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Characterization of sintered TiC–SiC composites

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Abstract

TiC–SiC composites were fabricated using TiC and SiC powders as starting materials at the range of 1650–2000 °C in Ar atmosphere by two-step method. In the first step, the ingots with intragranular SiC or TiC particle were prepared by arc-melting technique, subsequently, crushed and ground into TiC and SiC composite powders. In the second step, TiC–SiC composites were sintered using these as-prepared composite powders by SPS method. It was concluded that these TiC–SiC composites prepared by two-step method showed more excellent properties than that prepared by arc-melting technique. The hardness of the fabricated TiC–SiC composites was 25–27 GPa at the load of 0.98–9.8 N, which was obviously greater than that of arc-melting composites. The thermal conductivity of the TiC–SiC composites was 18–48 W K^{-1} m^{-1} at the range of 298–1273 K and slightly decreased with increasing temperature. The electrical conductivity of the composites was (2–5) × 10^5 S m^{-1} at the range of 298–1273 K and slightly decreased with increasing temperature.

Keywords: TiC–SiC composites; Two-step method; Microstructure; Arc-melting technique

1. Introduction

Silicon carbide has extremely high hardness and wear resistance, excellent corrosion, thermal shock and oxidation resistance, good high temperature strength, which allow the use of SiC for numerous structural and wear applications, e.g. heat exchanger, metal working parts, nozzles. However, the moderate fracture of toughness of SiC ceramic limits its use under severe conditions. Additions of TiC [1–7] particles were found to be effective in terms of increasing the fracture toughness of SiC ceramic. However, the TiC–SiC composites are difficult to be densified due to the low self-diffusion of SiC in TiC. The TiC–SiC composites are generally synthesized by the hot pressing [8] and SPS technique [9–11]. Moreover, the properties of composites strongly depend on their microstructure. The intragranular microstructure is effective in improving mechanical properties of composite [12]. However, the intragranular composites are difficult to be fabricated by above sintering method. The arc-melted method was one of the best techniques of the preparation of intragranular composites [13–15]. However, the as-prepared specimens have smaller size and are easily cracking. In this paper, the ingots with intragranular SiC or TiC particle, which were prepared by arc-melting technique, were crushed and ground into TiC and SiC composite powders. Subsequently, TiC–SiC composites were sintered using these as-prepared composite powders by SPS method and characterizations were investigated.

2. Experimental

An amount of TiC and SiC powders of high purity (99.9%) and small grain size (0.5–1 μm), produced by Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, were used as starting materials (source, chem.com., grain size). The powders were weighted and mixed in an agate mortar by adding a small amount of ethanol. The mixtures of powders, pressed into disks with 10 mm in diameter at 30 MPa, were melted and directionally solidified by an arc-melted method on the copper base in Ar atmosphere. The arc-melting ingots were crushed and ground into powders with the size of 1–10 μm in an agate mortar. The as-prepared TiC–SiC composite powders were filled into a carbon die for the SPS sintering at 1650–2000 °C, 80 MPa. The sintered TiC–SiC composites were cut into specimens of various sizes for measurement.

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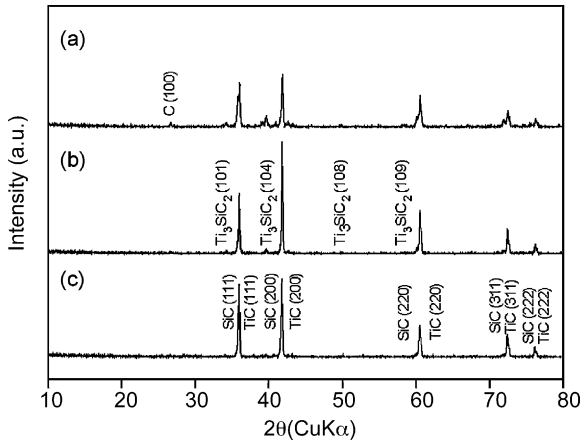

Fig. 1. XRD pattern of as-prepared composite powders at the composition of (a) 50TiC–50 SiC, (b) 70TiC–30 SiC, and (c) 80TiC–20SiC.

