STRUCTURE AND PROPERTIES OF INTERFACES IN INFILTRATED CARBON FIBER FELTS

B. Reznik[(a)], D. Gerthsen[(a)], M. Guellali[(b)], R. Oberacker[(b)], and M. J. Hoffmann[(b)].*

[(a)] *Laboratorium für Elektronenmikroskopie, Universität Karlsruhe (TH), Kaiserstraße 12, D-76128 Karlsruhe, Germany,* [(b)]*Institut für Keramik im Maschinenbau, Universität Karlsruhe, Haid-und-Neu Str. 7, D-76131 Karlsruhe, Germany*

**e-mail:reznik@lem.uni-karlsruhe.de*

Introduction

The microstructural characterization of interfaces yields important information with respect to the understanding of growth phenomena taking place during the carbon infiltration and the mechanical behavior of carbon fiber/carbon matrix composites [1]. The present work has been carried out to examine the nature of the fiber/matrix interface as well as interfaces between different matrix layers as a function of the bulk mechanical properties of the composites obtained by chemical vapor infiltration (CVI) of carbon fiber felts. The interfacial regions have been studied by transmission and scanning electron microscopy (TEM and SEM). The mechanical behavior of composites has been studied by three-point flexure testing.

Experimental

The examined composites are carbon fiber felts infiltrated by isothermal CVI at a temperature of 1100°C using a methane/hydrogen mixture of p_{CH4}/p_{H2} = 7:1 at total pressures of 20 (felt I) and 30 kPa (felt II) with bulk densities of 1.72 and 1.62 g/cm^3 [2]. The measurement of the flexural strength of the composites has been carried out in three-point bending mode on a universal testing machine (UTS, Germany) and is described elsewhere [3]. The fracture surfaces of the samples were examined with a high-resolution scanning electron microscope LEO 1530 with an Schottky field-emission gun. The TEM has been carried out in a Philips CM 200 FEG/ST electron microscope operated at 200 kV. The instrument is characterized by a Scherzer resolution of 0.24 nm and an information limit of 0.15 nm.

Results and Discussion

Fig. 1 demonstrates the microstructure of felt I. Fig. 1a is a SEM micrograph of the fracture surfaces after a bending test. The matrix layers (2,3) occupy distinctly other fracture plane levels compared to carbon fiber and the layer (1) around the fiber. Layers with smooth fracture surfaces cover the carbon fibers. The third layer exhibits a high surface roughness due to intensive fragmentation. The interfacial region between pyrocarbon matrix layers with different textures is presented in Fig. 1b. The FWHM of the arc of (002) reflections in selected area electron diffraction (SAED) patterns (inserts in Fig. 1b) were used to determine the texture of the different layers, i.e. the degree of alignment of the (002) basal planes parallel to the fiber surfaces [2]. The highly textured layer (3) contains numerous cracks, which are oriented parallel to the (002) carbon planes. Cracking is also observed inside the weakly textured layer (1) while the interfaces between the different carbon layers do not contain any cracks. The interface regions, which extend over a few nm, can be only roughly localized due to the very smooth transition between the different pyrocarbon layers [2]. The high-resolution TEM observation (Fig. 1c) shows the presence of a highly textured reaction layer at the interface between the fiber and layer (1) indicating a strong adhesion between fiber and matrix.

The matrix of felt II consists of 5 layers with alternating high and weak textures (Fig. 2a). The sliding between pyrocarbon layers is frequently observed in the form of a multiple „pull-out“. The interface between the fiber and the first matrix layer is displayed in Fig. 2b. A crack is located in the carbon matrix at some distance from the interface within the weakly textured area. As in the case of felt I a highly textured reaction layer is observed at the fiber/matrix interface (Fig. 2c). Based on the obtained data, a geometrical model for the interface between fiber and pyrocarbon matrix is proposed (Fig. 2b, inset). According to this model there are three distinct

areas: 1) a reaction layer with the (002) planes oriented parallel to the fiber surface 2) a weakly textured low density layer containing a crack, 3) a weakly textured layer with a higher density. Similar areas are found at the boundaries between layers with different textures (Fig. 1b) the area in the dashed square), where cracking occurs within single layers but not at interfaces. The same trend is also observed in pyrocarbon layers deposited on plane substrates in hot-wall reactors [5]. Since the quality of cross-sectional TEM samples depends significantly on the mechanical characteristics of the material to be prepared, the microstructure observed by TEM serves as an indicator of the stress-strain behavior of the observed areas [6]. The TEM images (Fig. 1b, Fig. 2b) illustrate that the investigated interfaces have a relatively high mechanical stability. In contrast, an extensive material disintegration takes place within the pyrocarbon layers (Fig. 2b).
According to the results of the three-point bending tests the felt I has a significantly lower flexural strength but a higher toughness than felt II [3,4]. The presented structural properties of the interfaces are applied for the explanation of the failure mechanisms taking place during flexural tests. It can be assumed that the non-brittle fracture behavior of the investigated composites is related to multiple crack deflections at interfaces between layers with a varying texture degree and the delamination microcracking within the highly textured pyrocarbon layer.

