

# IMPROVEMENT OF MECHANICAL PROPERTIES OF Y-TZP VIA CERIA ADDITION AND COLD ISOSTATIC PRESSING METHOD

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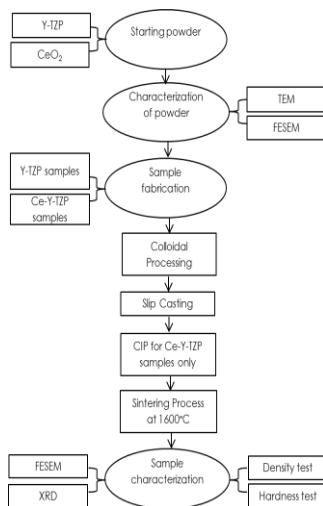
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## Graphical abstract



## Abstract

Fabrication and composition of tetragonal zirconia have been extensively studied to enhance its mechanical properties. The present study aims to investigate the mechanical properties of yttrium-stabilized zirconia (Y-TZP) with ceria addition consolidated via cold isostatic pressing (CIP). 3Y-TZP was prepared by slip casting. Another batch of samples was fabricated via slip casting with the addition of 5 wt% of ceria, followed by CIP. All samples were sintered at 1600 °C. Results showed that the density and hardness of Y-TZP increased with the addition of ceria and use of CIP. The density increased from 91.8% to 98% of theoretical density, and the hardness increased from 10.33 GPa to 14.14 GPa. Field-emission scanning electron microscopy (FESEM) images showed that Y-TZP with ceria and consolidated via CIP had more homogenous grain structure with lower porosity. The X-ray diffraction (XRD) analysis showed that the phase was 100% tetragonal for both materials. Ceria addition consolidated via CIP are an effective method to improve the mechanical properties of Y-TZP.

Keywords: Slip casting; cold isostatic pressing; ceria; zirconia

## Abstrak

Pembikinan dan komposisi tetragonal zirkonia telah dikaji secara terperinci untuk memperbaiki ciri-ciri mekaniknya. Kajian ini bertujuan untuk menyiasat sifat-sifat mekanik yttrium-stabil zirkonia (Y-TZP) dengan penambahan ceria yang dibentuk melalui penekanan isostatik sejuk (cold isostatic pressing, CIP). 3Y-TZP telah disediakan melalui penuangan buburan. Kumpulan sampel yang lain telah dihasilkan melalui penuangan buburan dengan penambahan 5 wt% ceria, diikuti dengan proses CIP. Semua sampel telah disinter pada suhu 1600 °C. Hasil kajian menunjukkan bahawa ketumpatan dan kekerasan Y-TZP meningkat dengan tambahan ceria dan penggunaan CIP. Ketumpatan meningkat daripada 91.8% kepada 98% daripada ketumpatan teori dan kekerasan yang meningkat daripada 10.33 kepada 14.14 Gpa. Medan pancaran mikroskop imbasan elektron (FESEM) menunjukkan imej Y-TZP dengan ceria dan dibentuk melalui CIP mempunyai struktur ira lebih homogen dengan keliangan yang lebih rendah. Pembelauan X-ray (X-ray diffraction, XRD) analisis menunjukkan fasa untuk kedua-dua bahan adalah 100% tetragonal. Penambahan ceria dan pembentukan melalui CIP merupakan kaedah yang berkesan untuk meningkatkan sifat mekanik Y-TZP.

Kata kunci: Penuangan buburan; penekanan isostatik sejuk; ceria; zirconia

## 1.0 INTRODUCTION

Tetragonal zirconia (TZP) is a ceramic material that has been widely applied as a biomaterial, particularly in dentistry as a restorative material. TZP has excellent mechanical and optical properties [1–3]. The composition of TZP significantly affects its properties. Common alkaline earth oxides, such as  $Y_2O_3$ ,  $CeO_2$ , and  $Al_2O_3$ , are added to zirconia to prevent or minimize tetragonal-monoclinic transformation, thereby enhancing the mechanical properties of tetragonal zirconia [4, 5]. Colloidal processing is an important and common fabrication method in ceramics processing. It produces homogenous dispersion of suspension that leads to samples with homogeneous microstructure and minimized microcracks and aggregates [6, 7]. Slip casting is employed to consolidate and shape the samples after suspension preparation via colloidal processing [8]. Slip casting is a suitable route in producing a defect-free homogeneous structure [9]. CIP is another technique to consolidate the samples. A high-dense material with uniform packing can be produced via CIP [10]. The present study aims to investigate the effects of ceria ( $CeO_2$ ) addition consolidated via CIP on the mechanical properties of yttrium-stabilized zirconia.

