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Influence of a multilayered matrix on the lifetime of SiC/BN/SiC minicomposites

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The mechanical behaviour at room temperature and the lifetime in air at 700°C under static loading of SiC/BN/SiC minicomposites have been investigated. The minicomposites consisted of a single tow of Hi-Nicalon fibres coated with a BN interphase and a CVI-SiC matrix. For few of them, a BN layer was introduced within the matrix. All the minicomposites were heat treated at high temperature to improve the BN crystallinity. In some cases, the BN interphase was submitted to a specific treatment before the infiltration of the SiC matrix, to further improve its crystalline state. The differences in interfacial zone, as assessed by TEM, were correlated with those in mechanical properties. A significant improvement of the mechanical behaviour at room temperature and the lifetime of the minicomposites with a multilayered matrix was observed. The multilayered matrix is efficient when a silica layer on both sides of the BN layer within the matrix is present.

1. Introduction

Multilayered interphases and matrices have been developed to improve the mechanical properties and the lifetime of ceramic matric composites (CMCs). Until now, the concept of multilayered matrix has not been developed very much [1–4]. On the other hand, multilayered interphases have been widely tackled, particularly $(PyC/SiC)_n$ sequences [5–9]. Only few applications of BN-based multilayered interphases have been reported [9, 10].

This concept applies only if the weakest zone within the multilayered interphase or matrix is located away from the fibre. The sequence of layers of different materials introduces interfaces which must play the role of a mechanical fuse preventing the catastrophic propagation of a crack. The path followed by the crack becomes tortuous, delaying the rupture of the fibres at room and high temperature. A strong bonding is therefore preferable close to the fibre [5]. In this work, only the role of a multilayered matrix has been studied.

The BN coatings as-processed from the (BCl_3, NH_3, H_2) gas system are only poorly crystallised and are consequently not stable at ambient temperature [11–14]. This instability results from an absorption of oxygen species in the form of O_2 and/or H_2O. To improve the crystallisation degree of the BN coatings, some authors have carried out post-processing heat treatments at higher temperature (1000 and 1200°C [14, 15], 1000–1700°C [12]). However, Lacrambe [12] has established that the efficiency of the heat treatment depends on the previous absorption of oxygen in the coating. It is worthy of note that the amount of oxygen absorbed by the boron nitride at room temperature is not completely removed after the heat treatment [14]. In this work, all the minicomposites were heat treated either as-processed, or after a specific treatment of the boron nitride. The conditions of the heat treatment have been chosen compatible with the stability of Hi-Nicalon fibres [9].

2. Experimental procedure

2.1. Materials

The SiC/SiC minicomposites with a BN interphase were prepared from Hi-Nicalon fibre bundles. In the as received state and when exposed to high temperatures, the Hi-Nicalon fibres display a superficial structural and chemical evolution which may affect their mechanical properties and particularly their lifetime in air at high temperatures. In order to improve the stability of the fibres, Bertrand *et al.* [9] have applied a heat treatment which prevents this drawback. This treatment leads to a decrease of the silicon concentration at the fibre surface. The thickness of the superficial carbon layer resulting from the local decomposition of SiC crystals, is only 10 nm thick but the silicon depletion stretches upon 80 nm. The removal of the carbon layer is necessary before the processing of the composites, as it may weaken the fibre/BN bonding and affect the lifetime of the composites at high temperature in air [16]. To this aim, the fibres have been specifically treated. The treatment conditions are based on previous studies, dealing with the chemical reactivity of various gaseous species with the fibre surface [17]. Auger electron spectrometry (AES) analyses were carried out after the treatment to confirm that the carbon layer initially present at the fibre surface was completely removed.

The BN interphase is processed by chemical vapour deposition (CVD) from the (BCl_3, NH_3, H_2) gas

TABLE I Description of the different types of minicomposites

	Sequence	Post-elaboration heat treatment	Specific treatment (Presence of oxygen in the coating)
Min1	Fibre/BN/SiC	No	No
Min2	Average BN thickness = 0.5 μm	Yes	No
Min3	Average BN thickness = 0.2 μm	Yes	Yes
MinA	Fibre/BN(1)/SiC/BN(2)/SiC	Yes	BN(1): No BN(2): Yes
MinB	Average BN(1) thickness = 0.2 μm Average BN(2) thickness = 0.5 μm	Yes	BN(2): No

mixture at low pressure ($P = 1.3$ kPa) and low temperature ($T = 800°$C) with a volume ratio Q_{NH_3}/Q_{BCl_3} higher than 1. In such processing conditions, the coatings are amorphous. A high degree of crystallisation can only be obtained after a post-processing heat treatment at a temperature chosen compatible with the stability of the substrate (Min2, Min3, MinA and MinB). In order to improve the efficiency of this heat-treatment a second specific treatment can be managed just after the deposition of the BN coating, in order to absorb oxygen (Min3 and MinA). The presence of a small amount of oxygen was seen to enhance the re-crystallisation by heat treatment [12].

The SiC matrix is processed by isobaric-isothermal chemical vapour infiltration (I-CVI) from the (CH_3SiCl_3 (MTS), H_2) gas mixture with a Q_{H_2}/Q_{MTS} volume ratio of 4.

