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ZTA Hardness Enhancement via Hot Isostatic Press Sintering

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Abstract. The present work mainly deals with the influence of Y_2O_3 stabilize ZrO_2 (YSZ) additions on the phases, microstructure and mechanical properties of Al_2O_3 which is known as zirconia toughened alumina (ZTA) through hot isostatic press (HIP) sintering. Different amounts of 8 mol% of yttria stabilize ZrO_2 (8YSZ) were added into Al_2O_3 and the amount of 8YSZ was varied from 0 wt.% to 25 wt.%. There is significant evidence that microstructural coarsening within ZTA was found to produce an improvement of hardness and fracture toughness. These improvements are the evidence for the toughening mechanism through the transition of tetragonal to monoclinic (t-m) structure provide by 8YSZ which is defined to optimize the materials and mechanical properties of Al_2O_3 ceramics. The 8YSZ grains and Al_2O_3 grains are impartially distributed among each other. The XRD patterns are corresponds to yttria doped zirconia $(Zr_{0.935}Y_{0.065})O_{1.968}$ peaks (ICDD files No. 01-078-1808) and Al_2O_3 (No. 00-010-0173). The existence of $(Zr_{0.935}Y_{0.065})O_{1.968}$ phase relates to the metastable tetragonal form of ZrO_2.

1. Introduction

The excellent properties such as good corrosion resistance, high hot hardness, and high chemical resistance make alumina (Al_2O_3) based ceramic a very promising candidate as material for implant and cutting tools applications. However, their application as cutting tools is restricted due to their brittleness [1–3]. Therefore, many researchers focus on their toughening improvement through secondary phase dispersion approach [4–9], however this approaches sacrificing their hardness for higher quality applications [3,4]. There are several factors that influence the deterioration of the hardness properties. For example, when 5 mol% of Y_2O_3 is added to ZrO_2 as stabilizer, certain amounts of lattice defects, e.g. oxygen vacancies formed in the ZrO_2 lattice [10]. Tekeli et al. [11] state that most promising results are reported from the addition of Al_2O_3 to the cubic YSZ known as zirconia toughened alumina (ZTA), where both enhanced densification behaviour and improvement in electrical conductivity is obtained together with improved mechanical strength. They found that 8 mol% of YSZ (8YSZ) will affect the sintering and grain growth behaviour of the alumina. Furthermore, they found that the alumina with 8YSZ achieved a density of 95% of its theoretical value. However, these findings shows that reduction of hardness value still remain as an issue due to an existence of porosity on the Al_2O_3 crystal lattice. An improvement of porosity related to densification of materials generated during the sintering process.

There are several categories of sintering such as solid state sintering, vitrification, viscous sintering and liquid state sintering [12]. Under some conditions, densification achieved by any of four categories of sintering might be inadequate. A solution to this problem is the application of external pressure during heating, giving the method of pressure sintering (of which hot pressing and hot isostatic pressing are common examples). Pressure sintering has the disadvantage of increased fabrication costs but effective when a microstructure of high density and fine grain size must be guaranteed. To distinguish it from pressure sintering, sintering performed without the application of an external pressure is usually referred as conventional sintering [13,14].

Hot isostatic pressing (HIP) has been used in powder metallurgical applications to densify the materials and for single crystals to reduce the porosity generated during the casting and homogenization processes. HIP can be applied to a wide range of metals, alloys and ceramics [15]. Furthermore, this process has been recognized to be an effective sintering technique to achieve full densification [13]. Porosity reduction by means of HIP has a significant beneficial effect on the certain applications [14]. In this paper, the effectiveness of using HIP towards the microstructure and hardness of alumina-8YSZ (ZTA) ceramic has been investigated. Therefore, the influence of HIP condition is believed to affect the microstructure by reducing the porosities and form densified ceramic.

