Mechanical Behavior of Non-brittle Ceramics C-SiC and SiC-SiC Composites

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ABSTRACT

2D C-SiC and 2D SiC-SiC composites reinforced with long fibers and within which the matrix is obtained by chemical vapor infiltration of Silicon Carbide (SiC) exhibit very high mechanical properties. Particularly, these composites are much less sensitive to thermal and mechanical shocks as well as thermomechanical fatigue than monolithic ceramics.

As regards their mechanical shock behavior, various characterization methods have been considered :

- at first, the methods commonly used for the characterization of the other materials such as refractory alloys and monolithic ceramics intended for thermomechanical applications.
These methods have been useful to compare these materials and to rank them.

- However, these methods are inaccurate to provide a meaningful toughness value for designing a component in terms of crack growth. Therefore more complex methods have been approached ; they are more suitable for composites which exhibit anisotropic characteristics as well as a non linear behavior due to multiple cracking.

KEYWORDS

Ceramic matrix composites (CMC); characterization; behavior under mechanical shock; toughness; rupture energy.

INTRODUCTION

The main difficulty with ceramic products prepared by the powder sintering process is their sensitivity to thermal and mechanical shocks. This brittleness is one of the main factors affecting their reliability and consequently their use in many fields.

The most frequently used method for mitigating this major disadvantage consists of adding fibrous reinforcement into the ceramics. The ceramic matrix composites thus obtained (CMC) belong to two main families:

- sintered ceramics reinforced with whiskers
- composites reinforced with long fibers within which the matrices are obtained by impregnation procedures using pre-ceramic precursors and/or by chemical vapor infiltration (CVI).

Among the latter, C–SiC and SiC–SiC composites are reinforced with carbon and silicon carbide fibers respectively, within which the matrix is obtained by chemical vapor infiltration. These composites currently have very high properties.
The purpose of this paper is to present results of mechanical shock and crack propagation resistance tests, obtained on these composites

MANUFACTURE

In this paper, we consider only 2D C–SiC composites, reinforced with TORAY–T300 high strength fiber–based fabrics and 2D SiC–SiC reinforced with NICALON–NL200 fiber–based fabrics made by NIPPON CARBON.

The composites with the properties indicated below were obtained by stacking dry fabric plies as indicated in the sketch on Figure 1. The warp and fill yarns are parallel to the sides of the plies, which are laid onto each other by rotating each ply 90° relative to the adjacent ply.

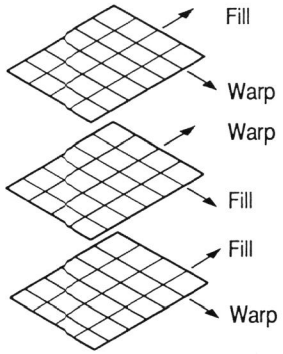

Figure 1. Laying up fabric plies for composites

The percentage by volume of fibers in the composites are as follows:

- 2D C–SiC : 45%
- 2D SiC–SiC : 40%

MAIN PROPERTIES

The reference axes adopted for property definitions are indicated on Figure 2.

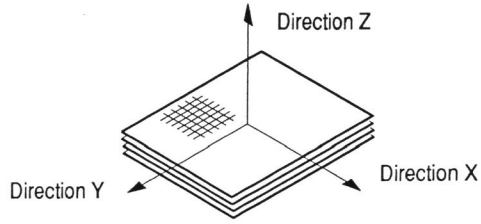

Figure 2 – Reference axes adopted for property definitions

The main properties of 2D C–SiC and 2D SiC–SiC composites are shown in Tables 1 and 2 respectively.

