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ScienceDirect
Ceramics International 37 (2011) 321–326
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

Novel fabrication of pressure-less sintering of translucent powder injection molded (PIM) alumina blocks

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Received 5 July 2010; received in revised form 30 July 2010; accepted 28 August 2010
Available online 25 September 2010

Abstract

A new method of fabricating translucent alumina brackets using powder injection molding (PIM) is reported. Alumina powder was mixed with MgO, La_2O_3, and Y_2O_3 to control grain size and porosity. The powders were mixed with a binder consisting of a mixture of paraffin wax and polyethylene in a 1:1 ratio to make feedstock for injection molding. The total amount of binder was limited to 14 wt% to minimize shrinkage and cracking after sintering. After injection molding, debinding was performed using the wicking method and samples were sintered in a vacuum at 1700 °C to achieve high density. Ultimately, translucent corundum was fabricated. The sintering additives resulted in a decrease in porosity and an improvement in translucency by promoting grain growth during pressure-less sintering. After sintering, Vickers hardness, bending strength, density, and transmittance of the fabricated parts were measured to show that those values were comparable to those of the commercially available dental brackets. Therefore, the translucent alumina block was successfully fabricated using PIM method to be potentially used as a dental bracket.

Keywords: Bracket; Powder injection molding; Sintering aids; Translucent alumina

1. Introduction

Most orthodontic appliances are made of metals, such as titanium, stainless steel, and nickel alloys, because these materials have good mechanical strength. However, metallic materials can be problematic due to their innate color and metallic allergy. Recently, the use of ceramic-based orthodontic appliances has been examined to overcome these issues. High-density, high-purity Al_2O_3 is being adopted in orthodontics because it has several beneficial properties, including excellent corrosion resistance, good biocompatibility, esthetic color, and good hardness characteristics. Single-crystal phase Al_2O_3 dental orthodontic appliances are superior to polycrystalline Al_2O_3 in terms of esthetics, discoloration, and customization. However, the use of single-crystal Al_2O_3 is limited because of its high processing cost.

This study is focused on α-Al_2O_3, and small amount of sintering additives were used as grain growth inhibitors. These additives are essential to achieve a very dense sintered body with a fine-grained microstructure [1]. Powder injection molding (PIM) technology was used to make polycrystalline alumina blocks and to reduce the machining sequences. PIM technology is an emerging technology for processing metal and ceramic parts. Mass production, cost-effective fabrication, and a near net-shape forming process for small, intricate, precise parts are possible using PIM [2]. The mixed powders were injection molded as blocks, which were debinded and sintered to obtain dense compacts of the desired material. Debinding can be done with thermal treatment, which extracts solvent or causes catalytic decomposition of the binder components. Moreover, while sintering at temperatures of 1600–1800 °C, the green body shrinks, producing a dense structure. Therefore, the sintering profile must be carefully controlled to avoid distortion and crack formation, caused by pores, resulted from extraction of the binder [3].

To be translucent, it is necessary to minimize residual pores in the ceramic body, as pores scatter light. Two methods have been used to eliminate residual pores in the fabrication of

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0272-8842/$36.00
doi:10.1016/j.ceramint.2010.09.011

translucent ceramics: one accelerates mass transport from particles to voids by sintering at high temperatures and by adding sintering additives and the other inhibits discontinuous grain growth, which can result in large grains with pores trapped inside. To achieve this, two sintering techniques have been developed to fabricate a translucent and dense ceramic body with a theoretical density: one technique uses high-temperature sintering with additives and the other uses pressure sintering, such as hot isostatic pressing (HIP) or hot pressing (HP), to promote diffusion. As HIP is not suitable for conventional mass production, a pressure-less sintering process using additives was studied. The fabrication of these translucent alumina blocks using PIM technology can be applied to the manufacture of orthodontic brackets.

There have been several methods introduced to produce translucent alumina blocks by controlling sintering profiles, sintering atmosphere, etc. [4–8]. In this study, unique way of fabricating translucent alumina block using PIM method was introduced and its effect of sintering additives on the translucency of alumina block was discussed.