The matrix of the minicomposites consists of a $(BN/SiC)_n$ sequence, with a thickness of the SiC layers of about 2 μm, whereas that of the BN layers ranges from 0.2 to 0.5 μm. The multilayered minicomposites of the present study display a rather simple architecture as the sequence number does not exceed two. The different minicomposites are described in more details in Table I.

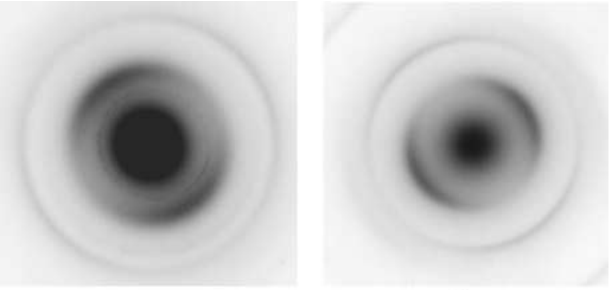
2.2. Microstructural characterisation

Transmission electron microscopy (TEM) analyses were performed with a Philips CM30ST with a resolution of 0.2 nm. They were performed according to conventional procedures, i.e., dark field, electron diffraction and high resolution modes. The samples were prepared following usual techniques (epoxy mounting, mechanical thinning, polishing and ion-milling with a Gatan Duo-mill). The development of an image analysis algorithm [18] has allowed the measurement of the local anisotropy OA (for the Orientation Angle). This technique is based on the high spatial resolution of the selected area electron diffraction technique (SAD). The misalignment of boron nitride layers affects the shape of the two 002 spots normally observed for hexagonal BN. Two arcs or even a complete ring appear for less anisotropic or isotropic materials, respectively.

2.3. Mechanical characterisation

The tensile and static fatigue tests were carried out using two house-made laboratory apparatus.

The minicomposites (with a 25 mm gauge length) were tensile tested at room temperature with a displacement rate of 50 μm·min^{-1}. The elongation was measured using two displacement sensors mounted on both opposite sides of the specimens. The stress was measured using a 500N load cell.



(a) (b)

Figure 1 SAD images of the BN interphase in the minicomposites: (a) Min2 and (b) Min3.

The minicomposites (with a 25 mm gauge length) were submitted to static fatigue tests at 700°C in ambient air. The minicomposites were attached with an alumina-based cement into two alumina tubes fixed in a cold part of the furnace. The whole minicomposite specimens were set in a relatively constant temperature zone, in the hot part of the furnace. The tensile load was applied once the temperature was reached.

3. Results and discussion

3.1. TEM characterisation

Selected area diffraction (SAD) managed on BN interphase in the Min2 and Min3 minicomposites exhibit broad and diffuse arcs. This means that BN is weakly crystallised and almost isotropic (Fig. 1). The Orientation Angle (OA) and the inter-reticular distance (d_{002}), measured from the SAD images, are presented in Table II. The crystallisation degree and the texture of the BN interphases of Min2 and Min3 minicomposites are only slightly different.

The bright edge observed on the dark field images along the fibre/BN interface as well as the (002) plane arrangement on the high resolution images evidence a more anisotropic BN layer directly in contact with the fibre (Fig. 2). Such features have already been observed for a BN processed from the (BF_3, NH_3, Ar) gas system. The (002) planes were observed to be parallel to

TABLE II Orientation Angle (OA) and inter-reticular distance (d_{002}) of the BN interphase in the minicomposites Min2 and Min3

