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Full Length Article

Preparation of silicon carbide/carbon fiber composites through high-temperature spark plasma sintering

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ABSTRACT

This study discusses the potentials of spark plasma sintering (SPS) integrated with high temperature process that can enable sintering of SiC/C_f composites without any sintering aids. The random distribution of carbon fibers was obtained through mixing composite components in ethanol by using a shaker mill for 10 min. The corresponding sintering process was carried out at 1900 and 2200 °C with 50 MPa pressure applied at maximum temperature. The results showed that 89 ± 0.9 and 97 ± 0.8% of the theoretical density can be obtained for sintering temperatures of 1900 and 2200 °C, respectively. The densification curves were plotted to monitor sintering behavior with punch displacement changes. The appropriate bonding between SiC particles and carbon fibers was detected using FE-SEM for sample which was sintered at 2200 °C. The clear maximum in hardness (2992 ± 33 Vickers), bending strength (427 ± 26 MPa) and fracture toughness (4.2 ± 0.3 MPa $m^{1/2}$) were identified for sample sintered at 2200 °C. XRD investigations supposed that SiC and carbon were the only crystalline phases in both sintered samples.

1. Introduction

For many decades, SiC has been used widely in ceramic industries for different applications [1,2]. Low density, thermal shock stability, high creep resistance, high strength at evaluated temperature, high hardness/modulus and also cost effectiveness of the SiC make it a promising choice in various applications such as rocket nozzles, engine flaps, and leading edges of spacecraft [3,4]. The main problem of ceramic base materials, especially covalently bonded ceramics, is low fracture toughness, high melting and reaction at high temperatures which make ceramic materials difficult to be sintered without sintering aids or additive [5]. Mostly, these sintering aids have low melting temperatures compared to ceramic matrices such as SiC, Si_3N_4, WC, etc. [6–9]. However, the recent progress in high-temperature processing provides the possibility to sinter some ceramic materials without sintering aids at high temperatures and by using pressure during these processes such as hot isostatic press (HIP), hot press (HP), SPS and pressure assisted microwave sintering [10].

Conventional processing methods for production of SiC/C composite in industrial scale are mostly based on infiltration methods including Chemical Vapor Infiltration (CVI), Liquid Silicon Infiltration (LSI), etc. [11,12]. Each method offer its own microstructure and final properties based on the processing parameters [13]. Although these techniques have applications in industrial scales, there is another chance to produce ceramic matrix composites such SiC/C or C/C composite with hand layer up arrange from initial elements and compounds during the whole time of powder production technique. In-situ producing methods, especially ceramic matrix composite (CMC) productions, offer unique advantages such as good time and energy efficiency in self-propagating high-temperature synthesis (SHS) [14]. On the other hand, there are some residual reaction products which are mostly not detected in many research works using conventional detection methods (i.e., SEM and XRD). Somehow, the small amounts of these materials can affect the mechanical performance of composites by making new bonds between particles. Therefore, the methods which are capable to use starting materials as final components of products can give proper properties without any reaction between starting materials [15].

Spark plasma sintering method offers unique advantages such as vacuum condition, applied pressure and fast heating rate using both pressure and high sintering temperature [16–19]. In HP and HIP methods, the heating source is produced by conventional heating principle, while in SPS, the neck growth is accelerated by creation of spark between particles [20]. These advantages make SPS method a powerful option to produce SiC/C ceramic matrix composites even without sintering aids and also open new the window to produce bulk composite materials and also final products with advanced cutting and shaping processes such as laser cutting process.

