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Infuluence of sintering technology on microstructure and mechanical properties of YAG/ZrB_2 ceramics

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Because ZrB_2 has some excellent physical performance and chemical stability, it has been widely applied in a lot of fields, but sintering of ZrB_2 is too difficult to be densified and easy oxidation at high temperature. To keep advantages and improve disadvantages of ZrB_2, the shell-core structure $A1_2O_3$-Y_2O_3/ZrB_2 composite powders are prepared by the co-precipitation methods, the high density YAG/ZrB_2 ceramics is prepared by the spark plasma sintering (SPS). The increasing ratio of mehanical properties is faster at sintering temperature from 1300 °C to 1700 °C, the increasing ratio of mehanical properties is faster under sintering pressure from 5 MPa to 20 MPa, the increasing ratio of mehanical properties is faster for holding time from 1 minute to 4 minutes. When the sintering conditons is 1700 °C, 20 MPa and 4 minutes, the Young's modulus and the fracture toughness are 430 GPa and 3.76 $MPam^{1/2}$, respectively. The YAG is coated on the surface of ZrB_2 crystal, that is to say, the YAG is show on the crystal boundary, which is help for the densification and the oxidation resistance at high-temperature condition.

Key words: ZrB_2, Microstructure, Mechanical property, Co-precipitation methods, Spark plasma sintering.

Introduction

Zirconium diboride (ZrB_2) has attracted substantial interest because of its extreme chemical and physical properties, such as, a high melting point, superior hardness and low electrical resistance. ZrB_2 has several applications such as for Hall-Heroult cell cathodes for electrochemical processing of aluminum, evaporation boats, crucibles for handling molten metals, thermowell tubes for steel refining, thermocouple sleeves for high-temperature use, nozzles, plasma electrodes, or as a dispersoid in metal and ceramic-matrix composites for heaters and igniters [1-4]. Yttrium aluminium garnet (YAG or $Y_3Al_5O_{12}$) adopts the cubic garnet structure, it is of great interest as a high-temperature engineering material, due to its high-temperature strength coupled with low creep rates, which indicates that YAG ought to be a suitable matrix and reinforcing material [5-8].

ZrB_2 has some extreme chemical and physical properties, however, it is difficult to sinter densification and easily oxide at high-temperature conditons. In order to show the advantage and improve the dis-advantage, the A1(OH)3-Y(OH)3 composite powder that the precursor of YAG was coated the surface of ZrB2 powder via the co-precipitation methods [9], the coated A12O3-Y2O3/ZrB2 composite powder was abtained to prepare the high-density YAG/ZrB2 ultra-high temperature with the shell-core microstructure. In this paper, the Infuluence of sintering technology on microstructure and mechanical properties of YAG/ZrB_2 ceramics prepared via SPS were investigated.

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Materials and Experiment

Commercially available ZrB2 powder (99.5% in purity) was used. Synthesized superfine Al2O3-Y2O3 composite powder with aluminum nitrate, yttrium nitrate and ammonia via the co-precipitation method was used. The Al2O3-Y2O3/ZrB2 composite powder was prepared with the Al2O3-Y2O3 composite powder and ZrB2 powder via a mechanical ball milling method. Then the Al2O3-Y2O3/ZrB2 composite powder was loaded into in a graphite mould with an inside diameter of 20 mm. The temperature was automatically raised to 600 °C, then monitored and regulated by an optical pyrometer aimed at the mould surface. A heating rate of 100 K · $minute^1$ between 600 °C and various chosen temperatures was performed, the graphite mould was removed, and YAG/ZrB2 ceramics were obtained. A process flow diagram is shown in Fig. 1. The YAG/ZrB2 ceramics sample is shown in Fig. 2.

Materials were sintered using the SPS method (Mode : SPS-1050, Japan), and the density was established with a precise electronic balance via the Archimedes' principle (Model : Sartorius BS210S, Germany) and compared to the YAG/ZrB2 theoretical density. Microstructuralanalysis was performed by scanning electron microscopy (SEM) (Model : JSM-5610LV, Japan).

