Available online at www.sciencedirect.com
ScienceDirect
Journal of the European Ceramic Society 28 (2008) 787–792
www.elsevier.com/locate/jeurceramsoc

The preparation and mechanical properties of the unidirectional carbon fiber reinforced zirconia composite

G.H. Zhou [a,b], S.W. Wang [a,*], J.K. Guo [a], Z. Zhang [a]

[a] *Shanghai Institute of Ceramics, Chinese Academy of Sciences, 1295 Dingxi Road, Shanghai 200050, PR China*
[b] *Graduate School of Chinese Academy of Sciences, Beijing 100049, PR China*

Received 14 March 2007; received in revised form 25 June 2007; accepted 6 July 2007
Available online 14 September 2007

Abstract

Unidirectional carbon fiber reinforced calcium stabilized zirconia composites (uni-C_f/ZrO_2) were prepared by slurry infiltration and hot-pressing method. The room temperature mechanical properties were investigated and the fracture features of composites were observed. A flexural strength of 588.0 MPa and fracture toughness of 15.4 MPa·$m^{1/2}$ parallel to the fiber direction for the composite hot-pressed at 1500 °C was attributed to the fiber pull-out. With increasing hot-pressing temperature from 1500 °C to 1650 °C, the relative density was augmented, but the mechanical properties of composites degraded gradually. Especially at 1650 °C, the flexural strength and fracture toughness decreased significantly to 173.2 MPa and 5.0 MPa·$m^{1/2}$, respectively. Thermodynamic calculation and XRD, TEM investigations showed that carbon fibers reacted with ZrO_2 to form ZrC phase at 1650 °C, and then formed chemical bonding and led to a strong interface between fiber and matrix, which resulted in the decrease of mechanical properties of the composite hot-pressed at higher temperatures. Moreover, the mechanical properties of carbon fibers degraded by the above reaction had also an adverse effect on the mechanical properties of the composite.

Keywords: Hot-pressing; Composites; Mechanical properties; ZrO_2; Carbon fiber

1. Introduction

Zirconia-based composites have demonstrated a wide range of attributes, including high melting point ($T_m \approx 2700$ °C) and stability, high strength and toughness, good heat resistance, unique wear resistance, interesting electronic properties such as fast ionic conductivity for structural and functional applications.[1,2] As well known, the problem of the low fracture toughness of ceramics can be overcome by designing and preparing composite materials reinforced with fibers, whiskers and particles. Up to now, surprisingly there are a few researches on ZrO_2–matrix composites reinforced with continuous fibers. Pujari and Jawed[3] reported chopped alumina fiber–TZP matrix composites prepared by a conventional powder metallurgy route. They found that the alumina fibers did result in a twofold increase in toughness with respect to the monolithic TZP but the failure mode remained brittle. In the meantime, Bender et al.[4] succeeded in preparing ZrO_2–SiO_2 and ZrO_2–TiO_2 matrix composites reinforced with uncoated or BN coated SiC fibers according to a liquid route (organometallic precursors). Their composites exhibited brittle failure when reinforced with uncoated SiC fibers due to a strong fiber–matrix bonding whereas they did behave in a non-brittle manner with BN-coated SiC fibers, the BN layer allowing the fiber pull-out to occur and preventing the fibers reacting with the matrix. Subsequently, Minet et al.[5] prepared ZrO_2-based composites by CVI densification from performs made of alumina and carbon fibers consolidated with a small amount of alumina, pyrocarbon or hex-BN. The composites mechanical properties exhibited similar to those already reported for the related C/SiC, C/B_4C, C/TiC or C/BN materials. Compared with the CVI procedure, the traditional slurry infiltration and hot-pressing method has some advantages such as low cost, short processing time and higher densification.

The aim of present work was to explore the potential of continuous carbon fibers reinforced zirconia-based composites (C_f/ZrO_2) for aerospace applications. Unidirectional carbon fibers reinforced zirconia composite was prepared by

* Corresponding author. Tel.: +86 21 52414320; fax: +86 21 52415263.
E-mail address: swwang51@mail.sic.ac.cn (S.W. Wang).

0955-2219/$ – see front matter © 2007 Elsevier Ltd. All rights reserved.
doi:10.1016/j.jeurceramsoc.2007.07.005

Table 1
The mechanical properties of ZrO_2 and C_f/ZrO_2 composite

	Temperature (°C)	Relative density (%)	Flexural strength (MPa)	Elastic modulus (GPa)	K_{IC} (MPa·m$^{1/2}$)
ZrO_2	1500	98.8	125.8 ± 12.1	117.3 ± 4.3	3.2 ± 0.1
C_f/ZrO_2	1500	95.4	588.0 ± 71.8	94.7 ± 1.5	15.4 ± 0.8
	1550	96.6	547.4 ± 38.8	90.3 ± 2.5	14.2 ± 1.1
	1600	96.8	508.3 ± 18.4	86.7 ± 3.2	12.3 ± 1.6
	1650	98.5	173.2 ± 2.0	75.0 ± 3.5	5.0 ± 0.3

slurry infiltration and hot-pressing sintering method. The effect of hot-pressing temperature on the mechanical properties of composite was investigated. The morphologies of composite fracture surface were observed. Transmission electronic microscope examined the microstructural features of fiber/matrix interface.

