A NEW PUSH-OUT PROCEDURE FOR THE EVALUATION OF INTERFACIAL PROPERTIES OF SIC/SIC COMPOSITES

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Abstract
A new procedure is proposed to perform push-out tests in SiC/SiC composites. To avoid the interphase damage resulting from mechanical polishing, the sample is prepared with the help of a ion slicing process. Initiation of debonding is detected on the mechanical response. A finite fracture mechanics approach allows to use the force at debonding onset to estimate the interfacial fracture energy.

1. Introduction
The mechanical behavior of fiber reinforced ceramic composites is closely related to their interfacial properties. It has been shown that optimal mechanical properties of ceramic matrix composites depend on the load transfer at the fiber/matrix interface [1]. The interfacial bond influences various features of the composite mechanical behavior, including non linear response and strength. It is thus necessary to evaluate the fracture energy of this interfacial bond in order to predict the mechanical behavior of the composite material.
The fibre push-out test is a simple method used to establish the properties which characterize the fibre/matrix interface under service loading conditions. For this purpose, a thin slice of the composite is cut normal to the fibre direction and a microindenter is used to push against the fibre end. Few papers report push-out studies on SiC/SiC composites [2]. Numerous difficulties are encountered with specimen preparation and testing, which were associated with the weak interfacial bonds and with the small diameter of the fibers. Studies on C/C composites deplored protuberant fibers that got out of their matrix sheath during metallographic preparation as a result of residual stress relaxation associated with the interface degradation [3]. The standard method requires a wedge-shaped specimen (Fig. 1). This geometry allows modifying the length of the indented fiber. The specimen is embedded in epoxy and polished using standard metallographic techniques. This polishing process is likely to damage the pyrocarbon interphase in the vicinity of the fiber ends.

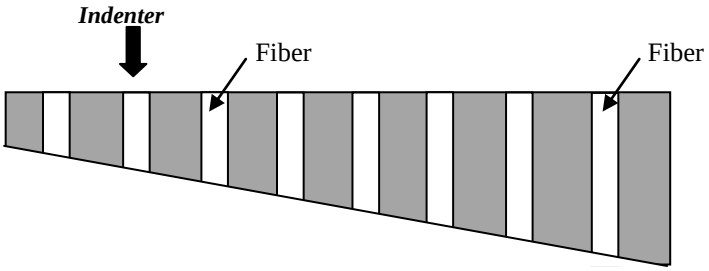

Figure 1. Schematic representation of the wedge-shaped specimen prepared by the mechanical polishing technique.

The purpose of the present paper is to propose a new procedure for the push-out test on SiC/SiC which includes i) a specimen preparation that could avoid or limit the damaging of the pyrocarbon interphase and ii) a dedicated numerical approach for extracting the interfacial fracture energy from the push-out data.

2. Experimental method

2.1 Material

The material was manufactured by Snecma Propulsion Solid (SAFRAN Group). It was fabricated by chemical vapor infiltration (CVI) of a preform of Nicalon fibers coated with a pyrocarbon interphase. The woven fiber architecture is based on an interlock weaving : layers of weft yarns are joined by the weaving of the warp yarns. The resulting composite was reinforced with a multi-layered matrix composed of boron carbide, silicium carbide and Si-B-C phases that ensure corrosion protection at high temperature.

2.2 Specimen preparation

A small sample (20 mm long and 1.7 mm wide) is first extracted from a plate of composite material. Then, 30 µm and 15 µm-roughed diamond coated disks mechanically thin the specimen using a MINIMET 1000 grinder polisher (Fig. 2). When the thickness of the specimen reaches 250 µm, it is sliced with a beam of argon ions (ion slicer JEOL EM-09100 IS). This operation requires height hours to reduce the thickness to 50 µm.

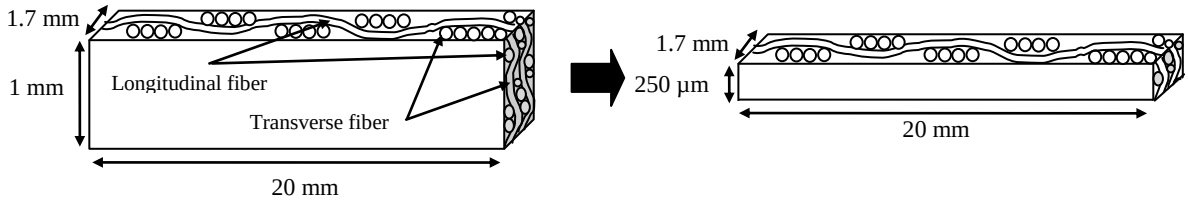

Figure 2. Mechanical thinning process.