## 2.0 EXPERIMENTAL

### 2.1 Materials

Two commercial nanosized powders were used in this study: Y-TZP (US Research Nanomaterials, Inc., Houston, TX, USA) and  $CeO_2$  (M K Impex Corp., Mississauga, Canada). Images obtained by transmission electron microscopy (TEM, Philips CM 12) show that the powder sizes of 3Y-TZP and  $CeO_2$  were approximately 20 nm and 70 nm, as shown in Figures 1 and 2, respectively. A cationic polymer polyethyleneimine (PEI, Sigma-Aldrich) was used as the dispersing agent in this study.

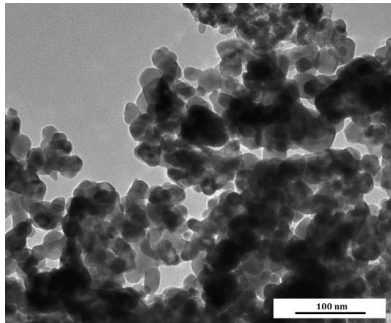


Figure 1 TEM image of 3Y-TZP

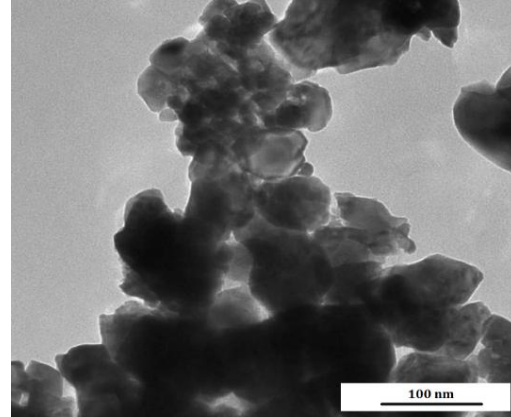


Figure 2 TEM image of  $CeO_2$

### 2.2 Y-TZP samples fabrication

3Y-TZP and ceria-zirconia composite disc specimens were fabricated via the colloidal process. The suspension was prepared with 10 vol% powder loading, that is, 3Y-TZP powder was added to distilled water. PEI (0.4 wt%) was then added as a cationic dispersing agent to the suspension, which was subsequently mixed for 45 min by using a magnetic stirrer. The suspension was then subjected to ultrasonic bath for 15 min. Afterward, the slurry was slip-casted into moulds for 24 h at room temperature. Finally, dried green bodies were sintered at 1600 °C using a furnace (CMTS Furnace-L 16).

### 2.3 Ce-Y-TZP Samples Fabrication

The same procedure as that for Y-TZP sample was followed to fabricate ceria-zirconia composite disc specimens. However, 5wt%  $CeO_2$  nanopowder was added to the suspension before slip casting. After slip casting, the casted green bodies were then subjected to CIP at 200 MPa for 1 min. The green bodies were later sintered at 1600 °C. The fabrication process and characterization of the samples are shown in Figure 3.

### 2.4 Sample Characterization

After sintering, density of the samples was measured using an electronic balance (Newclassic MSMettlerToledo). X-ray diffraction (XRD, D8 Advance, Bruker) was employed to determine the phases of both samples. The samples were ground and polished after mounting, and then Vickers hardness test (Shimadzu DUH-W201S) was conducted. Finally, FESEM with EDX was employed to characterize the microstructure of both materials.

