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Influence of Presintering Parameters on the Mechanical Properties of Presintered Dental Zirconia Block

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Abstract: This study aimed to investigate the effects of presintering parameters on the mechanical and microstructural properties of presintered zirconia block. The zirconia block was prepared using colloidal and cold isostatic pressing techniques and subsequently subjected to various presintering procedures at 850 °C, 900 °C, and 950 °C with two different heating rates (3 and 10 °C/min). All samples were evaluated in terms of hardness, density, and morphology. Results showed that the sample porosity decreased with increment in the presintering temperature. On the contrary, the Vickers hardness increased from 1.8 GPa at 850 °C to 2.7 GPa at 950 °C at 10 °C/min heating rate. The sintering temperature of 950 °C with 3 °C/min heating rate was considered the most suitable parameter among presintering procedures, with high theoretical density (96.2%) and appropriate hardness (1.27 GPa) that are within the range of commercial zirconia block hardness values for restoration milling procedure. Furthermore, to sinter the block samples successfully without considerable failure, the heating and cooling rates should be lowered to ≤3 °C/min. Therefore, on the basis of these results, the forming methods and the sintering temperature influence the resultant mechanical and microstructural properties of the final sample.

Keywords: Zirconia, pre-sintering, dental restorations, slip casting, cold isostatic pressing

1. Introduction

Zirconia, specifically 3 mol% yttria-stabilized zirconia (3YSZ), has exhibited rapid progresses in its use as a dental material because of its biocompatibility, good mechanical properties, and aesthetic relationship with the natural human teeth [1]. Nanosized zirconia, i.e., with a microstructural length or grain size of about 100 nm, has been specifically targeted recently by many investigators due to its excellent characteristics, specifically notable mechanical properties [2,3]. Furthermore, crack propagation in nanosized zirconia powders can be controlled, thereby improving the mechanical properties of their structures [4].

Zirconia dental restorations are commonly produced via two different machining techniques, i.e., either soft machining of presintered blanks or hard machining of completely sintered blanks [5]. Commercially fabricated zirconia blanks contain a binder that makes them suitable for pressing, which will be eliminated during sintering. The powder and binder mixture is subjected to cold isostatic pressing (CIP) to form a zirconia blank [6]. Nowadays, presintered zirconia blank/block machining has become increasingly popular because it is an easier and faster technique than hard machining due to the less friction during the milling process with minimal cutting tool [7]. Hard machining allows the clinician to obtain accurate anatomical prosthesis dimension, because it is not influenced by dimensional shrinkage. However, it has low machinability owing to the high hardness of the materials that sometimes may induce crack, encourage tetragonal-to-monoclinic (t–m) phase transformation, wear the milling tool, and consume too much processing time.

Amat et al. [8] found that the mechanical properties of zirconia block for dental restoration are improved through colloidal and CIP techniques. However, an appropriate presintering temperature that can be used to produce zirconia block with good strength and that can be mounted into jig without damage during machining is not yet completely studied. Therefore, the present study aims to determine the optimum presintering parameter, which can result in an acceptable hardness for machining. The highest densification and pore removal are also ascertained.

2. Materials and Methods

2.1 Material and slurry preparation

The zirconia nanopowder used in this study was 3YSZ (Inframat Advanced Materials, USA), which presents a specific surface area of 30–60 m^2/g. The

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zirconia powder was mixed with distilled water at solid loading of 10 vol%. Polyethyleneimine (Sigma-Aldrich, USA), a dispersant agent, was added into the suspension at 0.5 wt%. Nitric acid (HNO3) was also gradually added to the suspension to control the solution pH and ensure that the final pH was approximately 2. The suspension was constantly magnetically stirred for 15 min to aid the proper mixing of components. The suspension was subsequently placed in an ultrasonic bath for 10 min to break the agglomerates and milled using a Pulverisette-6 (Fritsch, Germany) planetary ball mill at 300 rpm for 120 min, with 10 mm-diameter zirconia balls as grinding media at a ball-to-powder ratio of 10:1 [8].

