of a fiber bundle in a carbon fiber reinforced carbon matrix composite. Carbon. 2000;38(15):2123-31.
[12] Stark H. Wavelets And Signal Processing: An Application-Based Introduction: Springer 2005.