EVALUATION OF MECHANICAL PROPERTIES OF SiC WHISKER REINFORCED IN-SITU COMPOSITES PREPARED BY CARBOTHERMAL REDUCTION OF SILICATE MINERALS

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Abstract

Al_2O_3-SiC_w and ZrO_2-SiC_w composite powders were prepared by carbothermal reduction of alumino-silicate minerals (kaoline and silimanite) and zircon respectively. The powders were hot pressed at 1750°C and 40 MPa pressure in argon atmosphere. Test specimens ($4 \times 6 \times 30$ mm^3) were made out of hot pressed discs for mechanical properties evaluation. The physical properties i.e. bulk density (B.D.), apparent porosity (A.P.) were measured by Archimedes principle. Young's modulus, fracture toughness and bending strength were measured by resonance frequency response, SENB technique and 4-point bend test respectively. Mechanical properties were found to be better in the case of composites prepared from silimanite and zircon due to optimum whisker content.

Introduction

It is well known that composite materials possess better mechanical properties compared to their monolithics[1-2]. These composite materials are generally prepared by physical mixing of the ingredients either in particulate or in whisker form. Due to the shape and size mismatch of the whiskers with the matrices, inhomogeneity prevails in the final composite body causing inferior mechanical properties. This problem can be overcome by in-situ preparation of the composites. For example, composites of SiC whiskers(w) reinforced in oxide matrices i.e. Al_2O_3, ZrO_2 etc. can be in-situ prepared by carbothermal reduction of cheap minerals such as kaolin, sillimanite and zircon. In a previous study the authors have presented the details of the carbothermic reactions leading to the formation of Al_2O_3 -SiC_w and ZrO_2 -SiC_w composites [3-4]. In this study, the physical and mechanical properties characterised from these composites are presented.

Experimental

Al_2O_3-SiC_w and ZrO_2 -SiC_w composite powders were obtained by carbothermal

reduction of (kaolin + carbon), (sillimanite + carbon) and (zircon + carbon) with C: SiO_2= 5:5 and at 1700°C/1h in N_2 atmosphere [3-4].

The excess carbon in the carbothermally reacted products was decarburised at 700°C/4h. 25-30 grams of decarburised product was cold compacted at 50MPa in a graphite die. After cold compaction the graphite tooling with the sample was as such introduced into the hot pressing furnace (Goliath type, stein Heurtey physitherm) and the temperature was uniformly raised upto 1100°C when it was back filled with argon gas to a pressure of 950 mbar. Temperature was raised to 1500°C and a pressure of 45 MPa was applied through the pressing rams. This pressure was maintained till the maximum temperature of 1750°C was reached. The rate of heating was 18°C/min throughout the heating cycle. After a dwell period of one hour at 1750°C /45 MPa pressure the system was left for cooling.

The hot pressed disks ($\varnothing$ = 35mm) recovered from the graphite die was ground flat on both sides to a thickness of 6mm. The disks were cut into specimen bars of 30 x 40 x 6 mm (2 bars) (for measurement of fracture toughness K_{IC}) and 30 x 40 x 3 mm (5 bars) for measurement of Young's modulus (E) and Flexural strength (σ_f).

Density Measurement

Dried specimen bars were used for hydrostatic density measurement by liquid immersion method. The samples were dried at 100°C/24 h, then weighed. A vacuum was created and bars were dropped into xylene and soaked for about 20 minutes. They were weighed in air & in water. The equation used for measurement of density & porosity are given below.

$$\text{Hydrostatic density (D)} = \frac{W_d \times \rho_w}{W_a - W_w}$$

$$\text{Open Porosity (\%)} = \frac{(W_a - W_d)\,\rho_w}{(W_a - Ww)\,\rho_x} \times 100$$

where

D	=	Hydrostatic density
W_d	=	Dry specimen weight
W_a	=	Wet xylene specimen weight in air
W_w	=	Wet xylene specimen weight in water
ρ_x	=	Xylene density (0.8802 g/cc)

Measurement of Young's Modulus (E)

The surfaces of the specimen bars 30 x 4 x 3 mm (surface perpendicular to the hot pressing axis) were mechanically polished in stages with 6 μm , 3μm diamond paste.