The transverse wave and the longitudinal wave in the

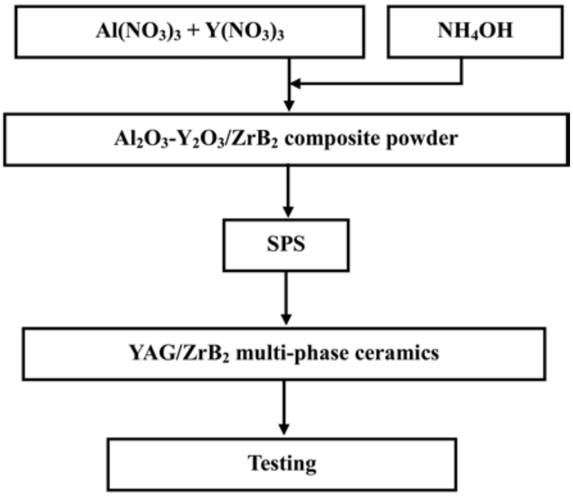

Fig. 1. The process flow diagram for preparing YAG/ZrB$_2$ ceramics.

Fig. 2. Picture of YAG/ZrB$_2$ ceramics sample.

sample were tested via the Ultrasonic pulse-echo method with Ultrasonic signal receiver (Model : Panametrics 5072PR, China), the Young's modulus of ceramics was counted with the formula (1).

$$E=\rho C_t^2\,\frac{3C_l^2-4C_t^2}{C_l^2-C_t^2} \tag{1}$$

where E is the Young's modulus of ceramics, ρ is the density of ceramics, C_t is the speed of transverse wave, C_l is the speed of longitudinal wave.

The fracture toughness of YAG/ZrB$_2$ ceramics were proformed via the indentation method with the micro-hardness instrument (Model: HX-1000, China), it is counted with the formula (2).

$$K_{IC}=0.016\left(\frac{E}{H_V}\right)^{\frac{1}{2}}\left(\frac{P}{C^{\frac{3}{2}}}\right) \tag{2}$$

where K_{IC} is the fracture toughness of ceramics, E is the Young's modulus of ceramics, H_V is the vickers hardness of ceramics, P is the pressure, C is the half length of crakle.

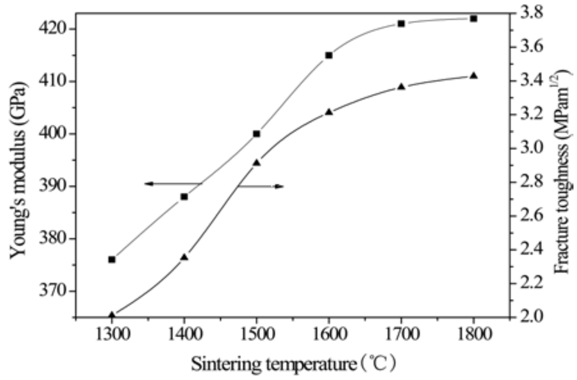

Fig. 3. Effect of sintering temperature on Young's modulus and fracture toughness of ceramics.

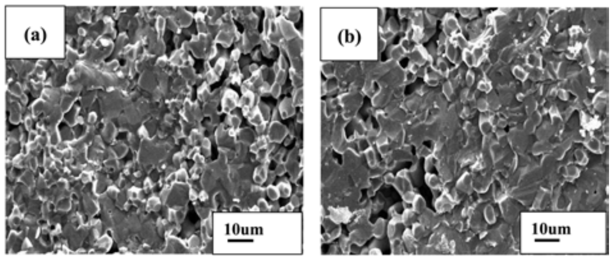

Fig. 4. SEM of ceramics under different sintering temperature (a-1500 °C and b-1700 °C).