2.2 Zirconia block consolidation and presintering procedure

The prepared slurry was poured into the plaster of Paris mold that formed into block with 40 mm diameter and 15 mm thickness as shown in Figure 1. The drying time of the block samples was approximately 1 to 2 days. The green body was placed in a rubber glove and vacuumed to help reduce the possibility of rubber glove leaking and oil-induced contamination of the sample in the CIP machine (Riken Seiki Co. Ltd., Japan). All the samples were pressed isostatically in a hydraulic oil medium, and the green bodies underwent this pressing process at 250 MPa for 2 min. Figure 2 shows the zirconia produced via slip casting and CIP technique. The pressed blocks were presintered in a high-temperature furnace (CMTS Furnace L16, Germany) at different temperatures (850 °C, 900 °C, and 950 °C) for 2 h with two different heating rates (3 and 10 °C/min) as shown in Figure 3.

2.3 Block characterization

The sintered densities of all samples were measured with an electronic balance (Newclassic MS Mettler Toledo, USA) using Archimedes' method. Distilled water was used as the medium. The theoretical density given by the supplier is 6.10 g/cm^3, which was used in measuring the relative density of each sample. Morphological analysis was conducted by thermally etching the samples at 1200 °C for 1 h. Afterward, field emission scanning electron microscopy (FESEM) was performed via FESEM (Zeiss Merlin, Germany). The hardness of the sintered samples was examined using a Vickers microhardness tester (ZHV10, Zwick, Ulm, Germany) at a load of 0.2 kgf and a dwelling time of 15 s.

3. Results and Discussion

3.1 Pre-sintered density

The presintered density data are presented in Figure 4. The relative density of a presintered zirconia sample is

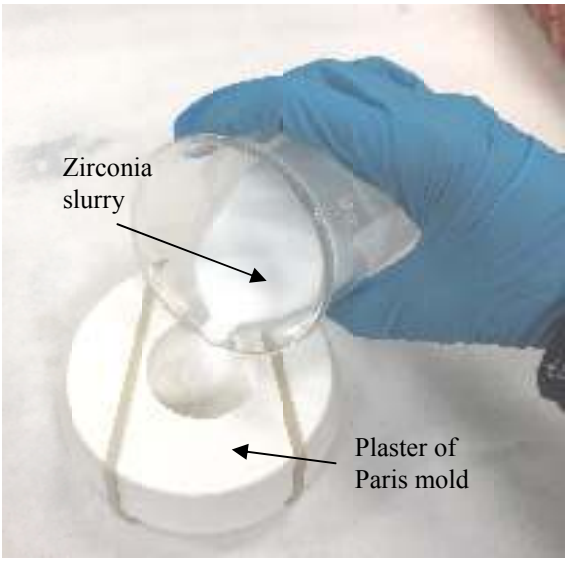

Fig. 1 Slip casting process of the zirconia block sample.

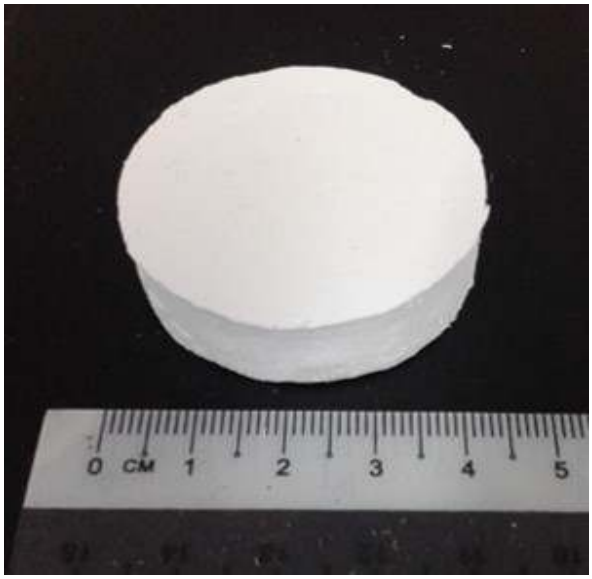

Fig. 2 Block sample produced through slip casting and CIP technique.

directly proportional to its porosity distribution; few pores in a body can result in approximately complete densification, which is close to achieving its theoretical density. The resultant graph shows that the difference in the density of samples sintered at 850 °C (94%) and 900 °C (94.3%) at a heating rate 3 °C/min was almost

negligible. Nevertheless, at 950 °C, the samples presented high density, with an average of 96.5%. The sample presintered at 10 °C/min heating rate displayed lower density than that presintered at 3 °C/min. This observation can be attributed to the slow rate of 3 °C/min, which enabled the good diffusion and binding of particles and subsequent pore minimization.