		23°C	1000°C	1400°C
SPECIFIC GRAVITY	$g.cm^{-3}$	2.1		
POROSITY	%	10		
TENSILE STRENGTH	MPa	350	350	330
ELONGATION (TENSILE)	%	0.9	0.9	
YOUNG'S MODULUS (TENSILE)	GPa	90	100	100
FLEXURAL STRENGTH	MPa	500	700	700
COMPRESSIVE STRENGTH (X–Y)	MPa	580	600	700
(Z)	MPa	420	450	500
SHEAR STRENGTH (XZ–YZ)	MPa	35	35	35
THERMAL DIFFUSIVITY (X–Y)	$10^{-6}, m^2sec^{-1}$	11	7	8
(Z)	$10^{-6}, m^2sec^{-1}$	5	2	2
EXPANSION COEFFICIENT (X–Y)	$10^{-6}, K^{-1}$	3		
(Z)	$10^{-6}, K^{-1}$	5		
SPECIFIC HEAT	$J.kg^{-1}$	620	1400	
TOTAL EMISSIVITY		0.75 – 0.80		

Table 1 – Main properties of 2D C–SiC

		23°C	1000°C	1400°C
SPECIFIC GRAVITY	$g.cm^{-3}$	2.5		
POROSITY	%	10		
TENSILE STRENGTH	MPa	200	200	150
ELONGATION (TENSILE)	%	0.3	0.4	0.5
YOUNG'S MODULUS (TENSILE)	GPa	230	200	170
FLEXURAL STRENGTH	MPa	300	400	280
COMPRESSIVE STRENGTH (X–Y)	MPa	580	480	300
(Z)	MPa	420	380	250
SHEAR STRENGTH (XZ–YZ)	MPa	40	35	25
THERMAL DIFFUSIVITY (X–Y)	$10^{-6}, m^2sec^{-1}$	12	5	5
(Z)	$10^{-6}, m^2sec^{-1}$	6	2	2
EXPANSION COEFFICIENT (X–Y)	$10^{-6}, K^{-1}$	3		
(Z)	$10^{-6}, K^{-1}$	2.5		
SPECIFIC HEAT	$J.kg^{-1}$	620	1200	
TOTAL EMISSIVITY		0.75 – 0.80		

Table 2 – Main properties of 2D SiC–SiC

BEHAVIOR UNDER MECHANICAL SHOCKS

In order to compare CMCs with other materials used in high temperature structural applications, we initially defined their properties using the same methods adopted for these materials.

Impact resistance by Izod testing

The resistance to breakage was measured by flexural shock according to ASTM D256-78 on notched specimens with dimensions and geometry as shown in Figure 3. The notch produces a stress concentration which results in a brittle fracture. The results are shown in terms of energy absorbed per unit of area broken.

The following ceramics where tested:

- CRYSTAR SiC (NORTON)
- KT SiC (CARBORUNDUM)
- REFEL SiC (B.N.F.L.)

Figure 3 shows the results for REFEL SiC only, which gave the highest of the three results.

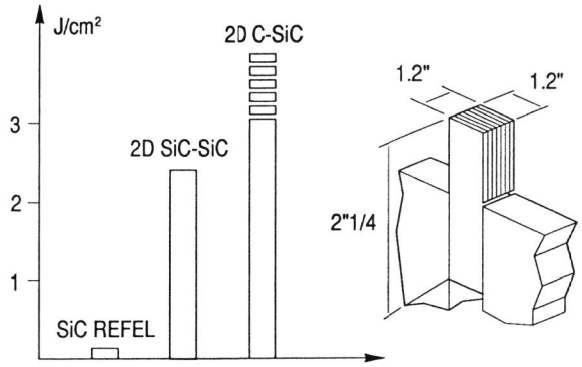

Figure 3 - Impact resistance by IZOD test

It can be seen that the values found for 2D SiC-SiC are about 17 times higher than those for the sintered ceramic. Those for 2D C-SiC where the highest. They are about 22 times higher than those for REFEL SiC. The CHARPY pendulum impact testing machine that we were using was limited to an energy of 4 J, and the test pieces did not break during the test carried out, even when repeated three times at maximum energy.

