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Full Paper

INFLUENCE OF PROCESSING ON MECHANICAL PROPERTIES OF 3Y-TZP FOR DENTAL APPLICATIONS

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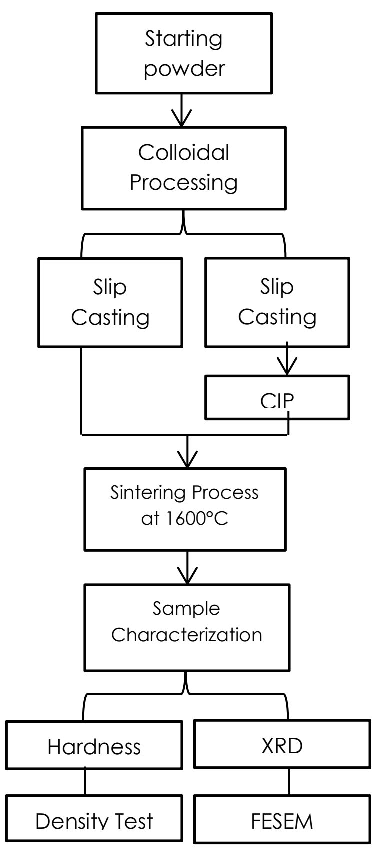
Graphical abstract



Abstract

Purpose: This study aimed to investigate the influence of processing on the mechanical properties of 3 mol% yttrium–tetragonal zirconia (3Y–TZP) for dental applications. In this study, cold isostatic pressing (CIP) was adopted as a second consolidation process to enhance the mechanical properties of slip-cast 3Y–TZP. Methods: Two batches were prepared. The first batch of 3Y–TZP suspension was fabricated via colloidal processing. Then, the suspension was subjected to the slip casting process. Simultaneously, the second batch was prepared via colloidal processing, followed by CIP. The specimens were sintered at 1600 °C. Sintered density, hardness, microstructure, and phase distribution were examined and analyzed. Results showed that the specimens fabricated via slip casting and CIP had the highest density of 99% of the theoretical density (6.1 g/cm3) and hardness of 14.4 GPa. The microstructure of the CIP samples was homogeneous with low porosity. According to X-ray diffraction examination, both batches exhibited a single phase (tetragonal phase). Conclusion(s): The density, hardness, and homogeneity of the microstructure of Y–TZP fabricated via slip casting and CIP improved. Using CIP as the second consolidation method improved the quality of green bodies.

Keywords: Cold isostatic pressing, mechanical properties, dental zirconia, agglomeration

Abstrak

Tujuan: Kajian ini bertujuan untuk menyiasat pengaruh kaedah pemprosesan terhadap sifat-sifat mekanik 3% mol ytria tetragonal zirkonia (3Y-TZP) untuk aplikasi gigi. Dalam kajian ini, penekanan isostatik sejuk (cold isostatic pressing, CIP) telah diguna pakai sebagai proses pengukuhan yang kedua untuk meningkatkan sifat-sifat mekanik 3Y-TZP yang telah menjalani penuangan buburan. Kaedah: dua kumpulan telah disediakan. Kumpulan pertama ampaian 3Y-TZP telah disediakan melalui pemprosesan berkoloid. Kemudian, ampaian itu telah menjalani proses penuangan buburan. Pada masa yang sama, kumpulan kedua disediakan melalui pemprosesan berkoloid, diikuti oleh CIP sebagai proses. Spesimen disinter pada suhu akhir 1600 ° C. Ketumpatan tersinter, kekerasan, mikrostruktur, dan taburan fasa telah diperiksa dan dianalisis. Hasil kajian menunjukkan bahawa spesimen direka melalui penuangan buburan dan CIP menunjukkan nilai ketumpatan iaitu 99% ketumpatan teori (6.1 g/cm^3) dan kekerasan (14.4 GPa) yang tertinggi. Mikrostruktur sampel CIP adalah homogen dengan keliangan yang rendah. Menurut pemeriksaan pembelauan X-ray, kedua-dua kelompok mempamerkan satu fasa (fasa tetragonal). Kesimpulan : Ketumpatan, kekerasan, dan kehomogenan mikrostruktur Y-TZP dibentuk melalui penuangan buburan dan CIP bertambah baik. Penggunaan CIP sebagai kaedah pengukuhan kedua tinggi meningkatkan kualiti jasad anum.

