



Short communication

Effect of oxygen partial pressure on temperature for onset of flash sintering 3YSZ

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ABSTRACT

In this paper, it is demonstrated that in an oxygen-enriched environment, the oxygen partial pressure affects the onset temperature of flash sintering of 3 mol% yttria-stabilized zirconia (3YSZ). Flash sintering experiments were performed with oxygen partial pressures in the range of 0.25–1 atm. The results indicate that the onset temperature increased by increasing oxygen partial pressure. According to the plots of the conductivity as a function of temperature, the oxygen partial pressure might affect the onset temperature, by changing the conductivity in the pre-flash stage. Combined to the analysis of the law of mass action, it was established that electron conduction might also represent a critical parameter during the pre-flash stage of flash sintering, excluding the oxygen vacancy conduction.

1. Introduction

In recent years, flash sintering has attracted much scientific attention [1–9] due to the dramatically lower furnace sintering temperature and shorter sintering time compared to the conventional field-assisted sintering technology. As an example, flash sintering has been adopted to sinter 3YSZ in a few seconds at temperatures as low as 850 °C [10]. Until now, several ceramic green bodies with various conductive behaviors have been sintered by using flash sintering. These included the following: a MgO-doped Al₂O₃ insulator [1], an undoped Y₂O₃ insulator [11], a Co₂MnO₄ electronic conductor [2], cubic and tetragonal yttria-stabilized ZrO₂ ionic conductors [9,12–15], a gadolinium-doped BaCeO₃ protonic conductor [3], a CeO₂ ionic conductor [4], a SrTiO₃ semiconductor [5], a polymorphic SiC semiconductor [6], a SnO₂ semiconductor [7], Al₂O₃/ZrO₂ composites [16–18], a SiC_w/ZrO₂ composite [19] and a Al₂O₃-Y₃Al₅O₁₂-ZrO₂ ternary eutectic oxide ceramic [20].

To intensively investigate flash sintering behavior, it is critical to examine the reason to cause this behavior. Up to date, several possible mechanisms have been proposed for the beginning of flash sintering, including local heating of grain boundaries [10], nucleation of avalanches of lattice defects [9,10,17], and thermal runaway of joule heating [21]. To be rigorous, the mechanism should consist of an abrupt non-linear increase in conductivity and for the rapid densification immediately following the flash. The conductivity is controlled by

the fastest-moving charge-carrying species, while rapid densification is controlled by the slowest-moving species. Unfortunately, none of these mechanisms reasonably explain all of the phenomena.

It is acknowledged that the onset of flash sintering should meet the following two criteria [8]: (i) the applied field must be greater than a critical value and (ii) the critical value of power dissipation in the sample must be achieved below a threshold temperature. The power dissipation in the sample depends on the current density and the applied field in flash sintering. Therefore, it seems that current flow through a sample constitutes a significant parameter for flash sintering, which is related to the electrical conductivity, which derives from ionic and/or electronic conductivity. Hence, flash sintering is strongly related to the charge carriers, contributing to the electrical conductivity in the sample. This includes vacancies, interstitials, electrons and holes.

Thus, current research was based on the design of an experiment to further investigate the relationship between the onset temperature of flash sintering and the partial pressure of oxygen in an oxygen-enriched environment. Main objective of the research was to define the function of conductive species at the initial stage of flash sintering. Results indicated that high partial pressure of oxygen would lead to a high onset temperature during flash sintering. It was demonstrated that electron conduction might have a significant contribution in the pre-flash stage of flash sintering, excluding the oxygen vacancy conduction.

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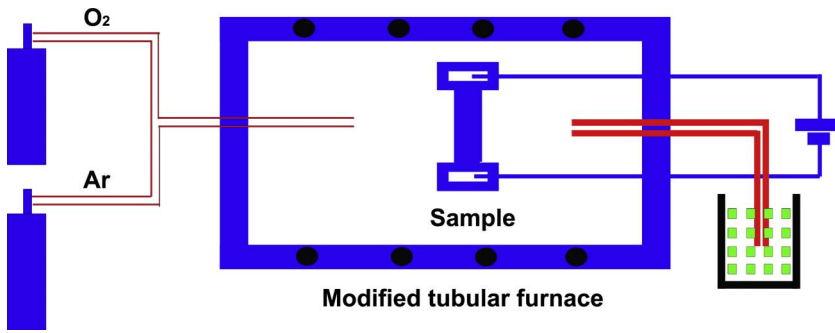


Fig. 1. Flash sintering apparatus using different atmospheres.

2. Experimental procedure

A diagram of the flash sintering apparatus is illustrated in Fig. 1. Samples were connected by using two platinum wires in a modified tubular furnace (GSL-1700-A2; KJ Group, Anhui, China). To improve the electrical contact between these platinum wires and the samples, Pt paste (PE-PT-7840; Sino-Platinum Metals Corp. LTD., Guizhou, China) was used. The furnace temperature was regulated to 1400 °C or less at a rhythm of 10 °C/min, until flash sintering occurred. The experiment was conducted by adopting different oxygen partial pressures: 0, 0.25, 0.4, 0.5, 0.6, 0.75, and 1 atm. For each given oxygen partial pressure, the temperature of onset of flash sintering, was recorded by adjusting the applied field at 80, 120 and 140 V/cm respectively for 3YSZ, by using a dc power source (Sorenson DLM-300; Sorensen, San Diego, CA). The power source was controlled by the limited current density after a power surge to prevent excessive joule heating on the specimen. Upon completion of flash sintering, the applied power on the specimen was interrupted. The specimen was then cooled to room temperature at a rate of 5 °C/min.

