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Sintering and Densification Behavior of Nb_2O_5 - doped Y-TZP Ceramics

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Abstract-- *The effect on densification and mechanical properties of 3 mol% Y-TZP ceramics doped with niobium oxide (0.1 wt. % to 1 wt. %), has been investigated in this study. Green samples were compacted by uniaxial pressing and cold isostatically pressed at 200 MPa. All samples have been sintered over the temperature range of 1250°C to 1500°C with a ramp rate of 10°C/ minute and 2 hours of holding time. The sintered bodies were examined to determine the phase content, bulk density, Vickers hardness and flexural strength of the Y-TZP material. The results showed that the addition of 1 wt. % Nb_2O_5 was effective in aiding densification (~99% theoretical density). It also improved the Vickers hardness (~13 GPa), when the samples were sintered below 1300°C compared to the undoped Y-TZP sintered at the same temperature. The Nb_2O_5 addition to the Y-TZP matrix also displayed an increment of flexural strength as the dopant content was increased.*

Keywords-- *Niobium Pentoxide; sintering behaviour; Y-TZP; Ageing*

I. Introduction

In 1999, Piconi and Maccauro [1, 2] pointed out that yttria-stabilized tetragonal zirconia (Y-TZP) has become increasingly popular as an alternative high mechanical and physical properties compared with other materials [3]. ZrO_2 is a polymorphic material that exists in three allotropes i.e. cubic (c), tetragonal (t), and monoclinic (m) symmetry and stable at high temperatures [4]. Y-TZP ceramics are used widely in many fields; creating machine parts or components, dental implants or even biomedical applications; out of which biomedical used zirconia would probably possess the best mechanical properties among all other single-phase oxide ceramics [5-7]. One of the major limitation of Y-TZP ceramics is aging or low temperature degradation. In order to suppress LTD in Y-TZP, the inclusion of sintering additive such as Nb_2O_5, CeO_2, Al_2O_3, MgO, etc [8]. Additives make Zirconia more stable, but may affect the mechanical and physical properties. Additionally, Y-TZP ceramics have been shown to have superior shock resistance over other ceramics such as mullite and alumina [9, 11], thus rendering this material suitable for high temperature applications [12, 13].

Yttria stabilized zirconia is also used as an electrolyte for ionic conduction application such as oxygen sensors [14-16]. Lange [17] concluded that the retention of the tetragonal phase depends on the density achieved during fabrication. The sintering temperature is a prime factor which affects the final density of the ceramic and affected the crystalline phase and microstructure of the sintered body and the heating rate during sintering does have some contributing effect on the densification rate of Y-TZP ceramics. Nano-particle clustering which increase with increasing volume fraction of nano-particle, led to a reduction in both the toughness and strength of the nano-structured ceramics [18-

20]. It was found that the larger the size of the clustered particle, the more defects it contains and this make it easier for a crack to pass through the clustered particle.

II. Experimental Techniques

A. Sample Preparation

Y–TZP powder will be mixed with various weight percentages of Nb2O5 (0.1- 1.0wt %) to produce mixture for each weight percentage of Nb2O5 respectively. Ethanol of 99.9% purity is added to the mixture in a high-density polyethylene (HDPE) bottle and ball-milled. The resulting mixture is then dried in an oven and screened to obtain a powder form precipitate. A uniaxial pressing method was used to produce the disc specimens 20 mm in diameter and bar specimens (32 mm x 13 mm x 6 mm). A pressure of 200 MPa was used to compact the powder into the desired disc and bar shape according to their respective dimensions. Samples were sintered in temperatures of 1200 °C, 1350 °C and 1500 °C. During the sintering process, a ramp-rate of 10°C / min was used for the heating and cooling process.

B. Characterization

Bulk density of the sintered specimen for this experiment was then determined by the water immersion technique which uses the Archimedes principle. In this process, the weight of the sample was first determined by suspending in the air. This process is then submerged under water to identify the weight in water. The density of the sample can be calculated by using the formula below.

$$\rho = \frac{ma}{ma - mw} \times \rho_w \quad (1)$$

The Vickers hardness test is used to determine the hardness of an object. A punch with a diamond pyramidal shaped tip which consists of a 136° tip is used to indent the polished sample. The indenter applies a force of 100N which leaves a diamond shaped dent on the surface of the sample. The Vickers hardness [21, 22] and fracture toughness are calculated based on the surface area of the indent can be determined by using the following formula:

$$Hv = \text{Constant} \frac{\text{test forcete}}{\text{indent diagonal squared}} = \frac{(1.854\ P)}{(2a)^2} \quad (2)$$

$$K_{IC} = 0.019P\frac{(E/H)^{1/2}}{C^{3/2}} \quad (3)$$

III. Results and Discussion

The effect of mechanical properties of Y-TZP (Yttria stabilized Tetragonal Zirconia Polycrystalline) doped with various amounts of Nb2O5 (Niobium Pentoxide) which would include 0.1 wt %, 0.3 wt %, 0.5 wt % and 1.0 wt % and sintered at temperatures of 1200°C, 1350°C and 1500°C were analyzed .