Impact behavior by instrumented Charpy testing

A special hammer in which a small piezo-electric pellet can be installed was fitted on the pendulum used during the tests described in the previous section. During the shock, the pellet outputs an electric charge proportional to the impact force. This charge is transformed to a voltage by means of an amplifier, and the voltage is stored by a transient recorder. Digital processing allows calculation of the following data items:

- the maximum force during the shock (Fmax)
- the elastic energy consumed before reaching the maximum force (E_e)
- the propagation energy consumed between the moment of the maximum force and complete rupture (E_p)
- the total energy ($E_t = E_e + E_p$), which can be compared to the energy reading on the dial (E_r)
- the various corresponding times (t_e, t_p and t_t).

This data, together with a curve of force against time, are output on a plotter.

Table 3 shows the mean results obtained for composites, and on sintered SiC (ROSENTHAL).

Figure 4 shows the dimensions of the test pieces used and the direction of the force.

Figure 5 is an example of a curve for a 2D SiC-SiC test piece.

	E_r (KJ.m^{-2})	E_t (KJ.m^{-2})	Fmax (N)
Sintered SiC	1.1	1.1	901
2D SiC-SiC	15.6	12.2	2215
2D C-SiC	56.7	51.7	1893

Table 3 - Test results

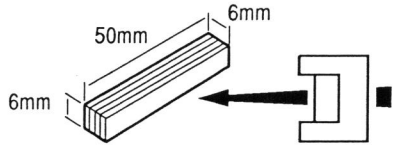

Figure 4 - Test specimen

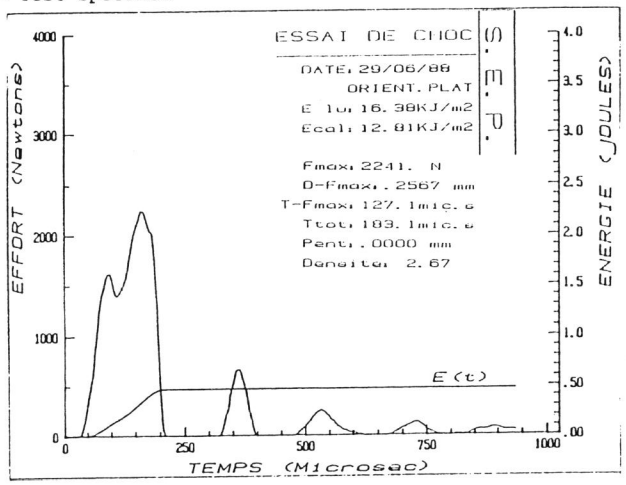

Figure 5 - Example of recording for 2D SiC-SiC specimens

In this type of test, fairly good agreement is observed between the measured and calculated energy values. The values found in the case of composites – and especially 2D C–SiC composites – are much higher than those for sintered SiC.

The curves for composites show a non–linear rise, typical of a non–brittle rupture, contrary to that of sintered SiC. The rupture surfaces of composite specimens are very irregular after the test.

RESISTANCE TO CRACK PROPAGATION

Sensitivity to notches and cracks is a material property which is helpful in understanding and making improvements in the mechanical performance, and may also be important in design. Conventional crack propagation resistance tests can be used to give the following two magnitudes: the critical stress intensity factor K_{IC} and the crack production energy per unit area G_{IC}; these two intrinsic properties are interdependent in conventional linear elastic fracture mechanics concepts.

Toughness:

.The concept of toughness involving linear elastic rupture mechanisms is only applicable to isotropic materials, and is therefore not strictly applicable to a composite. However, it does provide elements for comparison with available data for other materials.
.Measurements were taken on SENB type specimens up to 1400°C in a neutral atmosphere using the ASTM E-339 specification. Allowing for the high non–linearity of the stress-strain curves associated with propagation of the main crack, the selected parameter is defined as K_{1R}. It is expressed in $MPa\sqrt{m}$ and is calculated using the following formula:

$$K_{1R} = Sigma_R . Y(a_o) . \sqrt{a_o}$$

where:

$Sigma_R$ = the nominal stress equivalent to the maximum experimental load
a_o = the notch size
Y = a constant which depends on the specimen size and is calculated using the theoretical expansion proposed by B.GROSS and J.E. STAWLEY.