The phases of composites were determined by X-ray powder diffraction (Model D/MAX-3B, Rigaku, Japan) with Cu Kα radiation. The microstructure was observed by scanning electron microscopy (SEM, SX-40, Akashi Seisakushu). The hardness of the TiC–SiC composites were measured by the Akashi MVK-E Hardness Tester (Akashi Co., Tokyo, Japan) and calculated by taking the average value of 30 points in the composite indented at random. The electrical conductivities were measured by a dc four-probed method for rectangular specimens. The thermal conductivities were measured by a laser flash method using disk specimens in the temperature range between room temperature and 1023 K (TC-7000 Laser Flash Thermal Constant Analyzer, Japan).

3. Results and discussion

Fig. 1 shows XRD pattern of composite powders prepared by an arc-melting method at the composition of 80TiC–20SiC, 70TiC–30SiC and 50TiC–50SiC (mol%). The phase of composites had a certain Ti_3SiC_2 and C due to the reaction of TiC and SiC as well as raw materials, TiC and SiC phases. The content of the Ti_3SiC_2 and C increased with increasing the content of SiC due to more decomposing of SiC. Fig. 2 shows the microstructures of TiC–SiC composites sintered by two-step method at 1800 °C using 80TiC–20SiC, 70TiC–30SiC and

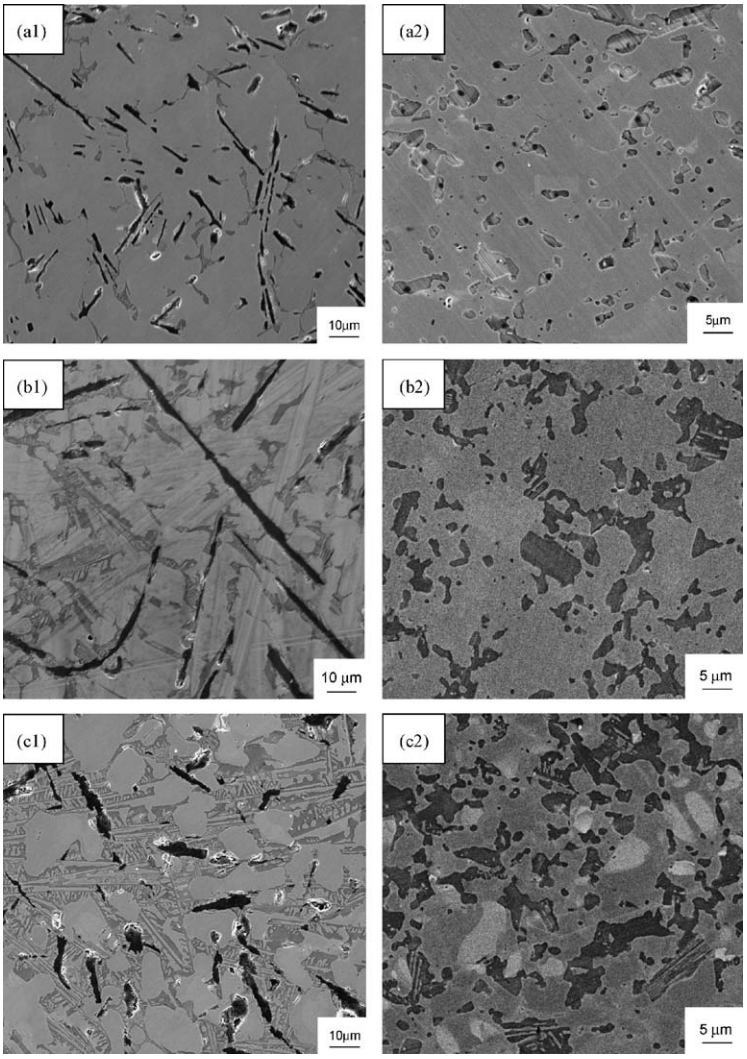

Fig. 2. SEM photograph of TiC–SiC composites prepared under the conditions of (a1) 80TiC–20SiC, arc-melting method, (a2) 80TiC–20SiC, two-step method, (b1) 70TiC–30SiC, arc-melting method, (b2) 70TiC–30SiC, two-step method, (c1) 50TiC–50SiC, arc-melting method, and (c2) 50TiC–50SiC, two-step method.