The initial ceramic powders were 3YSZ (Jirui Ceramic Technology Co., Ningbo, China). The powders were cold-pressed in a die at 160 MPa. The gauge section of the dog bones was 3.40 mm × 1.34 mm × 20 mm for 3YSZ. The compacted specimens were pre-sintered at 800 °C, using a ramp rate of 1 °C/min and that temperature was constant for 4 h. Subsequently, two holes of a diameter of 1.6 ± 0.1 mm were drilled on both ends of the specimen to connect it with the platinum wires. In addition, 3YSZ dense samples were also prepared using the same powder compacts by conventional sintering at 1500 °C for 2 h in a box furnace. The gauge section of the dense samples was 2.70 mm × 1.20 mm × 12.5 mm.

DC electrical conductivity for the 3YSZ green body and dense sample, was measured by using a multimeter (Keithley 2450). The conductivity test for 3YSZ was conducted in the temperature range of 500 °C and 1000 °C. Specific conductivity was calculated according to the following relation:

$$\sigma = \frac{I}{V} \frac{l}{wt} \quad (1)$$

where I equalled the measured current, V represents the applied voltage, l , w , and t correspond to the length, width, and thickness of the gauge section respectively. In addition, the conductivity plots for flash sintering were estimated according to the measured current and applied voltage by using equation 1.

3. Results and discussion

Fig. 2 presents typical estimated conductivity plots as a function of furnace temperature for different oxygen partial pressures at 140 V/cm. In this paper, the conductivity of green body corresponding to the onset temperature was defined to be 0.005 S cm⁻¹. According to Francis et al. [22], there is insignificant densification during the incubation time in flash sintering. Therefore, during the incubation stage, the geometry of

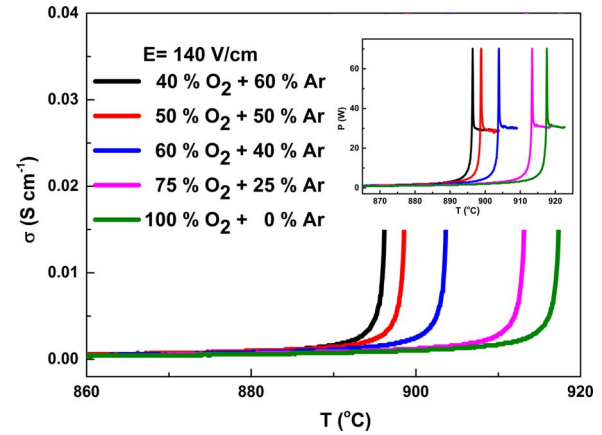


Fig. 2. Conductivity as a function of furnace temperature for different oxygen partial pressures at 140 V/cm. Inset: power dissipation versus temperature for different oxygen partial pressures at 140 V/cm.

the gauge section was simplified to be a constant. The curves obtained at all five values of oxygen partial pressure indicated the existence of a conductivity surge, similar to the characteristic behavior of flash sintering (see the inset in Fig. 1), as previously reported in the literature [22]. The curves also demonstrate that the onset temperature (the temperature when the surge occurred) of the samples increased by increasing oxygen partial pressure.

The onset temperature values of flash sintering 3YSZ for green body as a function of oxygen partial pressure are illustrated in Fig. 3. The onset temperature increased with the increase in the oxygen partial pressure. As an example, by applying the field of 140 V/cm, the onset temperature increased from 895 °C (0.4 atm) to 916 °C (1 Atm). To rule out the effect of the densification during flash sintering, the onset temperature values of flash sintering 3YSZ corresponding to the dense

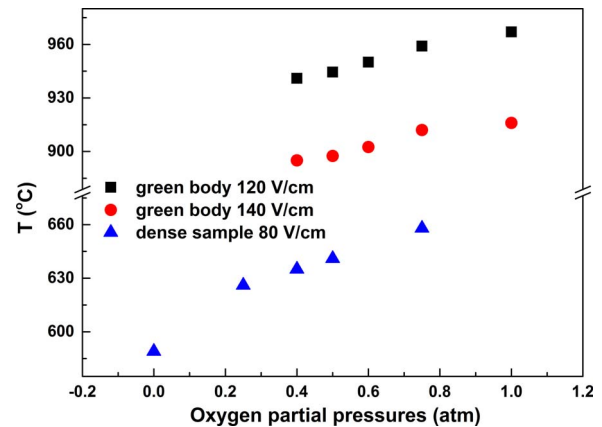


Fig. 3. The onset temperature of flash sintering as a function of the oxygen partial pressures.

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