International Journal of Current Science, Engineering & Technology
ISSN : 2581-4311
Original Research Article | Open Access | AMCT 2017 Malaysia | *Special Issue*

Microstructure and Hardness of Zirconia Toughened Alumina (ZTA) Ceramics by Hot Isostatic Press Sintering

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ABSTRACT. Mechanical properties of 8YSZ toughened alumina (ZTA) have been investigated. The compositions of 8YSZ as reinforce were varied from 0 wt.% to 25 wt.%. Both Al_2O_3 and 8YSZ powders was mixed and sintered at 1600 ºC for 1 hour using hot isostatic pressed sintering (HIP). The phases analysis were identified as corundum (Al_2O_3) and zirconium yttrium oxide ($Zr_{0.88}Y_{0.12}O_{1.94}$). The hardness values for ZTA ceramics produced through HIP sintering found to improve the hardness of ZTA ceramics. The Vickers hardness results show that 15 wt.% of 8YSZ produced the optimum hardness which is the highest value of 1706.74 HV. In addition, the percentage of porosity appears to be diminished (~30%).

Keywords: *Vickers hardness, Hot isostatic press (HIP), Porosity, Density;*

Received: *15.10.2017*, Revised: *15.12.2017*, Accepted: *30.02.2018*, and Online: *20.03.2018;*

DOI: *10.30967/ijcrset.1.S1.2018.135-140*

Selection and/or Peer-review under responsibility of Advanced Materials Characterization Techniques (AMCT 2017), Malaysia.

1. INTRODUCTION

The introduction of stabilize ZrO_2 into Al_2O_3 as a sintering aid has been long practiced for densification of Al_2O_3 [1-5]. The best way to stabilize the cubic zirconia phase by using additives like Y_2O_3, CaO, MgO and CeO_2 [6-8]. Amount of stabilizer plays an important role on size of tetragonal crystals which determines the critical stress required to activate the stress induced phase transformation. Increasing grain size yields a lower activation stress, which then leads to both larger transformed zones and act as shielding effects around a propagating crack [6].

However, while most of the researches have concentrated on toughening mechanism in order to improve the toughness of ZTA ceramics only but not to its hardness which is neglected their hardness properties. The approach under investigation here is the addition of intergranular phases of 8YSZ to limit grain growth as a first step and the application of external pressure from HIP as second step. 8YSZ is high temperature refractory ceramics composed of zirconium oxide stabilized with 8% yttrium oxide. Rahmawati et al. [11] reported that 8YSZ show homogeneous morphology and without provides a line crack. Meanwhile, 8YSZ is a cubic form and has no phase transformation during heating from room temperature up to 2500°C and cooling [12]. During heating, the methods of pressure sintering such hot isostatic pressing (HIP) are common examples. Although, HIP has the disadvantage of increased fabrication costs, but it is effective when a microstructure of high density and fine grain size must be guaranteed.

Therefore, the effectiveness of using combination of 8YSZ properties together with HIP sintering towards the microstructure and hardness of ZTA has been investigated. The porosity reduction by means of HIP has a significant beneficial effect on 8YSZ toughened alumina [13]. Thus, theinfluence of the combination process is believed to affect the microstructure by reducing the porosities and form densified ceramic.

2. MATERIALS AND METHODS

As starting powders, Al_2O_3 (99.0% purity) and commercially 8YSZ refer to 8 mol.% yttrium stabilized zirconia (94.6% purity) were used. The amount of 8YSZ was varied in different wt.% (0 to 25 wt.%) into the Al_2O_3 matrix by wet mixing method. The mixtures powders were uniaxial press at 10 MPa into cylindrical shapes mold with dimensions of Ø13 mm and 15 mm in height and the green body were HIP sintered at 1600 ºC in argon gases with 150 MPa for 1 hour. Pressureless sintering technique have been carried out at 1600 ºC for 4 hours soaking period as baseline samples for the purpose of improvement indicator. The density and porosity of sintered body were measured according to the ASTM C 830-00 test procedure. The phase identifications were analyzed by XRD, while microstructural investigation was performed using scanning electron microscopy (Hitachi TM3000 Tabletop SEM). The values of hardness were measured by a Vickers indentation technique tester (Shimadzu HSV-20) using a 30 kgf load.

3. RESULTS AND DISCUSSION

The Vickers hardness of HIP and pressureless sintered ZTA ceramic sample is shown in Fig. 1. The hardness values for HIP samples, trend is gradually increased from 1632.7 HV (0 wt.% of 8YSZ) and reach the highest peak at 1706.74 HV (15 wt.% of 8YSZ). However, further additions (20 – 25 wt.%) show the gradually decrease in hardness due to exceeding solubility limits, because of excessive 8YSZ addition in the ZTA composition. Rejab et al. [14] stated that, an exceeded limits of added materials can result in deterioration of mechanical properties.