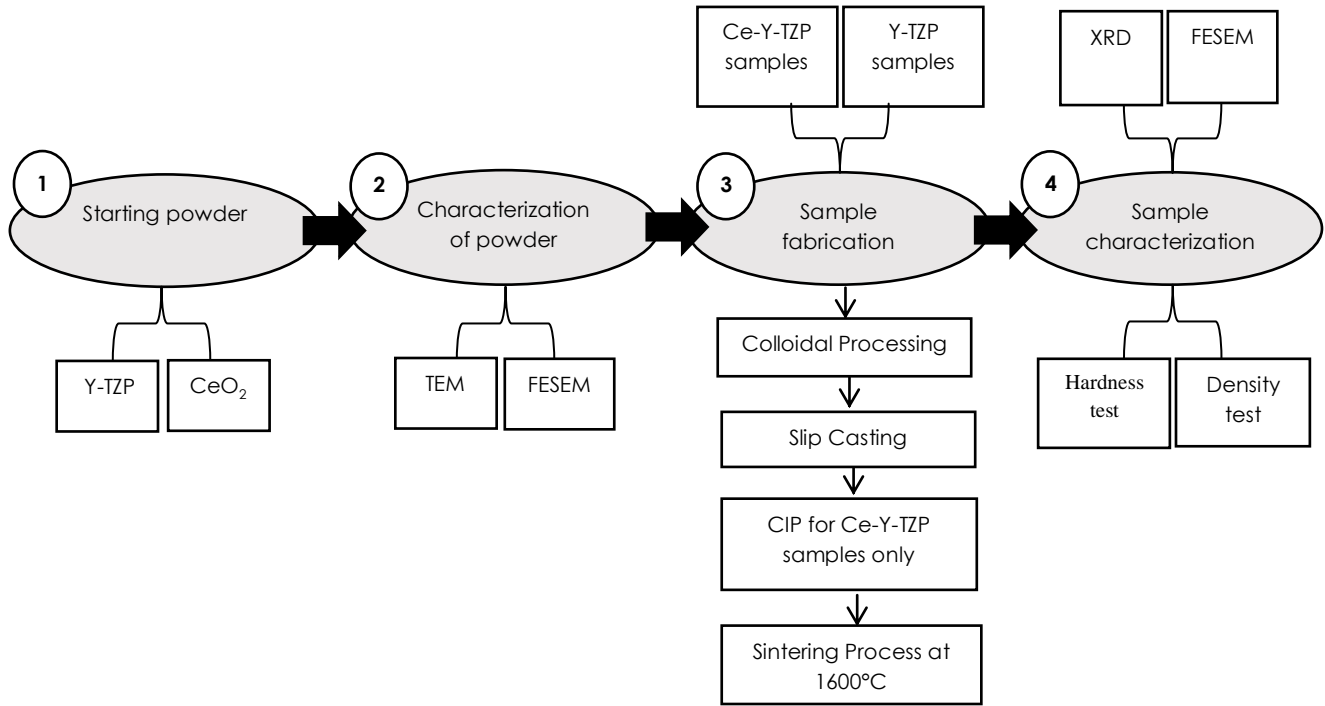


Figure 3 Fabrication and characterization of the samples.

### 3.0 RESULTS AND DISCUSSION

The FESEM micrograph of 3Y-TZP samples sintered at 1600 °C shows non-homogeneous microstructure with multiple porosity with different sizes. On the other hand, the micrograph of Ce-3Y-TZP sample sintered at the same temperature shows nearly no pores with homogenous microstructures. CIP can produce green bodies with high quality, which determines the homogeneity of microstructures of sintered samples [11]. FESEM micrographs of 3Y-TZP and Ce-3Y-TZP are shown in Figures 4 and 5, respectively.

Based on the density test using Archimedes' method, the sintered density of 3Y-TZP samples was 91.8% of the theoretical density, whereas the sintered density of Ce-3Y-TZP samples was 98% of the theoretical density. The presence of porosity was the main factor that decreased the sintered density of 3Y-TZP samples.