	OA (°)	d_{002} (nm)	L_c (nm)
Min2	94	0.369	1.2
Min3	74	0.359	1.3

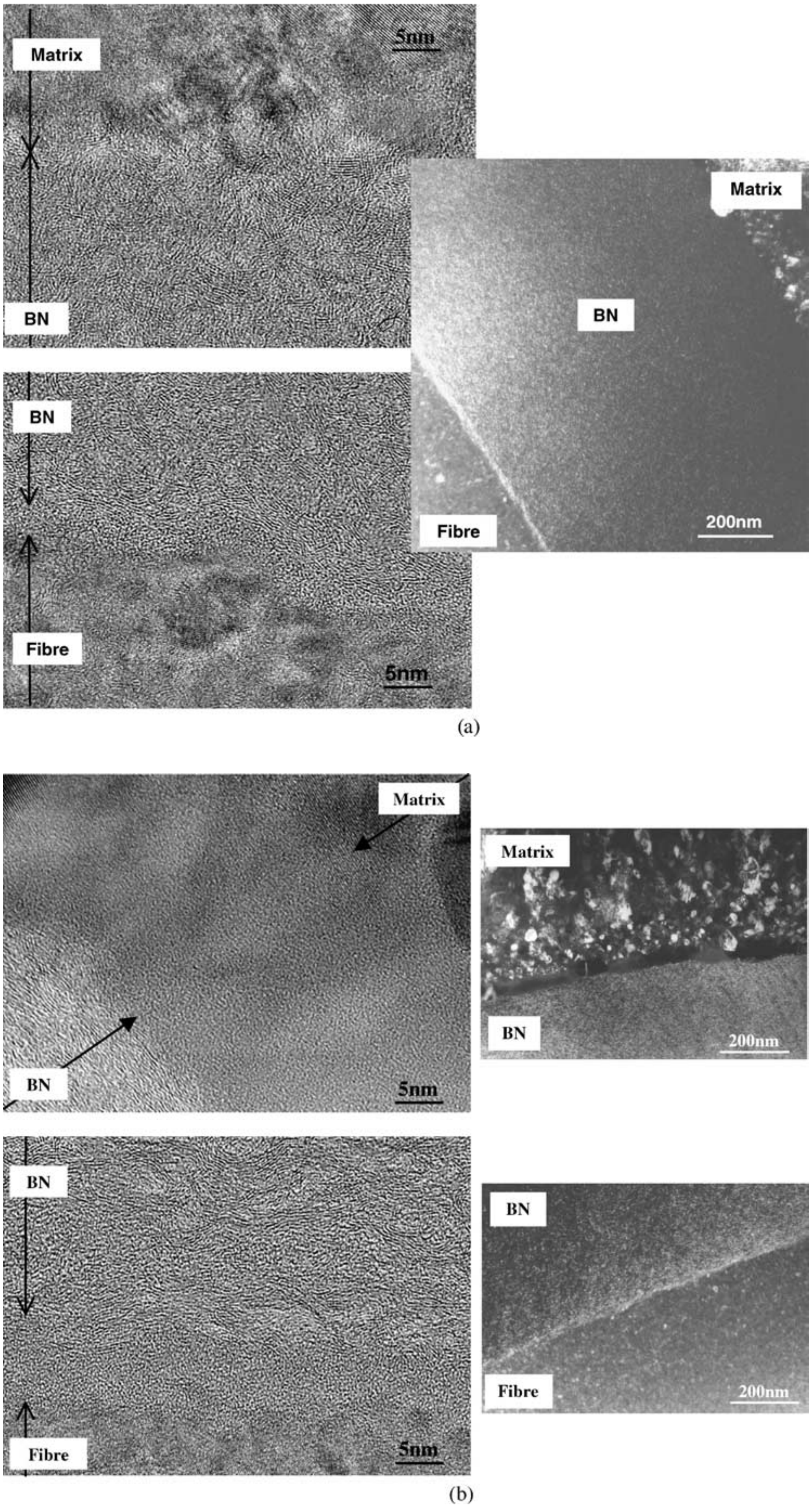


Figure 2 Dark field and high resolution images of the interfacial zone in the minicomposites: (a) Min2 and (b) Min3.

the fibre surface along the first 10 nanometers, while more irregularly oriented through the rest of the coating [19]. This feature has been attributed to a transitory growth mechanism related to a substrate effect. The BN planes were often found to be arranged parallel to each other and to the fibre surface when the substrate roughness is low, such as for example on silica. Such an organisation can be related to a 4 and 10 nm thick silica layer observed between the fibre and the BN coating, respectively in the Min2 and Min3 minicomposites. The major difference between the Min2 and Min3 minicomposites lies in the BN/matrix interface, which shows residual crystals within a thin silica layer for Min3 minicomposites. The presence of oxygen as silica at the fibre/BN (and BN/matrix interfaces in the Min3 minicomposites) results from the specific treatment of the boron nitride before the infiltration of the SiC matrix. The presence of these silica layers suggests a diffusion of oxygen towards both the interfaces during the post-elaboration heat treatment and its reaction with the SiC.

The BN layers of the MinA and MinB minicomposites, respectively, show the same characteristics as those of the Min2 and Min3 minicomposites previously described.

3.2. Tensile testing at room temperature

The tensile load/strain curves of each type of minicomposite are reported in Fig. 3.

The Min1, Min2 and Min3 minicomposites having a 0.5 μm thick BN interphase all show a similar non linear damageable behaviour. The presence of a plateau between the elastic zone and the final part of the curve (i.e., corresponding to a domain where the load is only supported by the fibres), suggests that the matrix cracking saturation is rapidly reached [20]. Such a mechanical behaviour can be associated to a relatively weak fibre/matrix bond [5]. Through the comparisons of the Min1 minicomposite with Min2, and the Min2 minicomposite with Min3, the post-processing heat treatment and the presence of oxygen in the coatings appears to lead to a wider plateau between the elastic and fibres loaded zones, which might be due to a lower load transfer along the debonded lengths.

In contrast, in the case of the Min1, Min2 and Min3 minicomposites with a thinner BN interphase, the load/strain curves still exhibit a non linear damageable behaviour but with a strongly reduced plateau. This feature can be assigned to a stronger fibre/matrix bond. Moreover, the changes in the mechanical behaviour resulting from both the post-processing heat treatment and the oxydation, are only very slight (contrarily to the series with 0.5 μm thick BN interphase).