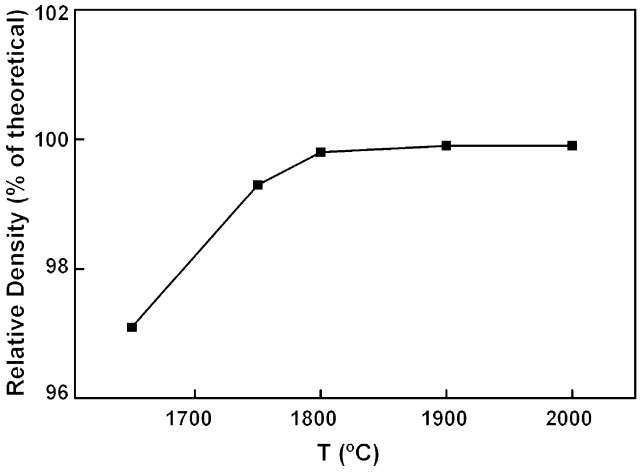

Fig. 3. Temperature dependence of relative density of the SPS-sintering 70TiC–30SiC composite.

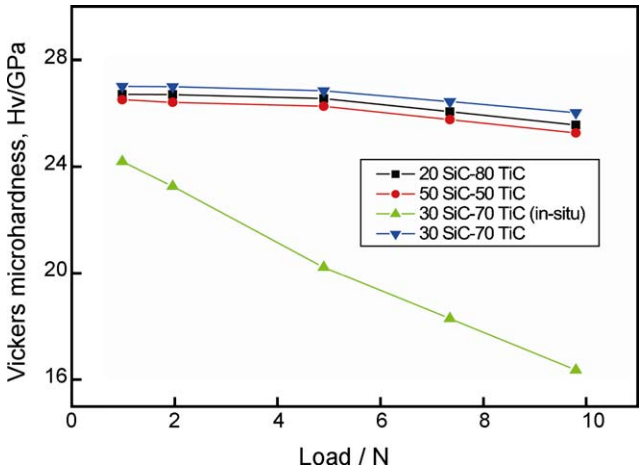

Fig. 4. Indenter load dependence of Vickers micro hardness of the SPS-sintering TiC–SiC composites at the composition of (a) 80TiC–20SiC, (b) 70TiC–30SiC, and (c) 50TiC–50SiC comparing with that of in situ 70TiC–30SiC.

50TiC–50SiC (mol%) composite powders as starting materials at 1800 °C, respectively, comparing with that by an arc-melting method. The arc-melting TiC–SiC composites showed intragranular microstructure where the spherical black SiC grains coexisted within the white TiC matrix, as shown in Fig. 2(a). However, there exist many elongated prismatic holes in the arc-melting TiC–SiC composites. The relative density of arc-melting 70TiC–30SiC (mol%) composite was only 94%. The TiC–SiC composites fabricated by two-step method were much denser than the corresponding arc-melting TiC–SiC composites with the same compositions, and kept the intragranular microstructure in the arc-melting TiC–SiC composites. Comparing with the arc-melting TiC–SiC composites the SPS-sintered TiC–SiC composites have no obviously increased