Kata kunci: penekanan isostatic sejuk, sifat mekanik, zirkonia pergigian, aglomerat

1.0 INTRODUCTION

Tetragonal zirconia has been extensively used as a biomaterial, especially in dentistry, for dental restorations such as crowns and bridges [1]. Tetragonal zirconia is highly biocompatible, with excellent appearance and high mechanical properties resulting from its transformation toughening mechanism [2]. Materials used in dental restorations must possess high mechanical properties to withstand specific conditions in the oral cavity, such as masticatory force. Dental materials, such as zirconia, must resist approximately 20 MPa of occlusion pressure around 3,000 times daily [3]. The use of nanosized powder is known to improve the mechanical properties of tetragonal zirconia.

Nonetheless, the high specific surface area of the fine particles promotes agglomeration and leads to defects such as porosity or voids. The microstructure defects decrease the mechanical properties [4]. In the same context, the raw material may contain defects, such as hard agglomerations [5]. These defects can cause early failure of dental restoration. Thus, the fabrication process plays a significant role in controlling the agglomeration and microstructure defects that degrade the mechanical properties of tetragonal zirconia. The variations in microstructural features include grain size, porosity, and yttrium distribution, as well as significantly affect the mechanical properties of zirconia [6].

Considering the aforementioned problems, this study conducted colloidal processing (slip casting). This common method can produce a suspension with excellent particle dispersion that can limit the microstructure defects [7–8]. For nanopowder zirconia, colloidal processing is suitable in reducing agglomeration [4]. Decreasing agglomeration produces excellent colloidal stability that results in sintered denes samples with homogenous microstructure. Cold isostatic pressing (CIP), another consolidation method, compacts the zirconia powder from all directions, thereby producing uniform green bodies with high density [9].

This study aimed to investigate the influence of processing on the mechanical properties of 3 mol% yttrium–tetragonal zirconia polycrystals (3Y–TZP) for dental application. CIP was used as a second consolidation process to enhance the mechanical properties of slip-cast 3Y–TZP for dental restorations.

2.0 METHODOLOGY

2.1 Materials

Nanosized yttrium-stabilized zirconia 3Y–TZP (US Research Nanomaterials Inc., Houston, TX, USA) was used in this study. The powder particle size was approximately 20 nm, as measured by transmission electron microscopy (TEM; Philips CM 12). Two batches of 3Y–TZP green bodies were prepared (Figure 1).

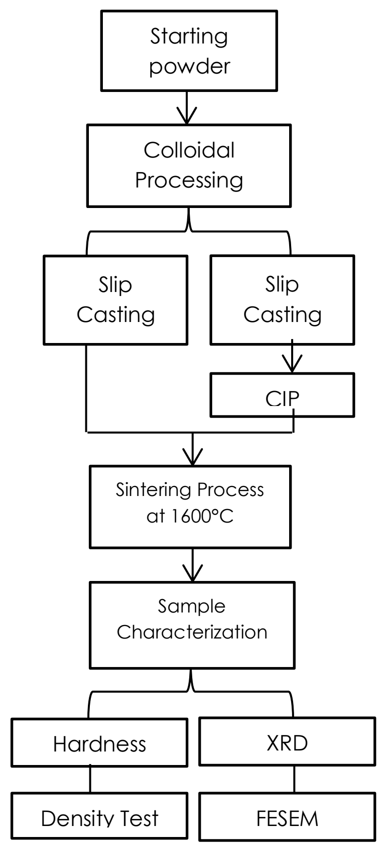


Figure 1 Flow diagram of sample preparation

2.2 Sample Preparation

The first batch of green bodies was fabricated via colloidal processing (slip casting). A suspension of 30 g powder in 70 mL distilled water was prepared. The suspension was adjusted to pH 2 using HNO_3. Then, 0.4 wt% polyethyleneimine (PEI) was added to the suspension as a cationic dispersant agent. The suspension was magnetically stirred for 30 min and placed in an ultrasonic bath for 15 min. Then, the suspension was magnetically stirred for 15 min for a second time. Thereafter, slip casting method for 24 h was employed as a consolidation and shaping method. Teflon molds 18 mm in size were used in this process.