Summary

1. Independent of the applied methane partial pressures a highly textured reaction layer is formed at the fiber/matrix interface.

2. Intensive cracking occurs in the pyrocarbon layers but not at the interfaces between pyrocarbon layers with different textures where a continuous texture transition is observed. The disordered structure in the weakly textured pyrocarbon appears to suppress the crack propagation at the interface with the highly textured pyrocarbon.

3. Taking into account that there is a comparably good adhesion between the fiber and matrix in both felts, the differences between the mechanical properties could be mainly related to the matrix architecture and the properties of interfaces:

- An increase of the flexural strength is observed in the composite with a pyrocarbon matrix consisting of five layers with alternating high and weak textures.
- At fiber-matrix interfaces the fracture occurs inside the weakly textured transitional layer with a low density, rather than at the interface.
- Crack deflection at interfaces between layers with different textures and within fragmented highly textured pyrocarbon are the two main cooperative energy dissipation mechanisms contributing to the toughness enhancement.

References

1. Fitzer E, Manocha LM. In: Carbon Reinforcements and Carbon/Carbon Composites, Berlin Heidelberg, Springer-Verlag, 1998, pp. 65-90.

2. Reznik B, Gerthsen D, Hüttinger K-J. Carbon 2001; 39 (2): 215-229.

3. Reznik B, Guellali M, Gerthsen D, Oberacker M, and Hoffmann M J. Materials Letters, 2001(submitted).

4. Guellali M, Reznik B, Gerthsen D, Oberacker R, and Hoffmann M J, (this conference).

5. De Pauw V, Reznik B, Kalhöfer S, Gerthsen D, and Hüttinger K-J, (this conference).

6. Reznik B, Kalhöfer S. Micron, 2001 (in press).

Acknowledgements

This research has been carried out in the Collaborative Research Center (Sonderforschungsbereich) 551 financed by the Deutsche Forschungsgemeinschaft. We thank Prof. K-J Hüttinger for the infiltrated samples.

