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Gas pressure sintering of Al_2O_3/TiCN composite

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Abstract

Al_2O_3/TiCN composites have been fabricated by gas pressure sintering, which overcomes the limitations of hot pressing. The densification behavior and mechanical properties of the Al_2O_3 gas pressure sintered with 30 wt.% TiCN at different temperatures have been investigated. The gas pressure sintered Al_2O_3–30 wt.%TiCN composite achieved a relative density of 99.5%, a bending strength of 772 MPa, a hardness of 19.6 GPa, and a fracture toughness of 5.82 MPa·$M^{1/2}$.

Keywords: B. Microstructure-final; C. Mechanical properties; D. Al_2O_3; Gas-pressure sintering

1. Introduction

Al_2O_3/TiC composites consist of small titanium carbide grains dispersed in an alumina matrix and have been used as excellent cutting tools for a long time [1–4].

The characterization of their mechanical properties has also been an interesting subject for research by noting the possible toughening effects resulting from the mismatch between the thermal and/or mechanical properties of the Al_2O_3 and TiC grains [5,6]. Up to now, most of the Al_2O_3/TiC composites have been fabricated by hot pressing which have many limitations for mass production. Instead, gas-pressure sintering is now widely used for manufacturing high-performance ceramics [7,8] because it combines the advantages of hot pressing, pressureless sintering and hot isostatic pressing. TiCN is very attractive for cutting tools due to its lower friction coefficient than TiC [9,10]. Based on these facts and background, the densification behavior and mechanical properties of the gas pressure sintered Al_2O_3/TiCN composite were investigated in the present study.

2. Experimental

The alumina powder used in the present study had purity of 99.5%, a mean particle size of 1 μm, and an alpha-phase of above 90%. $TiC_{0.7}N_{0.3}$ (1 μm, 99% purity) was used as an additive. To investigate the effects of the sintering temperature, the sintering temperature is 1700, 1750, 1800, and 1850 °C, respectively.

The powder mixtures consisting of 70 Al_2O_3 and 30TiCN were mixed and ball milled in alcohol for 24 h with alumina medium. The powder mixtures were then dry-pressed into bars in a steel die at 100 MPa. The compacts were placed in a graphite crucible and sintered in a graphite furnace at 1700–1850 °C for 60 min under an argon atmosphere of 60 atm. The bulk density was measured using the Archimedes' principle. Without being ground and polished, the sintered bar specimens, 5 mm × 5 mm × 30 mm in size, were used directly for 3-point bending strength measurements at a crosshead speed of 0.5 mm/min, with a bottom span of 24 mm. Fracture toughness was measured by the indentation method. The surface was polished for Vickers indentation, and a load of 5 kg was used for 15 s to induce indentation. Fracture surfaces were observed using scanning electron microscopy (SEM, Hitachi S520), The grain structure was further studied using transmission electron microscopy (TEM, Philips CM20). The TEM samples were prepared as

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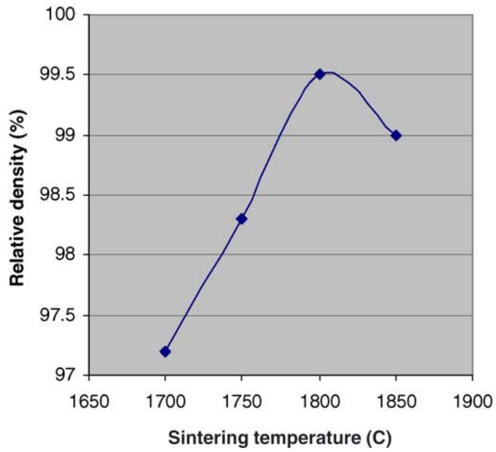

Fig. 1. Relative density as a function of sintering temperature for gas pressure sintered Al_2O_3–30 wt.%TiCN composite at a dwelling time of 60 min.