Results and Discussion

Infuluence of sintering temperature

The infuluence of sintering temperature on the mechanical properties of YAG/ZrB$_2$ creramcs is shown in Fig. 3, which indicates that the Young's modulus and the fracture toughness are increased with an increasing the sintering temperature, the increasing ratio of mehanical properties is faster at sintering temperature from 1300°C to 1700°C, the increasing ratio of mehanical properties is slower at sintering temperature from 1700°C to 1800°C. The major cause is that the increasing ratio of density is faster at sintering temperature from 1300°C to 1700°C (Fig. 4), which is main densification processing during the sintering temperature from 1300°C to 1700°C. However, the change of density is unconspicuous at sintering temperature from 1700°C to 1800°C. So the sintering temperature is decided at 1700°C.

Infuluence of sintering pressure

The infuluence of sintering pressure on the mechanical properties of YAG/ZrB$_2$ creramcs is shown in Fig. 5, which indicates that the Young's modulus and the fracture toughness are increased with an increasing the sintering pressure, the increasing ratio of mehanical properties is faster under sintering pressure from 5 MPa to 20 MPa, the increasing ratio of mehanical properties is slower under sintering pressure from 20 MPa to 30 MPa. The density of ceramics is increased with an increasing the sintering pressure, the major cause is

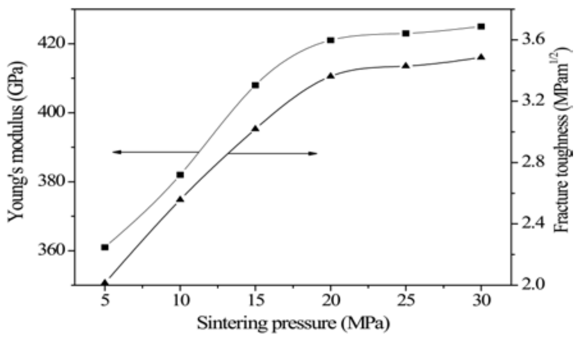

Fig. 5. Effect of sintering pressure on Young's modulus and fracture toughness of ceramics.

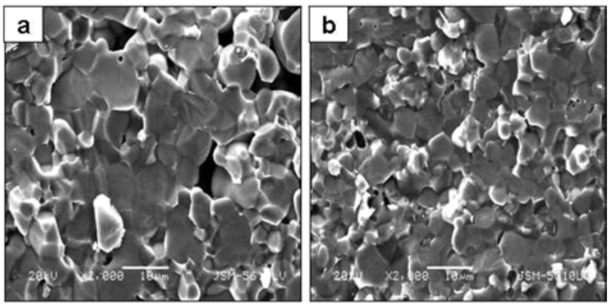

Fig. 6. Effect of sintering pressure on microstructure of ceramics (a-5 MPa and b-20 MPa)

that the increasing ratio of density is faster under sintering pressure from 5 MPa to 20 MPa (Fig. 6), which is main densification processing under sintering pressure from 5 MPa to 20 MPa, it make the pore of ceramics become few and small, these small pore can prevent the crackle expansionas in the ceramic body [10-11]. However, the change of density is unconspicuous under sintering pressure from 20 MPa to 30 MPa. So the sintering pressure is decided under the 20 MPa.

Infuluence of holding time

The infuluence of holding time on the mechanical properties of YAG/ZrB_2 creramcs is shown in Fig. 7, which indicates that the Young's modulus and the fracture toughness are increased with an increasing the holding time, the increasing ratio of mehanical properties is faster for holding time from 1 minute to 4 minutes, the increasing ratio of mehanical properties is slower for holding time from 4 minutes to 6 minutes. The density of ceramics is increased with an increasing the holding time, the major cause is that the increasing ratio of density is faster for holding time from 1 minute to 4 minutes (Fig. 8), which is main densification processing for holding time from 1 minute to 4 minutes. However, the change of density is unconspicuous for holding time from 4 minutes to 6 minutes. So the holding time is decided for the 4 minutes. When the sintering conditons is 1700°C, 20 MPa and 4 minutes, the Young's modulus and the fracture toughness are

