MECHANICAL RESPONSE AND RUPTURE MODES OF SiC/C CVD LAMELLAR COMPOSITES
M. Ignat, M. Nadal, C. Bernard, M. Ducarroir, F. Teyssandier

To cite this version:

HAL Id: jpa-00229555
https://hal.archives-ouvertes.fr/jpa-00229555
Submitted on 1 Jan 1989

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
MECHANICAL RESPONSE AND RUPTURE MODES OF SiC/C CVD LAMELLAR COMPOSITES

M. IGNAT, M. NADAL*, C. BERNARD, M. DUCARROIR* and F. TEYSSANDIER*

L.T.P.C.M.-E.N.S.E.E.G., Domaine Universitaire, BP. 75, F-38400
St Martin d'Hères, France
*I.M.P.-CNRS, Université Avenue de Villeneuve, F-66025 Perpignan, France

Résumé

Abstract
SiC/C lamellar composites were prepared by CVD under reduced pressure. Three point bending tests were performed on samples with different lamellar thickness, in order to estimate their mechanical behavior. This parameter seems to influence the properties of such composites. The results of the deformation tests were analyzed in terms of Griffith's approach or residual internal stress effects. The results are compared to previous data obtained on similar composites.

1 - INTRODUCTION

The increase of the mechanical properties of materials has been obtained in the last ten years by the development of composites, and by improving their method of obtention. For metallic composite materials, unidirectional solidification methods have led to the obtention of oriented single crystalline superalloys. Nowadays, the CVD techniques allow the preparation of fibrous or lamellar ceramic/ceramic materials. In the latter case, few studies have been carried out, although the interest for advanced materials has been emphasised by several authors /1,2/.

With metallic composites it has been clearly established that the role of interfaces is predominant. Detailed microstructural analysis showed that the controlling mechanisms fixing the transmission and the accommodation of plasticity are active at the interfaces /3,4/. In the case of similar ceramic/ceramic composites, the role of interfaces is also expected to be predominant, but principally with respect to the crack opening process during deformation. In fact, the interfaces will act as barriers to transversal cracks: stopping or deviating them. In several previous works /5,6/, it has been pointed out that deviating the cracks at weak interfaces will increase the global toughness with respect to the toughness of the single ceramic phases constituting a biphased material. In this paper our interest is focused on a lamellar ceramic composite which is constituted by the alternance of pyrocarbon and silicon carbide layers. From bending tests, its mechanical response and rupture modes are analysed. This chemical pair was chosen because the differences in properties of the constituents, the eased of deposition, and the existence of previous mechanical data for structural SiC-C composites.

2 - EXPERIMENTAL METHODS AND EXPERIMENTAL RESULTS

The deposits are carried out in a classical cold wall reactor by flow rate modulations of the gaseous vectors - Si(CH₃)₄ and C₄H₁₀ - under reduced pressure.

Figure 1 shows the microstructural perfection of the samples obtained in our apparatus. The mean macroscopic dimensions of the specimens were 35 x 10⁻³ [m] long, 4.6 x 10⁻³ [m] wide and their thickness was 0.34 x 10⁻³ [m] for sample 1 and 0.42 x 10⁻³ [m] for sample 2. On both samples, a three point bending test was performed. The distance between the supports of the bending device was 25 x 10⁻³ [m], and the displacement of the loading zone was adapted to a computer assisted testing machine.
The load cell used during the bending runs allowed an accuracy inferior to 0.2N. The two tests were performed with the same constant central loading displacement rate of \(5 \times 10^{-6}\) [m/sec]. The data acquisition (force-displacement) was carried out with a periodicity of four points per second. The recorded curves corresponding to the tests performed are shown in figure 2a and b. They correspond to samples, in which the lamellar thicknesses were different. For sample 1 it was \(4 \times 10^{-6}\) [m] and for sample 2 it was \(20 \times 10^{-6}\) [m]; for both samples, the volume fraction of the C and SiC phases was 50%.

Before the experiment, the testing device was laid down so that an instantaneous unloading was performed when the device detected a drop in stress of 0.25 N. Then the following unloading period was performed with the same displacement rate as the loading period \(5.10^{-6}\) [m/sec].