2. Experimental procedures

ZrO_2 powder (containing 6.6 wt.% CaO, ZrO_2 > 93 wt.% and average particle size around 2 μm), and PAN-based carbon fibers (4800 MPa average tensile strength, and 5 μm in diameter), were used as starting materials. The powders were mixed in deionized water with carboxymethyl cellulose (CMC) as a binder and *iso*-propyl alcohol as a dispersant, and then ball-milled with agate balls. The prepreg was prepared by infiltrating the continuous carbon fibers into the slurry and then dried, stacked in a graphite die and hot-pressed between 1500 and 1650 °C and 25 MPa in a Ar atmosphere. The content of carbon fiber was approximately 30 vol.% in the composites. For comparison, hot-pressed ZrO_2 specimen without reinforcement was also prepared.

Density measurements were performed based on Archimedes principle. The specimens were machined into bars of 36 mm × 4 mm × 3 mm parallel to the fiber direction to measure the flexural strength by the three-point bending method with a span of 30 mm and a cross-head speed of 0.5 mm/min, at room temperature in air. Single-edge notched-beam (SENB) samples were fabricated by notching the segments of tested flexure specimens with a 0.20 mm thick diamond wafering saw. The 30 mm × 6 mm × 3 mm SENB samples were tested in three-point loading with a span of 24 mm and a cross-head speed of 0.05 mm/min. Fracture toughness K_{IC} was calculated by the ASTME 399-74 formula.[6] The flexural strength and the fracture toughness measurements were conducted by Instron-5566 testing machine. Five specimens were tested for each sample.

Flexural strength was calculated by the following equation[7]:

$$\sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

where σ_f is the flexural strength (maximum stress at mid-span), P the applied load that leads the specimen to fail, L the support span, b the specimen width and, finally d is the specimen thickness.

Effective engineering modulus was obtained from the slope of the initial straight-line of the load–displacement curve by means of this equation[8]:

$$E = \frac{L^3}{48I}\frac{F}{\delta} \quad (2)$$

where F/δ represents the slope of the load–displacement curve and I is the cross-sectional inertia of the specimen.

The phase compositions of the samples were examined by X-ray diffractometer (D/max 2550 V). The fracture surfaces of the specimens were observed by scanning electronic microscope (SEM, Model JXA-8100, Jeol Co., Tokyo, Japan). The microstructural features of fiber/matrix interface were characterized using transmission electronic microscope (TEM, Model 200CX, Jeol, Japan).

3. Results and discussion

Table 1 lists the mechanical properties of ZrO_2 and C_f/ZrO_2 composite. When hot pressed at 1500 °C, it can be seen that flexural strength and fracture toughness are 125.8 MPa and 3.2 MPa·m$^{1/2}$, respectively, for ZrO_2 without the reinforcement of carbon fiber. While, flexural strength and fracture toughness of the composites were 588.0 MPa and 15.4 MPa·m$^{1/2}$, respectively, which were the five-fold that of ZrO_2. On the other hand, with the increase of the hot-pressing temperature from 1500 °C to 1650 °C, the mechanical properties decreased gradually though the relative density of the composite increased. For the composite hot-pressed at 1650 °C, the flexural strength and fracture toughness decreased sharply to 173.2 MPa and 5.0 MPa·m$^{1/2}$, respectively.

The typical morphologies of C_f/ZrO_2 composite specimens are shown in Fig. 1. It can be seen that the cracks are zigzag

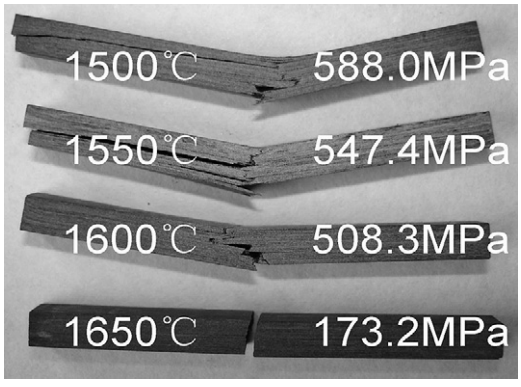

Fig. 1. Typical morphologies of C_f/ZrO_2 composite specimens after flexural test.