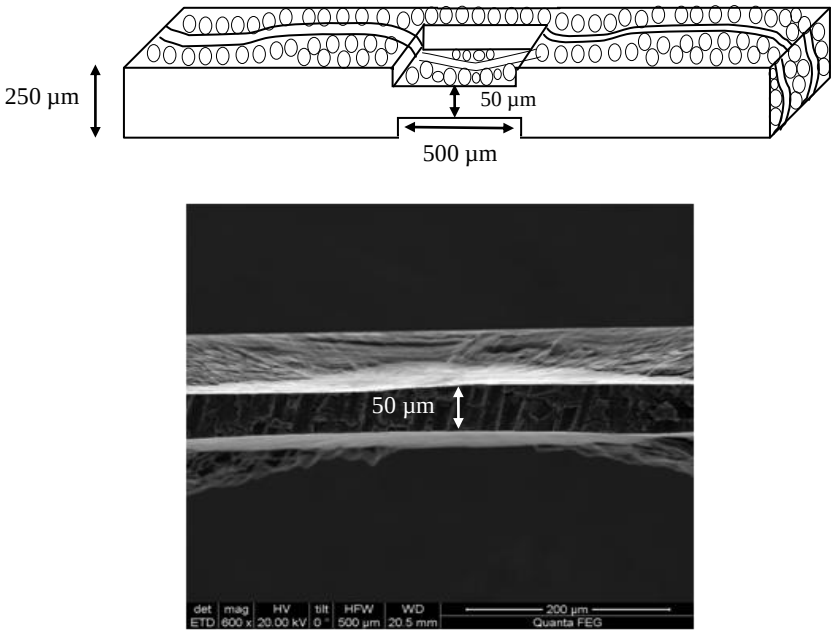

Figure 3. The composite sample after the ion slicing process.

The result is a small 50 µm thick and 500 µm wide area composed of two parallel and mirror-polished plans (Fig. 3). Thus, each fiber located within the polished area is 50 µm long.

2.3 Push-out test

The push-out tests are performed using the apparatus Nanotest Micromaterials NT600. For this purpose, the specimen previously described is fixed on the top of a cylindrical-shaped support whose top is grooved. The polished area of the specimen is placed on the top of the groove. The groove is wide enough (300 µm long) to facilitate fiber extraction (Fig. 4). An optical microscope is used to control specimen positioning and to determine the diameter of the tested fiber which is pushed under force control (loading rate : 0.5 mN/s) by a flattened conical diamond indenter mounted on a load cell. The diameter of the flat indenter in contact with the top of the fiber is 5.5 µm. All tests are performed in air at room temperature.

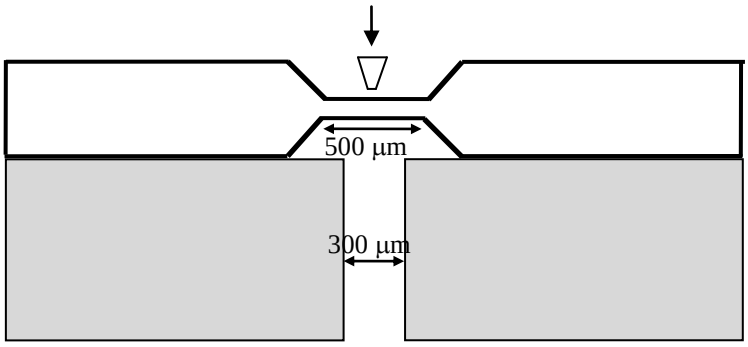

Figure 4. Geometry of the push-out test.

3. Results

Fig. 5 shows the surface of the tested area after the push-out tests. The push-out responses of three tests are plotted in Fig. 6. They exhibit an initial linear part corresponding to the elastic loading of the fiber with a bonded interface followed by a change of slope indicating the onset of debonding. A non linear response resulting from the progressive debonding of the interface is observed before reaching the plateau corresponding to the full debonded and sliding response. As mentioned by previous authors [4], this last part of the curve is not relevant of conditions experienced in cracking applications for brittle matrix composites which involve small amount of sliding.

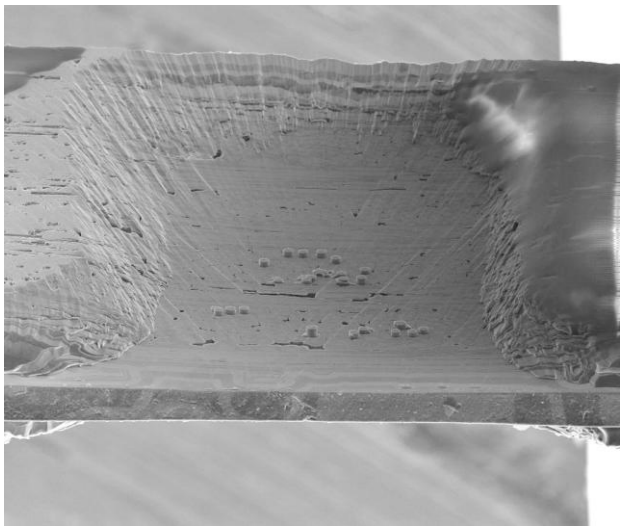

Figure 5. Micrograph showing fibers after push-out tests.

