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Feature article

Rapid heating of zirconia nanoparticle-powder compacts by infrared radiation heat transfer

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ABSTRACT

Homogeneous rapid sintering of nanoparticle powder compacts of yttria-stabilized zirconia was achieved by the radiation heat transfer. Green bodies were prepared by cold isostatic pressing (CIP) at various pressures providing different porosity of samples before sintering. Pressure-less sintering was performed in air at a heating rate of 100 °C/min up to the 1500 °C/1 min. Scanning electron microscopy, mercury intrusion porosimetry, and Archimedes technique were used to characterize the microstructure and to determine the density of the green and sintered bodies. Contrary to expectations, our results reveal opposite dependence of the green- and sintered densities on the CIP pressure. Since the whole sintering process does not exceed 10 min, to propose what processes are responsible for observed results, our attention is focused on the radiation heat transfer from furnace heating elements into the ceramics. Our arguments are supported by numerical calculations of the electromagnetic field enhancement in/between particles.

1. Introduction

Ceramic materials have generally unique mechanical (e.g. hardness, wear resistance, compressive strength, etc.) and functional (bioactivity/bioinertness, optical transparency, ionic conductivity, etc.) properties. To produce high-quality homogeneous and crack-free ceramic materials conventional sintering techniques have been used for many decades. From the physics point of view, these techniques are based on heat transfer from the heating elements into the heated material via radiation, convection, and conduction. However, a thermal expansion mismatch between places with different temperatures due to the low thermal conductivity and high thermal expansion can occur. Therefore, heating rates have to be small enough to prevent the formation of cracks within the sintered material due to strong negative temperature gradients when going from surface to deep in material. This brings huge energy consumptions and a relatively slow production. Therefore, great efforts have been made to develop novel techniques for sintering at higher heating rates that can ensure the absence of defects and gradients of material properties (density, grain size, etc.) inside the sintered bodies.

During last three decades spark plasma sintering (SPS) or pulsed electric current sintering (PECS) has been widely used for rapid sintering of ceramic materials [1]. However, there are still discussions on what kind of heating processes takes place during PECS. It was believed that together with Joule heating energy transfer from the pulsed electric current also activates ionization at the point contacts between particles [2]. Afterwards, Anderson et al. brought suggestion that the pulsed current supports the sintering effect by permanent cleaning of particle surfaces from oxides [3]. Plasma formation in compacted conductive powders treated by SPS was also observed [4]. Contrary to that, evidences that no plasma is present during SPS process were brought too [5]. Recently, rapid sintering of crack-free zirconia ceramics by pressure-less spark plasma sintering has been reported [6].

Besides SPS, microwave sintering, as another promising method of rapid sintering, has attracted attention. This technique is based on a strong absorption of the energy of electromagnetic field with frequencies from 300 MHz up to 300 GHz in the material [7]. However, surface heat losses result in opposite temperature gradients comparing with those by heating via conduction/convection.

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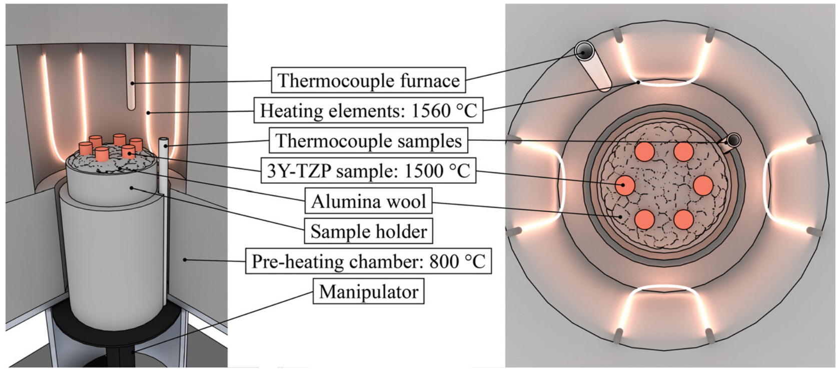

Fig. 1. Rapid sintering furnace with a connected pre-heating chamber. To minimize thermal conductivity from the sample holder the green bodies were supported with alumina wool.