The elastic zone is much larger than for the other minicomposites with the layering of the matrix (MinA and MinB minicomposites). Previous works [1, 5] have shown that a multilayered matrix can be an efficient way to improve the toughness of composite materials. The improved mechanical behaviour at room temperature of the MinA minicomposite, as compared with MinB, might result from the presence of an effective "mechanical fuse" within the matrix, i.e., a weak zone where matrix crack deflection can take place. Such a fuse can be managed by the occurrence of: (i) a highly anisotropic BN layer and/or (ii) a weakly bonded interfaces on both sides of BN interlayer. In both minicomposites, the interphases observed within the matrix have very similar microstructures and microtextures. The main difference is the presence of silica layers on both sides of the second BN layer located within the matrix of MinA minicomposite. It might have favoured deflections at the BN/SiO_2 interfaces in the second BN layer within the matrix of MinA. These silica layers are almost absent or too thin to weaken the chemical bonds along the corresponding interfaces in the MinB minicomposite.

3.3. Fracture surface analysis

All the different minicomposites show the same type of fracture surface : pull out with bare fibres, characteristic of a non brittle rupture and of a rather weak fibre/matrix bond (Fig. 4).

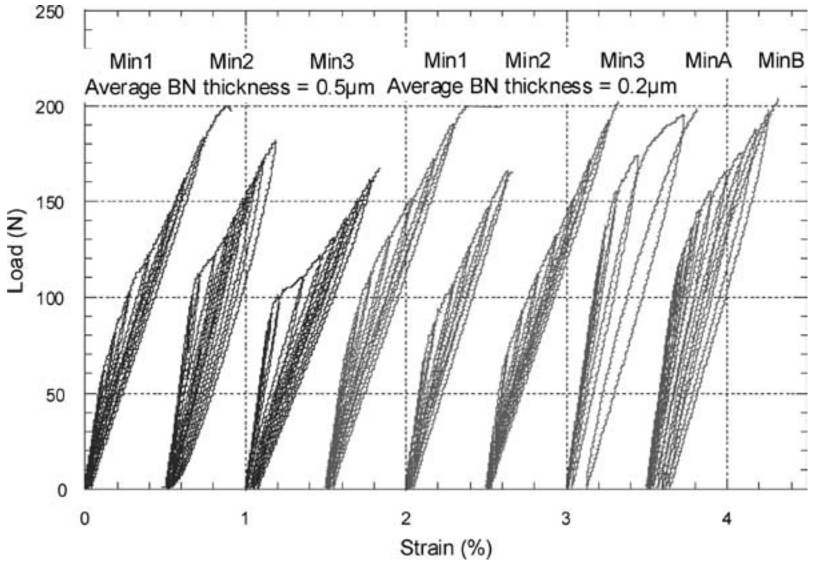
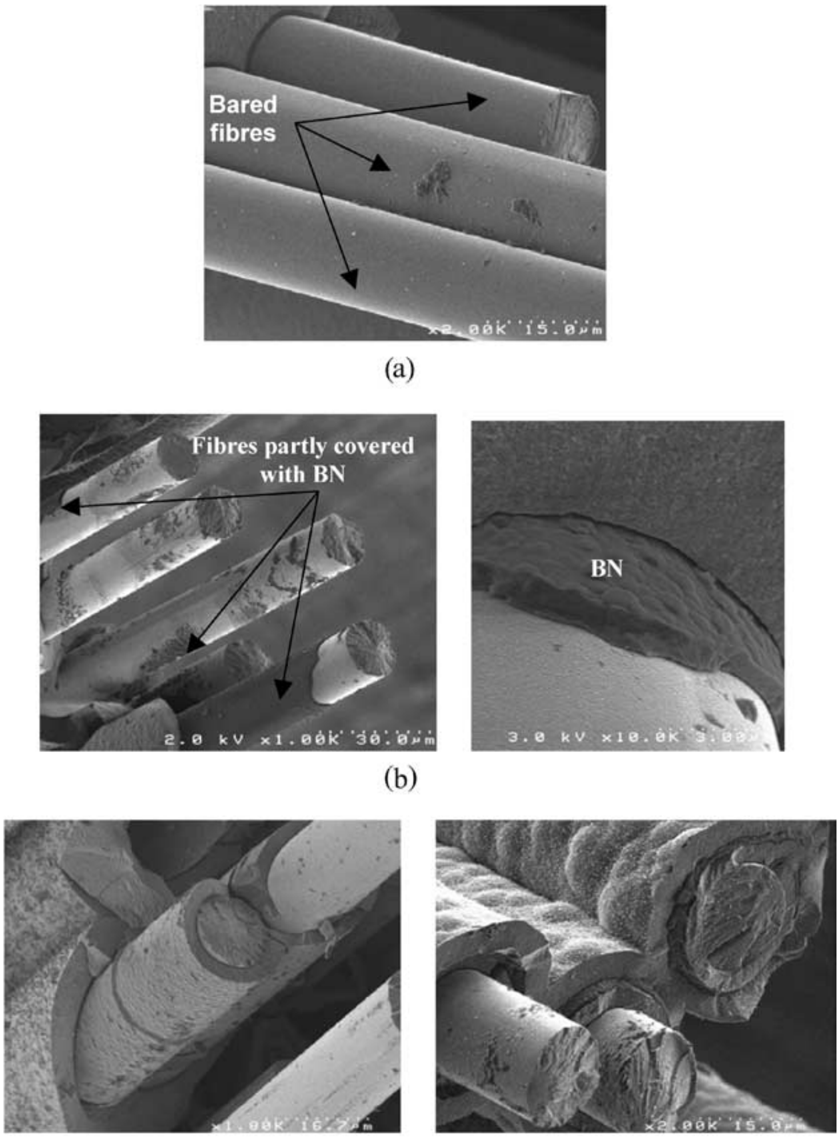


Figure 3 Load-strain curves of the different batches of minicomposites.



