Ceramics International ■ (■■■■) ■■■–■■■

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Tribo-mechanical characterization of spark plasma sintered chopped carbon fibre reinforced silicon carbide composites

Shivam Agarwal [a,b], Soumya Sarkar [b,*], Mitun Das [c], Amit Rai Dixit [a]

[a] Department of Mechanical Engineering, Indian School of Mines, Dhanbad, 826004 India
[b] Non-oxide Ceramics and Composites Division (NOCCD), CSIR-Central Glass and Ceramic Research Institute, Kolkata 700032, India
[c] Bio-Ceramics and Coatings Division (BCCD), CSIR-Central Glass and Ceramic Research Institute, Kolkata, 700032 India

ARTICLE INFO

Article history:
Received 4 June 2016
Received in revised form
25 August 2016
Accepted 25 August 2016

Keywords:
Composites
Fibres
Mechanical properties
Wear resistance

ABSTRACT

Short carbon fibre (C_f) reinforced silicon carbide (SiC) composites with 7.5 wt% alumina (Al_2O_3) as sintering additive were fabricated using spark plasma sintering (SPS). Three different C_f concentrations i.e. 10, 20 and 30 wt% were used to fabricate the composites. With increasing C_f content from 0 to 20 wt%, micro-hardness of the composites decreased ~28% and fracture toughness (K_{IC}) increased significantly. The short C_f in the matrix facilitated enhanced fracture energy dissipation by the processes of crack deflection and bridging at C_f/SiC interface, fibre debonding and pullout. Thus, 20 wt% C_f/SiC composite showed > 40% higher K_{IC} over monolithic SiC ($K_{IC} \approx 4.51$ MPa $m^{0.5}$). Tribological tests in dry condition against Al_2O_3 ball showed slight improvement in wear resistance but significantly reduced friction coefficient (COF, μ) with increasing C_f content in the composites. The composite containing 30 wt% C_f showed the lowest COF.

1. Introduction

Silicon carbide ceramic has been regarded as one of the most promising materials for high performance structural applications due to its low density, high strength and modulus, high hardness, better thermo-mechanical stability, creep resistance, chemical stability and thermal shock resistance [1–9]. Further, excellent wear resistance of SiC makes it an attractive material for high speed friction applications e.g. in automobile brake pads and clutches [4,14]. However, structural use of pure SiC is limited due to its low toughness that often leads to catastrophic failure during service [3]. To evade this issue, fabrication of fibre reinforced SiC composites has been an important topic of global research for quite a long time. However, unidirectional fibre reinforced SiC matrix composites are less attractive due to anisotropic mechanical as well as thermophysical performance even after processing through stringent manufacturing steps. Such property anisotropy can be avoided either using multi-directionally weaved fibre performs or simply through characteristic random distribution of short (chopped) fibres in the matrix phase [1,3–6,10]. Moreover, fabrication of short fibre reinforced composites is relatively easier [5,6]. C_f/SiC composites can be fabricated by chemical vapour infiltration (CVI), polymer infiltration and pyrolysis (PIP), hot pressing (HP) and liquid silicon infiltration (LSI) methods [1,11–15]. However, fabrication of dense components using these techniques generally involves high production cost because of long cycle time even for simple shapes [10]. In addition, if not properly managed, evolution of toxic gases can severely pollute the surrounding atmosphere. On the other hand, spark plasma sintering (SPS) is a technique that can rapidly densify C_f/SiC composites near to their theoretical density values at a fairly lower temperature and soaking time than those required in conventional sintering techniques [3,16]. However, considering the difficulties involved in solid state sintering of pure SiC and homogeneous dispersion of chopped C_f in SiC matrix, research work on solid state sintering of C_f/SiC composites and their property evaluation is still limited. In the present work, 7.5 wt% Al_2O_3 aided SiC composites reinforced with varying concentration of short C_f were fabricated using SPS. The effect of C_f concentration on microstructure, mechanical and tribological properties of the composites is being reported in the present article.