3.2 Vickers hardness

The hardness of a sintered 3YSZ body was significantly influenced with the sintering temperature (Figure 5), which was related to the resultant porosity distribution. The resultant Vickers hardness of the samples increased with increment in the sintering temperature. This result can be explained by that the pores on the grain bodies were reduced with the increased sintering temperature due to solid-state diffusion [9]. Thus, the relative density and hardness were increased. This result corresponds to that reported by Fan et al. [7] who also showed that hardness is directly proportional to the sintering temperature. The hardness of sample presintered at a heating rate of 3 °C/min was comparable to that of commercially available zirconia blank with a hardness range of 0.92–1.28 GPa [10]. All samples that presintered under a heating rate of 10 °C/min were not suitable for machining as the samples possessed hardness values that were more than the allowable value for machining; such values may also be detrimental to the properties of the final product and may deteriorate the cutting bur.

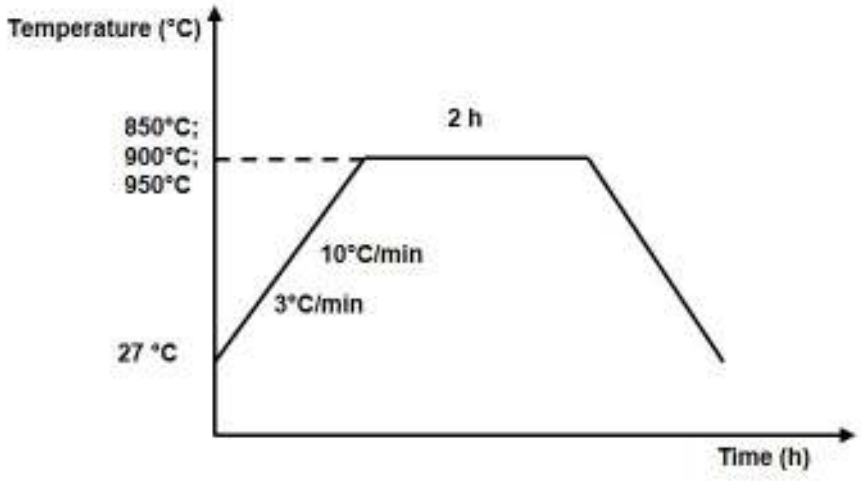

Fig. 3 Presintering profile of the zirconia block samples.

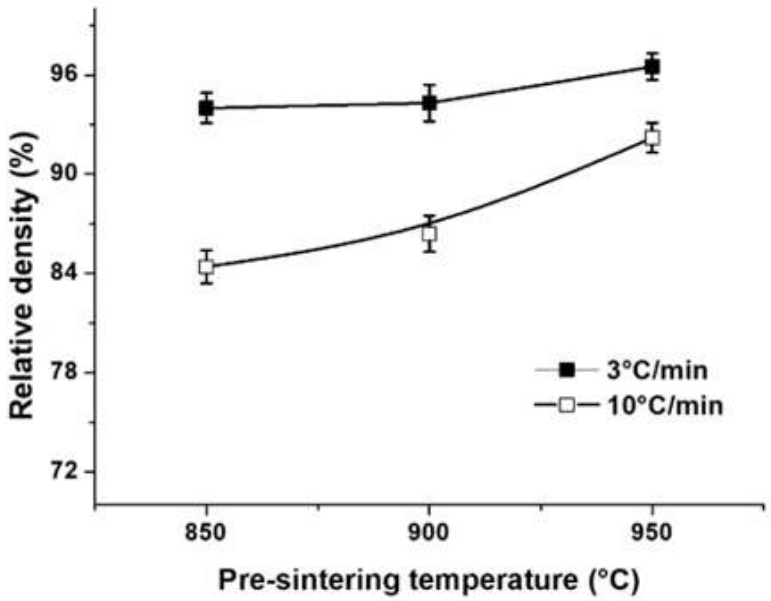

Fig. 4 Presintered densities of zirconia block at different presintering parameters