(a)

(b)

(c) (d)

Figure 4 Failure surface of the different types of minicomposites after tensile tests at room temperature: (a) Min2, (b) Min3, (c) MinA and (d) MinB.

The Auger electron spectroscopy (AES) analyses of bare fibres from the fracture surface of Min1 and Min2 minicomposites show the absence of BN at the surface of the fibres, suggesting that the fibre/matrix decohesion occurred along the fibre/BN interface.

The presence of BN is nearly systematically observed on the extracted fibres of the Min3 minicomposite, but only partly covering the fibres. The depth concentration profiles recorded from a zone of the fibre covered by the boron nitride show no significant amount of oxygen at the fibre/BN interface. The analysis of the bare fibre surface shows traces of boron and nitrogen as well as an excess of oxygen. This oxygen was probably absorbed by the boron nitride during the exposure to air after tensile test.

In contrast with the smooth surfaces of the CVD BN coatings, the surface of the boron nitride in the Min3 minicomposites (Fig. 4b) is much rougher in comparison with the as-processed BN [20]. Such a surface change probably results from the structural modifications occurring in confined space during the post-processing heat treatment in presence of oxygen. If this heat treatment is managed before the matrix deposition, this phenomenon disappears. This mechanism might involve the volatilisation of gaseous oxygen rich species confined by the SiC matrix.

The fibre/matrix decohesion occurs in all these materials either at the fibre/BN interface or at the BN/matrix interface. The weak bond along the BN/matrix interface in Min3, probably results from the structural ordering of BN or/and from the lower debond energy due to the presence of a silica layer between the interphase and the matrix. In the first case, the interphase volume decreases radially, due to the BN re-ordering. That would stress the fibre in compression, reinforcing the fibre/BN bond while weakening the BN/matrix bond. In the second case, the BN/matrix decohesion would simply result from a weakening of the BN/matrix bonding by the presence of a thick silica sublayer which does not exist in Min1 or Min2. The double deflection observed from the BN/matrix interface to the fibre/BN interface probably results from the high roughness of the surface of the BN coatings, impeding the sliding between the BN interphase and the SiC matrix. Conversely, the high anisotropy of BN at fibre/BN interface while decreasing the friction forces, would tend to promote the

TABLE III Interfacial shear stress calculated from the opening of hysteresis loop using the Lamon-Rebillat-Evans micromechanical model of the different types of minicomposites

	Average BN thickness = 0.5 μm			Average BN thickness = 0.2 μm				
	Min1	Min2	Min3	Min1	Min2	Min3	MinA	MinB
τ (MPa)	85 ± 37	67 ± 13	52 ± 4	220 ± 56	149 ± 17	212 ± 60	48 ± 2	48 ± 5

sliding between fibre and matrix. Also, the decrease of the thickness of the interphase improves the load transfer between the fibre and the matrix, limiting the sliding, in particular at the BN/matrix interface. The matrix cracks are consequently more rapidly deflected from the BN/matrix interface towards the fibre/BN one, resulting in a decrease of length of the BN patches still attached to the extracted fibres (pull out).

In the case of the MinA minicomposite, deflections occur at the fibre/BN interface as well as within the matrix. The lengths of the extracted fibres, which are either bared or covered with the first SiC layer, are respectively below 50 and 30 μm.

In the case of the MinB minicomposite, the decohesions are mainly observed at the fibre/BN interfaces. The lengths of the extracted bared fibres are about few tenth of μm. On the other hand, the fibres covered with the first SiC layer are extracted on larger lengths. These observations suggest that for a given BN interphase, the fibre/BN bond is weaker than the matrix/BN bond, probably because of different roughness and chemical composition of the fibre and matrix surfaces. The differences in length of the extracted fibres covered with the first SiC layer in the MinA and MinB minicomposites, tend to corroborate the assumption that the specific treatment of boron nitride may have weakened the bonds at both interfaces of the boron nitride and therefore increased the ability of the BN interphase to deflect matrix cracks.

3.4. Crack spacing

The minicomposites with a 0.5 μm thick BN interphase was measured with a relatively high crack spacing ($\geq$130 μm), probably resulting from a weak load transfer between the fibres and the matrix (in agreement with the long debonding lengths). The minicomposites with a 0.2 μm thick BN interphase have a lower crack spacing ($\leq$120 μm). This is expected because the load transfer between fibre and matrix is increased due to a lower thickness of the BN layer. This decrease of the crack spacing with the thickness is more significant for Min3 minicomposites (compared to the decrease with thickness in the Min2 series). This is suppose to be related essentially to the amount of silica at the interfaces in Min3, which is able to weaken the fibre/matrix bond and which is more abundant when BN is 0.5 μm thick.