A. Bulk Density

The effect of sintering temperature and percentage content of Nb_2O_5 on the bulk density of the samples can be seen in Figure 1. The results revealed that relative density 98% of theoretical (i.e ≥ 5.96 Mg m^{-3}) could be obtain in Y-TZP sintered at low temperature, 1500°C, with the additions of From the figure it was observed that the sample containing ≥ 0.3 wt. % Nb_2O_5. It was found that the bulk density of the samples for all weight percentages increases as the sintering temperature increases.

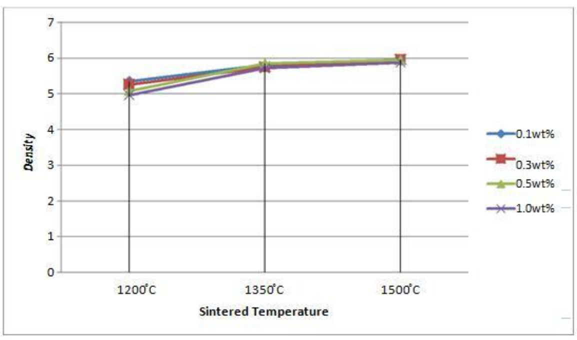

Figure 1. The effect of sintering temperature and percentage content of Nb_2O_5 on the bulk density of the samples.

B. Dimensional Shrinkage

Figure 2 and 3 showed the linear shrinkage and volumetric shrinkage of the Nb_2O_5 doped Y-TZP. From these figures, it is noticed that the percentage shrinkage of all the samples containing varying amounts of Nb_2O_5 increases as the sintering temperature increases. The volumetric shrinkage of 0.5 wt % sintered at temperatures ranging from 1200°C to 1350°C shows an increase in shrinkage from 63.98 % to 71.82 % which gives a difference in increment of 8.28%. It was also can be deduced that the sintered temperature is also inversely proportional to the level of porosity therefore as the sintering temperature increases, the level of porosity in the samples will drop.

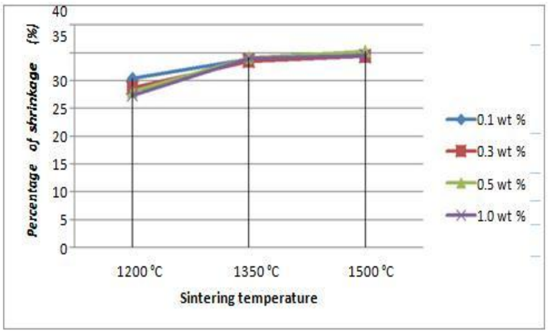

Figure 2. The effect of sintering temperature on the linear shrinkage based on the weight percentage of Nb_2O_5.

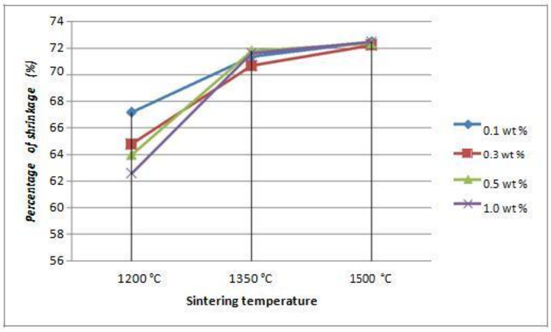

Figure 3. The effect of sintering temperature on the volumetric shrinkage based on the weight percentage of Nb_2O_5.

C. Vickers Hardness

The effect of sintering temperature and Nb_2O_5 on the vickers hardness of Y-TZP is shown in Figure 4. The results clearly show the beneficial effect of Nb_2O_5 in improving the hardness of the Y-TZP at low sintering temperature. The samples with 0.1 wt % possess the highest hardness values at 1500°C sintered temperatures which is 23.95 GPa. Similar observation was noted for the hardness trend of 0.3 wt % and 1.0wt % Nb_2O_5-doped Y-TZPs with increasing sintering temperature. The increasing in hardness for samples sintered at high temperature can be attributed to the increased proportion of transformable tetragonal phase and associated pseudo plasticity.