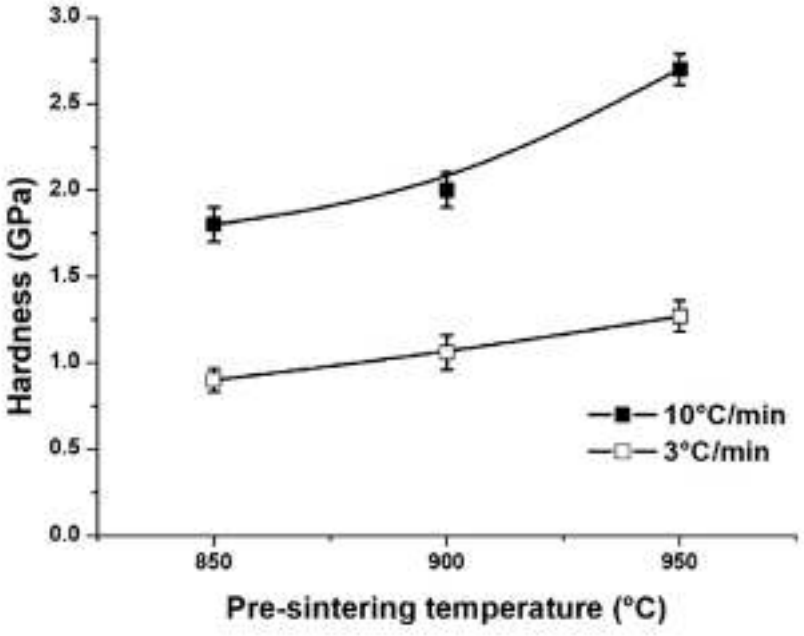

Fig. 5 Vickers hardness of presintered zirconia block at different presintering parameters

3.3 Morphological characterisation

Figure 6 display the resulting surface imaging of the samples presintered at 850 °C, 900 °C, and 1000 °C. The porosity difference between the samples presintered at 850 °C and 900 °C was relatively small. Considerable variation existed between the porosities of these two samples and that sintered at 950 °C. This trend is in agreement with the theory that porosity reduces with increased sintering temperature [11,12]. The porosity difference between the samples at heating rates of 3 and 10 °C/min was significant. Less pores were observed in the microstructure of sample sintered at 3 °C/min, especially at 950 °C, which can be attributed to that volume expansion occurs in the grains during the tetragonal-to- monoclinic transformation [5].

The mechanical properties of 3YSZ were strongly influenced with its grain size, and a range of sintering temperature was most likely to influence grain size [13]. According to Trunec et al. [11], when a 3YSZ body is sintered to a minimum theoretical density of 99%, the grain sizes are increased with long sintering times and high sintering temperatures [11]. This trend is observed only in completely sintered bodies. This study also showed that the presintered bodies present no grain size formation but particle formation because they are yet to reach the consolidation temperature. However, when machining a dental framework, the presintered block must possess appropriate hardness and robustness to enable gripping in a jig during machining. Thus, in the present study, the samples presintered at 950 °C with heating rate of 3 °C/min showed a good potential for further machinability test. Their shrinkage upon complete sintering can also be investigated so that the machined framework can fit into the desired final size of dental restorations.

Conclusions

The samples presintered with a heating rate of 3 °C/min exhibited higher relative density than that of sample presintered at 10 °C/min, but they reached no complete densification yet. The shrinkage upon final sintering stage and the hardness values of all samples at a heating rate of 3 °C/min can be potentially further studied for machining test. This research showed that the heating and cooling rates should be reduced (from an initial rate of 10 °C/min to 3 °C/min) to provide grains with sufficient time and space to transition from the tetragonal phase to the monoclinic one.

