

Influence of sintering technology on microstructure and mechanical properties of ZrB₂-YAG-Al₂O₃ ceramics

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Abstract. ZrB₂, YAG and Al₂O₃ are widely applied because of some excellent performances, but ZrB₂ is easily oxidized in the high-temperature air. To keep advantages and improve disadvantages of ZrB₂, the shell-core structure Al₂O₃-Y₂O₃/ZrB₂ composite powders are prepared by the co-precipitation methods, the high density ZrB₂-YAG-Al₂O₃ ceramics is prepared by the spark plasma sintering (SPS). The mechanical properties of prepared ceramics under the 20MPa sintering pressure and 4min holding time are shown, which indicates the mechanical properties are increased with increasing sintering temperature, and the increasing ratio of mechanical properties is faster at sintering temperature from 1300°C to 1700°C. When the sintering condition is 1700°C, 20 MPa and 4 minutes, the Young's modulus and the fracture toughness are 421.8 GPa and 12.23 MPam^{1/2}, respectively. The reinforced phase (YAG-Al₂O₃) is coated on the surface of ZrB₂ crystal, that is to say, the YAG-Al₂O₃ is show on the crystal boundary, which is help for the densification and the mechanical properties.

Introduction

ZrB₂ is posed particular interest due to the unique property combination of high refractoriness, high-electrical and high-thermal conductivity, chemical inertness against molten metals or nonbasic slags and good thermal shock resistance [1], which is became candidates for hightemperature applications where corrosion-wear-oxidation resistance is demanded. ZrB₂ poses several applications such as Hall-Heroult cell cathodes for electrochemical processing of aluminium, evaporation boats, crucibles for handling molten metals, thermowell tubes for steel refining, thermocouples sleeves for high-temperature uses, nozzles, plasma electrodes, or as dispersoid in metal and ceramic-matrix composites for heaters and igniters. In addition, ZrB₂ is a metallic conductor with electrical resistivity comparable with those of their parent metals. This permits to produce complex components at reduced shaping costs. ZrB₂, because of its higher costs of production, has found a limited number of applications [2].

Yttrium aluminum garnet (YAG) with a chemical composition of Y₃Al₅O₁₂ is an important advanced structural and functional material. Because of its relatively stable lattice structure and large thermal conductivity, it is used as a host for solid-state laser materials in luminescence systems and in the window material for a variety of lamps. YAG also has great potential application as a high-temperature engineering material because of its good high-temperature strength as well as its superior creep resistance [3-4].

To keep advantages and improve disadvantages of ZrB₂, the shell-core structure Al₂O₃-Y₂O₃/ZrB₂ composite powders were prepared by the co-precipitation methods, the high density ZrB₂-YAG-Al₂O₃ ceramics is prepared by the spark plasma sintering (SPS) and the microstructure and mechanical properties were investigated.

Materials and experimental

Analytical grade of aluminum nitrate, yttrium nitrate, ammonia and commercially available ZrB_2 powder (99.5% in purity) were used. ZrB_2 particles were coated with Al_2O_3 - Y_2O_3 composite particles via the co-precipitation method. ZrB_2 -YAG- Al_2O_3 ceramics were prepared using coated Al_2O_3 - Y_2O_3 / ZrB_2 shell-core composite particles on the surface of ZrB_2 particles with spark plasma sintering (SPS).

ZrB_2 -YAG- Al_2O_3 ceramics were prepared with the SPS (Mode: SPS-1050, Japan). Phase analysis was identified by X-ray powder diffraction (XRD) (Model: D/Max-RB, Japan). Microstructure analysis was performed by scanning electron microscopy (SEM) (Model: JSM-5610LV, Japan). Mechanical properties of ZrB_2 -YAG- Al_2O_3 ceramics was tested.