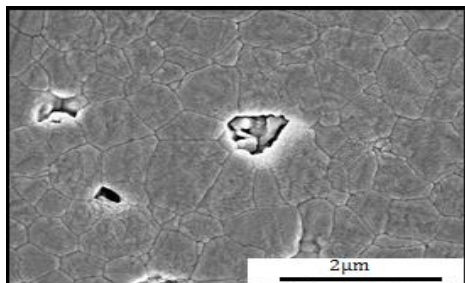


Figure 4 FESEM image of 3Y-TZP

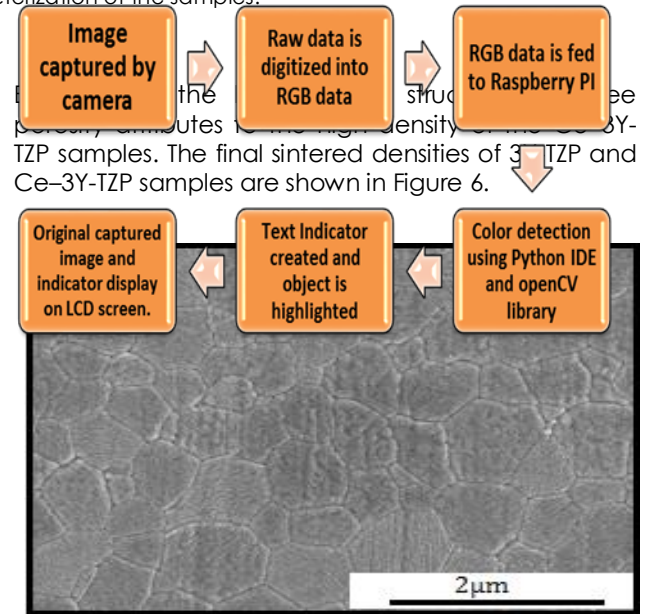


Figure 5 FESEM image of Ce-Y-TZP

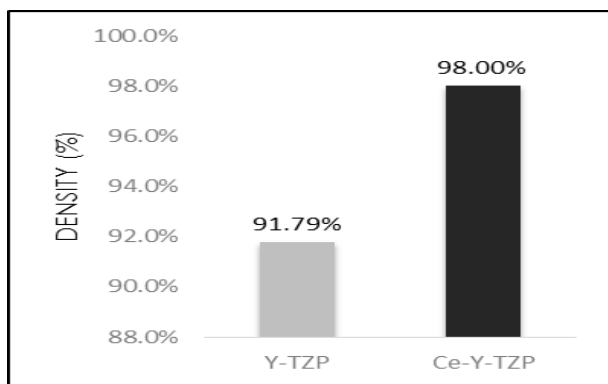


Figure 6 Sintering density of Y-TZP and Ce-Y-TZP

The results of Vickers hardness test show that the average hardness value of 3Y-TZP was 10.33 GPa, whereas the average hardness value of Ce-3Y-TZP was 14.14 GPa. These results are consistent with the density test and FESEM results. The hardness was controlled by the homogeneity of the microstructure and density of both materials. At any rate, the excellent packing efficiency of Ce-3Y-TZP samples that were subjected to slip casting and CIP as consolidation stages lead to the increase in hardness values. The average hardness values of 3Y-TZP and Ce-3Y-TZP samples are shown in Figure 7.

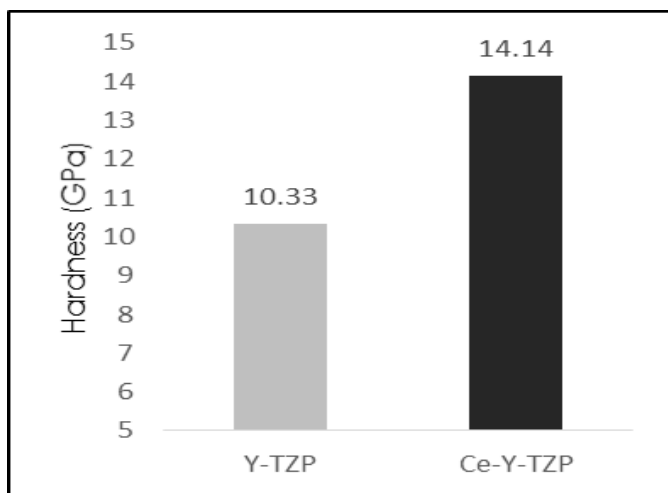


Figure 7 Hardness values of Y-TZP and Ce-Y-TZP.

The Figure 8 of EDX by FESEM shows good distribution of 3Y-TZP and CeO<sub>2</sub> in the Ce-Y-TZP sample. More than one factor can lead to this excellent distribution. This result can be attributed to the slip casting technique, which can enhance the distribution during the suspension stage [10]. CeO<sub>2</sub> can dissolve in the crystal structure of zirconia [4, 5]. XRD results show that the phase was 100% tetragonal for both materials.