Acknowledgments

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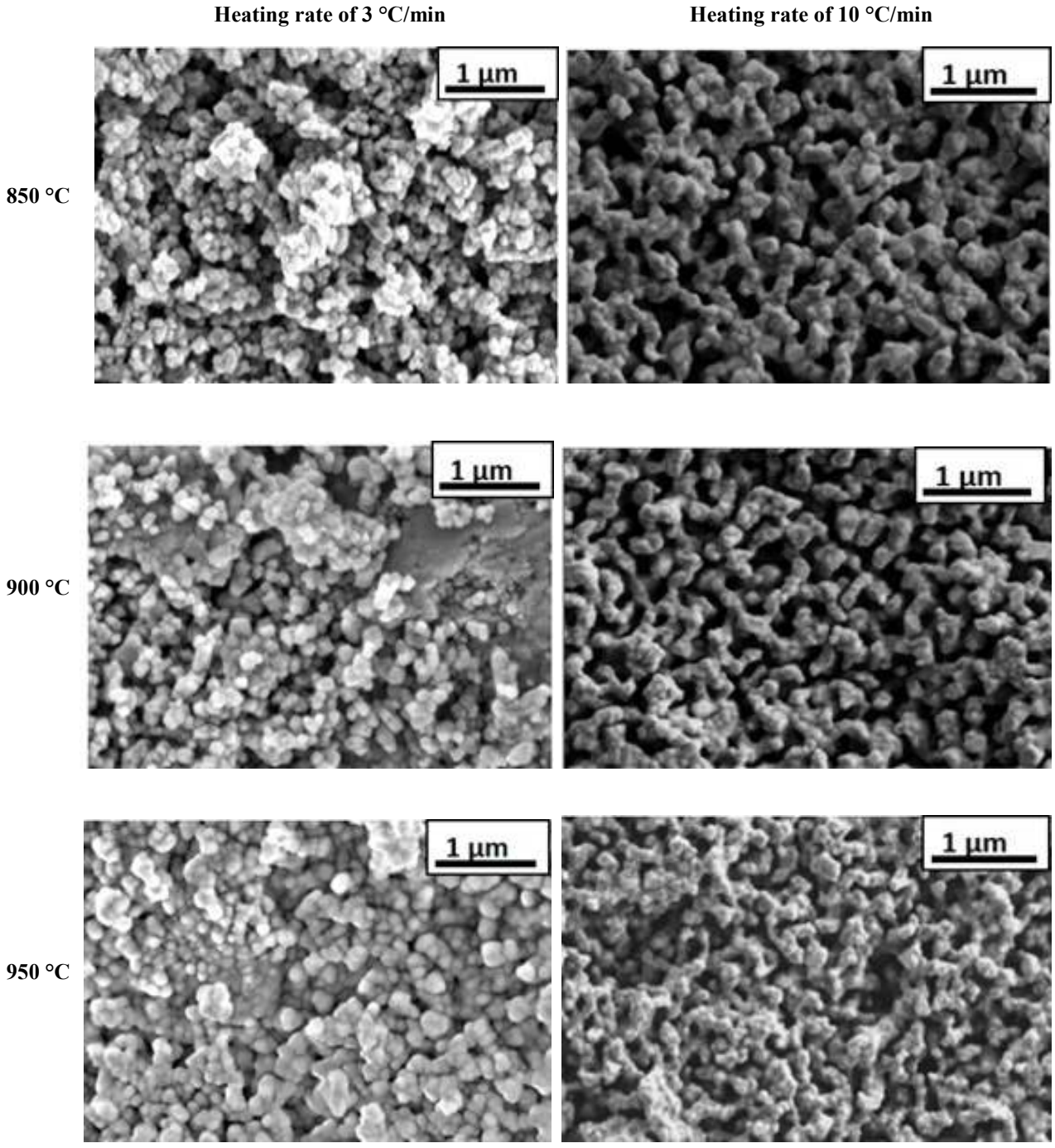
Heating rate of 3 °C/min
Heating rate of 10 °C/min
850 °C
900 °C
950 °C
1 µm

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