.The values of K_{IR} are only comparative values of the K_{1C} toughness values indicated in the case of monolithic ceramics, and should be used by default.

.These tests have the advantage of being simple and that they can be implemented at high temperatures on small specimens.

Figure 6 indicates the values of K_{1R} found for composites, and the envelope of the K_{1C} curves for monolithic ceramics. (Data from CARBORUNDUM, ROSENTHAL and NORTON, on SiC and $SI_3 N_4$).

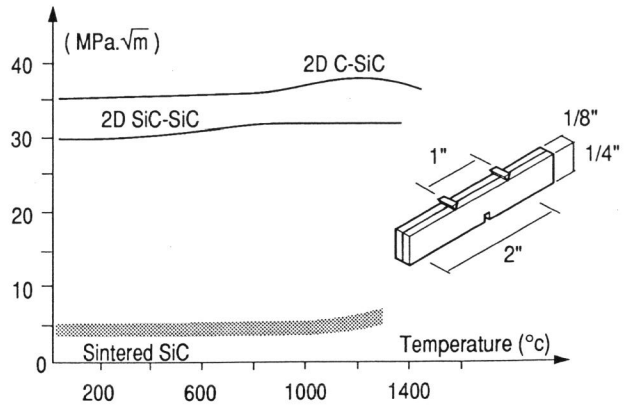

Figure 6 – Fracture toughness versus temperature

It can be noted that the values on composites are much higher than those on monolithic ceramics. These values are also maintained up to at least 1400°C (maximum temperature in the tests performed). They are also very much higher than those for other CMCs reinforced with whiskers and obtained by sintering processes. The literature indicates that values for the latter are only between 6 and 10 MPa $\sqrt{m}$.

R curves:

There are two main reasons for the inapplicability of the proposed tests for measuring the fracture toughness parameter:

- firstly, the substantial sub–critical crack growth which occurs prior to the maximum load
- secondly, the fracture of composites is associated with a large fracture process zone around the crack tip.

A more suitable property for composite materials is the crack propagation energy, and especially the variation of resistance to propagation depending on the development of the crack.

These plots are called R–curves or Resistance curves, and in the case of linear elastic materials can be calculated from the corresponding strain energy release rate G. Using the compliance unloading to evaluate the crack extension ($a_{i+1}=a_i+\Delta a$), the energy G at each unloading can be expressed as:

$$G_i = \frac{P_i^2}{2} \frac{dC(a_i)}{dA}$$

where dC/dA is the rate of change of compliance with respect to crack growth and P_i is the load of the i^{th} loading.

The R curves for C–SiC and SiC–SiC composites are obtained using this method on CT specimens, and the results are shown in Figure 7.

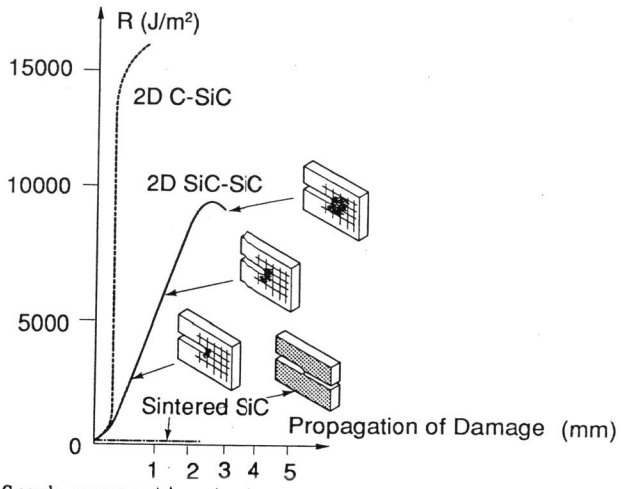

Figure 7 – Crack propagation test

These tests demonstrate the existence of three distinct areas associated with different states of material damage at the bottom of the crack and the propagation of the macrocrack.

