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The fracture behaviour of SiC reinforced SiAlYON ceramic matrix composites

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ABSTRACT :

This paper presents the results of a study carried out at the AEROSPATIALE Joint Research Center on SiAlYON Matrix Composites reinforced with 0, 10, 20 and 30% of SiC whiskers.

Firstly the test methodology for measuring the K_{Ic} is described. The SENB (Single Edge Notched Beam) specimen pre-cracked in compression cycling is compared to the Chevron Notched bend bar test. In both cases the fracture toughness is measured in a bend test more adapted for tests at elevated temperatures.

Then the crack propagation at elevated temperature of the composite for the four SiCw volume fractions is described and discussed in relationship with the rheological behaviour of the matrix and the volume fraction of reinforcement.

Keywords : Ceramic Matrix Composites - SiC - Whisker - SiAlYON - Fracture Behaviour

1. INTRODUCTION

For certain applications involving high temperature, ceramics exhibit a combination of attractive properties : low density, chemical inertness, high elastic modulus and resistance. Nevertheless, the widespread use of ceramics has up to now been limited due to their brittleness and their correlated poor reliability in strength. Many studies have been carried out to try to improve this behaviour so as to allow the use of these materials on structural applications. Considerable progress has been made in the composition and microstructure design of ceramics. However the most important results have been obtained through the conception of composite materials. In particular, SiC whiskers reinforcement has been experimented with success on alumina and silicon nitride [1,2]. The main objective is then to significantly increase the fracture behaviour in the composite while maintaining the attractive high temperature properties of the matrix. The critical stress intensity factor (K_{Ic}) is considered as one of the most important fracture properties of ceramics. So it is the main point to have a viable, simple and reproducible methodology for measuring the real fracture toughness of the ceramic or the composite and to understand perfectly what happens in the material when a crack propagates as a function of the temperature and thus of the rheology of the matrix.

This paper presents the results of a study carried out on SiAlYON matrix composites reinforced by 0, 10, 20 and 30% v / o of SiC whiskers.

2. FRACTURE TOUGHNESS MEASUREMENT

Many test methods of measurement of the fracture toughness on ceramics have been evaluated recently but they lead to results which are not in perfect agreement. Among them we selected two, both based on the bending test, easiest to proceed and well adapted for tests at elevated temperature, but using two different kinds of specimen. These are the Chevron Notched Beam and the Single Edge Notched Beam

(SENB) precracked in cyclic compression. These two test procedures were compared on different ceramics and ceramic matrix composites.

As it is difficult to create sharp reproducible precracks in brittle materials the Chevron Notched Beam has been developed and studied as an interesting alternative for ceramics [3-4]. The main interest of this specimen is that it does not require any precracking. Due to the specific geometry of the machined notch the crack, which appears at the vertex of the chevron for a small load level, then slows down and stabilizes; as a matter of fact, for a given load the K factor decreases as the crack propagates and the crack tip dimension is increasing. An increase of load is then needed to make the crack further grow. The specimen rupture directly gives the fracture toughness associated with a stable crack. Besides the maximum load measurement is enough to get the K_{Ic} : no measurement of the real crack length is necessary. However, this test procedure can only be applied to materials with flat or nearly flat crack growth resistance curves.

Although getting a stable precrack on brittle materials is merely impossible with conventional methods, EWART and SURESH [5] observed that it was possible to develop a stable crack in polycrystalline alumina using a compression cycle loading on a SENB specimen. The fatigue crack develops along the plane of the notch, in the direction normal to the compression axis, with a decreasing speed and finally stops. This method may then be considered for precracking specimens that shall be used for K_{Ic} or even R-Curve measurements, presuming that the crack length may be measured or calculated.

Table 1 reports the results obtained with the two methodologies on several ceramics and ceramic matrix composites (SiAlYON with 10, 20 and 30 wt % of SiC_w reinforcement).

		Si ₃ N ₄	SiAlYON	10% SiC _w	20% SiC _w	30% SiC _w
CHEVRON	nber spec.	16	6	5	6	6
	K_{Ic} (MPa√m)	7.1	5.3	5.4	5.2	4.9
	stand. dev.	0.2	0.1	0.1	0.05	0.1
SENB precracked	nber spec.	5	8	7	9	7
	K_{Ic} (MPa√m)	7.2	5.4	5.9	5.7	5.4
	stand. dev.	0.1	0.2	0.3	0.3	0.2

Table 1 : comparison of the fracture toughness values obtained with the two methodologies

For each test the scatter band is narrow, which corresponds to one of our objectives. In all cases the fracture toughness obtained with the SENB is greater than that measured with the Chevron Notch although the difference is small ($\leq 0,5$ MPa√m). This difference may be related to the fact that the precracking operation in the SENB may induce residual stresses which delays the catastrophic propagation.

On the other hand the Chevron Notch method requires a stiff test machine. As a matter of fact, if the energy stored in the testing machine is too high the crack, as it starts propagating does not stabilize. The rupture mode is unstable and the K_{Ic} values calculated from this test are overestimated. In this study, with our particular specimen geometry, we have observed up to 30% of unstable rupture. This suggests that a minimum of number specimen has to be broken to obtain a sufficient number of valid results. The values calculated from unstable tests have not been reported in table 1.