Simultaneously, another batch was prepared through colloidal processing (slip casting). Then, a CIP machine (Riken Seiki, Japan) was used at 200 MPa for 1 min in the second consolidation process. The samples were placed in gloves to maintain dryness. Subsequently, samples were immersed in fluid and uniform hydrostatic pressure was applied from all directions. By using a furnace (CMTS Furnace-L 16), the specimens of both batches were sintered at 1600 °C as the final sintering temperature for 2 h of soaking time. The sintering process scheme is explained in Figure 2.

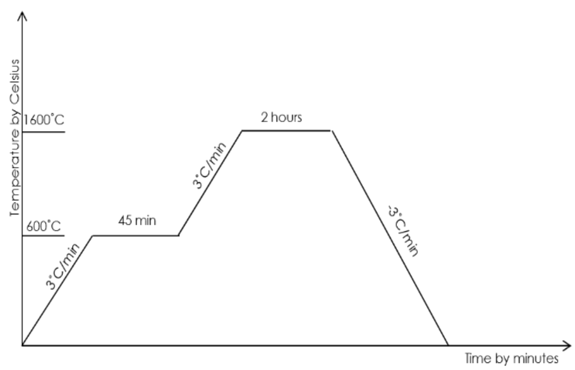


Figure 2 The sintering process scheme

2.3 Sample Characterizations

Finally, sintered density was measured using a density meter (Newclassic MS, Mettler Toledo, Columbus, OH, USA). Meanwhile, hardness values were measured by a Vickers hardness tester (Shimadzu DUH-W201S). Field-emission scanning electron microscopy (FESEM; Hitachi SU8000, Hitachi, Tokyo, Japan) was conducted to measure the microstructure. Finally, phase distribution was examined and analyzed in both batches using X-ray diffraction (XRD; D8 Advance, Bruker, Billerica, MA, USA). Existing peaks spectra within the range of 5–80°2θ.

3.0 RESULTS AND DISCUSSION

3.1 Density

Figure 3 shows the density results in which the second batch of samples fabricated via slip casting and CIP achieved the highest sintered density at 99% of the theoretical density (6.1 g/cm3). High density signifies that the material possesses high mechanical properties. The densification of samples is correlated with microstructure porosity. Porosity decreases ceramic density; therefore, the mechanical strength of the final product deteriorates. However, the first batch with slip casting without CIP exhibited low sintered density at 92% of the theoretical density.

The combination of slip casting and CIP produced high-denes green bodies because the colloidal processing (slip casting) partially reduced the agglomerates and the CIP produced excellent particle packing. This multi-stage process was completed by firing operation under a suitable sintering temperature at 1600 °C. The method yielded excellent diffusion of grains with complete growth, which resulted in a homogeneous microstructure with no pores and voids [10]. Consequently, the sintered samples achieved full density.

3.2 Hardness

Hardness was also measured in consideration of its importance in the clinical success of dental crowns and bridges. Zirconia in the oral cavity comes in contact with mechanical and chemical agents that may create irregularities on surfaces [11]. This phenomenon increases the adhesion of microorganisms and protects them from forces of salivary inflow and oral hygiene [12]. The surface roughness generated by mechanical wear may increase the degradation process [13]. Furthermore, the chemical degradation occurs on the surface because of the pH level of the oral cavity [14]. Thus, all these specific conditions and their effects on the surface of materials can lead to failure of zirconia as a restorative material.

Figure 4 presents the hardness values of the samples subjected to slip casting with CIP. These samples achieved the highest hardness value of 14.4 GPa. Similar to the sintered densities, the hardness values of the samples subjected to slip casting without CIP were lower (9.8 GPa) than those of the other samples. The results of the hardness test corresponded with the density results probably because of the combination of slip casting and CIP.