2. Materials and methods

2.1. Powder preparation

The powders used in this study were high-purity AKP-50 alumina powder (α-Al_2O_3) with a particle size of 100–300 nm (Sumitomo Chemical, Tokyo, Japan). The amounts of magnesium oxide (MgO), yttrium (III) oxide (Y_2O_3), and lanthanum (III) oxide (La_2O_3) were varied as sintering agents to study their effects on translucency of the sintered alumina parts [9]. Table 1 shows the compositions of the ceramic parts.

The powders were mixed in ethanol using a stirring bar under infrared light for drying. Then, the powders were filtered through a 100-μm sieve. Paraffin wax and polyethylene were used as binders for feedstock fabrication. Paraffin wax softens as the temperature increases and melts at temperatures greater than 60 °C. As polyethylene softens at temperatures greater than 80 °C and becomes a liquid, a mortar and pestle were used to mix the powders to fabricate 50 g of feedstock at 120 °C. The percentage of binder to the total composition was approximately 14 wt%, which is the lowest possible percentage for mixing to minimize shrinkage during debinding and sintering, while enabling some mixing fluidity during injection molding. The paraffin wax gives fluidity to the feedstock during injection molding and the polyethylene consolidates the powders to maintain the shape of the feedstock. A 1:1 ratio of paraffin wax to polyethylene was found to be optimal. After mixing the binders and powders, injection molding was performed at a pressure of 50 bar at temperatures of 148–160 °C using Ministar VMP-43 molding equipment (ANC, Ansan, Korea).

Table 1
Powder preparation.

Sample	Additive composition	Sintering profile
A	0.25 wt% MgO	1
B	0.25 wt% MgO + 0.01 wt% La_2O_3 + 0.01 wt% Y_2O_3	
C	0.25 wt% MgO	2
D	0.25 wt% MgO + 0.01 wt% La_2O_3 + 0.01 wt% Y_2O_3	

2.2. Debinding

The injected parts must be debinded to achieve a high density of the desired material. To avoid large numbers of pores and cracks, the debinding process was carried out using the wicking method, and this was followed by the debinding profile, as shown in Fig. 1.

Wicking is a method to remove the binders after injection molding of the sample. Wicking provides Al_2O_3 bedding to surround the injection molded sample for heat treatment. As the surrounding temperature increases, the binders on the surface of the injection-molded sample melt and pores are formed. These pores enable elimination of binder inside the sample, as the powder bed is linked to the pores. Moreover, the powder bed absorbs binder vapors immediately, which prevents the pores from connecting and increasing in size [10].

2.3. Sintering

Before the main sintering, the debinded samples were presintered at 800 °C in air for 50 h. Then, high-temperature sintering was carried out using a vacuum furnace (TM-14-16; Thermonik, Tokyo, Japan) at 1700 °C for 2 h with a pressure of 2.0×10^{-5} Torr. During sintering, thermal treatment at 1300 °C to promote diffusion of MgO into the alumina grain boundaries was done to prevent the irregular grain growth. The sintering profile was adjusted to vary hold times at 1300 °C to examine the effects of MgO on grain growth, as shown in Fig. 2. The hold time for sintering profile #2 was 1 h longer than that of profile #1. 2 samples for each profile were prepared with total of 4 samples.

After sintering, the Vickers hardness and bending strength were measured using Vickers hardness tester and UTM (Universal Testing Machine), respectively. Additionally, density was measured by Archimedes' method and the structure

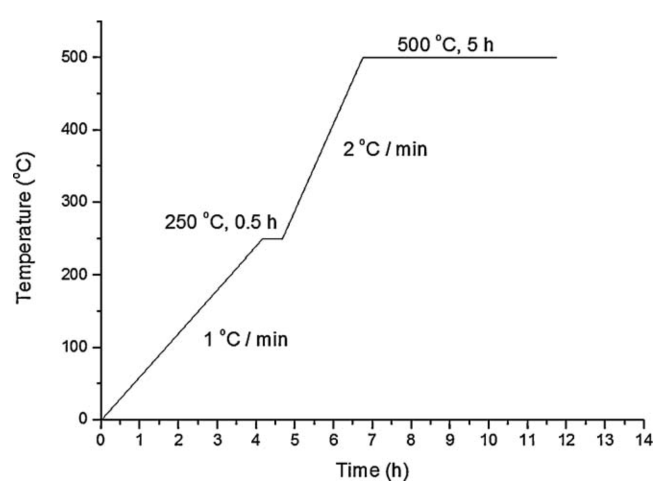

Fig. 1. Debinding profile.