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ScienceDirect

Ceramics International 41 (2015) 7910–7915

CERAMICS INTERNATIONAL

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Microwave sinter forging of alumina powder

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Received 8 January 2015; received in revised form 18 February 2015; accepted 24 February 2015
Available online 4 March 2015

Abstract

Microwave sintering under load is expected to be a promising technique to process ceramics with fine microstructure. This paper presents a new setup allowing sinter forging ceramic compacts in a 2.45 GHz single-mode microwave cavity. This setup has the following features: maximum temperature of 1600 °C, heating rate between 1 and 250 °C min^{-1}, maximum stress of 50 MPa applied upon an 8 mm diameter sample. A specific protocol has been defined to calibrate the pyrometer used to measure the sample temperature. Alpha-alumina compacts have been microwave sinter forged under various stresses in the range 0–30 MPa. The results are compared to those obtained in a conventional furnace under 0, 4 and 8 MPa. Final axial and radial shrinkages are identical with the two techniques. The final relative density of the material is not affected by load and is equal to 0.94 and 0.96 in microwave sinter forging and conventional sinter forging, respectively. Two assumptions are proposed to explain this difference: a microwave effect and a temperature discrepancy. Finally the load does not significantly affect grain growth.

Keywords: A. Sintering; Microwaves; Forging; Pyrometer; Alumina

1. Introduction

Ceramics are widely used in the aeronautic and biomedical fields, to manufacture thermal barriers, gas burner nozzles, ballistic protection, implants, prostheses, and functionalized biomaterials [1–4] for examples. Rapid manufacturing of dense ceramics with fine microstructure and high mechanical properties is of great interest for such applications. Hot pressing, hot isostatic pressing, sinter-forging, spark plasma sintering [5], flash sintering and microwave sintering are some of the techniques proposed as alternatives to conventional sintering to improve ceramic processing and properties. Microwave sintering is particularly relevant because it allows rapid volumetric heating with high production rate and low energy consumption. This technique has been used to sinter ceramics, such as Al_2O_3 [6–14], ZrO_2 [15,16], Al_2O_3/ZrO_2 composites [17,18], and hydroxyapatite [19]. Several studies tried to compare microwave and conventional sintering of pure alumina. However, comparison of one study with another in terms of final density and microstructure is difficult, because different powders or heating cycles are used. Some authors [13,14] found that the densities obtained after microwave sintering of pure alumina are equal to densities after conventional sintering. Other authors [6,7,12–14] show that densities of doped alumina are higher with microwave sintering than with conventional sintering. Moreover, Xie et al. [6,7] found that the grain size is larger in microwave sintering than in conventional sintering for alumina while the same authors [6] found the opposite for zirconia.

Several processes involve the application of a load upon the compact during sintering. If the compact is inside a die, the technique is called hot pressing. It is called sinter forging when the lateral surfaces of the compact are free. Hot pressing allows manufacturing high density materials with fine microstructure. The main disadvantages of this process are its high cost and limited capacity to fabricate complex shape parts [20,21]. Sinter-forging is a reference process to reduce flaws and grain size of ceramics and to increase their strength and toughness

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http://dx.doi.org/10.1016/j.ceramint.2015.02.130
0272-8842/

[22–26]. The load applied on the sample allows reducing sintering temperature [24,27,28] and generates a crystallographic texture [29,30]. To the current knowledge of the authors, no study refers to the manufacturing of ceramics by using the combined benefits of microwave sintering and loading. This is the topic of the present paper.

This paper describes an original setup designed to perform sinter-forging in a single-mode cavity microwave furnace. For accurate measurement of the sample temperature during sintering in the cavity, a specific calibration procedure is presented. The setup is then used to study the behavior of a nanosize α-alumina powder during microwave sinter forging. The obtained results (deformation, density, grain size) are compared to those obtained in conventional sinter forging experiments.

2. Experimental methods

2.1. Sample preparation

Commercial α-Al_2O_3 BMA15 powder produced by Baikowski is used in this study. The BET specific surface area is 16 $m^2\ g^{-1}$ and the average crystallite size of the powder is 100–150 nm. Cylindrical compacts are fabricated by uniaxial cold pressing in a 8 mm diameter steel die at 200 MPa. To reduce the friction coefficient between the powder and the steel die and thus facilitate the compression, stearic acid has been sprayed on the die surface. After pressing, the green compacts have been cleaned at 600 °C for 2 h. The compacts finally have a green relative density of $52 \pm 1\%$ (3987 g/cm^3 alumina theoretical density), a diameter $\varnothing_0 \approx 8$ mm and an initial height $H_0 \approx 8$ mm. $\varnothing_0$ and H_0 are accurately measured for every compact.

