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Preparation of Al_2O_3–$Y_3Al_5O_{12}$–ZrO_2 eutectic ceramic by flash sintering

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ABSTRACT

Al_2O_3–$Y_3Al_5O_{12}$–ZrO_2 ternary eutectic oxide ceramic was prepared by flash sintering the corresponding powder. The highly dense sample was obtained at relatively low temperature and very short time with the assist of dc electric field. As-prepared ceramics well preserve the phase composition and microstructure of the original eutectic powder. The ceramic exhibits relatively high hardness and high fracture toughness. These results clearly demonstrate the feasibility of employing the flash-sintering technique to fabricate eutectic ceramics.

Eutectic oxide ceramics have attracted extensive recent attentions due to their excellent fracture strength/toughness, thermal stability and creep resistance at elevated temperatures [1]. These properties, attributed to the strongly eutectic interfaces between the different phases [1], make the materials very promising for high-temperature structural applications such as turbine blades. So far, eutectic ceramics are primarily prepared by directional solidification techniques, including Bridgman [2], laser floating zone [3,4], edge-defined film-fed growth [5], micro-pulling-down [6,7], laser zone remelting [8,9], and selective laser melting methods [10,11]. However, these directional methods are very time-consuming and difficult to fabricate large and complex parts. In order to overcome these problems, researchers have tried to make eutectic ceramics with indirect methods. For examples, the ceramics formed by directional methods were first crushed into powder, and then sintered to obtain bulk ceramics using conventional sintering [12], hot pressing [13], or spark plasma sintering (SPS) [12,14,15]. Although the indirect methods can be used to prepare large/complex parts, they usually require high temperatures and/or high pressures.

Recently, flash sintering, in which some oxide ceramics can be sintered in very short time at relatively low temperatures with the assist of applied electric field, has been developed [16]. Compared to other sintering methods, flash sintering exhibits many advantages such as low sintering temperature and short time [16–21]. While flash sintering technique has been applied to many oxides, such as zirconia and alumina [17,21], it has not been tried on eutectic ceramics yet. In this paper, we report the preparation of Al_2O_3–$Y_3Al_5O_{12}$ (YAG)–ZrO_2 ternary eutectic ceramic by flash sintering the corresponding powder. We have demonstrated that the ceramic can be consolidated by flash sintering to very high density, and exhibits reasonably good mechanical properties.

The starting Al_2O_3–YAG–ZrO_2 eutectic powder used here was prepared in our lab by laser zone remelting technique described previously [22]. In brief, the high purity nanopowders of Al_2O_3, Y_2O_3 and ZrO_2, at the molar ratio of Al_2O_3:Y_2O_3:ZrO_2 = 65.8:15.6:18.6, were uniformly mixed together using high-energy balling. The powder mixture was pressed into a cylindrical-shaped sample, which was then pressureless sintered at 1500 °C for 2 h. The sample obtained was converted to eutectic ceramic by laser zone remelting technique, in which a section of the sample was rapidly melted by the CO_2 laser beam. The melting zone moved along with the motion of the laser; meanwhile the previously melted section was solidified. When the laser beam moved from one end of the sample to the other, the entire sample was converted to the eutectic ceramic. The ceramic was finally ball-milled into the eutectic powder.

The obtained eutectic powder was pressed into a rectangular-shaped green body at a uniaxial pressure of 10 MPa. The green body was then pressureless sintered at 1300 °C for 2 h. After pre-sintering, the green body density is 3.09 g/cm^3. The dc (direct current) voltage was applied onto the sample via Pt wire which was enlaced on both ends of the sample. After the voltage was stabilized, the temperature was increased at a heating rate of 10 °C/min until the occurrence of the flash sintering. Upon completion of flash sintering, the power to the sample was disconnected. At the same time, the sample was cooled down to room temperature at a cooling rate of 5 °C/min.

The density of the sintered samples was measured using the samples cut from the gauge section by the Archimedes method with reagent-

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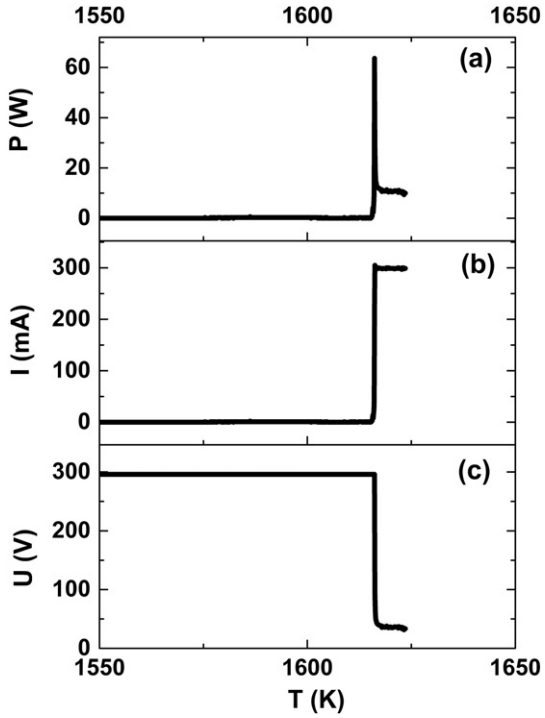

Fig. 1. Plots of power dissipation (a), current (b) and voltage (c) as a function of temperature for initial applying voltage of 495 V/cm and current limit of 0.3 A.

grade water as the medium. The crystal structure of the samples was identified by X-ray diffractometer (Rigaku D/max-2400, Tokyo, Japan) using CuKa ($k = 1.54$ Å) radiation. The microstructure of the sintered eutectics was observed by scanning electron microscopy (SEM, S-4700; Hitachi, Tokyo, Japan) on polished and thermally etched surfaces. The hardness and fracture toughness were determined using indentation technique (HMV-G20ST; SHIMADZU, Tokyo, Japan) on the polished surface of the composite, using the indentation load of 9.8 N for 15 s. More than ten indentations were made on each sample to obtain reliable data. The indentation size and crack length were measured from SEM images.