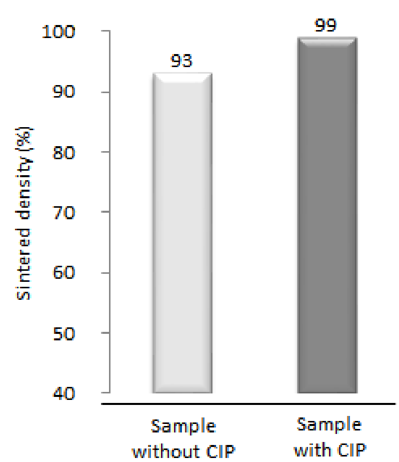


Figure 3 Density of Y-TZP samples with and without CIP

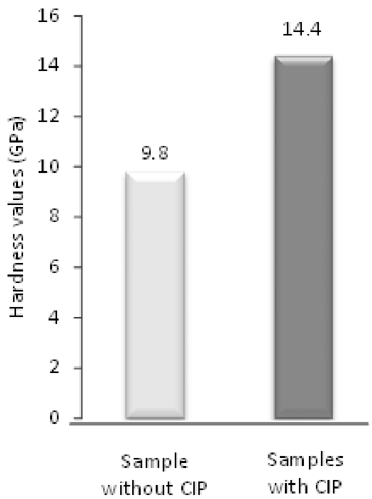


Figure 4 Hardness of Y-TZP samples with and without CIP

3.2 Microstructure

The microstructure of slip casting with CIP samples had a more homogeneous structure and nearly no pores compared with the samples without CIP (Figures 5). The homogeneous and compact microstructure improved the properties of 3Y–TZP in terms of hardness and density as mentioned in the preceding subsection of this paper [15–16].

The grains of the samples fabricated via slip casting and CIP became larger and packed after being subjected to the high and suitable conventional sintering temperature of 1600 °C as the final sintering temperature, which eliminated the porosity and other defects such as the agglomerations [17–18]. The microstructure of the slip-cast samples revealed a high degree of porosity and voids. Inhomogeneous structure was observed because of the variation in grain size. The agglomerations were also apparent, although the colloidal processing can limit the microstructural heterogeneities and produce excellent suspension stability as mentioned in this paper [7–8]. The presence of flows in the raw materials, such as the hard agglomerations (Figure 7), can reduce the suspension stability and degrade the performance of tetragonal zirconia [19].

CIP as a dry shaping method was used as the second consolidation method after the wet shaping method (slip casting) with the second batch. CIP compacted the slip-casted samples from all directions by applying uniform hydrostatic pressure. This compaction resulted in compact samples with homogeneous particle packing. Uniform particle packing is a major factor considered in successful ceramic processing [20]. As shown in the FESEM micrographs, the reduction of aggregates and other defects using the slip casting process was insufficient. By contrast, CIP enhanced the microstructure of the samples that were first subjected to slip casting. The improvement was due to the excellent particle packing.