The samples were controlled by Scanning Electron Microscopy (S.E.M) before the test and carefully observed after. Figure 3a and b presents the most striking features. For sample 1 (figure 3a), the only damage observed after the test was localized on the outer layer and in the central part of the sample, below the load application zone. A transversal crack was observed, and this unique crack intercepts the first interface producing a crack opening at the interface. These were the only observations giving evidence that the sample was submitted to a test. For sample 2 (figure 3b), it was surprising to observe no outer layer rupture, but on the contrary strong evidence of damage: cracks in the lamellae and at the interfaces. Debonding of lamellae and intense intralamellar transversal and longitudinal cracks, particularly in the SiC lamellae were observed. Near the outer layer of this sample, a large longitudinal crack remains after the test. Contrarily to sample 1 which showed no macroscopic permanent deformation, sample 2 presented a macroscopic curvature after the bending test.

3 - PREDICTIVE CALCULATIONS AND EXPERIMENTAL BEHAVIOUR

Due to the lack of information on this type of material, some simple predictive calculations were performed. These calculations can give an idea about the potential composite behaviour, they were based on the rule of mixtures formalism. The Young modulus and the maximum strength were then calculated (relations [1] and [2]), assuming that:
- the elastic response of each phase followed Hooke's law.
- the geometry and volume fraction of the constitutive phases are well defined.
- there is perfect bonding at the interface so that the strain in each phase are equal when considering the parallel model or the stress in each phase are equal when considering the series model (these models correspond to the extreme cases when the applied force is parallel or perpendicular to the interfaces).

The equations for the Young modulus are:

- parallel model : \(E = V_1 E_1 + V_2 E_2\) \([1]\)
- series model : \(\frac{1}{E} = \frac{V_1}{E_1} + \frac{V_2}{E_2}\) \([2]\)

The equations for the maximum strength are:

- parallel model : \(\sigma_R = V_1 \sigma_1^R + V_2 \sigma_2^R\) \([3]\)
- series model : \(\frac{1}{\sigma_R} = \frac{V_1}{\sigma_1^R} + \frac{V_2}{\sigma_2^R}\) \([4]\)

In these equations the subindex \(i = 1\) or \(2\) refers to each phase (C and SiC); \(V_i\) corresponds to the volume frac-
tion, $E_i$ is the Young modulus and $\sigma_{R,i}$ the rupture strength of the phases. The values used in our calculations and the results with this predictive formalism are reported in Table I.

Table 1: Results of calculations with a rule of mixture formalism: bulk values are taken from references /7/ and /8/.

<table>
<thead>
<tr>
<th></th>
<th>$E$ [GPa]</th>
<th>$\sigma_R \cdot 10^3$ [MPa]</th>
<th>Volume fractions [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>SIC</td>
<td>250</td>
<td>2.5</td>
<td>100</td>
</tr>
<tr>
<td>C</td>
<td>180</td>
<td>18</td>
<td>100</td>
</tr>
<tr>
<td>Composite (parallel)</td>
<td>215</td>
<td>10.25</td>
<td>50/50</td>
</tr>
<tr>
<td>Composite (series)</td>
<td>209</td>
<td>4.39</td>
<td>50/50</td>
</tr>
</tbody>
</table>

From the three point bending tests, we deduced a flexural Young modulus ($E_F$) and an apparent yield stress ($\sigma_y$); these parameters are presented in Table II.

For the apparent yield stress ($\sigma_y^a$), we used the relation /5/:

$$\sigma_y^a = \frac{3 F_I D}{2 b e^2}$$  \[5\]

$F_I$ is the maximum loading force; $D$ the distance between the supports in the bending test ($25 \, 10^{-3} [\text{m}]$); $b$ the sample width and $e$ its thickness.

The Young modulus from the flexural test was given by the equation /6/:

$$E_F = \frac{D^3 \Delta F}{4 b e^3 \Delta l}$$  \[6\]

In which, the $\Delta F/\Delta l$ term corresponds to the force-displacement gradient when the loading path is linear.
4 - DISCUSSION

From these first results, several points may be emphasised and discussed. Firstly, for this ceramic composite predictive calculations based on the bulk properties of the carbon and silicon carbide constitutive phases (mixture rules formalism), do not represent the behaviour of the composite. Results of predictive calculations are one or two orders of magnitude higher than the values calculated with experimental data (see Table I and II).