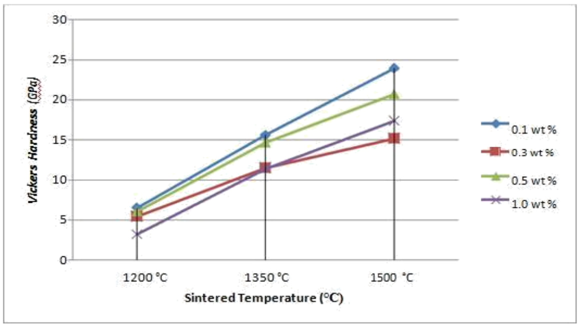

Figure 4. The effect of sintering temperature and percentage content of Nb_2O_5 on the hardness of the sample.

D. Flexure Strength

The effect of sintering temperature and percentage content of Nb_2O_5 on the flexure strength of the samples can be seen in figure 5. The samples containing 0.5 wt % of Nb_2O_5 shows a vast increment of flexure strength from 1.76 MPa to 5.2 MPa at sintering temperatures 1200 °C to 1500 °C. The highest value of flexural strength is showed when 1.0wt% MgO is added at sintering temperature 1350°C. The high flexural strength generated is probably due to the reduced grain size of the size, hence reducing thermal stress as well as improving strength.

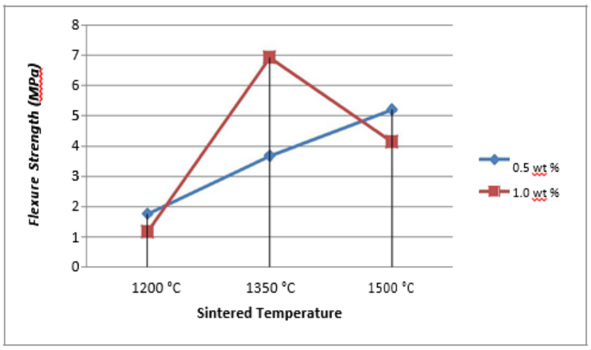

Figure 5. The effect of sintering temperature and percentage content of Nb_2O_5 on the flexure strength of the sample.

E. Fracture Toughness

The variation in the fracture toughness of Nb_2O_5-doped Y-TZP is shown in figure 6. The samples with the highest fracture toughness are that samples containing 0.5 wt % Nb2O5 that were sintered at temperatures 1200°C and 1500°C, both samples attain a value of 7.31 MPa. The reason for these two samples possessing the same toughness value could be due to the grain growth during the sintering process. If there are large grain growths present in the sample, the fracture toughness would decrease.

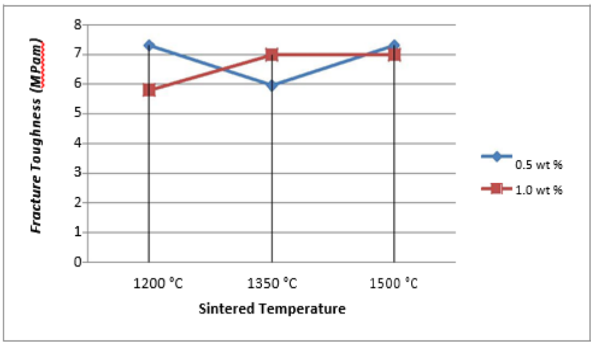

Figure 6. The effect of sintering temperature and percentage content of Nb_2O_5 on the fracture toughness of the sample.

IV. Conclusion

The results of mechanical properties of Y-TZP doped with various amounts of Nb2O5 (Niobium Pentoxide) presented and discussed. The effect of different mass percentage of Nb_2O_5 and sintered temperatures were investigated. As the temperature increases, the level of shrinkage will also increase. The highest shrinkage level is found at 1500°C. The samples show an increasing trend in bulk density as the sintering temperature increases where the level of porosity decreases, therefore the samples with the highest density value can be found at the sintering temperature of 1500°C. The hardness values of the samples are proportional to the increase of sintering temperature. The beneficial effect of Nb_2O_5 doping in suppressing the hydrothermal degradation of Y-TZP ceramics has been revealed. In particular, the addition of 0.1 wt% Nb_2O_5 was found to be most effective in ageing induced phase transition.

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