



Role of conduction and convection heat transfer during rapid crack-free sintering of bulk ceramic with low thermal conductivity



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ABSTRACT

Rapid sintering is nowadays a domain of novel methods such as spark plasma sintering (SPS) or flash sintering. These methods deal with special heating and, therefore, it is difficult to describe obtained results by a conventional pressure-less sintering mechanism. This work deals with specially designed pressure-less rapid sintering furnace, which allows heating rates on level of hundreds degrees per minute. Sample with volume over 30 cm³ from low thermally conductive tetragonal zirconia stabilized with 3 mol% Y₂O₃ (3Y-TZP) was rapidly sintered to relative density of 86% without crack formation. Experimental data were used for numerical calculations of conduction/convection heat transfer. Obtained results reveal that the maximum temperature in the sample does not exceed 1200 °C if only heating by conduction and convection is considered. Our results indicate that during rapid sintering of low thermally conductive materials radiation heat transfer is dominant in both conventional and SPS conditions.

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1. Introduction

Rapid pressure-less sintering of ceramics with low thermal conductivity such as tetragonal yttria-stabilized zirconia is usually limited by slow heat transfer. Conventional pressure-less sintering allowed heating rate only 7 °C min⁻¹ during sintering of tetragonal zirconia stabilized with 3 mol% Y₂O₃ (3Y-TZP) to prevent crack formation [1]. An alternative rapid sintering technique, hybrid microwave heating allows heating rate of 20 °C min⁻¹ without any observed damage to the 3Y-TZP samples [1]. Faster heating rates during the conventional rapid heating can lead to temperature gradients and, therefore differential densification, low final density, gradient structure, or a specimen crack formation. These difficulties can be overcome by pressure assisted rapid sintering techniques, such as the spark plasma sintering (SPS) method [2] or high-frequency induction heated sintering (HFIHS) [3]. Both techniques permit heating rates up to 200 °C min⁻¹ or even higher without a crack formation of zirconia ceramics, but the applied mechanical pressure during sintering influences a possible crack formation.

However, our previous work revealed an unique possibility of sintering crack-free 3Y-TZP ceramics by pressure-less SPS with heating rates up to 500 °C min⁻¹ with no observable grain size

gradients [4]. Presented results seem to be in contradiction with density gradient previously observed during typical fast pressure-less sintering of zirconia ceramics, which indicate a requirement of high thermal conductivity for rapid sintering in pressure-less conditions [5]. However, several reports confirmed unique behavior of various materials during pressure-less SPS. Strong necking of porous materials have been observed for calcium phosphate [6] and silicon nitride [7]. Densification rate comparable with microwave sintering was reported for ZnO₂ fine powder [8], but presence of carbon lead to formation of surface patterns as an evidence of surface reaction of ZnO. The term “SPS conditions” includes environment with presence of strong electromagnetic field, low gas pressure (approximately 5 Pa), and presence of carbon. The fast radiation heat transfer is a possible explanation of unique sintering performance of SPS and, therefore, it is desirable to perform rapid sintering in a conventional furnace.

However, maximum heating rate in conventional high-temperature furnaces does not allow rapid sintering. Therefore, a modification of the heating process is required to achieve heating rate 100 °C min⁻¹ typical for SPS. Outside SPS furnace there are three possible heat transfer mechanisms conduction/convection and radiation, while generally applied slow heating of zirconia ceramics favors heat conduction/convection. However, the influence of a heat transfer mechanism on rapid sintering of ceramic materials with low thermal conductivity is not clear, and it has main influence on sintering and a crack formation.

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Aim of this work is to inspect the role of conduction and convection heating during rapid sintering of zirconia ceramics in conventional pressure-less conditions. Rapid heating of a large sample (over 30 cm^3) of 3Y-TZP was performed in especially designed furnace to prove that the crack-free sintering can be performed also under conditions different from SPS. The main difference between the SPS conditions and our design of the experiment consist in the heat transfer by conduction and convection into the sample. Therefore, heating by conduction and convection

was numerically simulated (COMSOL Multiphysics Software) with input data based on experimental results.

2. Experimental

Cylindrical zirconia samples were prepared by compaction of green powder of commercially available 3 mol.% Y_2O_3 partially stabilized ZrO_2 powder (3Y-TZP, average mean particle size 80 nm, Tosoh Co., Tokyo, Japan). Ceramic powder (92 g) was placed in the cylindrical rubber mold and consequently compacted by pressure



Fig. 1. Schematic of the furnace with the moving bottom table used for controlled insertion of the sample into the hot zone. Right down corner: the sample after rapid sintering.

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