2. Experimental Method

The composition of 8 mol% of yittria stabilize zirconia (8YSZ) was varied from 0 wt.% to 25 wt.%. The starting materials was mixed continuously for 8 hours and dried in oven at 90 °C for 24 hours [3]. The mixture is then subsequently hydraulically pressed into cylinder mould (Ø 13 mm) at 10 MPa to form green bodies. The pressed samples were sintered in the hot-isostatic pressing (American Isostatic Presses, INC HP630) at 1600 °C for 1 hour soaking time under 150 MPa using Ar gas. The pressureless sintering was carried out to perform a comparison between the two techniques of the same mate- rials. The analysis of structure was carried out by X-ray diffraction (XRD) using the Bruker D8 Advance with CuKa radiation (40 kV, 30 mA) diffracted beam monochromator, using a step scan mode with a step size of 0.1° in the range of 20°–80° of 2ϴ°. Field emission scanning electron microscopy (Carl Zeiss, Germany) was employed to study the microstructure of the grinded and polished samples. Sample grinding process was executed using Metkon 120, 240, 500, 800 and 1200 grit alumina abrasive grade papers. The samples were polished by using Imptech grinder polisher with 1 μm and 0.05 μm Al_2O_3 particle as polishing medium. The density and porosity values were obtained according to the ASTM C 830-00 test procedure. The Vickers hardness test (Shimadzu HSV-20) was carried out to obtain both Vickers hardness values.

3. Results and Discussion

ZTA contains yittria stabilize zirconia particles that increase its fracture by microcracking and by stress induced tetragonal-to-monoclinic phase transformation. Figure 1shows the stacking of XRD analysis for Al_2O_3 ceramics mixed with various amount of 8YSZ (0 wt.% to 20 wt.%). The patterns are correspond to yttria doped zirconia $(Zr_{0.935}Y_{0.065})O_{1.968}$ (ICDD files No. 01-078-1808) and corundum (ICDD files No. 00-010-0173) phases. A quantitative evaluation of phase analysis was performed using the EVA software at 2ϴ° = 17° - 89° and recorded in Table 1. The addition starts at 5 wt.% 8YSZ which shows 98% corundum and 2% of 8YSZ. As the addition increase to 10 wt.% of 8YSZ the value of corundum decrease to 95% while the 8YSZ (wt.%) increase to 5%. In this work, the presence of $(Zr_{0.935}Y_{0.065})O_{1.968}$ phase plays significant roles in determining the properties on Al_2O_3 ceramics. XRD analysis demonstrates the presence of monoclinic ZrO_2 crystal structure, it is an unavoidable phenomena and the small amount of monoclinic ZrO_2 would not influence the composition overall. Additionally, a work directed by Azhar et al. [16] expressed that no less than 15% of monoclinic stage will dependably be available. In this work, $(Zr_{0.935}Y_{0.065})O_{1.968}$ phase proved to preserve fine grains as shown in Figure 2 which is related to the grain boundary inhibition. This finding is in good agreement

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with previous works [17] which is report that grain boundary inhibition can be due to the pinning effect, which is associated with particles locations at grain boundaries.