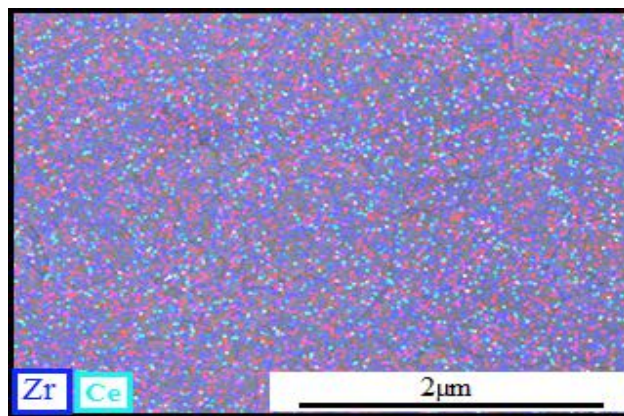


Figure 8 EDX by FESEM for Ce-Y-TZP sample

## 4.0 CONCLUSION

Ce-Y-TZP samples formed by slip casting and CIP had more homogenous grain structures with less porosity than 3Y-TZP samples formed by slip casting alone. Results show that the density of Ce-Y-TZP increased from 91.8% to 98% of the theoretical density and the hardness increased from 10.33 GPa to 14.14 GPa. XRD results show that the phase was 100% tetragonal for both materials. Thus, slip casting accompanied by CIP as the second consolidation technique can enhance the homogeneity of microstructure of zirconia, thereby increasing the mechanical properties.

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## References

- [1] Zarone, F., Russo, S., Sorrention, R. 2011. From Porcelain-Fused-To-Metal To Zirconia: Clinical And Experimental Considerations. *Journal Of Dental Materials*. 27: 83-96.
- [2] Pilathadka S., Vahalová D., Vosáhlo T. 2007. The Zirconia: a New Dental Ceramic Material. An Overview. *Prague Medical Report*. 108: 5–12.
- [3] Garvie, R. C., Hannink, R. H. J., Pascoe, R. T. 1975. Ceramic Steel?. *Nature*. 258: 703-704.
- [4] Manicone, P. F., Iommetti, P. R., Raffaelli, L. 2007. An Overview Of Zirconia Ceramics: Basic Properties And Clinical Applications. *Journal Of Dentistry*. 3: 819 – 826.
- [5] Goff, J. P., Hayes, W., Hull, S., Hutchings, M. T., Clausen, K. N. 1999. Defect Structure Of Yttria-Stabilized Zirconia And Its Influence On The Ionic Conductivity At Elevated Temperatures. *Phys Rev B*. 59(22):14202–19.
- [6] Noor Faeizah Amat, Andanastuti Muchtar, Mariyam Jameelah Ghazali, Norziha Yahaya. 2014. Suspension

- Stability And Sintering Influence On Ytria-Stabilized Zirconia Fabricated By Colloidal Processing. *Ceramics International*. 40: 5413-5419
- [7] Chuin Hao Chin, Andanastuti Muchtar, Che Husna Azhari, Masfueh Razali, Mohamed Aboras. 2015. Optimization Of Ph And Dispersant Amount Of Y-TZP Suspension For Colloidal Stability. *Ceramics International*. 41(8): 9939-9946
- [8] Abi, C. B., Emrullahoglu, O. F., Said, G. 2013. Microstructure And Mechanical Properties Of Mgo Stabilized Zr<sub>02</sub>-Al<sub>2</sub>O<sub>3</sub> Dental Composites. *J. Mech. Behav. Biomed. Mater.* 18: 123-131.
- [9] Chevalier, J. and Gremillar, L. 2009. Ceramics For Medical Applications: A Picture For The Next 20 Years. *J. Eur. Ceram. Soc.* 29:1245-1255.
- [10] Mouzon, J., Glowacki, E., Oden, M. 2008. Comparison Between Slip-Casting And Uniaxial Pressing For The Fabrication Of Translucent Ytria Ceramics. *J. Mater Sci.* 43: 2849-2856.
- [11] Henderson, R.J., Chandler, H.W., Akisanya, A.R., Barber, H., Moriarty, B. 2000. Finite Element Modelling Of Cold Isostatic Pressing. *Journal of the European Ceramic Society.* 20:1121-1128.