We have demonstrated that the eutectic powder can be flash sintered, evidenced by the presence of an abrupt increase in power dissipation, as well as quick densification. Fig. 1a shows a typical curve of power dissipation as a function of temperature. It is seen that when the applied electric field is 495 V/cm, the abrupt increase in the power dissipation occurs at about 1345 °C. Fig. 1b and c are plots of current and voltage as a function of temperature, respectively. It is seen that the current passing through the sample is very low before the onset of flash sintering, and then suddenly increased to the preset limit of 0.3 A at the flash-sintering onset temperature. Meanwhile the electric filed retains the same as 495 V/cm before the onset of the flash sintering, and rapidly drop to a much lower level to maintain the current (the power supply system is preseted so that it will be switched automatically to current control mode when flash-sintering occurs); indicating there is a sudden decrease in the resistance of the sample at the onset of the flash sintering. The same sintering procedure was also carried out at the applied electric field of 1000 V/cm and preset current limit of 0.15 A, in which the flash-sintering was found to occur at lower temperature of 1258 °C. This is consistent with previous report that flash sintering temperature decreased as the applied electric field increased [23].

The microstructure of the obtained ceramics was characterized using scanning electron microscopy (SEM). Fig. 2 compares the microstructure of the samples prepared at different conditions. It can be seen that the sample sintered at lower current limit (1000 V/cm–0.15 A, Fig. 2a, b) shows much more pores than that sintered at higher current (495 V/cm–0.3 A, Fig. 2c, d). The density measurement also indicates that the density (4.40 g/cm^3) of the sample sintered at 0.3 A is

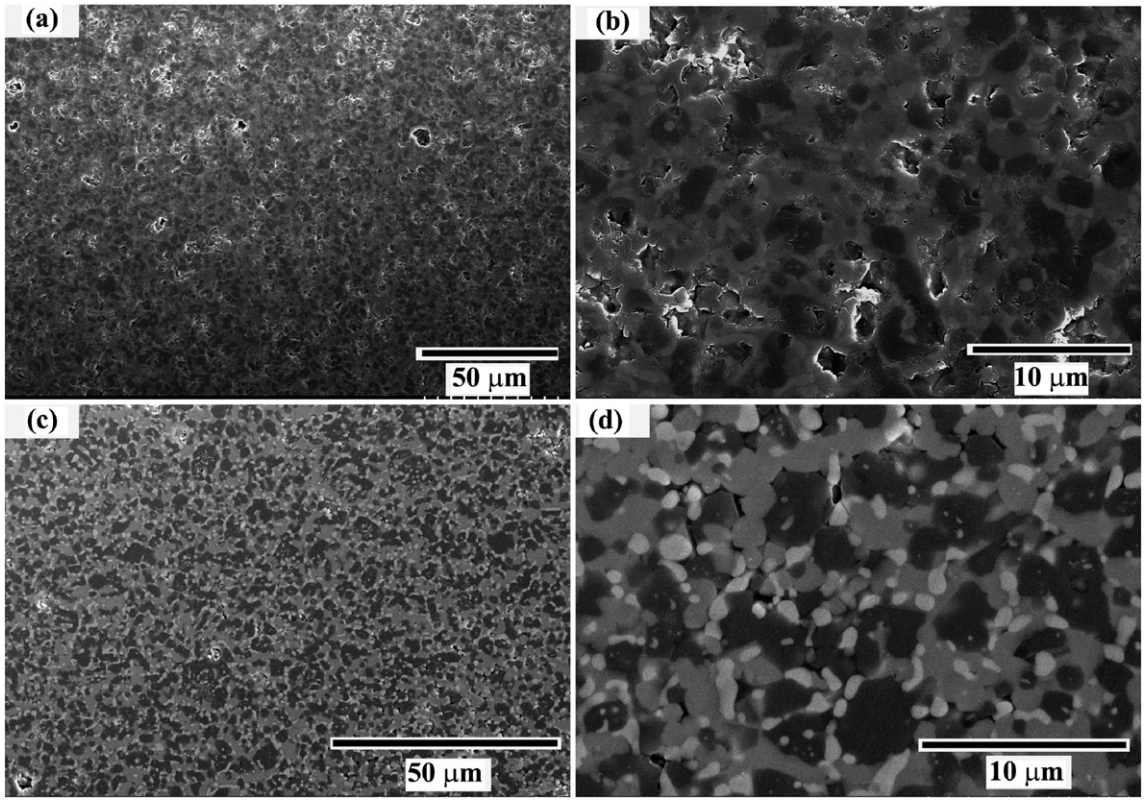

Fig. 2. SEM images showing the microstructures of the samples flash-sintered at 1000 V/cm–0.15 A (a, b) and 495 V/cm–0.3 A (c, d).