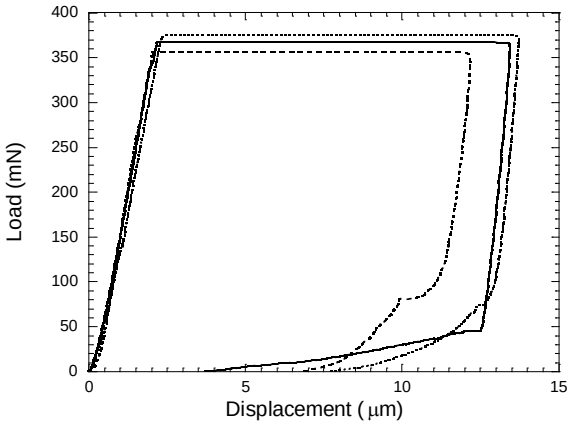

Figure 6. Force / displacement curves of push tests performed on three fibers.

Due to the complex stress state generated by the push-out loading, a numerical procedure is required to estimate the interfacial fracture toughness from the push-out data. Finite elements computations are performed with the help of a cell representative of the fiber embedded in the composite. Fig. 7 depicts the axisymmetric representative cell which includes a fiber surrounded by a matrix layer and inserted within an equivalent material.

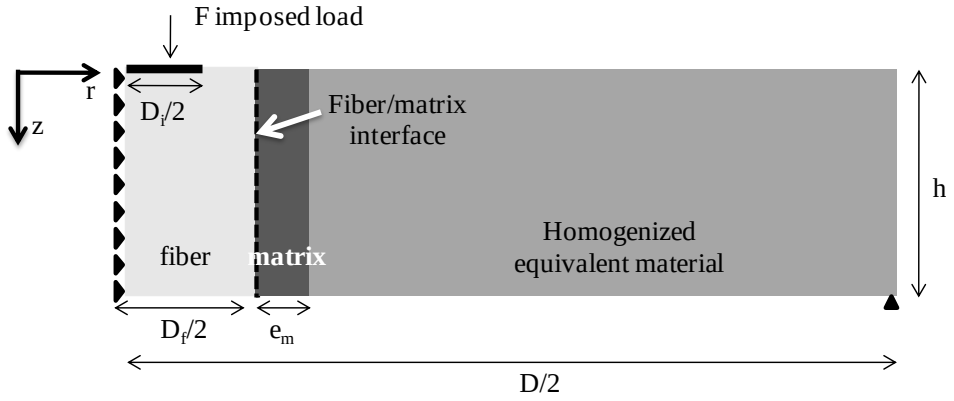

Figure 7. Axisymmetric representative cell.

The length and radius of the cell are measured relatively to the push-out specimen with $h = 50$ μm and D = 500 μm. The fiber radius is $D_f = 14.5$ μm. The axial displacement of one node at the right bottom of the cell is set to zero and a load F is imposed on the top of the fiber for $r \le D_i/2$ to simulate the indentor loading. The Young modulus and Poisson ratio of the isotropic fiber and matrix constituents are taken as $E_f = 200$ GPa, $\nu_f = 0.12$, $E_m = 400$ GPa, $\nu_m = 0.2$. The modulus E_h of the surrounding equivalent material is adjusted to fit the initial slope of the experimental response. To describe the onset of debonding, use is made of a finite fracture mechanics approach based on a coupled criterion (CC) which combines a stress and an energy condition [5].

The stress condition requires that the radial stress $\sigma_{rr}(z)$ along the fiber/matrix interface is greater than the interfacial strength σ_i^c with:

$$\sigma_{rr}(z) = k_{rr}(z)\frac{F}{\pi\left(D_f^2/4\right)} \ge \sigma_i^c \qquad (1)$$

Fig. 8a plots the normalized radial stress along the interface. The highest value of k_{rr} is observed near the surface at the top of the fiber for $z < 2$ μm. Debonding is thus likely to occur from the top of the sample.

The energy condition states that the incremental energy release rate reaches the interfacial fracture toughness G_i^c with:

$$G_i^{inc}(d) = A_i^{inc}(d)\frac{F^2}{\pi^2 E_i \left(D_f/2\right)^3} \tag{2}$$

where the interfacial modulus E_i is given by $\frac{1}{E_i} = \frac{1}{2}\left(\frac{1-\nu_f^2}{E_f} + \frac{1-\nu_m^2}{E_m}\right)$ and d is the debonding length (starting from the top of the specimen)**.** $A_i^{inc}(d)$ is the normalized incremental energy release rate which is plotted in Fig. 8b and reveals a maximum value A_i^* for $d = d^*$ with $d^* = 1$ µm.