2. Experimental

2.1. Raw materials and composite fabrication

For the matrix phase, *M*-15 grade SiC powder of *Carborundum Universal Pvt. Ltd., India* having > 98 wt% purity (predominantly α-phase) and specific surface area of 16 m^2/g was used as the raw

* Corresponding author.
E-mail address: soumya@cgcri.res.in (S. Sarkar).

http://dx.doi.org/10.1016/j.ceramint.2016.08.155
0272-8842/

Table 1
Nomenclature, composition and properties of the spark plasma sintered SiC and C_f/SiC composites.

Specimen ID	Composition	TD (g/cc)	RD (%)	AP (vol%)	COF (μ)	Wear Rate (x 10^{-6} mm^3/N-m)
M15	> 98 wt% SiC	–	–	–	–	–
S0	SiC + 7.5 wt% Al_2O_3	3.247	98.46	0.67	0.52	2.50 ± 0.44
S10	(*S0*) + 10 wt% C_f	3.093	96.48	0.19	0.38	2.60 ± 0.47
S20	(*S0*) + 20 wt% C_f	2.976	94.56	0.32	0.37	2.27 ± 0.24
S30	(*S0*) + 30 wt% C_f	2.883	92.23	1.48	0.19	2.25 ± 1.02

material. Average particle size (i.e. d_{50}) of SiC powder, measured using a laser diffraction particle size analyser (*LS 13 320 MW, Beckman Coulter, USA*), was found to be ~0.55 μm. Reactive polycrystalline Al_2O_3 (*A-16-SG*, purity > 99.5 wt%, *Almatis Alumina Pvt. Ltd., India*) having d_{50} of 0.4 μm was used as the sintering additive [17,18]. C_f (*KGF-200, C-203S, Kureha Corporation, Tokyo, Japan*) having average diameter and length of 14.5 μm and 3 mm, respectively, was used as the reinforcement. Initially, the 7.5 wt% Al_2O_3/SiC batch was prepared using 3 h magnetic stirring of SiC and Al_2O_3 powders in ethyl methyl ketone followed by drying in an air oven at ~75 °C. In the next step, appropriate amount of short C_f was dispersed in isopropyl alcohol (IPA) by 1 h ultrasonication (*VCX1500, Sonics and Materials Inc., USA*) at 900 W. Finally, the dispersed C_f and requisite amount of Al_2O_3/SiC powder mixture were attrition milled for 3 h in IPA using Al_2O_3 grinding balls (ϕ=3 mm). The C_f/(7.5 wt% Al_2O_3/SiC) mixture was then dried in the oven at ~75 °C and sieved through a 60 mesh B.S. sieve. Four different batches containing 0, 10, 20 and 30 wt% fibre were prepared using the above steps. Compositions and nomenclature of the studied batches are given in Table 1. All batches were then sintered in a *HP-D-25* (*FCT Systeme GmbH, Germany*) SPS furnace at 1800 °C under 50 MPa uniaxial pressure with 20 min dwell time. A ramp rate of 100 °C/min was used during the entire SPS cycle. The specimens were compacted in a graphite mould having inner diameter of 20 mm. The SPS processed pellets were ~20 mm in diameter and ~6 mm in thickness.

2.2. Material characterisations

Physical properties of the sintered specimens were measured using *Archimedes* water immersion technique. Phase analyses were carried out in an X-ray diffractometer (*X'pert Pro MPD, PANalytical, Netherlands*) using CuK_α radiation in 20–80° of 2θ values. Sintered specimens were polished and etched in Murakami solution. The specimens were viewed through a scanning electron microscope (SEM, ProX, Phenom-World BV, The Netherlands). Matrix grain size values of the studied specimens were evaluated using the SEM micrographs. More than hundred different grains were measured at five different spots. Vicker's microhardness values of the sintered specimens were evaluated using a hardness tester (*402 MVD, Wolpert-Wilson, Germany*) at 9.8N load for 10 s. For averaging purpose, 15 indents were made on each of the studied specimens. Indentation K_{IC} values of the specimens at 9.8N were evaluated using direct crack measurement method applying Niihara et al. [19] formulae:

$$^{Niihara}K_{IC}=0.0181\times E^{0.4}\times H^{0.6}\times\frac{a}{(c-a)^{0.5}}\text{(Palmqvist crack)} \quad (1a)$$

$$^{Niihara}K_{IC}=0.0667\times E^{0.4}\times H^{0.6}\times\frac{a^2}{(c)^{1.5}}\text{(Mediancrack)} \quad (1b)$$

where, E=Young's modulus, H=Hardness, a=half of indentation diagonal, c=crack length.

2.3. Tribological study

Unlubricated tribological properties of SPS processed C_f/SiC composites and pure SiC were evaluated at room temperature (RT) using a ball-on-disc tribometer (*Nanovea, USA*) according to ASTM standard G-99-95a [20]. Wear tests were performed under 10N normal load using Al_2O_3 ball (ϕ=3 mm) as the counter body. A sliding speed of 60 mm/sec and sliding distance 500 m were used. After the wear test, natures of the scar profiles were obtained using a contact type surface profilometer (*Talysurf i120, Taylor Hobson, USA*). Finally, wear rate (W_R) was calculated using the following expression:

$$W_R=\frac{V}{Ps} \quad (2)$$

where, V=scar volume (mm^3), P=normal load (N), s=total sliding distance (m) [1,7]. To trace out the effect of C_f addition on the wear characteristics of pure SiC, worn surfaces of the samples were viewed through scanning electron microscope (*ProX, Phenom-World BV, The Netherlands*).

3. Results and discussion

3.1. Densification behaviour

Table 1 shows theoretical density (TD), relative density (RD) and apparent porosity (AP) values of the SPS processed SiC and C_f/SiC composites. TD values of the composites were determined using "rule of mixture". For this purpose, density values of SiC, Al_2O_3 and C_f were taken as 3.2, 3.97 and 2.1 g/cc, respectively, [21,22]. It was observed that while, the pure SiC specimen attained the highest density (RD ~98.5%), RD of the composites decreased with increasing C_f content. The 30 wt% C_f/SiC composite attained ~92% of its TD (Table 1). As far as literature data on densification trend of short C_f reinforced SiC composites was concerned, Tang et al. [1] obtained 96% and 92% RD values of hot-pressed C_f/SiC composites containing 42 and 53 vol% short fibres, respectively. Using SPS processing at 1650 °C under 25 MPa uniaxial pressure with 3 min dwell, Ding et al. [3] obtained the highest BD of 2.83 g/cc for a 20 vol% short C_f/SiC composite that was ~13.5% lower than that obtained for pure SiC sintered only at 1550 °C under 15 MPa pressure. Furthermore, according to the report by Li et al. [5], RD values of 2.5, 5 and 10 wt% short C_f reinforced SiC composites were found to be 97%, 96% and 93%, respectively, even after pressureless sintering at 2100 °C for 1 h in Ar atmosphere [5]. Therefore, densification of present SPS processed short C_f/SiC composites was on the higher side of available literature data not only as referenced above but also for the composites reported by others [6,10,16,23–28]. Furthermore, other researchers have also reported significant reduction in matrix densification at increased C_f concentration as observed in the present study [3,5,24,26]. Addition of C_f significantly restricted mass transport through grain boundary regions during the sintering process that resulted in lower RD of the composites. In addition to that, Yang et al. [24] reported that for C_f/SiC composites, while, the planes containing C_f significantly

Please cite this article as: S. Agarwal, et al., Tribo-mechanical characterization of spark plasma sintered chopped carbon fibre reinforced silicon carbide composites, Ceramics International (2016), http://dx.doi.org/10.1016/j.ceramint.2016.08.155