CONGRES DE METALLURGIE DES POUDRES

(Société Française de Métallurgie et de Matériaux -SF2M)

6 au 9 juin 1994

Centre de Congrès de Paris - CNIT La Défense

Titre

Metallic-fibre-reinforced ceramic-matrix composite

F.Prevost, G.Schnedecker, M.Boncoeur

CEA.Saclay - DTM/CEREM/CE2M/SERC, 91191 Gif/S/Yvette Cédex (France)

Résumé :

Une grille en fil de métal réfractaire tissé est incorporée dans une céramique oxyde grâce à la méthode de projection plasma, pour réaliser des plaques de composites. Au cours des tests mécaniques, le composite suit un mode de fracture pseudo-ductile, alors qu'à l'origine la céramique a un comportement fragile.
On obtient une contrainte à la rupture plus élevée, et le composite reste d'un seul tenant même lorsque la déformation est importante.
De plus, le procédé ne nécessite aucun traitement thermique supplémentaire.

Abstract: A refractory metal wire cloth is embedded in an oxide ceramic matrix, using a plasma spraying technology, in order to make composite plates. When mechanically tested, the composite fails with a pseudo-ductile fracture mode whereas the ceramic alone is originally brittle. It exhibits a higher fracture strength, and remains in the form of a single piece even when straining is important. No further heat treatment is needed after the original processing to reach these characteristics.

1. CONCEPTION OF THE COMPOSITE

1.1. Oxide ceramics

The optimisation of the mechanical properties of oxide ceramics is of prime interest. Indeed, these materials have interesting basic properties: no microstructural modifications up to temperatures higher than 1000 °C (1200 °C for the ceramic used for this study, Yttrium Oxide Y_2O_3), reduced chemical reactivity, oxidation resistance, hardness, and good resistance to creep. However, their rather low ultimate strength (about 60 MPa in three-point bending for Y_2O_3 produced by plasma spraying), and their tendency to fail by brittle fracture restrict their use as structural materials. Hence the idea of associating oxide ceramics with a tougher constituant to try and modify these characteristics, and enlarge the possibilities of employing these materials[1].

1.2. Metallic structure

The idea of reinforcing such a ceramic by metallic fibres led to a composite which would benefit from the properties of both materials and, principally, would follow a pseudo-ductile fracture mode. Preliminary experiments and results from other studies induced us to choose a high ductility refractory metal with a thermal expansion coefficient matching that of the ceramic matrix (9.10^{-6} K^{-1} for Y_2O_3), so as to ensure compatibility. Moreover, a bi-directional reinforcement seemed more reliable. Thus, we used a Ta-10%W woven wire cloth, with wires 0.80 mm in diameter and opening 2.35 mm in width. Its thermal expansion coefficient is about 7.10^{-6} K^{-1}, slightly lower than that of Y_2O_3: tensile stresses will develop in the metal rather than in the ceramic, which is better, to avoid weakening the latter. The characteristics of the grid were chosen for the following reasons: the wires must be sufficiently large in diameter in order to intercept cracks developping in the matrix, and the opening not to small, so that the cloth should not act as a separation layer between the upper and lower parts of the plate.

1.3. Processing of the ceramic-metal composite

In order to obtain an intimate embedding of the fibres in the Y_2O_3 matrix, plasma spraying of ceramic powder onto the grid was chosen to process the composite. The ceramic matrix is, then, regularly generated through several passings, with a high density (9.5% of closed porosity and no open porosity), and the thicknesses available for the plates range from a few tenths of millimeter to several millimeters. The coating adhesion of the ceramic to the fibres can be controlled by modifying the parameters of the plasma gas: then, the fusion of the powder particles and their speed when reaching the cloth are increased or decreased, and so the internal structure of the deposition changes, as well as the structure of the interface. Thus, plates 2.5 to 3.0 mm thick were processed; this corresponds to fibre contents ranging from 13 to 11%. The grid is at the same distance from the upper and the lower surfaces of the plate.