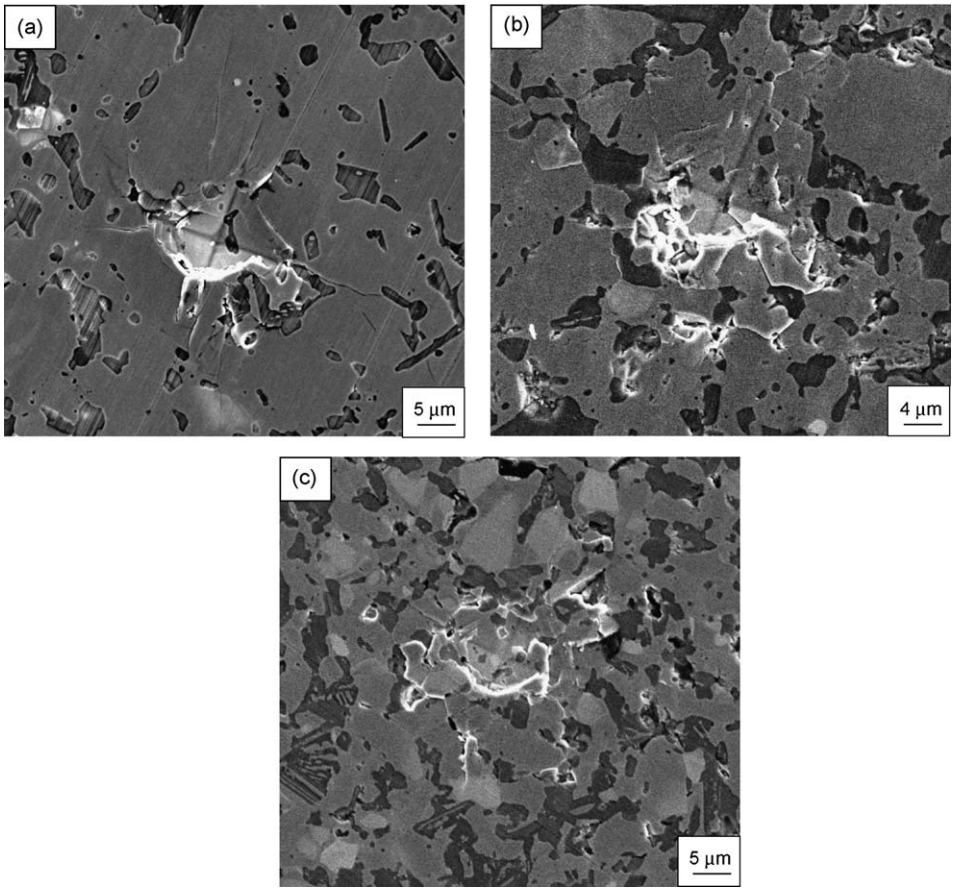

Fig. 5. Indentation of Vickers micro hardness of the SPS-sintering TiC–SiC composites in 4.9 N loading at the composition of (a) 80TiC–20SiC, (b) 70TiC–30SiC, and (c) 50TiC–50SiC (mol%).

in the grain size and SiC has more regular morphology. Moreover, a little WC phase has been found in the microstructure of arc-melting 50TiC–50SiC composite, which is due to the dissolution of electrode W in the melting composite. However, WC could not be found in the 70TiC–30SiC and 80TiC–20SiC composites, which suggested that TiC–SiC composites were difficult to be melted with the increase of SiC content. Fig. 3 shows temperature dependence of the relative density of 70TiC–30SiC composite in the sintering temperature of 1650–2000 °C. The relative density of 70TiC–30SiC increased with increasing temperature. The ingots became full dense when sintering temperature was about 1800 °C.

Fig. 4 shows the indenter load dependence of Vickers microhardness of the TiC–SiC composites fabricated by two-step method and arc-melting technique. The hardness of the SPS-sintering TiC–SiC composites was about 25–27 GPa, which slightly increases with decreasing the indenter load. The hardness of SPS-sintering TiC–SiC composites increased with increasing the content of SiC from 20 to 30%, but decreased with increasing the content of SiC from 30 to 50%. The former is due to the higher hardness of SiC than TiC, the later is because there existed WC grains with lower hardness. Moreover, the hardness of SPS-sintering TiC–SiC composites was obviously bigger than that of arc-melting composite. The hardness of TiC–SiC composites was related to the density as well as the composition. Full densification of TiC–SiC composites favors the increase of the hardness. Fig. 5 shows the indentation of Vickers micro hardness of the SPS-sintering TiC–SiC composites at the composition of (a) 80TiC–20SiC, (b) 70TiC–30SiC, (c) 50TiC–50SiC (mol%). The crack propagation path was deflected in the intergranular mode with the evidence of the transgranular mode. This result was almost consistent with results of the sample sintered at 2000 °C [8], in which the crack traveled around the TiC. These results indicate that there exists a weaker coherent layer between TiC and SiC grains in the composites such as carbon or Ti_3SiC_2 layer, which increase the toughening mode of TiC–SiC composites [16]. Fig. 6 shows the temperature dependence of electrical conductivity for the TiC–SiC composites fabricated by two-step method. The electrical conductivity of TiC–SiC composite slightly decreased with increasing temperature showing metallic behavior. The electrical conductivity of TiC–SiC composite was about (2–5) $\times 10^5$ S m^{-1} at room temperature, which was greater than a value of SiC and TiC. These results suggest that there exist carbon and Ti_3SiC_2 phases with higher electrical conductivities in the boundary of grains. The electrical conductivity of TiC–SiC composite slightly decreased with increasing the content of SiC because of the higher electrical conductivity of SiC than that of TiC. Fig. 7 shows the temperature dependence of thermal conductivity for TiC–SiC composite. The thermal conductivity of TiC–SiC composite was 18–48 W K^{-1} m^{-1} at the room temperature and slightly decreased with increasing temperature. The thermal conductivity of TiC–SiC composite was between that of TiC and SiC.

