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Evaluation of Nd₂O₅ Doped Y-tzp Using two step Sintering Method

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ABSTRACT

Background: An investigation was carried out to evaluate the mechanical properties of Nd₂O₅ doped 3Y-TZP using the two-step sintering method. A pressure less sintering technique was used to sinter samples over a temperature range of 1300°C to 1500°C with a ramp rate of 10°C/ minute and a holding time of 2 hours. Sintered bodies were tested to determine the bulk density, hardness, Young's Modulus and fracture toughness. The results show that bulk density, hardness and fracture toughness was enhanced by the addition of 0.3 wt. % Nd₂O₅. The two step sintering method was also prominent in improving Young's Modulus. An increasing trend is seen from 0.3 wt. % up to 0.5 wt. % addition of Nd₂O₅.

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INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is known to possess unique properties such as high melting point, chemical inertness, biocompatibility and excellent mechanical properties (Jia Lin *et al.*, 2012) and (S. Ran *et al.*, 2007). The biocompatibility, high strength and fracture toughness of Y-TZP ceramics make it suitable for structural and biomedical applications. (Chih-Liang Yang *et al.* 2005), (Xiao Huang, 2008) and (J. Vleugels *et al.*, 2002)

Yttria-tetragonal zirconia polycrystal (Y-TZP) compared to other ceramics possess an ability to absorb energy from propagating crack, which prevents further crack development (Tao Xu *et al.*, 2004), (Shubin Wang *et al.*, 2013) and (G.A. Gogotsi 2012). This phenomenon is known as transformation toughening. In this mechanism, the energy absorbed by the zirconia matrix in the vicinity of the propagating crack is consumed by the tetragonal (t) grains to transform to the monoclinic (m) symmetry which is accompanied by approximately 3 to 4% volume expansion (J. Vleugels *et al.*, 2002), (D. Casellas *et al.*, 2001), (Mahmood Mamivand, 2014) and (E. Apel 2012).

However, Y-TZP suffers from a limitation known as low-temperature degradation (LTD). F. Zhang *et al.* (2013), S. G. Huang *et al.* (2005) and Peter Tatarko *et al.* (2014) observed that Y-TZP ceramics exhibit slow t-m phase transformation, beginning at the surface and proceeds to the formation of microcracking. Also, they reported that the mechanical properties of the material face a major decrease due to the poor resistance of the material to the effects of humid atmospheres at temperatures ranging anywhere from 60 to 500°C.

Experimental Techniques:

Two powders were prepared for this experiment; the 3 mol% of yttria-stabilised zirconia powder as the main powder and neodymium oxide (Nd₂O₅) as the dopant. Compositions were prepared via ball-milling in ethanol using zirconia balls as the milling media. The resulting slurry was oven dried, sieved and then uniaxially pressed (3 discs and 1 bar for each profile) at 0.3MPa. The samples were cold isostatically pressed at 200 MPa before being sintered at temperatures ranging from 1200°C to 1500°C, at 10°C/min ramp rate and a holding time of 2 hours.

Characterization:

The bulk density of the sintered samples was determined based on Archimedes' Principle using the water immersion method (Mettler Toledo AG204). The Vicker's hardness (Hv) and fracture toughness (K_{IC}) was measured on polished samples using the Vicker's indentation method, where a load of 100N was applied for 10

seconds to the polished samples. Vicker's hardness, H_v was calculated using the following equation (A. Mohajerani 2012).

$$H_v = 1.854P / a^2 \quad (1)$$

P is the applied load and a is the indent half diagonal (Huang, 2005). The K_{IC} was computed according to the equation derived by Antis *et al.* which was recently modified by (Kaliszewski, *et al.* 1994) (Mary S. Kaliszewski *et al.*, 1994).

The Young's modulus test was conducted on the bar samples by impulse excitation technique using the commercial testing instrument (GridoSonic: MK5 "Industrial", Belgium (ASTM 1998)).

RESULTS AND DISCUSSIONS

The variation of bulk density for 3Y-TZPs with different amounts of Nd_2O_5 sintered at a temperature range of $1300^\circ C - 1500^\circ C$ is shown in Figure 1. A decreasing trend is observed for amounts $>0.3wt\%$ Nd_2O_5 with increasing sintering temperature. The temperatures ranging between $1300^\circ C$ to $1400^\circ C$ were found to be the most profound sintering temperature as samples sintered within this temperatures display an increasing trend. This can be due to the over-stabilized phase condition where the phase takes the transformation from tetragonal to cubic phase.

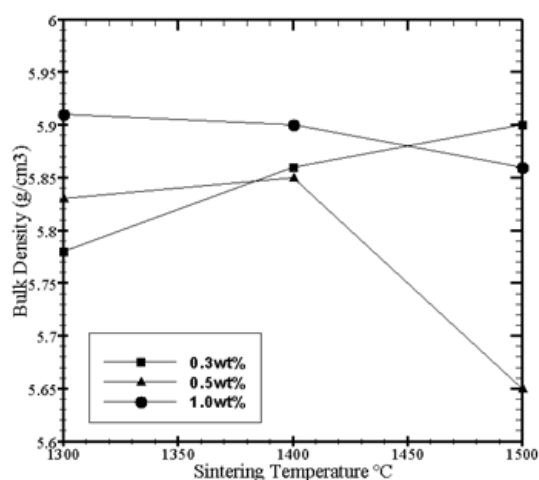


Fig. 1: Effect of sintering temperature and Nd_2O_5 addition on the Bulk Density of Y-TZP.