For both the MinA and MinB minicomposites, the crack spacing was measured from the outer SiC matrix layer and found close to 150 μm.

3.5. Interfacial shear stress (τ)

The interfacial shear stresses were calculated by using the Lamon-Rebillat-Evans micromechanical model [21] (based on the analysis of the hysteresis loop opening before the rupture) (Table III). The τ values obtained show that the decrease of the thickness of the BN interphase from 0.5 to 0.2 μm leads to a significant strengthening of the interfacial load transfer.

It is worthy of note that for equivalent BN thicknesses, the τ values obtained for these minicomposites are higher than those reported by (i) Bertrand [9] for a 0.15 μm thick BN interphase ($\tau \approx 40$ MPa) and (ii) by Morscher *et al.* [22] for a 0.5 μm ($\tau = 15$ MPa). The low τ values reported by these two authors is well explained by the presence of a thin carbon layer at the surface of the Hi-Nicalon fibre this carbon layer is observed on the surface of the debonded lengths. This carbon layer generally appears during the processing of the minicomposites. In the present study, the treatments of the Hi-Nicalon fibres before the processing of the minicomposite allow to remove such a superficial carbon layer on the fibre. As a result, the load transfer between the fibres and the matrix was strongly improved.

The τ values obtained in the case of the MinA and MinB minicomposites are suprisingly low and even lower than for conventional minicomposites. These τ values are not consistent with (i) the load-strain curves and (ii) the particularly complex dissipating process of the energy expected for multilayered composites. One of the reason for that discrepancy is the fact that matrix cracking saturation is never reached during damaging until the rupture, as shown from the continuous variation of τ values.

3.6. Static fatigue results

The lifetime tests were carried out on three samples for each type of minicomposites. The average value is reported in Table IV, except when the scattering of the data was important. For applied load values slightly beyond the elastic limit, the minicomposites were only slightly damaged and the resulting rupture times, extremely

TABLE IV Lifetimes of the different types of minicomposites in air at 700°C

	Average BN thickness = 0.5 μm			Average BN thickness = 0.2 μm		
	Min1	Min2	Min3	Min3	MinA	MinB
Load (N)	100				120	
Lifetime (h)	25–75.5	0.3–4.5	21	133.5	72	22.5

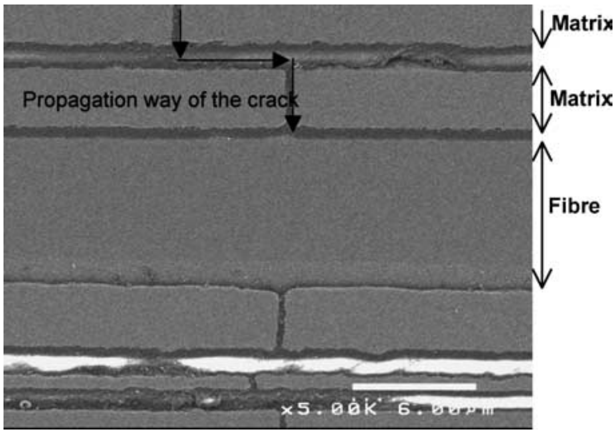


Figure 5 SEM observation of a matrix crack deflection after tensile test in the minicomposite MinA.

long. To assess the effect of the matrix crack opening on lifetime, the minicomposites were tested under higher loads (100N). The elastic limit of the multilayered minicomposites being higher than that of the other minicomposites, they were tested under a 120N load.

For equal BN thicknesses, the following features could be noticed:

(i) the lifetime of the minicomposites Min2 was observed to be shorter than that of the minicomposites Min3. The tensile testing study evidenced that the Min3 minicomposites show a rather poor load transfer (but at room temperature). On the other hand, their BN interphase is slightly more crystallised than in the Min2 minicomposites. Such a slight difference in the microstructure alone probably does not explain such a large lifetime difference. The existence of a boron-rich silica layer in Min3 may have a healing behaviour already at 700°C, increasing the diffusion path of oxygen towards the fibres.

(ii) the lifetime of the Min1 minicomposites is longer than that of the Min2 minicomposites. The difference between those two materials is the post-processing heat treatment of Min2. This treatment is supposed to debond the matrix and weaken the load transfer. As a result, large crack opening enhance the oxygen diffusion at the fibre surface in Min2. Comparatively, Min1 minicomposite has an improved lifetime which result in less opened matrix cracks due to a more efficient fibre/matrix load transfer.

(iii) the lifetime of the Min1 minicomposites is also longer than that of the Min3 minicomposites. This means that the effect of the post processing treatment for Min3 which is not applied for Min1, is highly detrimental for the protection of the interface.

The decrease of the interphase thickness strongly improves the lifetime of the material. The minicomposites with a 0.2 μm thick interphase indeed show a strengthened load transfer between the fibres and the matrix, limiting the opening of the matrix cracks. This feature is in agreement with the work from Leparoux *et al.* [15], who have also reported that the lifetime of SiC/SiC composites depends on the thickness of the BN interphase.