There were attempts to solve this problem by a microwave hybrid heating: the ceramic powder is simultaneously heated by a conventional heating procedure to balance both gradients [8,9]. Furthermore, in case of many ceramic materials electromagnetic absorption strongly increases with temperature which leads to better coupling of microwaves with the material. For instance, according to Zhang et al. yttria-stabilized zirconia demonstrates a significant increase of ionic conductivity above 800 °C [10]. Another way how microwaves may interact with the ceramic material is a non-thermal acceleration of mass transport resulting in a significant increase of vacancy diffusion [11]. This phenomenon is called the "microwave ponderomotive effect".

In this paper, we report our experimental results on homogeneous rapid pressure-less sintering of nanoparticle powder compacts of yttria-stabilized zirconia using only radiation heating from elements heated up to 1833 K (frequencies of the electromagnetic radiation from 30 THz to 300 THz). Contrary to expectations, these results reveal an opposite dependence of the green- and sintered densities on the pressure of cold isostatic pressing under which the green bodies were prepared. The whole sintering process took less than 10 min which is not sufficient for conduction/convection heating of the ceramic powder up to a desired temperature, as we have recently demonstrated [12]. Therefore, to provide the physical insight into basics of this sintering process, we focused our attention on the radiation heat transfer from furnace heating elements into the ceramics. Thus, to elucidate what processes may take place, calculations of near (or localized) electromagnetic field induced in/between particles are performed. Considerable enhancement of the local electromagnetic field in the vicinity of nanoparticles (e.g. nanospheres, nanorods, nanoantennas, etc.) is one of intensively studied topics by plasmonics [13]. Several analytical approaches to determine the field near an irradiated nanoparticle of various shapes have been suggested, see e.g. Refs. [14,15]. However, here we use the finite element numerical calculations to map the field between ceramic particles. Results of these calculations indicate weakening of the field enhancement at the points of contact during development of bridges between them. Furthermore, the influence of the field enhancement between particles on contamination of their surface is discussed as well.

2. Experimental

2.1. Green body preparation

Samples were prepared from a commercially available nanoparticle powder of tetragonal zirconia with 3 mol% Y_2O_3 (3Y-TZP), the average green particle size was 80 nm (grade TZ-3Y, Tosoh Co., Japan). Before the sintering process the powder was formed into green bodies by compacting it with uniaxial pressing at 25 MPa followed by cold isostatic pressing (CIP) at four distinct pressures: 50, 100, 200, and 300 MPa. By this procedure the disk-shaped green bodies (weight about 6 g, diameter ca 15 mm, thickness 6.2–7.0 mm depending on the powder compaction) were fabricated. Organic compounds being present in the powder "as received" were burnt out in air at 600 °C for one hour.

2.2. Sintering process

To minimize the thermal conductivity from the sample holder all green bodies were supported with alumina wool. Firstly, several green bodies were put into the pre-heating chamber connected to the special fast heating furnace (Clasic Ltd., Czech Republic) (see Fig. 1) both containing atmospheric air. Since the onset temperature for sintering of 3Y-TZP is higher than 870 °C [16], the temperature of green bodies in the pre-heating chamber was set to approximately 800 °C to suppress the sintering process here. From the pre-heating chamber the green bodies were lifted via a manipulator to the furnace where the final sintering temperature was set to 1500 °C with a dwell time of 1 min. Both the heating- and cooling rates were 100 °C/min (controlled by a thermocouple). Therefore, the duration of the entire pressure-less sintering process was shorter than 10 min.

2.3. Evaluation of properties of green bodies and sintered ceramics

The Archimedes technique (EN 623-2) was used to determine the densities of the green bodies and sintered material. All values of the relative density were calculated with respect to a value of 6.08 g/cm^3 being the theoretical density of tetragonal zirconia 3Y-TZP. The pore size distribution was evaluated by mercury intrusion porosimetry (Pascal 440, Porotec, Germany). The green and sintered grain size was determined by using scanning electron microscopy (SEM), Lyra 3 XMH (TESCAN Ltd., Czech Republic).

3. Results

3.1. Pore structure of the green bodies

As expected, the green bodies prepared by CIP with lower pressures exhibited a higher number of larger pores, as it can be seen from the pore-size distribution in Fig. 2(c). During detailed inspection of SEM images we focused our attention to pores with dimensions comparable or slightly lower than the average particle size (80 nm). These pores are formed by clustering 5 or 6 particles touching each other. The respective SEM images are shown in

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