2.2. Microwave sinter forging (MWSF) setup

The experiments have been performed in a 2 kW, 2.45 GHz single mode cavity microwave furnace provided by SAIREM [16]. It includes a high voltage power supply linked to a magnetron that delivers a variable forward power. A rectangular wave-guide of 86.36 mm × 43.18 mm section allows the transport of the microwave radiation to a rectangular TE_{10p} cavity. This resonant cavity is closed by a coupling iris (a vertical slot in a copper sheet) on the magnetron side and by a reflector (short-circuit piston) on the other side. Sintering tests are performed in air. A prescribed heating cycle is processed by continuous manual adjustment of the length of the cavity through the position of the short-circuit piston, as this length controls the electric field distribution and thus the power dissipated in the sample.

The specific setup designed to realize MWSF is sketched in Fig. 1. Fig. 1a shows the front face of the setup and Fig. 1b shows its crosscutting top view. The green sample is positioned upon a fixed rod composed of a dense alumina punch of 10 mm diameter aligned with a water-cooled steel rod. An identical rod (alumina punch plus cooled steel rod) can translate in the upper column of the microwave cavity. A lever arm pushes on the upper rod and transmits a constant force to the sample. With the developed device, a maximum force of $F = 2500$ N can be applied. An external linear transducer allows recording the upper steel rod displacement and following the axial strain of the sample.

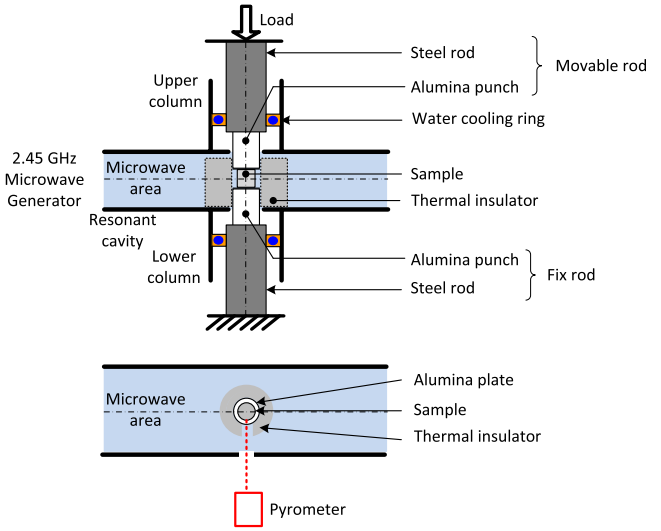

Fig. 1. Schematic front (a) and top views (b) of the microwave sinter forging setup.

We do not know the precise origin of the piece of alumina from which punches were fabricated. As this material showed good coupling properties with the microwaves we decided to use it for the punches. Its composition was analyzed and the presence of potassium, sodium, chlorine and calcium residues was found. We believe that these residues are responsible for the interaction of the material with microwaves. However, as the potassium is supposed to improve the sticking of the sample with the punches during sintering, a pure alumina plate of 0.5 mm thickness is inserted between each punch and the sample. A porous thermal insulator, composed of alumina and mullite fiber, with a maximum working temperature of 1600 °C (Fiberfrax Duraboard 1600), is placed around the sample and the punches. Sample temperature is measured with a bichromatic pyrometer through a hole in the cavity and the thermal insulator (Fig. 1b). The pyrometer calibration is presented in the following subsection. Considering the sintering configuration, it is assumed that hybrid heating occurs during sintering, i.e., the sample is heated both by direct coupling with the microwaves and by radiative and conductive transfer from alumina punches.

2.3. Pyrometer calibration

A bichromatic pyrometer using wavelengths $\lambda_1 = 1.28$ μm and $\lambda_2 = 1.65$ μm is used to measure sample temperature. The ratio $k = \varepsilon_1/\varepsilon_2$, where ε_1 and ε_2 are the apparent emissivities of the sample for wavelength λ_1 and λ_2, respectively, has to be estimated in a thermal configuration corresponding to the one found in the microwave cavity. Fig. 2 shows a photo and a schematic cross section of the setup used to calibrate the