The BN interphases located within the matrix of both the MinA and MinB minicomposites have only a slightly different crystallisation degree. The higher lifetime of the MinA minicomposite cannot be explained only by the better oxidation resistance of this interphase. On the other hand, the observation of the matrix cracks on the polished longitudinal sections of the tested specimens showed that the oxygen path towards the fibres is longer in the case of the MinA minicomposite, as the cracks are generally deflected by the second BN layer within the matrix (Fig. 5).

The multilayered minicomposites have a longer lifetime than the other minicomposites.

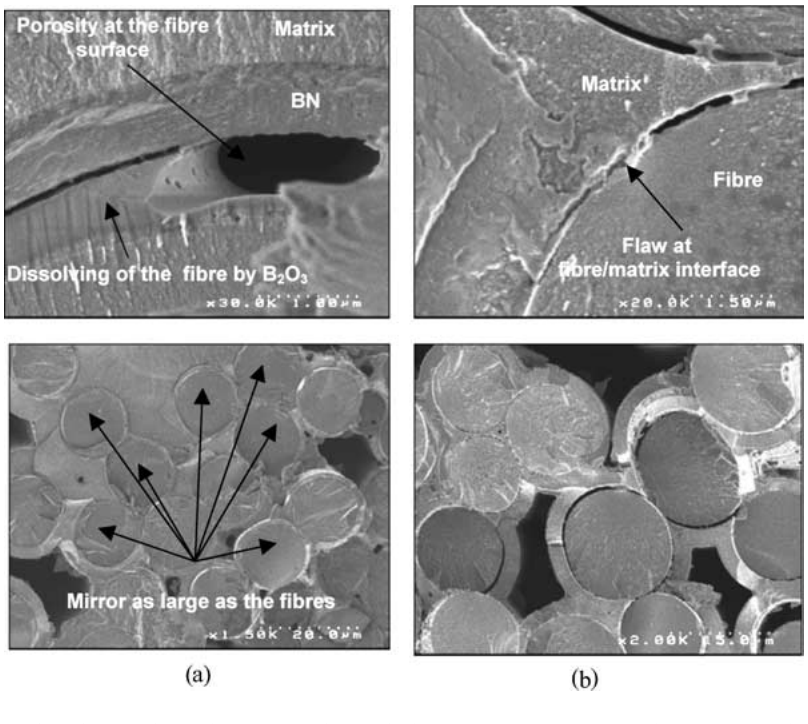


Figure 6 SEM observations of minicomposite surface failure after static fatigue tests at 700°C in ambient air: (a) Min3 with a 0.5 μm thick BN interphase and (b) Min3 with a 0.2 μm thick interphase.

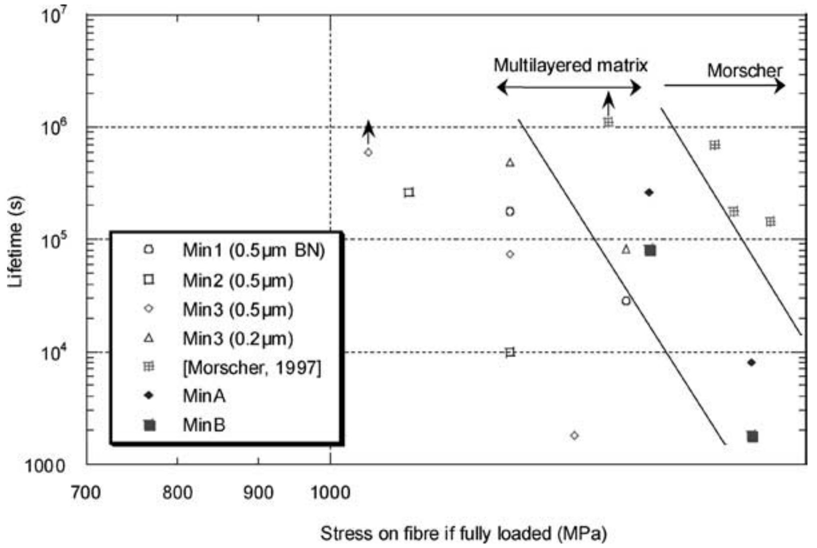


Figure 7 Comparison of lifetimes in static fatigue in air at 700°C, between the present study and results from Morscher [23].

The SEM observations of the rupture surface of the tensile tested Min3 minicomposites show that the fracture surfaces of the fibres are in the same plane as that of the matrix (Fig. 6). Moreover, this rupture surface is characterised by a large number of mirrors with a size close to the section of the fibres, suggesting a subcritical cracking mechanism or a rupture of the fibres at very low stresses. The BN interphase is only partly oxidised. The flaws responsible for the failure of the fibres are generally surfacial. They may have been created by the reaction between the boron oxide and the silicon carbide and/or by the formation of silica bridges between the fibres and the matrix. These features lead respectively to the degradation of the fibres strength and to stress concentrations at the fibre surface. They result in the early rupture of the fibres and, consequently, in the failure of the minicomposites (for applied loads well below what the fibres are able to support at ambient temperature).