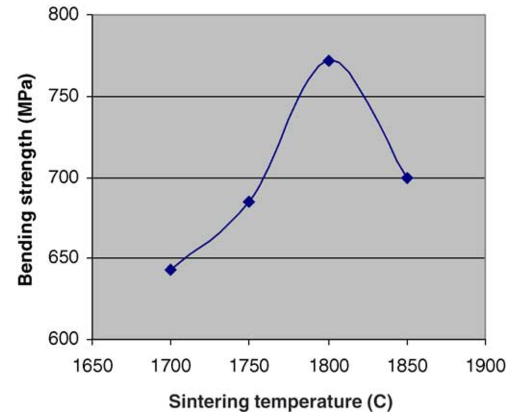

Fig. 2. Bending strength vs. sintering temperature.

following procedure. Slices of materials ~1000 μm thick were cut from the sintered bars. These slices were ground with diamond wheel to ~200 μm thick, and were then polished to ~120 μm thick. Discs with a diameter of 3 mm were cut from the polished slices using a supersonic drill with water and silicon carbide grinding medium. A Gatan dimple grinder was used to polish each side, leaving a central area ~20 μm thick. Finally the dimpled region was thinned to electron transparency using a Precision Ion Polishing System (Gatan 600, USA).

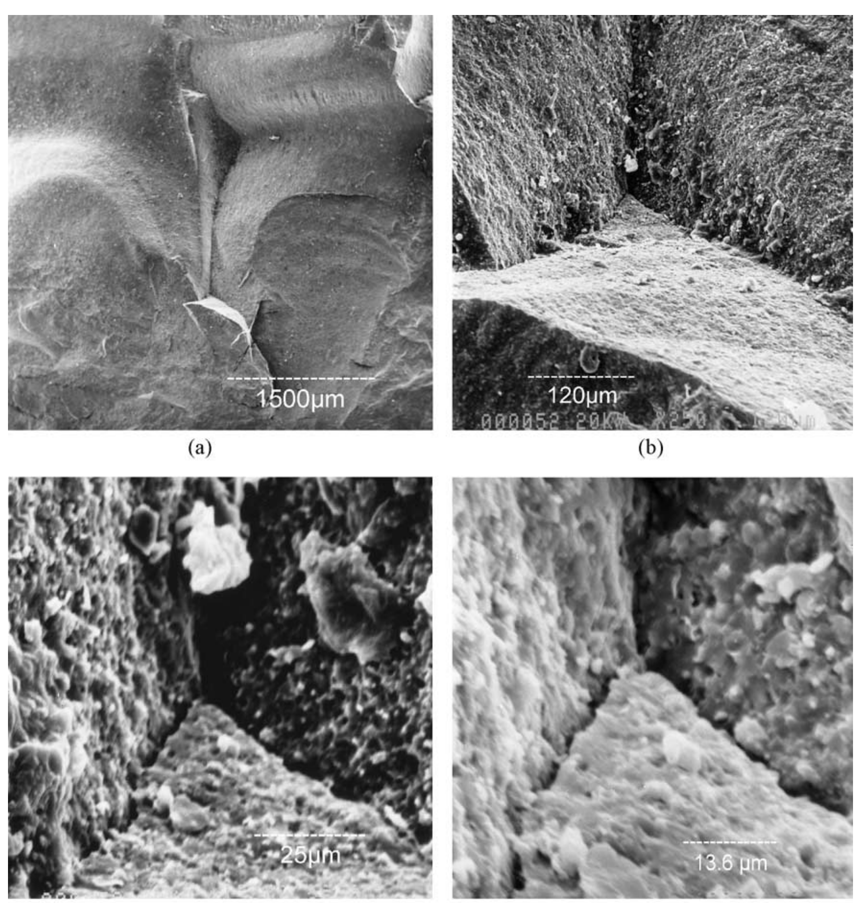

Fig. 3. SEM micrograph of the fracture surface of the Al_2O_3–30 wt.%TiCN composite gas pressure sintered at 1800 °C for 60 min.

3. Results and discussion

3.1. Densification behavior

Fig. 1 shows the relative density as a function of sintering temperature, for gas pressure sintered Al_2O_3–30 wt.%TiCN composite at a dwelling time of 60 min. The relative density initially increased from 97.3% to 99.5% with increasing the sintering temperature from 1700 to 1800 °C, the optimal sintering temperature is at 1800 °C, which corresponds to the highest relative density, and then decreased to 99% with increasing the sintering temperature to 1850 °C. 99.5% theoretical density obtained is excellent for gas pressure sintered Al_2O_3–30 wt.%TiCN composite.

3.2. Mechanical properties

Fig. 2 and shows the relations of bending strength versus sintering temperature. The trend of the curve is the same with Fig. 1. It is reasonable that the higher the relative density, the higher the strength. The strength increased from 643 to 772 MPa, the highest strength, with increasing sintering temperature from 1700 to 1800 °C, and then the strength decreased to 700 MPa with increasing temperature to 1850 °C. The relations of toughness and hardness versus sintering temperature are almost the same with Fig. 1 and Fig. 2. The highest toughness was 5.85 $MPa{\cdot}M^{1/2}$ and the highest hardness is 19.6 GPa, which corresponded to the highest strength and highest relative density for the samples gas pressure sintered at 1800 °C.