Secondly, our experiments tend to suggest an increase in the flexural strength when the lamellae are thinner. This behaviour has already been established for unidirectionally solidified metallic composites /9/ and is correlated with a Hall Petch type relation : \( \sigma \propto \lambda^{-1/2} \), \( \sigma \) is the strength and \( \lambda \) the lamellar thickness. But this is not a general behavior for ceramic composites : for example, in the TiC/TiB\(_2\) PVD lamellar composite, with a 50 % volume fraction of each phase when its lamellar thickness decreases from 5 \( 10^{-6} \) [m] to 1 \( 10^{-6} \) [m], no Hall Petch effect is detected /10/. On the contrary, for the Ni/TiC composite (82% volume fraction of Ni phase), when the lamellar thickness decreases from 8 \( 10^{-6} \) [m] to 2.10\(^{-6} \) [m], the strength of the material increases following a Hall Petch relation /11/.

In our case on the one hand, it appears that the lamellar thickness affects the strength; but on the other hand, the lamellar thickness should also affect the deforming mechanisms. As a matter of fact even if deformed with exactly the same experimental parameters, both samples show totally different deformation curves and totally different deformation microstructures.

From these remarks, stands out the third point of our discussion, which is an attempt to characterize the composite rupture behaviour. This attempt is supported by Griffith's approach /12/ when considering the results obtained with sample 1; while for sample 2 we based our analysis on the effects of residual internal stresses.

Taking into account Griffith's earlier works /12/, and assuming that the loading and unloading paths are linear and correspond to full elastic behaviour, a strain energy release rate term can be calculated with the relation

\[
G = \frac{\Delta U_{el}}{S} \quad [7]
\]

\( \Delta U_{el} \) is the elastic energy associated to the deformation cycle :

\[
\Delta U_{el} = (F_1 - F_u) \cdot d \quad [8]
\]

in which \( d \) is a recoverable displacement, \( F_1 \) and \( F_u \) are the maximum loading and unloading forces.
Finally S represents the crack surface originated during the deformation cycle, and which releases the elastic energy $\Delta U_{el}$. From the recorded experimental curve of sample 1 (Figure 2a), the values of $F_i$, $F_u$ and $d$ are directly deduced. They are 12 N, 7.7 N and $0.25 \times 10^{-3}$ m respectively.

The crack surface value is hard to evaluate directly from the SEM observations: the crack follows a transversal path on the outer layer (maximum tensile stress) then deviates at the first interface. Its total extension corresponds approximately to ten times the lamellar thickness. As the sample's span is $4.6 \times 10^{-3}$ m, it results a crack surface of $1.84 \times 10^{-7}$ m$^2$. We must insist that the value of the crack surface is extrapolated directly from surface observations by SEM; therefore they must be considered with care.

With the above mentioned parameters, the strain energy release rate term $G$ is: $6.25 \times 10^3$ [Joules/m$^2$]. As the sample was instantaneously unloaded when a drop of 0.25 N was detected, it seems reasonable to associate the strain energy release rate to a critical value for crack opening in the material. Moreover our value is of the same order of magnitude as the energy values of the work of fracture detected for C/C and SiC/SiC laminar composites: $5.3 \times 10^3$ [Joules/m$^2$] and $3.4 \times 10^3$ [Joules/m$^2$] respectively /13/. Finally, taking into account these considerations and assuming that a plane stress condition prevails during the crack opening process, an apparent fracture toughness value can be deduced from the following relation /12/:

$$ K_{IC}^{app} = (G/E)^{1/2} $$

with $E = 65 \times 10^9$ [Pa] (see table II) and $G = 6.25 \times 10^3$ [Joules/m$^2$], the apparent fracture toughness of our composite (sample 1) is: $20 \times 10^6$ [Pa.m$^{1/2}$].

For laminar composites reinforced by fibers with a sintered or CVD matrix the fracture toughness parameters given by Sakai /13/ for C/C and SiC/SiC composites are respectively $11.5$ [MPa.m$^{1/2}$] and $8.46$ [MPa.m$^{1/2}$]. For fibrous C/SiC composites, the toughness parameter deduced by Gomina et al /14/ from the analysis of three point bending tests was $15$ [MPa.m$^{1/2}$]; which is of the same order of magnitude as our apparent value. On the contrary, the toughness value determined for other composites having an aluminum metal matrix and reinforced with SiC fibers and Al$_2$O$_3$ dispersed particles /15/ is lower: $7$ [MPa.m$^{1/2}$].