Table 1: Phase percentages of Al_2O_3 ceramics with the addition of 8YSZ

8YSZ (wt.%)	Corundum (%)	YSZ (%)
0	100	0
5	98	2
10	95	5
15	93	7
20	92	8
25	90	10

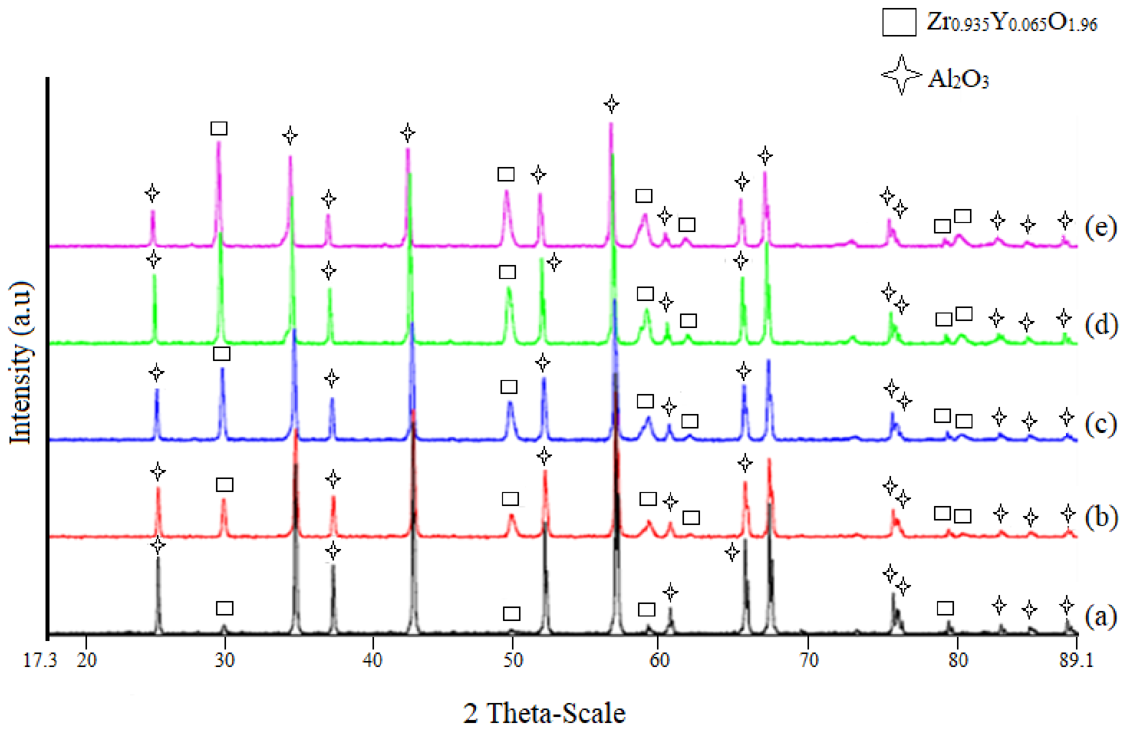

Figure 1. X-ray diffraction patterns of the sintered Al_2O_3 based ceramics added 8YSZ, (a) 5 wt.%, (b) 10 wt.%, (c) 15 wt.%, (d) 20 wt.% and (e) 25 wt.%.

Based on Figure 2, the SEM micrograph for $(Zr_{0.935}Y_{0.065})O_{1.968}$ phase showed the bright grains appeared as small rounded grains and the microstructure of Al_2O_3 with the dark grains and round shapes. There are some large Al_2O_3 grains that are formed due to the coalescence of the grains, which is related to grain boundary migration [18]. The result of grain sizes (Figure 3) were found to be decreased from 14 µm to 5 µm with various 8YSZ additions on Al_2O_3 ceramics, respectively. The significant role of $(Zr_{0.935}Y_{0.065})O_{1.968}$ phase, are more efficient in pinning the grain boundaries towards the grain growth. In the present work, further 8YSZ additions in alumina ceramics show that the distribution of the particles is very uniform and combine with each other to form an overall. Basically, reduce pore and gap exist. Therefore the structure becomes compact, and crystal grains are refined with the adding of 8YSZ, thus mechanical properties of these ceramics are far better than those of pure alumina ceramics. Hardness of ceramics is usually affected by the microstructural features such as multiphases, grain size, porosity and grain boundary constitution [19].

The effect of various amount 8YSZ additions on the Vickers hardness and porosity is shown in Figure 4. It is observed that the percentage of the porosity dropped, the hardness of the materials is increase accordingly. At the beginning, 5 wt.% 8YSZ has 1650.21 HV and 19.48% porosity. The value of hardness is increase to the highest point which is 1706.74 HV as the percentage of porosity decreased to the lowest point to 1.94% for 15 wt.% 8YSZ addition. At composition higher that 15 wt.% 8YSZ, the value of hardness decreased as the percentage of porosity increased. At 15 wt.% of 8YSZ addition, the grains starts to growth and the 8YSZ start to diffused inside the alumina and gradually filled these pores. Therefore, with the less amount of porosity, the ZTA able to increase the hardness at the optimum composition which is 15 wt.% 8YSZ [20]. However, further addition of 8YSZ will make the Al_2O_3 ceramic become more brittle with the addition of more than 20 wt.% of 8YSZ such as at 25 wt.% . These increase the percentage of porosity up to 15% and reduce the hardness to 1448.9 HV (13% reduced).