Young's modulus was measured using a dynamical method with Grindosonic apparatus. The distance between the supports for the bars was adjusted. The specimen bar in the apparatus was slightly hit with a ball hammer and the constant minimum resonance frequency displayed was noted. The fundamental resonance frequency of the tested bars was related to Young's modulus as a function of the bars dimensions. The Young's modulus was assessed by the following relation.

$$E = 0.94478(M / b)(L / W)^3 f^2$$

where,

E = Young's modulus (GPa)
M = Mass of specimen (g)
b = thickness (mm)
L = length (mm)
W = Height of the specimen (mm)

$f = \frac{2 \times 10^6}{R}$, where R is the Resonance frequency.

Measurement of Flexural Strength (σ_f)

Specimen bars (30 x 4 x 3 mm) with surface polished in the same manner (for Young's modulus measurements) were used in a 4-point bending machine (Schenk Trebel and Instron. Type) which allows a load of 50000 N. A maximum of 3 bars per sample were tested. The peak load of failure was noted when the cross head speed of 0.5 mm / min was maintained. The following relationship was used to obtain the fracture strength.

$$\sigma_f = (3/2)P(L1 - L2)/ bW^2$$

where

σ_f = Flexural strength (MPa)
P = Applied load (N)
L1 = Distance between bottom supports (mm)
L2 = Distance between top span (mm)

Measurement of the Fracture Toughness K_{IC}

Specimen bars (30 x 4 x 6 mm) were polished to mirror finish by mechanical polishing of surface in stages upto 0.3μm alumina suspension finish. Fracture toughness (K_{IC}) was determined by single edge notch beam (SENB) technique. A notch was made at the centre to less than half the width of the specimen bar and the depth of the flaw made was measured microscopically. The flaw depth to width ratio ≤ 0.4 was verified and confirmed [5].

The notched specimen bars were loaded upto failure in the same four point bending machine (Schenck Trebel W.Germany) at room temperature maintaining the cross head speed at 0.5 mm/min. The K_{IC} values were obtained using the formula

$$K_{IC} = \sigma_f Y \sqrt{a}$$

where

K_{IC} = fracture toughness MPa $\sqrt{m}$
σ_f = flexural strength (MPa)
a = depth of the critical flaw (in meters)

Geometric factor for the case of four point bending

$$Y = 1.99 - 2.47\,(a/w) + 12.97\,(a/w)^2 - 23.7\,(a/w)^3 + 24.8\,(a/w)^4$$

where

a = the exact depth of notch (critical flaw) (in mm) in the specimen
w = the width of the specimen (in mm)

The Young's modulus(E), the fracture strength (σ_f) and the fracture toughness K_{IC} for the different hot pressed samples have been summarised in the table [2].

Results and Discussion

The theoretical density of the hot pressed composites derived from kaolin precursor is found to be low. This is obvious due to higher SiC whisker loading (45%) since kaolin ($Al_2O_3.2SiO_2.2H_2O$) contains two moles of silica compared to one mole of silica in sillimanite ($Al_2O_3.SiO_2$). The lower mechanical properties of the kaolin based composites are due to low density and higher whisker content. However, both sillimanite and zircon based composite (which have near optimal whisker loading) have produced better strength and mechanical properties.

Conclusion

From the results of mechanical properties characterisation, it is concluded that

composites prepared by in-situ whiskerisation can produce better mechanical properties if whisker content is optimum.

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Table 1
Physical Properties of composite specimens made from different precursors

Precursor composition	Reaction condition	Final product composition	%SiC in sample (Theoretical)	Density g/cc	Total porosity %	%Theoretical density
	Powder			Hot pressed body		
Kaolin + Carbon	1750°C N_2 /h	α-Al_2O_3 + SiC_w + mullite(traces)	44.0	3.23	0.150	84.0
Sillimanite + Carbon	1750°C Ar/h	α-Al_2O_3 + SiC_w	28.2	3.56	0.030	96.0
Zircon + carbon	1750°Cr N_2 /h	m-ZrO_2 + SiC_w	24.5	4.45	0.020	90.0

Table 2
Mechanical properties of composite specimens made from different precursors

Precursor composition	Reaction condition	Final product composition	%SiC in sample (Theoretical)	Young's modulus E(GPa)	Fracture Strength σ_f(MPa)	Fracture toughness (MPa $\sqrt{m}$)
	Powder			Hot pressed body		
Kaolin + Carbon	1750°C N_2 /h	α-Al_2O_3 + SiC_w + mullite(traces)	44.0	220 230	100 112	5.10 4.58
Sillimanite + Carbon	1750°C Ar/h	α-Al_2O_3 + SiC_w	28.2	200 208	220 216	8.75 8.67
Zircon + carbon	1750°C N_2 /h	m-ZrO_2 + SiC_w	24.5	200 204	250 244	7.03 7.04