At this point, the comparison among the different values of toughness seem to confirm the rate expected for regular distributed interfaces in this type of materials: deviate and stop crack opening. For sample 2, this approach is unrealistic: the Force/Displacement curves are not linear (loading/unloading paths), and a permanent displacement remains at the end of the test. Furthermore an evaluation of the global rupture surface after the test is impossible. Meanwhile an attempt to explain the origin of the severe observed damage by considering the internal residual stress effects can be developed. This attempt is based on the following relation which has been often used for to estimate the order of magnitude of the thermal residual stress in a layer /18/:

$$ \sigma_{th} = \frac{\Delta \alpha \cdot E \cdot \Delta T}{(1 - \nu)} $$

$\sigma_{th}$ is then the thermal residual stress developed in a layer having a young modulus $E$ and a coefficient of Poisson $\nu$. $\Delta T$ is the temperature variation of the sample and $\Delta \alpha$ is the linear thermal expansion mismatch between the considered layer and an adjacent one constituted by a different phase.

For our thicker samples the calculated residual thermal stress, in a SiC layer can reach 1.4 [GPa] (calculated with $E_{SiC} = 250$ [GPa], $\nu = 0.2$, $\Delta T = 1473$ [K] and $\Delta \alpha = 3.1 \times 10^{-6}$ [K$^{-1}$]).

Now considering these thermal stresses full elastic the corresponding stored energy $U_{el}$ in a lamellae of unit surface area and having a thickness $\lambda$, can be calculated by the relation /17/:

$$ U_{el} = \frac{\sigma_{th}^2}{2} \frac{\epsilon (1 - \nu)}{E} $$

In the case of sample 2, with this relation, we obtain for the SiC lamellae: $125$ [Joules/m$^2$].
Compared to the above mentioned works of fracture energies for a transversal rupture in C/C and SiC/SiC laminar composites, our value is one order of magnitude lower. Instead of that, we may point out that an interlaminar work of fracture close to our value was measured for the SiC/SiC laminar composite: 140 Joules/m² (13). Then if as observed in the case of in situ metallic lamellar composites, the lamellars faulted regions (bends, lamellar ends, kinks, ...) can act as stress concentrators /3,4/; nearby such regions interlaminar crack opening could occur so to release the high level of residual thermal stresses.

Further deformation of the material presenting thick lamellae would then be controlled principally by the coalescence of cracks. This last assumption is supported by our microstructural observations and also by the low level of work of deformation deduced from the curves corresponding to the test performed on sample 2: approximatively 0.28 \(10^{-3}\) Joules, reminding that for sample 1 we obtained: 1.15 \(10^{-3}\) Joules.

5 - CONCLUDING REMARKS

In spite of the restricted number of experiments, the results on this type of lamellar ceramic composites are somewhat promising. With respect to the crack opening process, improvement is obtained when comparing the apparent fracture toughness value to values for other ceramic composites.

Our results seem to prove that thinner lamellae and numerous interfaces stop transverse crack opening. The influence of lamellar thickness and lamellar perfection appear to be crucial to the properties of the investigated composite. Even if it is still not clear whether or not the material follows a Hall Petch type relation, thicker lamellae increase the residual stress effects catastrophically.

Further investigations may clarify the mechanical behaviour in function of other parameters.

REFERENCES


"Proceedings of the Conference on "In situ Composites" - (September 1979) - Lakeville N MAB (1973) 1.


12/ GRIFFITH, A.A

13/ SAKAI, M., TAKEVCHI, S., FISCHBACH, D.B., BRADT, R.C.
Proceedings fo the Conference of Ceramic Science- University of California, Berkeley- (July 1986)

14/ GOMINA, M., THEMINES, D., CHARMANT, J.L., OSTERSTOCK, F.

15/ SKINNER, A., KOCJAK, M.J., LAWLEY, A.

16/ HOFFMAN, R.W.

17/ PRESCOTT, J.J.

Figure 1 Micrograph of the lamellar C/SiC CVD composite. The layers are perpendicular to the length of the sample.
Figure 2 (a) and (b): Force/Displacement curves. The dotted lines correspond to linear fitting. Curve (a): sample 1. Curve (b): sample 2.
Figure 3 (a) and (b): S.E.M micrographs after the tests. The wide open arrow shows the approximate loading direction; the large black arrows in (a) show the crack opening in the outer layer; the small arrows show crack openings in (a) and (b); on micrograph (b), the small white arrow points to a wide longitudinal crack; the dotted lines schematize the sample contours.