JOURNAL OF MATERIALS PROCESSING TECHNOLOGY 209 (2009) 2446–2452

journal homepage: www.elsevier.com/locate/jmatprotec

Improving sinterability of ceramics using hybrid microwave heating

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ARTICLE INFO

Article history:
Received 18 February 2008
Accepted 24 May 2008

Keywords:
Microwave sintering
Ceramics
Thermal residual stress
Pore ratio
Hardness

ABSTRACT

Microwave processing, as a new method for sintering ceramics, has key advantages such as increased heating rate, uniform heating and reduced cost compared to conventional methods. It is generally accepted that microwave sintering can improve the macroscopic mechanical performances of ceramics, however, the performances of microwave-sintered ceramics on the microscopic scale are rarely investigated. In the present study, the ceramics are sintered by hybrid microwave sintering (HMS), which combines the characteristics of microwave heating and conventional heating. To evaluate the homogeneous performance of the sintered ceramics, the behaviors of thermal residual stress distribution in the microwave-sintered and conventionally sintered ceramics were investigated by X-ray diffraction technique. The thermal residual stress investigation shows microwaves can sinter ceramics in entire volume while offering improved mechanical properties. Subsequently, the distribution behaviors of pore ratio and hardness in the ceramics were investigated, respectively. The experiment results confirm that the sinterability of ceramics is homogenously improved by hybrid microwave sintering.

1. Introduction

Microwave heating is a process in which the materials couple with microwaves, absorb the electromagnetic energy volumetrically, and transform it into heat. This is different from conventional methods such as electric heating or vapor heating, in which heat is transferred between objects by the mechanisms of conduction, radiation and convection. In conventional heating, the material's surface is first heated followed by the heat moving inward. This means that there is a temperature gradient from the surface to the inside. However, microwave heating generates heat within the material first and then heats the entire volume (Yadoji et al., 2003). This heating mechanism is advantageous to rapid heating rates, uniform heating, selective energy absorption, high efficiency and reduced costs (Yadoji et al., 2003; NMAB, 1994; David et al., 2000). In recent years, microwave heating has been well employed in the sintering and joining of ceramics. It has been demonstrated that microwave sintering has the potential of enhanced densification and suppressed grain growth due to a fast heating rate and apparent low-temperature firing (Tsay et al., 2004; Clark et al., 1997). In the previous studies (Gupta and Wong, 2005; Mizuno et al., 2004; Travitzky et al., 2000), researchers have evaluated the advantages of the performance of microwave-sintered ceramics just on a macroscopic scale, focusing on tensile strength, elastic modulus, toughness and so on, and found overall mechanical performance is improved by microwave sintering. However, the performances of microwave-sintered ceramics on a microscopic scale are rarely investigated. In addition, the sinterability of ceramics

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doi:10.1016/j.jmatprotec.2008.05.037

is generally evaluated by measuring the relative densification. It should be noted that densification is difficult to estimate at a local scale, which leads to difficulties in characterizing the homogenous sinterability of microwave-sintered ceramics (Boch and Lequeux, 1997).

In the present study, Al_2O_3 ceramics are sintered by hybrid microwave heating and conventional heating method, respectively. The behaviors of thermal residual stress and the pore ratios were investigated, and utilized to characterize hybrid microwave-sintered specimens and conventionally sintered specimens. The characteristics of two-directional hybrid microwave sintering (HMS) were also confirmed by using thermal residual stress and the pore ratio distribution. Otherwise, Vickers hardness (HV) distribution was also investigated. The advantages of the performance of hybrid microwave-sintered ceramics are evaluated on the microscopic scale, and the experimental results confirm that the sinterability of ceramics is improved by hybrid microwave sintering.