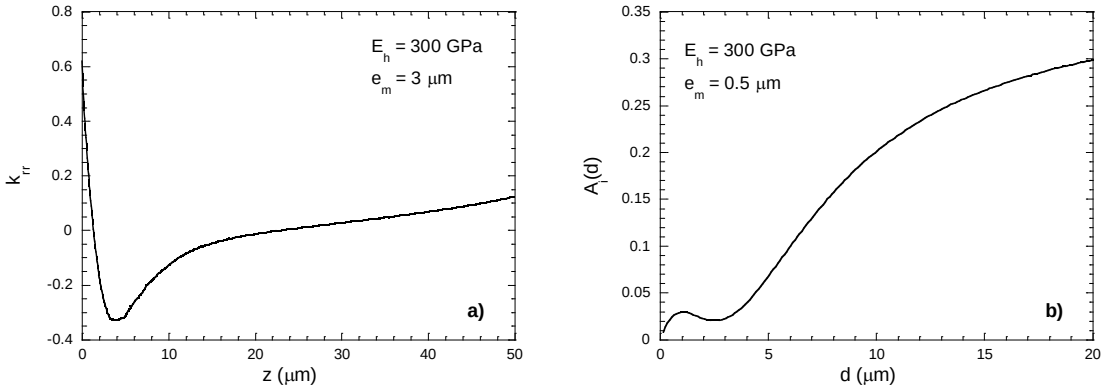

Figure 8. Normalized coefficients : a) $k_{rr}(z)$, b) $A_i^{inc}(d)$.

It was shown in a previous study [6] that d^* is the nucleation length predicted by the CC provided the interface is weak enough with:

$$\sigma_i^c \le \sigma_i^* = \sqrt{2\frac{E_i G_i^c}{D_f A_i^*}} k_{rr}\left(d^*\right) \tag{3}$$

In this case, the force F^* at onset of debonding only depends on G_i^c with:

$$F^* = \sqrt{\frac{\pi^2 E_i D_f^{\,3}}{8A_i^*} G_i^c} \tag{4}$$

Relation (4) thus allows to estimate the interfacial toughness G_i^c with the help of the force F^* (as provided by the experimental data) and A_i^* (as evaluated from the finite element analysis).

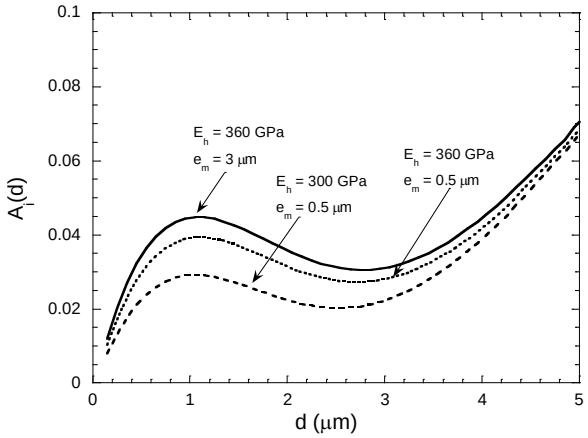

Figure 9. Normalized coefficients $A_i^{inc}(d)$.

This approach is thus used to analyze the three push-out responses plotted in Fig. 6. It is first found that E_h ranges between 300 and 360 GPa. The force F^* is detected from the first loss of linearity of the mechanical response. As shown by Fig. 9, the value of A_i^* depends on the modulus E_h and the thickness e_m of the matrix layer. Selecting $0.5\mu m \le e_m \le 3\mu m$ and $300\ \text{GPa} \le E_h \le 360\ \text{GPa}$ leads to interfacial toughness values within the range 2-6 Jm^{-2} and interfacial strengths smaller than 160-200 MPa (Table 1). As expected, those values characterize a weak fiber/matrix interface which results from the presence of the pyrocarbon interphase.

Test n°	F^* **(mN)**	G_i^c **(Jm^{-2})**	σ_i^* **(MPa)**
1	263	2.0-3.0	160
2	330	3.2-4.8	200
3	362	3.8-5.8	220

Table 1. Load at debonding onset and interfacial properties extracted from the push-out responses.

Conclusion

A new procedure for push-out test in SiC/SiC composites is proposed. The specimen preparation uses an ion slicer device to obtain a thin specimen (50 μm) with perfectly polished surfaces and non damaged interphases. To determine the interfacial fracture energy, the push-out test is modeled with a finite element analysis. The estimated stress distribution shows that the debonding initiates from the top of the specimen. A finite fracture mechanics approach is then used to describe the initiation of debonding. It is shown that this analysis allows to derive the interfacial fracture toughness from the value of the force recorded at onset of debonding.

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