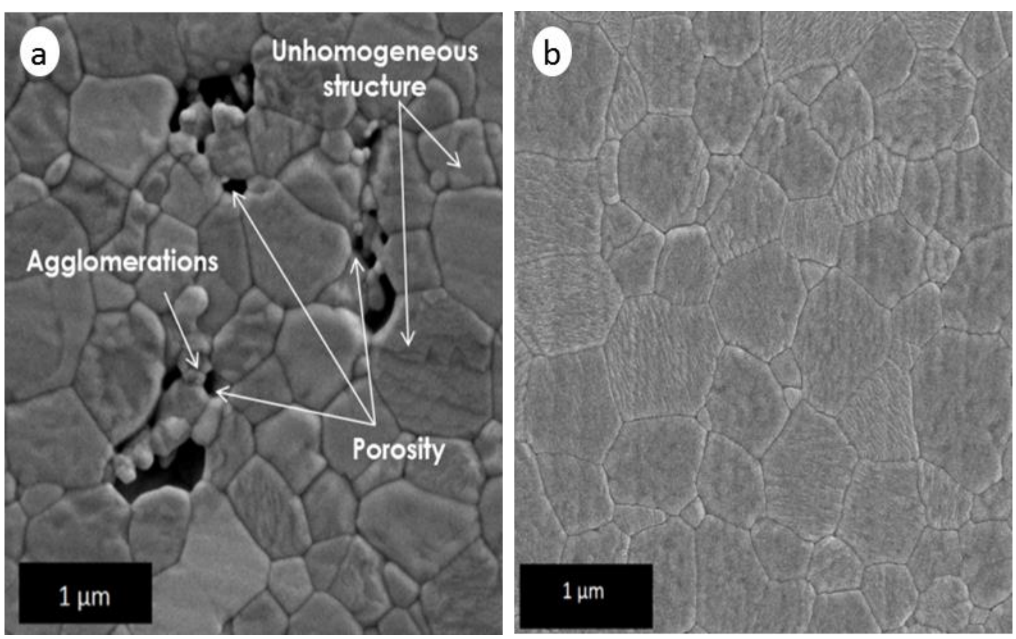


Figure 5 FESEM micrograph of 3Y-TZP without CIP (a) and with CIP (b)

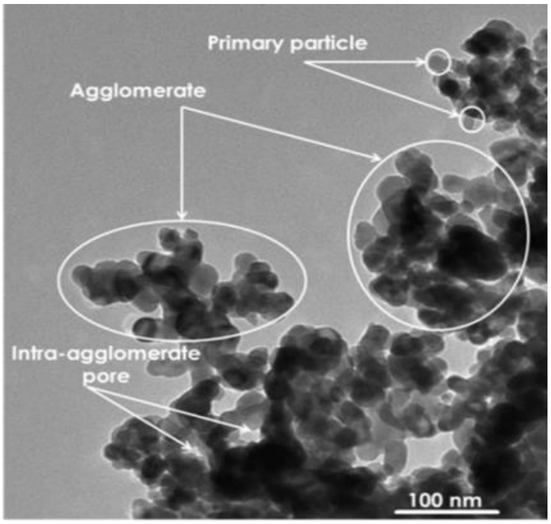


Figure 6 Morphology of 3Y-TZP powder under TEM

3.3 XRD results

According to XRD results, Figure 7 shows that only the tetragonal phase was observed in all Y–TZP samples, and no monoclinic phase was observed. The fabrication process, final sintering temperature, and duration of this process affected the phase content of zirconia [21].

High mechanical properties were usually found in tetragonal zirconia because of the stress-induced transformation toughening [22].

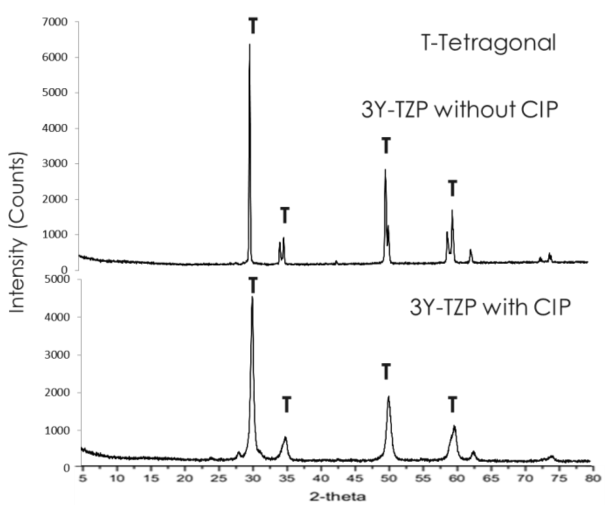


Figure 7 XRD results of Y-TZP samples with and without CIP

4.0 CONCLUSION

The results of the study demonstrated the influence of processing on the mechanical properties of 3Y–TZP. The density, hardness, and homogeneity of the microstructure of 3Y–TZP were significantly improved by slip casting with the CIP method. Thus, CIP proved suitable as the second consolidation method after slip casting and successfully improved the quality of green bodies and the mechanical properties of sintered 3Y–TZP.

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