Bull. Mater. Sci., Vol. 25, No. 6, November 2002, pp. 493–495. © Indian Academy of Sciences.

A novel technique for fabrication of near-net-shape CMCs

A DEY, M CHATTERJEE*, M K NASKAR, S DALUI[†] and K BASU[‡]

Sol-Gel Division, [†]Composite Division, Central Glass and Ceramic Research Institute, Kolkata 700 032, India

[‡]Regional Research Laboratory, Bhopal 462 026, India

Abstract. A sol–gel vacuum infiltration technique has been developed for the fabrication of near-net-shape ceramic matrix composites (CMCs) using discontinuous mullite fibre preform with 15 vol.% of fibre content and $ZrO_2 \cdot 10$ wt.% Y_2O_3 sol as the infiltrant. Effect of sol viscosity, number of infiltration and calcination temperature on physico-mechanical properties of fabricated CMCs were examined. Characterization of the fibre preform, matrix material (in the form of ceramic specimen without fibre) and the developed CMCs were performed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD indicated the presence of cubic (*c*) and tetragonal (*t*) zirconia in both the CMCs and the ceramic specimens calcined even at 1400°C. Flexural strength of the CMCs and the ceramic specimens (calcined at 1400°C), determined by the three-point bending test, was found to be about 14 mPa and 1·40 mPa, respectively. SEM indicated multiple fracture of the matrix which gave rise to pseudo elasticity. This is also evident from the load-displacement curve of the three-point bend test. SEM studies also indicated fibre pull-out in the fracture surface of the CMCs.

Keywords. Sol–gel; vacuum infiltration; fibre reinforced ceramic matrix composites.

1. Introduction

Ceramic fibre reinforced ceramic matrix composites (CMCs) have attracted attention now-a-days and may be considered as promising alternative to monolithic ceramics (brittle) for obtaining structural materials suitable for engineering applications (Gu *et al* 2000). As the sol–gel method of producing CMCs has specific advantages (Russel-Floyd *et al* 1993), an attempt has been made, in the present investigation, for the development of a technique for the fabrication of near-net-shape CMCs using discontinuous mullite fibre preforms and zirconia–yttria ($ZrO_2 \cdot 10$ wt.%Y_2O_3) sol (Chakrabarty *et al* 2001).

2. Experimental

The as-received discontinuous mullite fibre preforms with 15 vol.% fibre content and dimensions of ~ 100 mm (diameter) × 10 mm (thick) [manufactured by the Orient Cerwool Limited, Lakhtar (Gujarat)] were cut into small pieces of approximate dimensions 40 × 7 × 6 mm and activated at 500°C for 1 h in air. The parent zirconium acetate sol of composition $ZrO_2 \cdot 10$ wt.% Y_2O_3 and viscosity 2·8 ± 1 mPa s was prepared by the precipitation–peptization technique (Chakrabarty *et al* 2001). From this sol, other sols of viscosities 40–60 mPa s were prepared by evaporation and were used as infiltrants. The vacuum infiltration technique (Russel-Floyd *et al* 1993) was followed for the fabrication of CMCs. Table 1 summarizes the experimental conditions used for the fabrication of CMCs. Further, rectangular bars (ceramic specimen without fibre) of approximate dimensions 41 × 9 × 8 mm were fabricated under an isostatic pressure of 1·5 ton using oxide powder of – 300 mesh (calcined at 1000°C), obtained from the $ZrO_2 \cdot 10$ wt.% Y_2O_3 sol as prepared above, and calcined at 1400°C for 1 h. The fabricated materials were characterized by XRD (Model: Philips PW 1730), SEM (Model: Leo 400c) and three-point bending strength measurement using Instron Universal Testing machine (Model: 5500 R). Each strength datum is an average over 6 samples.

3. Results and discussion

Table 1 presents the characteristics of the CMCs obtained in the present investigation. In the present process, multiple infiltration cycles followed by intermediate heat-treatment at 800°C was adopted to minimize matrix cracking caused due to large shrinkage during drying of the infiltrated materials. The final calcination temperature of 1400°C for 1 h, in the range 1000–1400°C, was found to be the optimum. Comparing the results of run nos. 1, 2 and 3 of table 1, it is clear that with the increase in the no. of infiltrations, keeping other parameters unaltered, although the overall strength of the infiltrated material increases (run nos. 2 and 3), they fail to exhibit the reinforcement characteristics (figure 1a). This may be due to the development of strong interaction between the fibre and the excess matrix material for excess loading. On the other hand, the pseudo-elastic behaviour of the

*Author for correspondence

product of run no. 1 is evident from figure 1b. Figures 2 and 3 represent the SEM of the as-received (uninfiltrated) fibre preform and the fracture surface of the product of run no. 1 of table 1, respectively. Figure 3 clearly indicates fibre pull-out in the CMC. The reinforced fibres acted as crack arresters which prevented the propagation of cracks.

XRD of the CMCs fabricated at 1400°C confirmed the presence of both *c*- and *t*-ZrO_2 along with the mullite phase, without any formation of monoclinic ZrO_2 polymorphs. Obviously, the Y_2O_3 additive in the matrix materials helped to retain *t*-ZrO_2 at 1400°C by inhibiting grain growth (Chakrabarty *et al* 2001). Under identical conditions of heat-treatment, the ceramic specimens, without any fibre-reinforcing materials, exhibited a flexural strength of about 1·40 mPa (Load at break: 24·12 N). This low value of the flexural strength (figure 1c) supports the necessity of mullite fibre reinforcing materials in the specimens, inspite of the retainment of *c*-/*t*-ZrO_2 in the matrix (identified by XRD).

Table 1. Characteristics of CMCs obtained under different experimental conditions.