2. CARACTERISATION AND INTERPRETATION

2.1. Tensile test results

Tensile tests were carried out on the composites perpendicular to the plane of the grid on square specimens 10 mm wide stuck on holders. These tests aimed at characterising the matrix/fibre interface. Strong bonds between both would induce a rupture of the composite within the ceramic matrix; weaker bonds would result in the decohesion of the composite at the metal/ceramic interface with a fracture strength more or less important according to the quality of the adhesion. Numerical results from these tests were compared and the confrontation with further results proved that a relatively weaker interface is better to improve the mechanical properties of the composite and for the fibres to act as toughening agents.

2.2. Bending test results

Samples 12 mm wide and 3 mm thick were tested in three-point bend using a 50 mm span and a cross-speed of 0.5 $mm.mn^{-1}$, at room temperature, in order to characterise the mechanical behaviour of the composite. Charge was recorded related to displacement, and examinations of the material were made during the tests; the samples were also observed in light microscopy and in scanning electron microscopy. The composites with a strong interface revealed no improvement compared to non-reinforced ceramic samples: the cracks run through the wire cloth and the composite fails by brittle fracture.
For the others, major improvements were obtained:

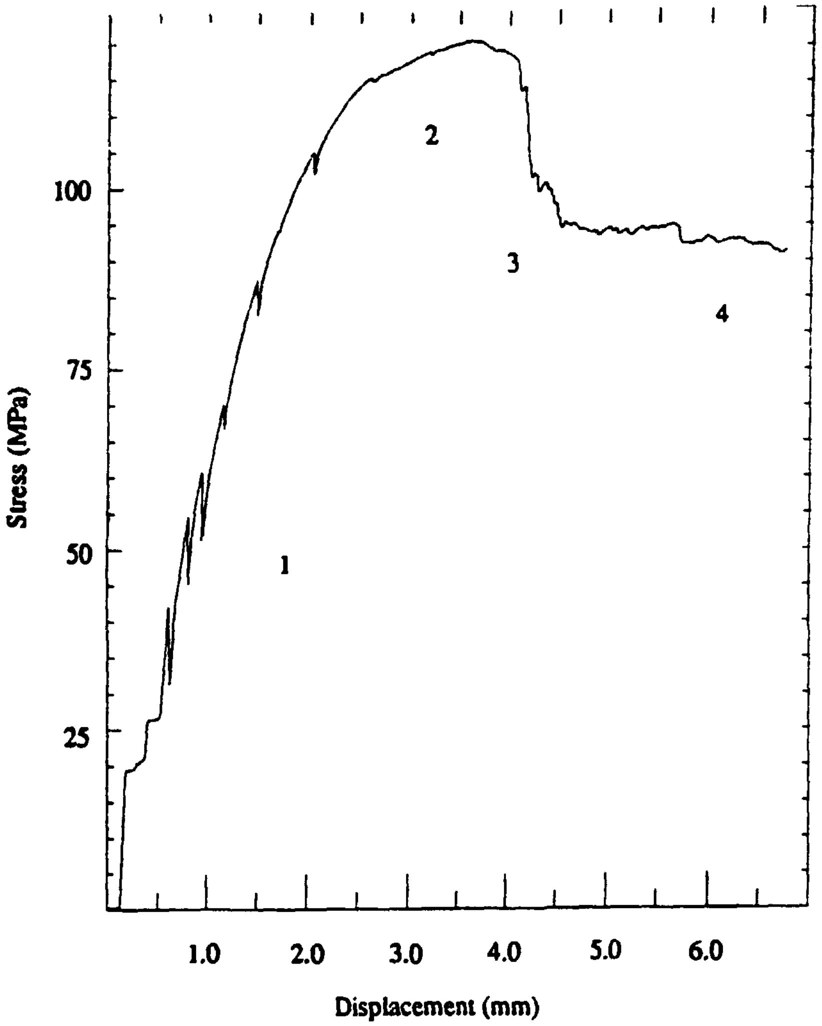
a) a higher fracture strength: an average of 120 MPa, against 60 MPa for Y_2O_3 alone;

b) a progressive propagation of the cracks in the composite, first perpendicular to the surface, then with a deflection in the plane of the grid;

c) a fracture mode which is no longer brittle but pseudo-ductile *(see stress-displacement graph below)*. This fracture mode is not the superposition of that of the ceramic and that of the metal, but is really new and specific to the composite. This fracture mode is said to be pseudo-ductile, and not ductile, on the one hand because in the first stage of the rupture (corresponding to the quickly increasing part of the graph), irreversible cracks occur in the matrix; and on the other hand, in the last but one stage, there is a fall by 10% in stress, corresponding to cracks eventually passing through the wire cloth.

d) cohesion of the composite still ensured by the wire cloth even after fracture: even cracked, the ceramic matrix still grips the wires, and the plate is still a whole piece.