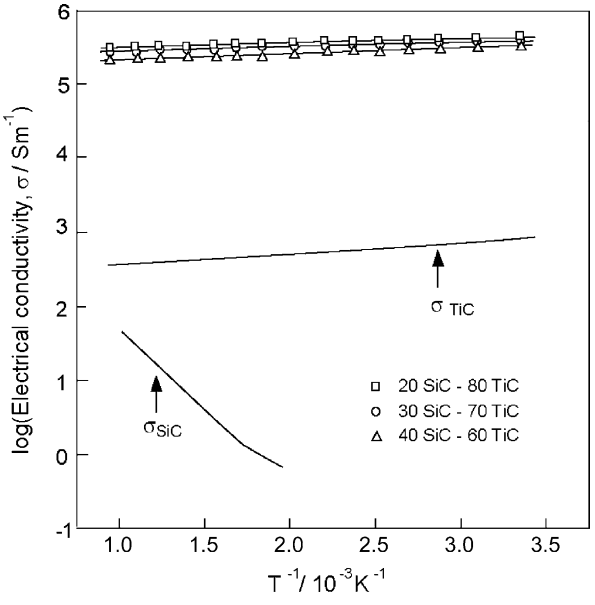

Fig. 6. Temperature dependence of electrical conductivity of the SPS-sintering of TiC–SiC composites.

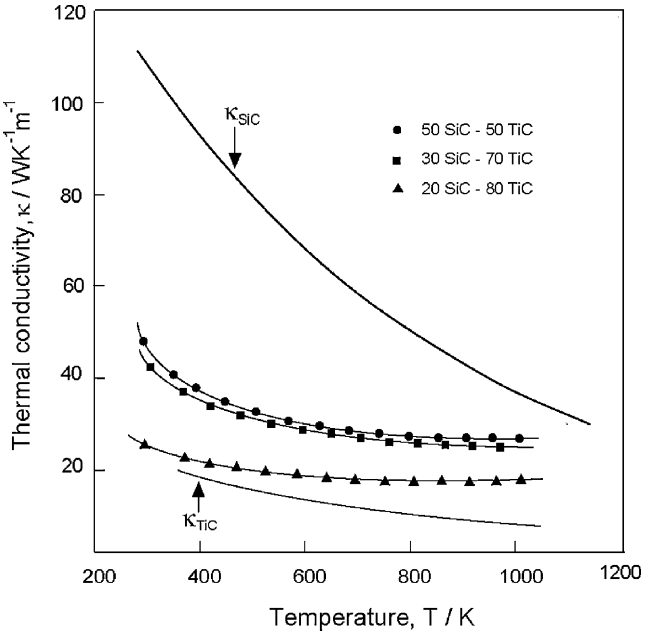

Fig. 7. Temperature dependence of thermal conductivity of the SPS-sintering TiC–SiC composites.

4. Conclusion

The TiC–SiC composites fabricated by two-step method have bigger density comparing with the arc-melting TiC–SiC composites. The hardness of the SPS-sintering TiC–SiC composite was 25–27 GPa. The electrical conductivity of SPS-sintering TiC–SiC composite was about (2–5) $\times 10^5$ S m^{-1} at the range of 298–1273 K. The thermal conductivity of SPS-sintering TiC–SiC composite was 18–48 W K^{-1} m^{-1} at the range of 298–1273 K.

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