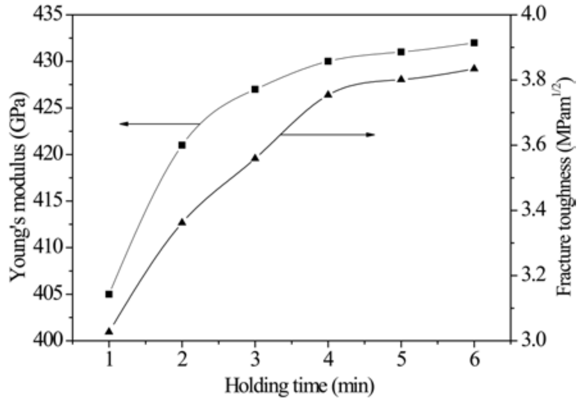

Fig. 7. Effect of holding time on Young's modulus and fracture toughness of ceramics

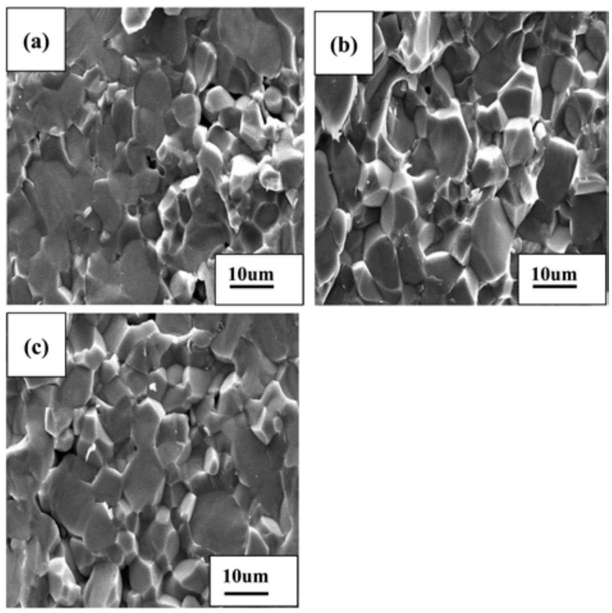

Fig. 8. SEM of ceramics under different holding time (a-2 min, b-4 min and c-6 min).

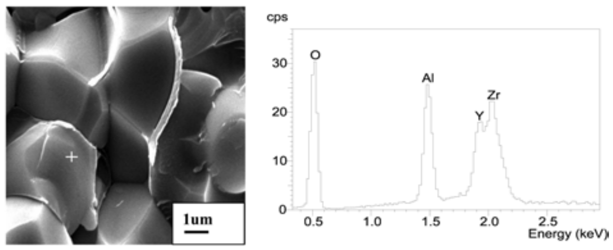

Fig. 9. SEM and EDS of ceramics sintered under 1700 °C, 4 min and 20 MPa.

430 GPa and 3.76 MPam1/2, respectively. The micro-structure of coated YAG/ZrB_2 multi-phase ceramics sintered under 1700°C, 4 min and 20 MPa is shown in Fig. 9, the EDS show that the YAG is coated on the surface of ZrB_2 crystal, that is to say, the YAG is show on the crystal boundary, which is help for the densification and the oxidation resistance at high-temperature condition.

Conlusions

The increasing ratio of mehanical properties is faster at sintering temperature from 1300°C to 1700°C, the increasing ratio of mehanical properties is faster under sintering pressure from 5 MPa to 20 MPa, the increasing ratio of mehanical properties is faster for holding time from 1 minute to 4 minutes. When the sintering conditons is 1700°C, 20 MPa and 4 minutes, the Young's modulus and the fracture toughness are 430 GPa and 3.76 $MPam^{1/2}$, respectively. The YAG is coated on the surface of ZrB_2 crystal, that is to say, the YAG is show on the crystal boundary, which is help for the densification and the oxidation resistance at high-temperature condition.

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