The variation of Young's Modulus (E) of sintered samples with increasing sintering temperature is shown in Figure 2. The major effect of Nd_2O_5 in enhancing the matrix stiffness of Y-TZP can be seen particularly when sintered at low temperature, at $1300^\circ C$, where 0.3wt% and 0.5wt% samples reached almost the theoretical value of the Young's modulus. The Young's modulus of 0.3wt% Nd_2O_5 -Y-TZP increased gradually as the sintering temperature increased but started to drop after $1400^\circ C$ whereas in contrast the samples with $\geq 0.3wt\%$ showed no decrease.

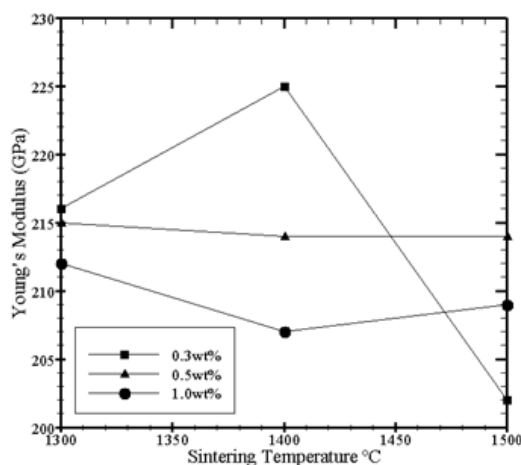


Fig. 2: Effect of sintering temperature and Nd_2O_5 addition on the Young's modulus of Y-TZP.

Figure 3 shows the effect of fracture toughness of the Nd₂O₅-Y-TZP samples where it is seen that the additions of niobium oxide have an effect on the fracture toughness of 3Y-TZP. There was a remarkable enhancement in fracture toughness of the 1.0wt% Nd₂O₅-Y-TZP samples compared to <1.0wt% samples sintered at 1300°C. The samples with 1.0wt% Nd₂O₅-Y-TZP attained a value of 6.9 MPam^{1/2} while the samples with <1.0wt% Nd₂O₅-Y-TZP only managed values of about 6.7 MPam^{1/2}. Generally, high fracture toughness would indicate that the (t) tetragonal grain was in a metastable state and responded immediately to the stress field of propagating crack, such as induced during the indentation test.

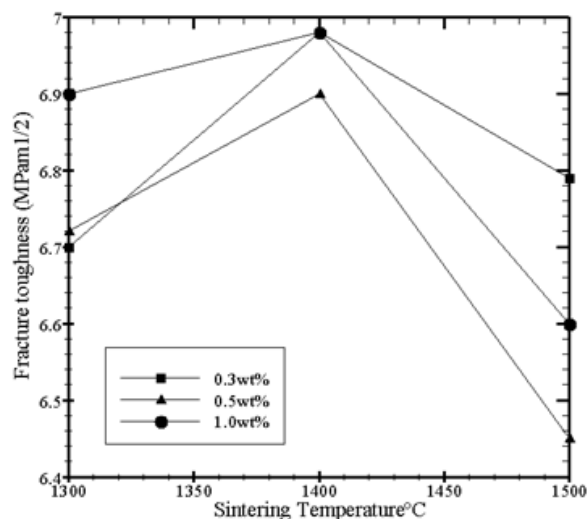


Fig. 3: Effect of sintering temperature and Nd₂O₅ addition on the fracture toughness of Y-TZP.

The effect of sintering temperatures and Nd₂O₅-Y-TZP samples on the Vickers hardness of Y-TZP is shown in Figure 4. The lowest value was of the 1.0wt% Al₂O₃-Y-TZP hardness was around 13.6 GPa when sintered at 1300°C which surpassed the theoretical hardness value while other doped samples showed good hardness values too. The hardness values increased gradually till 1400°C and dropped significantly upon further firing at 1500°C. Good hardness values were obtained at lower sintering temperature.

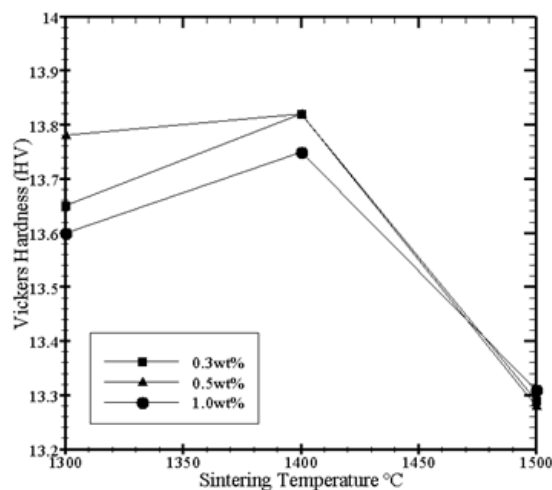


Fig. 4: Effect of sintering temperature and Nd₂O₅ addition on the hardness of Y-TZP.

Conclusion:

The current work has shown that the addition of neodymium oxide up to 0.3 wt% with Y-TZP ceramics was found to be beneficial as a sintering aid as the density was improved to ~5.9 g/cm³ (~98% of the theoretical density) when sintered at 1400°C. Doping Nd₂O₅ into 3Y-TZP also enhanced other mechanical properties; Young's modulus was enhanced to ~225 GPa, fracture toughness was increased to ~6.9 MPam^{1/2} and the hardness was increased to ~13.8 GPa. Experimental results do not support with the other researchers' work which state that best sintering temperature would be between 1500°C-1550°C. The reason is due to the starting

powder which having very high agglomeration rate and affects the experiment during the green body preparation.

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