It must be noted that all these results are unchanged after a heat-treatment at 1000°C during 100 hrs. This treatment is meant to relax the residual stresses due to processing; this tends to prove that these stresses are insignificant compared to the ones resulting from external load and related to crack propagation. The as-processed material does not need any heat-treatment, unlike other metal/ceramic assemblies.



1. Initiation and propagation of cracks within the lower part of the ceramic matrix.

2. Deflection of the cracks along the grid and fibre pullout.

3. Transmission of the cracks to the upper part of the composite plate.

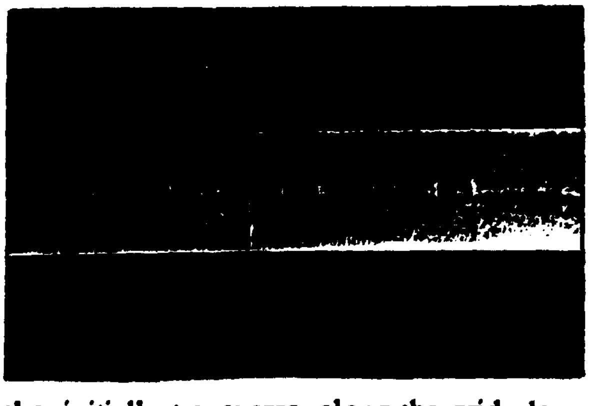
4. Resistance of the cracked composite still in a whole piece.

Pseudo-ductile fracture mode underlined on this stress/displacement graph recorded during a three-point bend test.

2.3. Modelisation of the mechanical behaviour during fracture (3-point bend initiated)

The following statements are based on observations of the samples during and after the tests, on theoretical data established previously[2], and on the results given above.

First, the crack initiates in the matrix, due to external stress, and propagates (perpendicular to the cloth) up to the metallic phase. There, it is momentarily halted, because the relatively weakly bonded fiber acts as a hole in the matrix and diffuses the stresses at the crack tip, making them lower than the fracture strength. It is a first stage of fracture energy dissipation. Then, when the interface is relatively weak, this crack is deflected away from its initial direction, thus prevented from passing through the cloth (that occurs with a strong interface and leads to brittle fracture). So, cracks follow the plane of the grid for two or three meshes *(see photo below)*.



Deflection of cracks, initially transverse, along the grid plane on a section of composite plate which was partially put through a three-point bend test.

This deflection is due to interfacial shear along the wires and constitutes the second stage of energy dissipation.

Then, as the stress is partially transferred to the fibres, a sligth fibre/matrix debonding enables fibre pullout, third stage of energy dissipation, by frictional resistance. Only one part of the matrix, under the grid, is fractured, whereas the upper layer is still intact.

Thanks to these phenomena, the fracture energy rises and the fracture strength is significantly increased. Further more, the ductile fibres bridge the fracture planes, as they do not break, enhancing the toughness and preventing the composites from falling into pieces. This is true even if the cracks go on propagating again perpendicular to the cloth after being deflected along it, when further straining is applied, because the weak interface is resistant enough to ensure a permanent cohesion.

Moreover, the reinforcing structure is bi-directional and woven: the fibres can support elongation and slide within the cloth, but stress transmission and friction occur where the wires intersect. This is another source of energy dissipation, and an additional element of cohesion; and no direction is weaker than another.

It is thanks to four main structural designs: a ductile metallic layer, its wire-shape geometry, a weak interface with the ceramic matrix, and a woven structure, that the composite undergoes a fracture mode which is no longer brittle but pseudo-ductile, and exhibits a good cohesion.

References

Research reports: [1] J-D.Lulewicz, G.Schnedecker, M.Boncoeur: Exemple de composite à matrice oxyde et renfort métallique: Y_2O_3/Nb (Note technique CEA/DTA/CEREM, NT 92/010, 20/08/92).

Books: [2] K.K.Chawla, Composite materials (Springer-Verlag, 1987), chap. 4, 7, 12.