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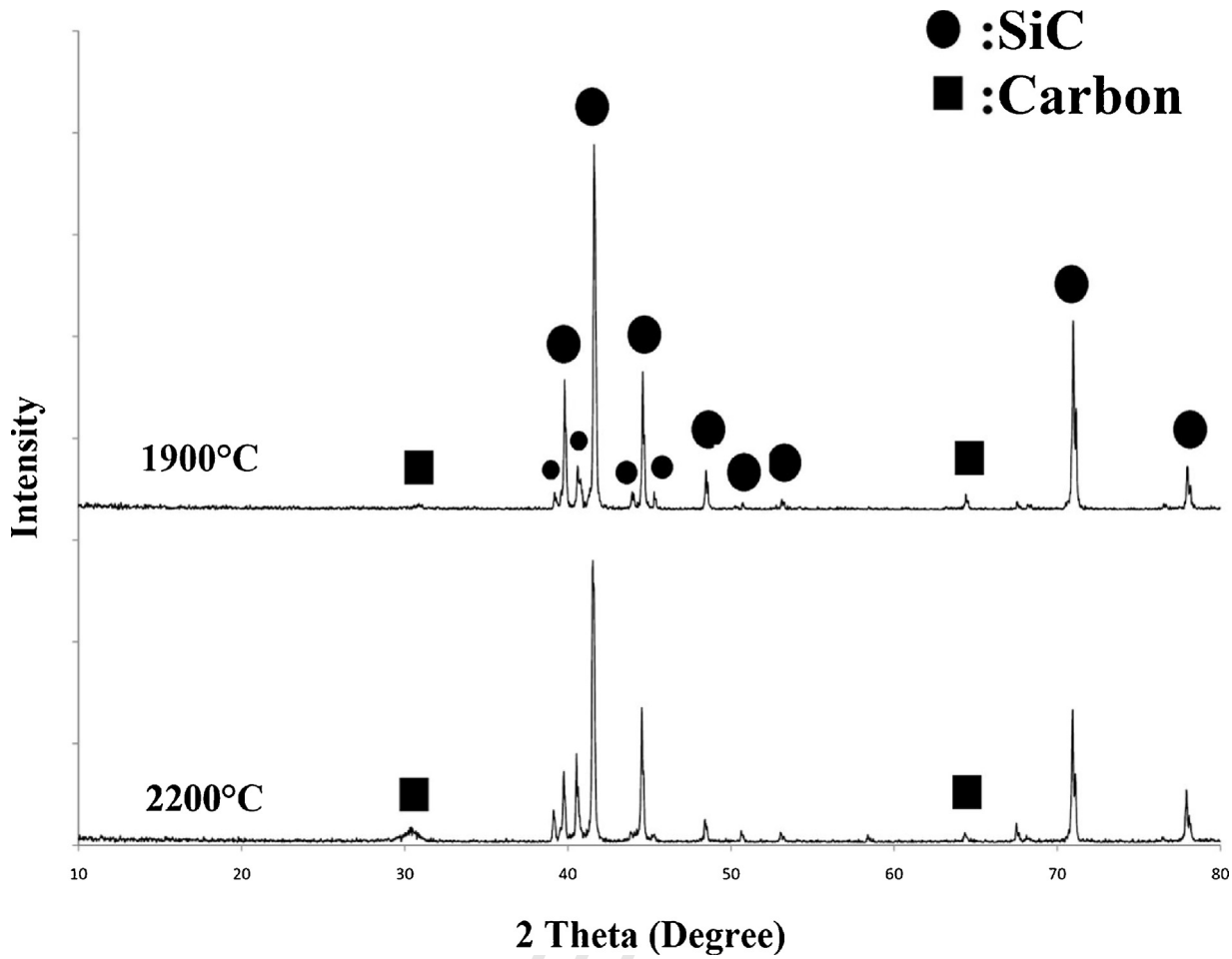

Fig. 1. XRD patterns of SiC/C composites sintered at temperatures of 1900 and 2200 °C.

In the present work, we report a new route to produce SiC/C ceramic matrix composites from carbon fiber and SiC nano powders as starting materials. For better understanding the effects of processing parameters on final properties of the composite, the sintering behavior, microstructure and mechanical properties of produced specimens have been investigated.

2. Experimental procedures

SiC nano powders (Alfa Aesar-average particle size of 50 nm) and carbon fibers (5 μm diameter) were used as starting materials. 1 wt.% carbon fibers which were 30 μm in length were cut and the separation of carbon fibers from each other was performed with a high energy ultrasonic in ethanol. Then, SiC powders were added to carbon fibers and ethanol and the mixture was inserted into Spex mixer mill for 10 min to enhance random distribution of carbon fibers within SiC powders. After mixing stage, the mixture was dried at 80 °C on the hot plate. After drying, the mixed powders were inserted directly into a graphite mold with 30 mm diameter and graphite foils (1 mm thickness). The sintering processes were performed using SPS (20T-10, China) with an initial pressure of 10 MPa under vacuum condition (15 Pa) and followed by increasing pressure during the process (final pressure of 50 MPa) and finished at sintering temperatures of 1900 °C and 2200 °C for about 8 and 6 min as holding time. After removing the surface layer from the sintered disks by grinding, the phase identification was carried out using XRD (Philips X0 Pert System) with a Co k_α (λ = 1.789 Å) radiation source and an image-plate detector over the 2θ range of 10–85° in reflection geometry. After cutting samples into 5 × 5 × 25 mm dimensions (using a Charmilles Robofill 310 wire EDM), the three point bending strength measurements were examined by Santam-STMm 20 (Iran) machine. The bulk density of sintered samples was measured using the Archimedes' principle. Microstructure characterization of sintered samples was carried out using Field Emission Scanning Electron Microscopy (FE-SEM) (MIRA 3 TESCAN, Czech Republic) equipped with an Energy DispersiveSpectrometer (EDS). The Vickers hardness measurements were performed on the polished surface of the specimens with 10 successive indentation tests (Koopa universal hardness tester, model: UV1, Iran) under the load of 30 Kgf for 10 s at room temperature. The corresponding diagonals of the indentation and crack sizes were measured using the optical microscope and FE-SEM observations. The fracture toughness (K_{IC}) was calculated as below [21]:

$$K_{IC} = 0.18(P/c^{1.5})(E/Hv)^{0.5}$$

where P is the load (N) and c is half of the average crack length, Hv is Vickers hardness and E is Young's modulus which is calculated from the rule of mixture.

3. Results and discussion

Fig. 1 shows XRD patterns of specimens sintered at different temperatures (1900 and 2200 °C). As it can be concluded from Fig. 1, the only crystalline phases are SiC and carbon with high and low intensity, respectively, which is predictable from the used starting materials. Considering the vacuum condition of the spark plasma

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