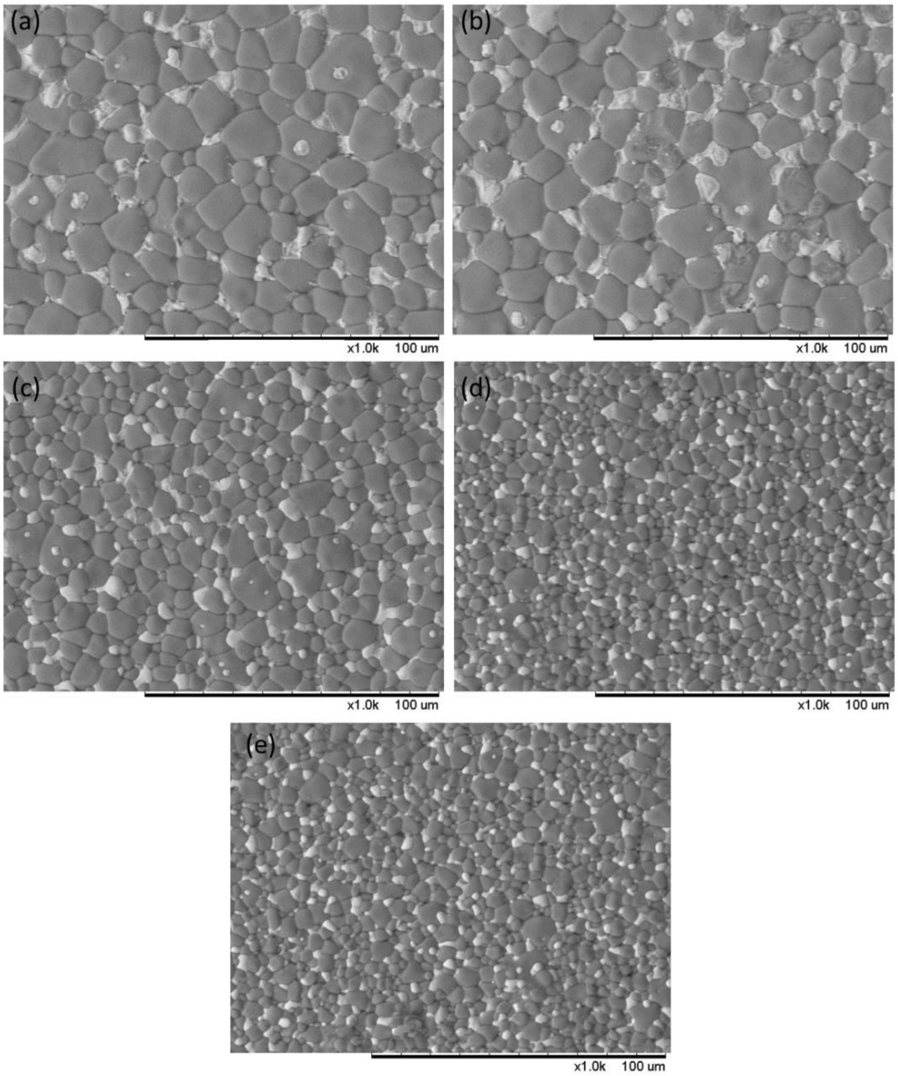

Figure 2. SEM micrograph corresponding to the various additions of 8YSZ on Al_2O_3 ceramics, (a) 5 wt.%, (b) 10 wt.%, (c) 15 wt.%, (d) 20 wt.% and (e) 25 wt.%.