Results and discussion

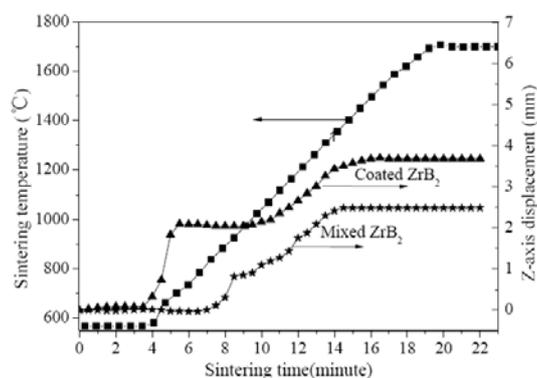


Fig.1 Sintering shrinkage curve of ZrB_2 -YAG- Al_2O_3 ceramics

The sintering curves for preparing 80wt% ZrB_2 -YAG- Al_2O_3 multiphase ceramic materials from different composite raw materials with the spark plasma sintering technique are shown in Fig.1. The Z-axis displacement of spark plasma sintering shows the shrinkage state of a ceramic body during the sintering process, when the value of the Z-axis displacement increases, this indicates the ceramic body is shrinking. The Z-axis displacement shows the shrinkage state of coated Al_2O_3 - Y_2O_3 powders and mixed YAG powders, respectively. The composite powders with Al_2O_3 - Y_2O_3 coated show a rapid shrinkage displacement from 700°C to 1000°C, where the second biggish shrinkage displacement also is shown from 1000°C to 1600°C, the Z-axis displacement is not varied basically above 1600°C. The sintering temperature curve is broken down into four parts, which include the preheating process below 700°C, the reaction process from 700°C to 1000°C, the sintering process from 1000°C to 1600°C, and the adjustment of the microstructure above 1600°C. The densification occurs mainly during the reaction process and sintering process, that is to say, the sintering processing is shown from 700°C to 1600°C. YAG is produced from 700°C to 1000°C[5-6]. The reaction temperature is lower than the 1100°C for synthesizing YAG powders from Al_2O_3 - Y_2O_3 composite powders, because ZrB_2 particles are changed electrically during the entire sintering process, which produces a plasma among ZrB_2 particles to purify the nearby particle surfaces and increase the sintering activity.

However, the Z-axis displacement for powders with YAG- Al_2O_3 added shows a lower shrinkage displacement below 1000°C, a larger shrinkage displacement is shown from 1000°C to 1600°C, the Z-axis displacement is not varied basically above 1700°C. The Z-axis displacement adding the different powders both show the same shrinkage state from 1000°C to 1600°C, YAG melts above 1000°C, the temperature is lowered because of the action of the plasma. Because the results of the two routes both show the Z-axis displacement is not varied basically above 1700°C, a sintering temperature of 1700°C is chosen for preparing high density ZrB_2 -YAG- Al_2O_3 multiphase ceramics (Fig.2), the microstructure of ZrB_2 -YAG- Al_2O_3 multiphase ceramics are shown in Fig.3, which indicates the density of ZrB_2 -YAG- Al_2O_3 ceramics with Al_2O_3 - Y_2O_3 composite powder coated is higher than that of ZrB_2 -YAG- Al_2O_3 ceramics with YAG- Al_2O_3 powder mixed (Fig.3-a, b). The reinforced phase of ZrB_2 -YAG- Al_2O_3 ceramics with Al_2O_3 - Y_2O_3 composite powder coated is

shown on the grain boundary (Fig.3-c), however, the reinforced phase of ZrB_2 -YAG- Al_2O_3 ceramics with YAG- Al_2O_3 composite powder mixed is shown among the crystal grain (Fig.3-d).

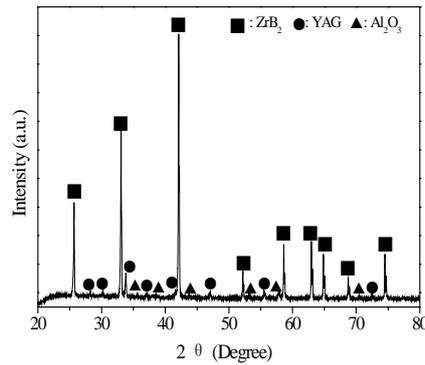


Fig.2 XRD of ZrB_2 -YAG- Al_2O_3 ceramics with Al_2O_3 - Y_2O_3 composite powder coated