Contrarily to what was expected for the SiAlYON matrix composites, the addition of SiC whiskers does not lead to a significant increase of the fracture toughness. These results may be explained by the fact that the materials exhibit a very strong interface between the matrix and the whiskers. This is confirmed by the SEM examination of the fracture surfaces which evidence an intergranular failure mode without any whisker pull out. The size of the whiskers may also be a concern : studies on alumina and silicon nitride matrix composites have shown the importance of the size and the aspect ratio of the whiskers for toughness [6-7].

2. HIGH TEMPERATURE TOUGHNESS

For the tests at elevated temperature, only the "SENB precracked" has been used. Two temperatures, 1250°C and 1300°C, have been chosen for this investigation. The results of fracture toughness tests are

reported in tables 2 and 3. Further to the examination of the fracture surfaces, two values of stress factor intensity have been considered : one calculated with the initial crack length and the fracture load has been called K_{Ii} , the other, calculated with the length of the crack after stable propagation and the fracture load, has been called K_{Iapp} (apparent fracture toughness). As the fracture toughness is a criterion of sudden crack propagation, it seems more sensible to use this last value.

% SiCw	Nber spec.	Vt (mm/min)	K_{Ii} (MPa√m)	stand. dev.	K_{Iapp} (MPa√m)	stand. dev.
00	4	0.1	4.7	0.2	6.2	1.1
10	4	0.1	6.3	0.2	6.8	0.3
20	4	0.1	6.2	0.7	6.9	0.9
30	6	0.1	4.9	0.2	-	-

Table 2 : Fracture toughness at 1250°C for the different SiAlYON

% SiCw	Nber spec.	Vt (mm/min)	K_{Ii} (MPa√m)	stand. dev.	K_{Iapp} (MPa√m)	stand. dev.
00	4	0.1	4.0	0.7	5.6	1.4
10	3	0.1	6.7	0.3	10.2	0.3
20	3	0.1	6.6	0.7	9.2	0.7
30	4	0.1	7.8	0.3	9.2	0.7

Table 3 : Fracture toughness at 1300°C for the different SiAlYON

The comparison with the room temperature tests realised on the same batches is given in figures 1 and 2. Above 1250°C a difference between reinforced and unreinforced materials is observed, whereas at room temperature the addition of SiC whiskers did not lead to a change of the fracture toughness.

At 1250°C, the 30% SiC whiskers composite keeps its linear elastic and brittle behaviour. For the other materials, a stable crack growth prior to fast fracture is observed on the fracture surfaces. This propagation is larger for the unreinforced SiAlYON than for the composites with 10 and 20% of SiCw. (0.15 to 0.43 mm for the SiAlYON, and 0.9 to 0.27 mm for the 10 or 20% SiCw/SiAlYON). In all cases, the calculated stress intensity factors are superior than those at room temperature.

At 1300°C, all the materials exhibit stable crack growth before fracture. On the load-displacement curves, the elastic strain is followed by a non linear part. Obviously, the stable propagations observed are more important at 1300°C than at 1250°C (0.66 to 1.58 mm for the SiAlYON, 0.13 to 0.46 mm for the 10%, 0.55 à 1.22 for the 20%, and 0.8 to 0.91 mm for the 30 %). The fracture toughness of the 30% SiC(w)/SiAlYON increases to the same level as the 10 and 20% SiC composites.

Figure 1 : Comparison of the K_{Ii} values at 1250°C and 1300°C with the K_{Ic} at room temperature

Figure 2 : Comparision of the K_{Iapp} values at 1250°C and 1300°C with the K_{Ic} at room temperature

These results are related to the fact that beyond 1250°C, the matrix becomes viscoplastic. The increase of K_{Ic} is due to the softening of the second phases of the materials. On the fracture surface, subcritical crack growth prior to fast fracture is observed. In this stable propagation part, the fracture is intergranular. It can be shown that the importance of this zone depends on the loading rate. Higher is the loading rate, smaller is the extension of the stable crack.

The difference between unreinforced and reinforced materials indicates that the whiskers hinder the grain boundary sliding and the crack growth. Unfortunately, the fracture surfaces are oxidized which does not allow to confirm the contribution of the whiskers.

3. CONCLUSION

A complete study of SiAlYON matrix composites reinforced with different volume fractions of SiC whiskers have been achieved. Two fracture toughness testing methods have been compared : the Single Edge Notched Beam precracked in cyclic compression and the Chevron Notched bend bar. At room temperature no enhancement of toughness is obtained with the reinforcement, certainly due to a strong whisker / matrix interface. At temperatures over 1250°C the addition of 10 to 30% of SiC whiskers to the SiAlYON matrix leads to a significant improvement of the fracture toughness of the materials. This effect is attributed to the fact that, at this temperature, the transition temperature of the second phase is reached, the matrix becomes viscoplastic and the whiskers hinder the deformation of the material.

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