Run no.	Sol viscosity (mPa s)	Final calcination temperature (°C)	Load at break (N)	Flexural strength (mPa)	Remarks
1.	$60 \pm 1(1)^{\dagger}$, $40 \pm 1(2)^{\dagger}$	1400	28·96	5·21	Good surface (CMC character)
2.	$60 \pm 1(5)^{\dagger}$	1400	265·91	11·49	Good surface (ceramic character)
3.	$60 \pm 1(1)^{\dagger}$, $40 \pm 1(4)^{\dagger}$	1400	301·76	13·95	Good surface (ceramic character)

†Number in the parenthesis indicates the no. of infiltration.

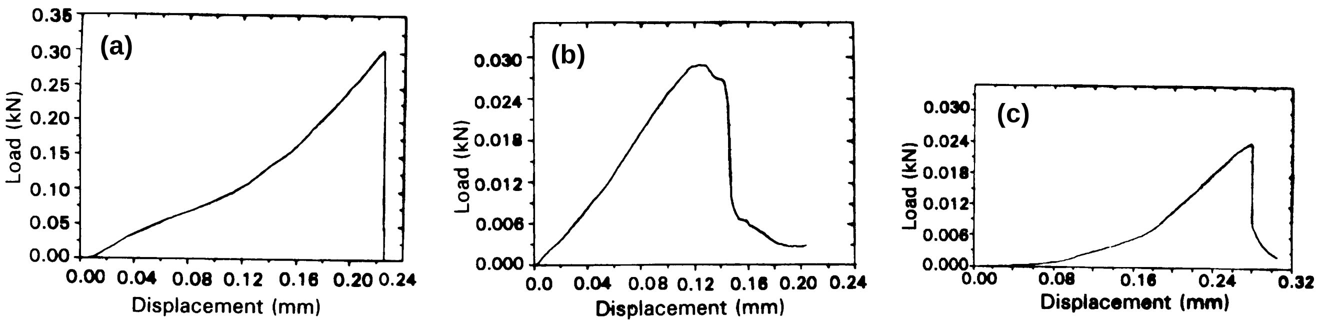

Figure 1. Load displacement curves of (**a**) the product of run no. 3 of table 1, (**b**) the product of run no. 1 of table 1 and (**c**) the ceramic specimen (without any fibre reinforcing material).

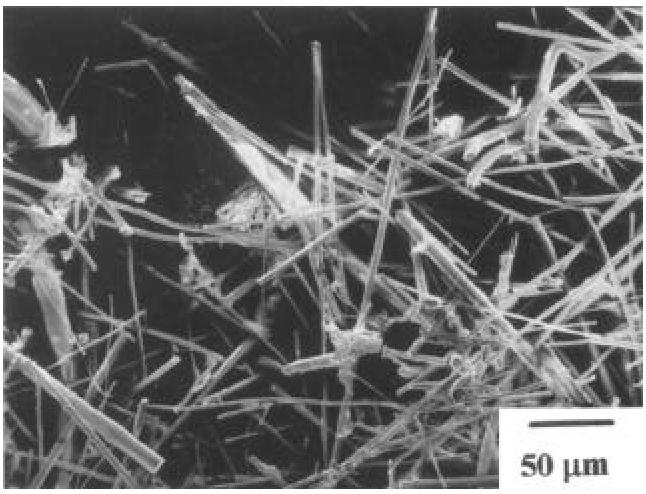

Figure 2. SEM of the as-received (uninfiltrated) mullite fibre preform.

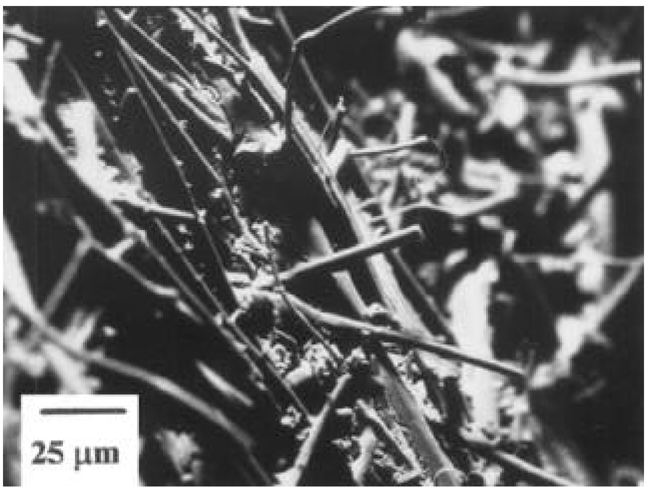

Figure 3. SEM of the fracture surface of the product of run no. 1 of table 1 showing fibre pull-out.

4. Conclusions

A sol–gel vacuum infiltration technique has been developed for the fabrication of near-net-shape CMCs with pseudo-elastic character using discontinuous mullite fibre preform with 15 vol.% of fibre content and $ZrO_2 \cdot 10$ wt.% Y_2O_3 sol as the infiltrant.

Acknowledgements

The authors thank Dr K K Phani, Composite Division, CGCRI, Kolkata, for providing valuable suggestions throughout this work and colleagues of the X-ray and SEM Sections for material characterization. Partial financial assistance from the Aeronautics Research and Development Board (AR&DB), Ministry of Defence, Govt. of India, is also thankfully acknowledged.

References

Chakrabarty P K, Chatterjee M, Naskar M K, Siladitya B and Ganguli D 2001 *J. Eur. Ceram. Soc.* **21** 35

Gu X, Trusty P A, Butler E G and Ponton C B 2000 *J. Eur. Ceram. Soc.* **20** 675

Russell-Floyd R S, Cooke R G, Laurie J and Hammett F W 1993 *J. Am. Ceram. Soc.* **76** 2635