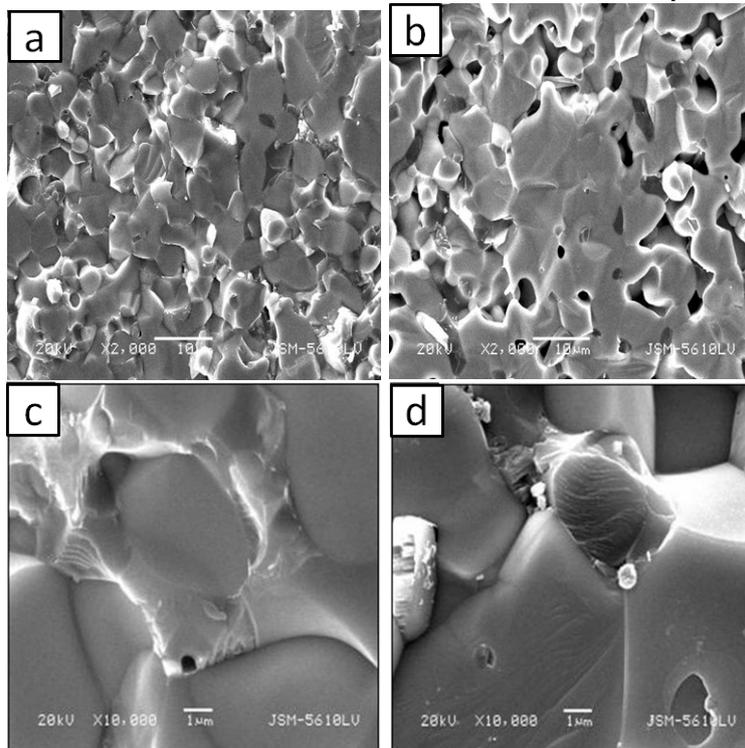


Fig.3 SEM of ZrB_2 -YAG- Al_2O_3 ceramics (a, c-Coated powders, b, d-Mixed powders)

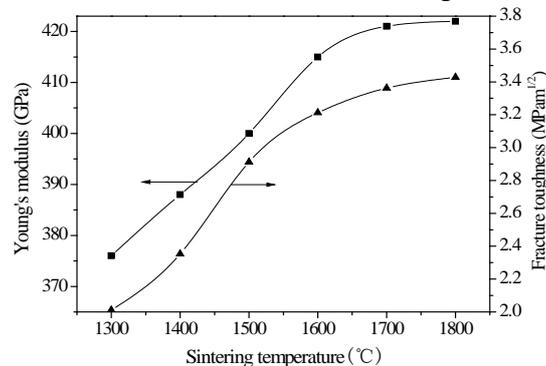


Fig.4 Effect of sintering temperature on Young's modulus and fracture toughness of ceramics

The mechanical properties of prepared ceramics under the 20MPa sintering pressure and 4min holding time are shown in Fig.4 and Fig.5, which indicates the mechanical properties are increased with an increasing sintering temperature, and the increasing ratio of mechanical properties is faster at sintering temperature from 1300°C to 1700°C. Because the main desifacation process is produced through the sintering temperature range, the density of ceramics is increased mainly, the Young's modulus and the fracture toughness are 421.8 GPa and 12.23 MPam^{1/2}, respectively. When the

sintering temperature is overed 1700°C, the Young's modulus and the fracture toughness are increased less, the sintering temperature is decided at 1700°C.

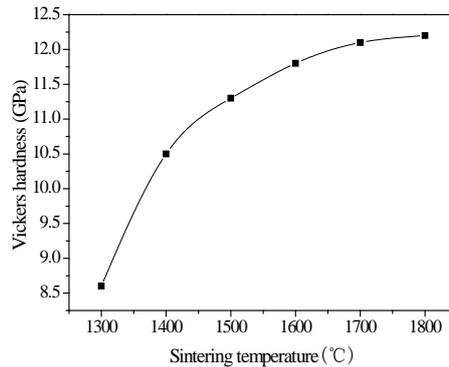


Fig.5 Effect of sintering temperature on hardness of ceramics

Conclusion

Through the all results and discussions, the conclusions are the 80wt%ZrB₂-YAG-Al₂O₃ multiphase ceramic materials posing different composite raw materials with the spark plasma sintering technique were successfully prepared. The mechanical properties of prepared ceramics under the 20MPa sintering pressure and 4min holding time are shown, which indicates the mechanical properties are increased with increasing sintering temperature, and the increasing ratio of mechanical properties is faster at sintering temperature from 1300°C to 1700°C. When the sintering conditons is 1700°C, 20 MPa and 4 minutes, the Young's modulus and the fracture toughness are 421.8 GPa and 12.23 MPam^{1/2}, respectively. The reinforced phase (YAG-Al₂O₃) is coated on the surface of ZrB₂ crystal, that is to say, the YAG-Al₂O₃ is show on the crystal boundary, which is help for the densification and the mechanical properties.

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