3.3. Microstructures

Fig. 3a shows a typical SEM micrograph of the fracture surface of Al_2O_3–30 wt.%TiCN composite sintered at 1800 °C for 60 min in argon at 60 atm.

The fracture surface is complex which often means high strength by absorbing more fracture energy; low strength often leads to simple fracture surface. The pictures at higher magnification (Fig. 3b–d) show that trans-granular fracture is predominant, which is the characteristic of high fracture strength.

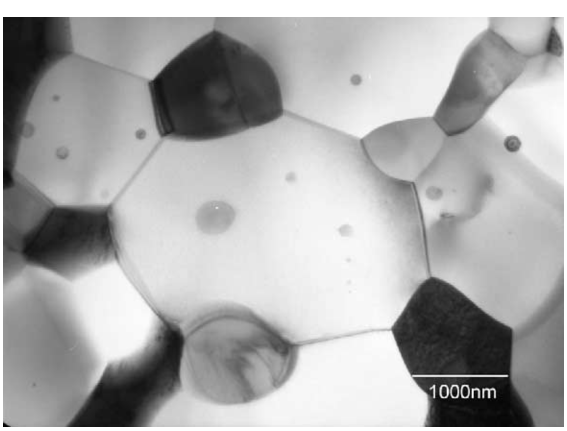

Fig. 4. TEM micrograph showing the microstructure of the Al_2O_3–30 wt.%TiCN composite gas pressure sintered at 1700 °C for 60 min.

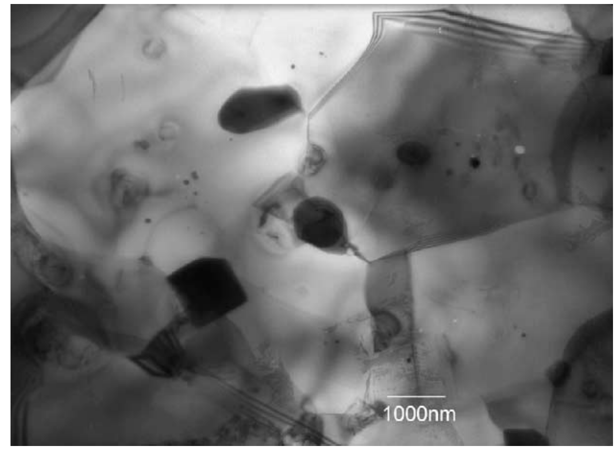

Fig. 5. TEM micrograph showing small TiCN particles dispersed in Al_2O_3 grains.

A typical TEM micrograph of the Al_2O_3–30 wt.%TiCN composite sintered at 1800 °C for 60 min in an argon atmosphere of 60 atm is shown in Fig. 4. The large bright irregular grains are Al_2O_3; the average grain size is about 3 μm. The small dark TiCN grains sized about 1 μm located at grain boundaries and triple points of alumina grains. Considering the particle size of 1 μm of the Al_2O_3 and TiCN powder used in the present study, Al_2O_3 grains grew about three times and little grain growth occurred for TiCN during sintering. It was observed that some very small TiCN particles in nanometer scale dispersed in Al_2O_3 grains (Fig. 5), which suggested that some TiCN might dissolve in Al_2O_3 matrix at high temperature during sintering and reprecipitated in Al_2O_3 grains after cooling down. The sintered Al_2O_3–30 wt.%TiCN composites were almost completely free from abnormal grain growth. All these resulted in high strength and toughness for the Al_2O_3–30 wt.%TiCN composite in the present study.

4. Conclusion

Al_2O_3–30 wt.%TiCN composite have been fabricated successfully by gas pressure sintering. The optimal sintering temperature is at 1800 °C, which corresponds to the highest relative density and mechanical properties. The gas pressure sintered Al_2O_3–30 wt.%TiCN composite achieved a relative density of 99.5%, a bending strength of 772 MPa, a hardness of 19.6 GPa, and a fracture toughness of 5.82 $MPa{\cdot}M^{1/2}$. Little grain growth occurred for TiCN during sintering while Al_2O_3 grains grew about three times from 1 to 3 μm.

Acknowledgements

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