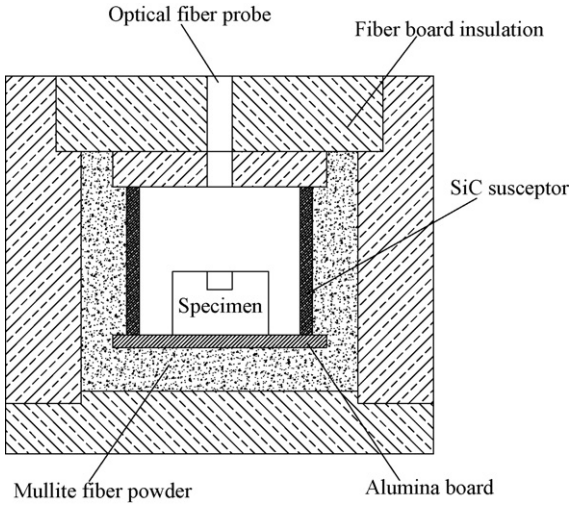

Fig. 1 – Hybrid microwave heating system.

2. Experiment

2.1. Sample preparation

High-purity Al_2O_3 (purity > 99.5%) powder was used as a raw material. The $MgO–Al_2O_3–SiO_2$ system sintering additives, which consist of 21.5 wt.% MgO, 61 wt.% SiO_2, and 17.5 wt.% Al_2O_3, were employed to control the microstructure and decrease the sintering temperature (Singh, 1981; Nakajima and Messing, 1998). The average size of the powders is about 1 μm. Al_2O_3 powder and sintering additives were mixed with organic binder, and consolidated by uniaxial pressing into disks (35 mm diameter × 15 mm thickness) at 130 MPa. The green compacts had a density of approximately 60%.

2.2. Hybrid microwave sintering and conventional sintering

Compacts formed above were sintered by microwave and conventional processing respectively. The 2.45 GHz microwave sintering system, which consists of a rectangular multimode cavity, a continually adjustable power supply (0.5–2.7 kW) and an insulation system, was used for microwave sintering experiment. Due to its low dielectric loss factor at room temperature and lengthy heating time, Al_2O_3 is difficult to heat via microwave radiation (Peelamedu et al., 1999; Zhao et al., 2000). However, Al_2O_3 begins to absorb microwave energy intensely after arriving the coupling temperature about 700 °C. Therefore, SiC is used as a susceptor in the microwave furnace to raise the temperature of Al_2O_3 to microwave coupling temperature because of its high dielectric loss factor and excellent refractory properties (Peelamedu et al., 1999; Zhao et al., 2000). The microwave sintering technique using susceptors is termed "hybrid microwave sintering" (Zhao et al., 2000). To enhance the sintering efficiency, a special hybrid heating system was used. SiC plates with thicknesses of 6 mm were set around the Al_2O_3 sample to initially heat it at a relative lower temperature. The temperature of specimens was measured by a far infrared fiber optic pyrometer with a range of 550–2000 °C. The arrangement of the sample, the SiC susceptors and the thermal insulations are shown in Fig. 1. In the conventional sintering experiment, the electric heating furnace was used, and a heating rate of 5 °C/min and a holding time of 4 h at 1480 °C were employed in this work.

2.3. Thermal residual stresses measurement

The thermal residual stresses in the specimens caused by the sintering were measured by X-ray diffraction (XRD) (Suzuki and Tanaka, 1999). The most important conditions for X-ray stress measurement are summarized in Table 1 (Suzuki et al., 1989).

Residual stress is obtained from a gradient of a straight-line obtained from $\sin^2\psi$ diagram. The $\sin^2\psi$ diagram is drawn by measurement of the Bragg angle at 2θ every tilt angle ψ. In plane stress state, the stress σ can be expressed as

$$\sigma = KM\,(\mathrm{MPa}) \tag{1}$$

$$K = -\frac{E}{2(1+\upsilon)} \times \cot\theta_0 \times \frac{\pi}{180}\,(\mathrm{MPa}/deg) \tag{2}$$

$$M = \frac{\partial(2\theta)}{\partial(\sin^2\psi)}\,(deg) \tag{3}$$

Table 1 – Conditions for X-ray stress measurement

Characteristic X-ray	Fe-Kα
Diffraction plane	2 1 10
Filter	Mn foil
Tube voltage	30 kV
Tube current	10 mA
Stress constant	−458 MPa/deg