- A small vertical area limited by the initial damage generation threshold.
- A second area in which there is a rapid increase in energy; this area represents the start of damage.
- A third area associated with a reduced increase in resistance R, and combined with a reduction of load. At this stage, a macrocrack is initiated which propagates within the specimen, and during its propagation it creates a constant dimension micro-cracked process area.

For the orientation of the layers in the specimen considered within this document, scanning electron microscopy has shown that the main micro-mechanical mechanisms contributing to these high values of R were the rupture in the matrix parallel to the longitudinal fibers, the loss of fiber-matrix bond and fiber rupture.

The objective of this method is to define a single crack propagation criteria for a given material. It has been established that this procedure cannot be very accurate for CMCs since it ignores the effects of observed non-linear deformation. A formalization was proposed by M. WECHARATANA and S.P. SHAH : it takes account of the sometimes large residual strains observed after cycling on the various test specimen configurations.

However, the major importance of inelastic mechanisms makes it necessary at the present time to perfect suitable test methods and experimental operating procedures, always adding a global energy approach to cracking in the materials. The methods proposed by S.I. GARWOOD and M.SAKAI use this reasoning. The definition of test specimens and the mechanism for the transposition of high temperature tests form a major theme in learning about CMCs. For this reason, SEP has asked several university laboratories to think about this matter.

CONCLUSION

The various methods used to compare C-SiC and SiC-SiC toughness to that of monolithic ceramics are all leading to a same conclusion :

long fiber reinforced composites constitute a new family of materials exhibiting a toughness fairly superior to that of any other ceramic product available today. 2D C-SiC toughness also is superior to that of 2D SiC-SiC.

These results have been evidenced by the mechanisms successively encountered throughout the progressive rupture of those composites which all absorb an important energy :

- pre-cracking in overstressed zones.
- partial debonding between fiber and matrix followed by a load transfer to the fiber.
- fiber breakage followed by a fiber pull out from the surrounding matrix.

A new testing method more adapted to composites has to be developed ; it would provide more meaningful toughness values for designing as well as life prediction under stress for the resulting parts.

REFERENCES

- L. HERAUD, P. SPRIET
"High toughness C-SiC and SiC-SiC composites in heat engines". Int. Conf. on whiskers and fiber toughened ceramics OAK RIDGE (USA) - June 1988

- L. HERAUD
"New horizons through carbon-carbon and ceramic-ceramic materials" - Ceramics' 88 - NAGOYA (JAPAN) - March 1988

- T. MACKE, J.M. QUENISSET
"Impact behavior of ceramic matrix composites ". ICCM VI - ECCM II - LONDON (U.K) - July 1987

- A. MELCHIOR, M.F. POULIQUEN, E. SOLER
"Thermostructural composite materials for liquid propellant rocket engines" - 23rd Joint Propulsion Conf. - SAN DIEGO (USA) - July 1987

- G. FANTOZZI
"Comportement mécanique des céramiques composites à fibres et dispersoïdes" Matériaux Mécanique Electricité N° 421, Juin-Juillet 1987

- B. GROSS, J.E STANLEY - Tech. Note NASA - TN - D - 2603 (janv. 1965)

- M. WECHARATANA and S.P SHAH "Slow crack growth in cement composite" proceedings of ASCE, J. of Structural Division

- S.J. GARWOOD, J.N. ROBINSON, C.E. TURNER
"the measurement of crack growth resistance curves (R-curves) using J.integral" Int. J. Fract., 1975, V-11, P. 528.530

- M. SAKAI, K. URASHIMA, M. INAGAKI
"Energy principle of elastic plastic fracture and its application to the fracture mechanics of polycrystalline graphite"
J.Am. Cram. Soc., 1983,66,868