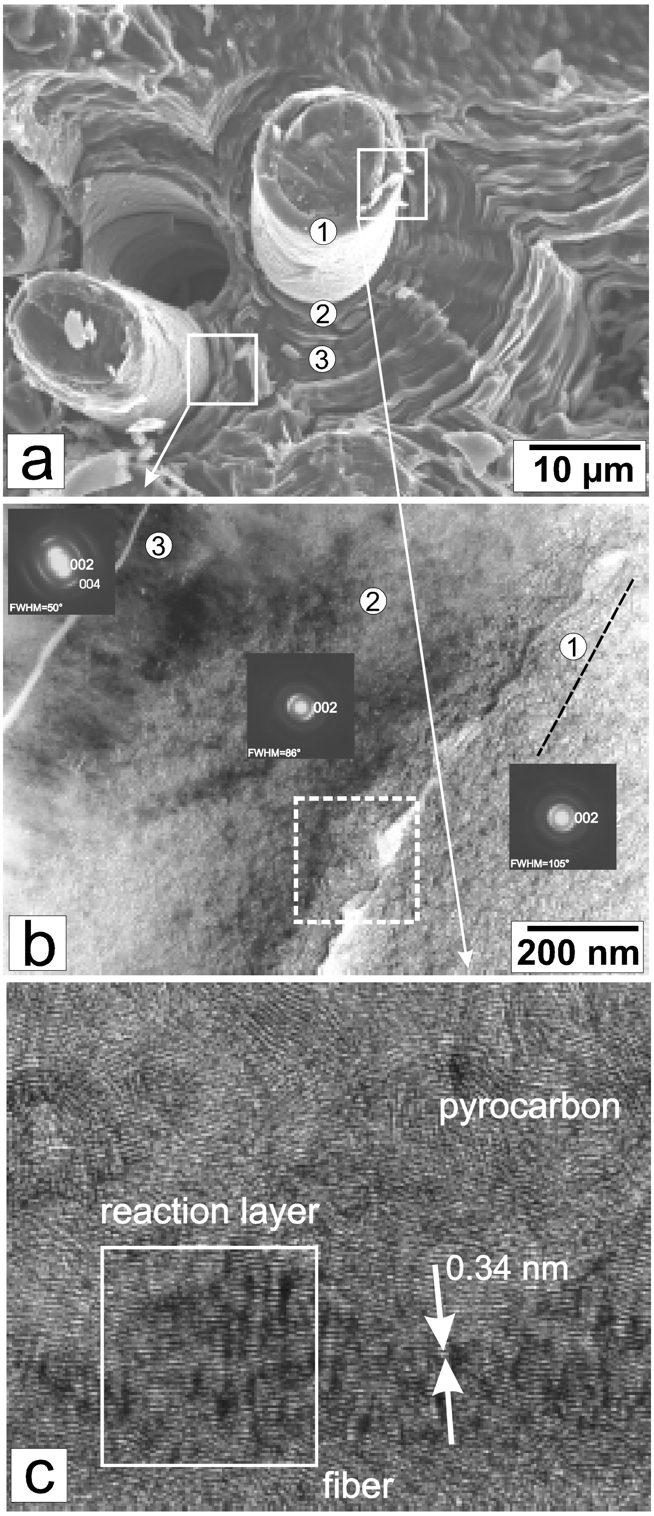

Fig. 1. Microstructure of the felt I. a) SEM image showing the ring-shaped laminar layers with a smooth (1,2) and rough (3) fracture surface. b) TEM cross-sectional image of the interfacial region between pyrocarbon layers with different textures. Insets: SAED patterns. The dashed line marks the orientation of the fiber surface. The dashed square contains a transition zone with a crack within the weakly textured pyrocarbon layer 1. c) HRTEM image of the interface area between the fiber and the weakly textured pyrocarbon layer 1 showing the formation of a highly textured reaction layer. The dominant lattice fringes correspond to the (002) basal planes with a distance of 0.34 nm.

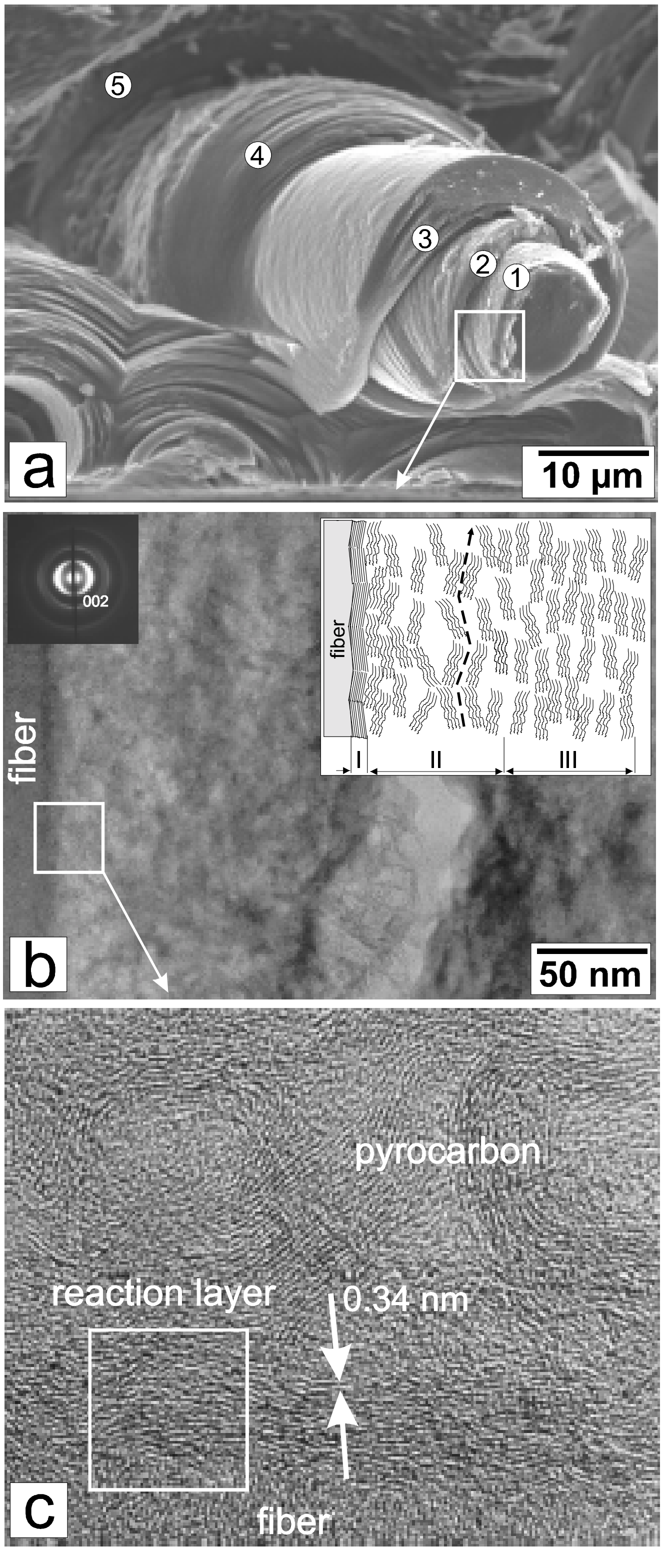

Fig. 2. Microstructure of the felt II. a) SEM image showing the sliding of pyrocarbon layers relative to each other. b) TEM cross-sectional image of the interfacial region between the pyrocarbon matrix and fiber surface. Left inset: SAED pattern of pyrocarbon. Right inset: schematic representation of the fiber/matrix interfacial region displaying three distinct features: 1) highly textured layer, 2) weakly textured layer with a low density containing a crack, 3) weakly textured layer with higher density. c) High-resolution TEM image of the contact area showing the formation of highly textured reaction layer at the fiber surface.