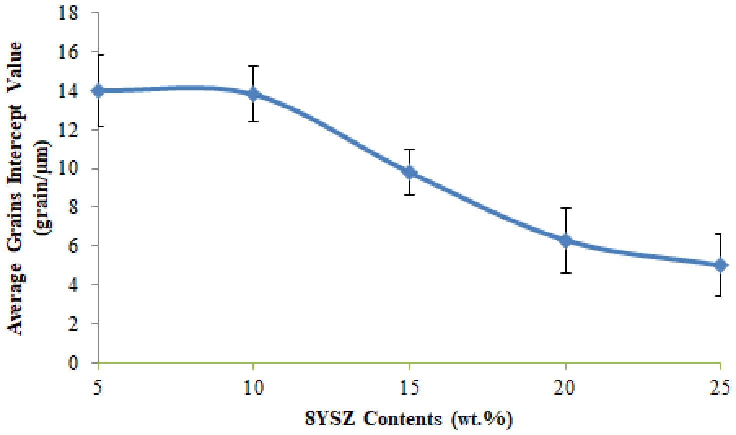

Figure 3. Average grains intercept value for Al_2O_3 ceramics with various 8YSZ contents.

Table 2 shows the optimum values of Vickers hardness reported from previous authors and their similar corresponding compositions of YSZ addition in Al_2O_3 ceramics. Hossen et al. [21] and Kwon et al. [22] found that sintering of 20 wt.% of 8YSZ with conventional method (presureless sinter) at 1600 °C shows maximum hardness are 1175 HV and 1193 HV, respectively. The current work shows that by using HIP the maximum hardness are 1537.26 HV which is significantly improved (~23%). By comparing previous findings and the current work, it's proved that the effectiveness of using HIP towards the Vickers hardness of Al_2O_3 ceramics has been achieved.

Table 2. Authors and their corresponding compositions of YSZ involved in Al_2O_3 ceramic

References	YSZ (wt.%)	Firing Method (1600 °C)	Vickers Hardness
[23]	20	Conventional	1175 HV
[22]	20	Conventional	1193 HV
Current	20	HIP	1537.26 HV

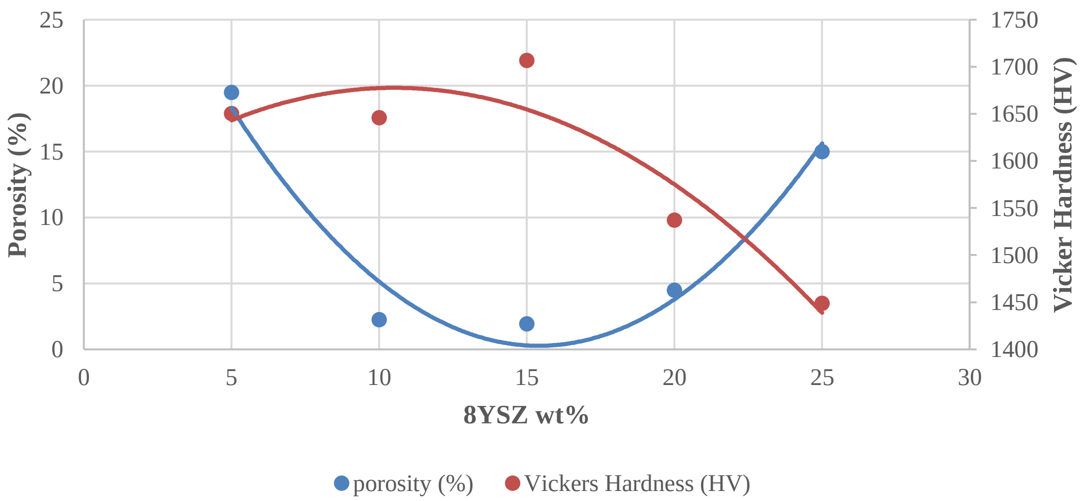

Figure 4. Relationship between hardness and percentage of porosity.

4. Conclusion

The values of the Vickers hardness for the toughened Al_2O_3 ceramics were controlled through addition of different quantities of 8YSZ. Superior properties of ZTA ceramics achieved by adding 15 wt.% of 8YSZ. The ceramics exhibited the highest Vickers hardness (1706.74 HV), and the lowest porosity (1.94%). The presence of 7% of $(Zr_{0.935}Y_{0.065})O_{1.968}$ phase is considered as the major contributor to optimize the mechanical properties of Al_2O_3 ceramics. Therefore, by using hot isostatic press sintering (HIP) technique proved to improve the hardness properties of Al_2O_3 ceramics.

Acknowledgments

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