As expected, the minicomposites having a 0.5 μm BN interphase show a smaller number of silica bridges than those with a 0.2 μm interphase. Less silica is needed to fill in the interfacial space when debonded. The formation of silica bridges is thus easier. The fibre mirrors observed for the latter are smaller (the larger mirrors being observed at the periphery of the minicomposite). Some volume flaws are still observed as in the as-received fibres.

The lifetime of the present materials have been compared with those of the SiC/BN/SiC minicomposites studied by Morscher [23], using the concept of the equivalent stress (i.e., the stress that would be applied to the fibres if the whole matrix was unloaded). In Morsher's works, the BN interphase was processed at 1400°C from (BCl_3, NH_3, Ar) gas system. The thickness of the boron nitride interphase ranged from 0.5 to 3 μm for the minicomposites studied by Morscher. The coherent domains of this interphase are characterised by a L_c value of 3.5 nm and a d_{002} inter-reticular distance value of 0.344 nm [24]. These structural properties have been obtained by XRD whereas they have been estimated by TEM in the present study. It is therefore difficult to compare the two materials, as the values given by TEM are generally larger for d_{002} and lower for L_c than those given by XRD.

The lifetime of the minicomposites studied by Morscher are longer than those of the present study (Fig. 7). From what is known from their processing conditions and structural properties, these materials are not expected to have a better crystallised BN interphase than those of the present study. Moreover, the interfacial shear stress of these materials ($\tau = 30$ MPa, as measured by indentation) is low, a feature not surprising because of the large interphase thickness. The lifetime of the minicomposites studied by Morscher may appear surprisingly long, while considering such a low interfacial shear stress and poor crystallisation degree. However, Morscher reported that the matrix cracks were found hardly opened after testing. This feature suggests that an effective healing of the matrix may have occurred, favoured by the silicon excess of the matrix. This hypothesis might explain the fact that the interphase was less damaged at 700°C than at 500°C (a too low temperature to allow the matrix crack healing).

4. Discussion-conclusions

The issue addressed in this work is the efficiency of BN coatings as interphase for SiC/SiC composites in replacement of pyrocarbon. When deposited in conditions compatible with Hi-Nicalon fibre stability, boron nitride does not result in a high degree of crystallisation and anisotropy. This poor structure was suspected to be responsible for the low efficiency of BN as interfacial material. In this work a specific treatment of BN and a post-processing heat treatment were developed to improve the crystallisation degree and the anisotropy of the interphase. First, Min1 series of minicomposites is produced as a reference, consisting of a CVI deposit of BN interphase and silicon carbide matrix in the same batch. Min2 was processed as Min1 but with an additional post-processing heat treatment to improve the crystallinity. But it was shown by TEM that the crystallinity is only slightly improved by this way. Thus during the processing, a treatment is added just after BN deposit to incorporate some oxygen. Min3, with this additional treatment, was characterised with TEM: there was no strong improvement. But Min3 has shown unexpected effects as the formation of a silica layer on both side of the BN layer. Silica is supposed to be a reaction layer formed on both sides of the BN interphase (e.g., residual SiC crystals form islands in the silica on matrix side). The mechanical behaviour at room temperature is changed by these treatments : the elastic limit is increased but the non-linear part still exhibits a plateau, typical of a weak interface. Matrix crack saturation is reached sooner. τ, the interfacial shear stress, decreases while the crack spacing keeps almost constant above 130 μm. All the data are consistent. Fracture surface analysis in Min3 series exhibits some pull-out with the fibres covered with BN, contrarily to Min1 or Min2 (pull-out with bare fibres) demonstrating the role of silica sublayers as mechanical fuse. The static fatigue is managed at a load of 100N, a temperature of 700°C under air. First, it is seen than Min2 lifetime drops regarding Min1 reference. This is attributed to the post-processing heat treatment known to open a large interfacial decohesion between the fibre and the matrix where oxygen diffuse readily during the test. Min3 is again equivalent to the reference. This was attributed to the closing of the interfactial decohesion (silica reaction layer) and also to the shift of the location of the weakest zone to the BN/matrix interface where the thick silica layer may act as glass healing material.

In order to balance the heat treatment effects and to strengthen the fibre/matrix bond, the thickness of the boron nitride interphase was efficiently reduced (0.2 μm instead of 0.5 μm). When BN thickness is reduced the load-strain plots exhibit the same trend: it results in a higher fibre/matrix transfer efficiency. This was evidence by a longer convex-up plot and a reduced plateau in the non-linear zone. The second point is the attenuation of both the effect of post-processing treatment (Min2) and oxygen content in Min3. It strongly improves the lifetime.

Finally, the multilayering of the matrix, even with a simple architecture, leads to an improvement of the mechanical properties at room temperature (e.g., higher elastic limit, deflection within the matrix) and also at high temperature (e.g., longer lifetime at a more severe stress). It was deduced that BN deposited within the matrix acted as an efficient mechanical fuse, especially when a silica layer was present on both sides of the BN layer in the matrix (MinA), in other words when a weakest debonding zone is managed far from the fibre/matrix interface.

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