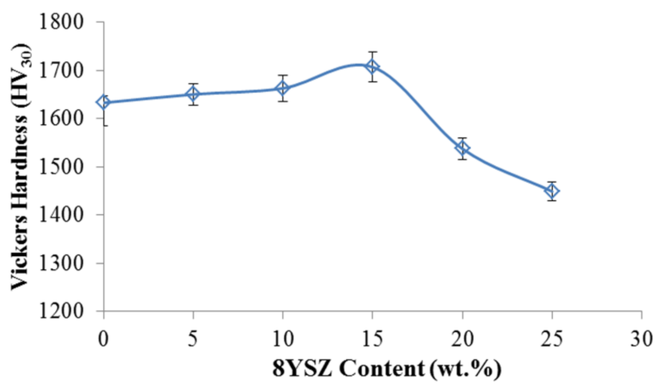

Fig. 1 Vickers hardness values of HIP sintering as a function of 8YSZ content

Table 1 shows XRD results of various 8YSZ added in Al_2O_3 matrix. Fig. 2 shows that overall peaks patterns were identified as corundum (α-Al_2O_3) with ICSD file number 98-001-7443 and zirconium yttrium oxide ($Zr_{0.88}Y_{0.12}O_{1.94}$) ICSD file number 98-003-2793. Table 1 detailed the phase's percentages for overall composition. These results proved that toughening mechanism provide by $Zr_{0.88}Y_{0.12}O_{1.94}$ phase reach a limit at 8.1% for 15 wt.% addition. Further increased of $Zr_{0.88}Y_{0.12}O_{1.94}$ phase deteriorate the hardness of ZTA ceramics. To further appreciate the role of the both phases as obtained by XRD analysis, the grains shape distribution of those phases in the microstructure were revealed by SEM analyses.

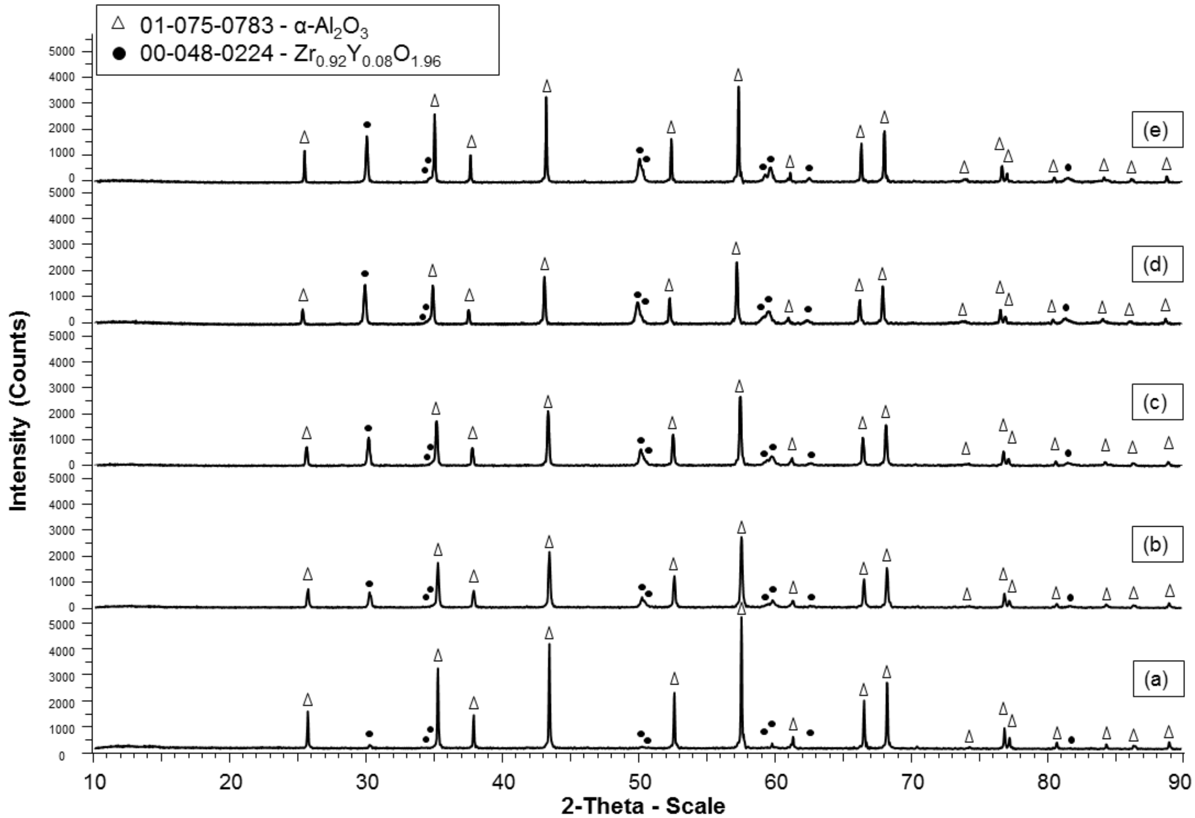

Fig. 2 XRD pattern of the Al_2O_3 added 8YSZ (a) 5 wt.%, (b) 10 wt.%, (c) 15 wt.%, (d) 20 wt.% and (e) 25 wt.%

Table 1 Phases percentages of various 8YSZ toughened Al_2O_3

8YSZ	Phase Percentages	
	Al_2O_3	$Zr_{0.88}Y_{0.12}O_{1.94}$
5	99.6	0.4
10	97.7	2.3
15	91.9	8.1
20	86.0	14.0
25	83.3	16.7

Based on micrograph structure shown in Fig. 3, there are two distinct size and shade. ZrO_2 has lighter grains, smaller in size and discoidal shapes while Al_2O_3 refer to darker grains, larger in size and tabular shapes. The size of ZrO_2 is smaller in size because the material used is 8YSZ which is relatively smaller than Al_2O_3. The microstructures are more isotropic and homogenous. Smuk et al. [15] reported that HIP capable to eliminate large pores by diffusion and grain arrangement. Therefore, the sintered body fabricate by HIP capable to produce almost zero porosity bodies [16-20]. However, abnormal growth such as platelet grains occurs among ZrO_2 grains; they can be seen obviously in samples with 25 wt.% of 8YSZ addition. The deformation in grain shape of ZrO_2 could be due to exceeding solubility limits, because of excessive of 8YSZ addition in the Al_2O_3 matrix.

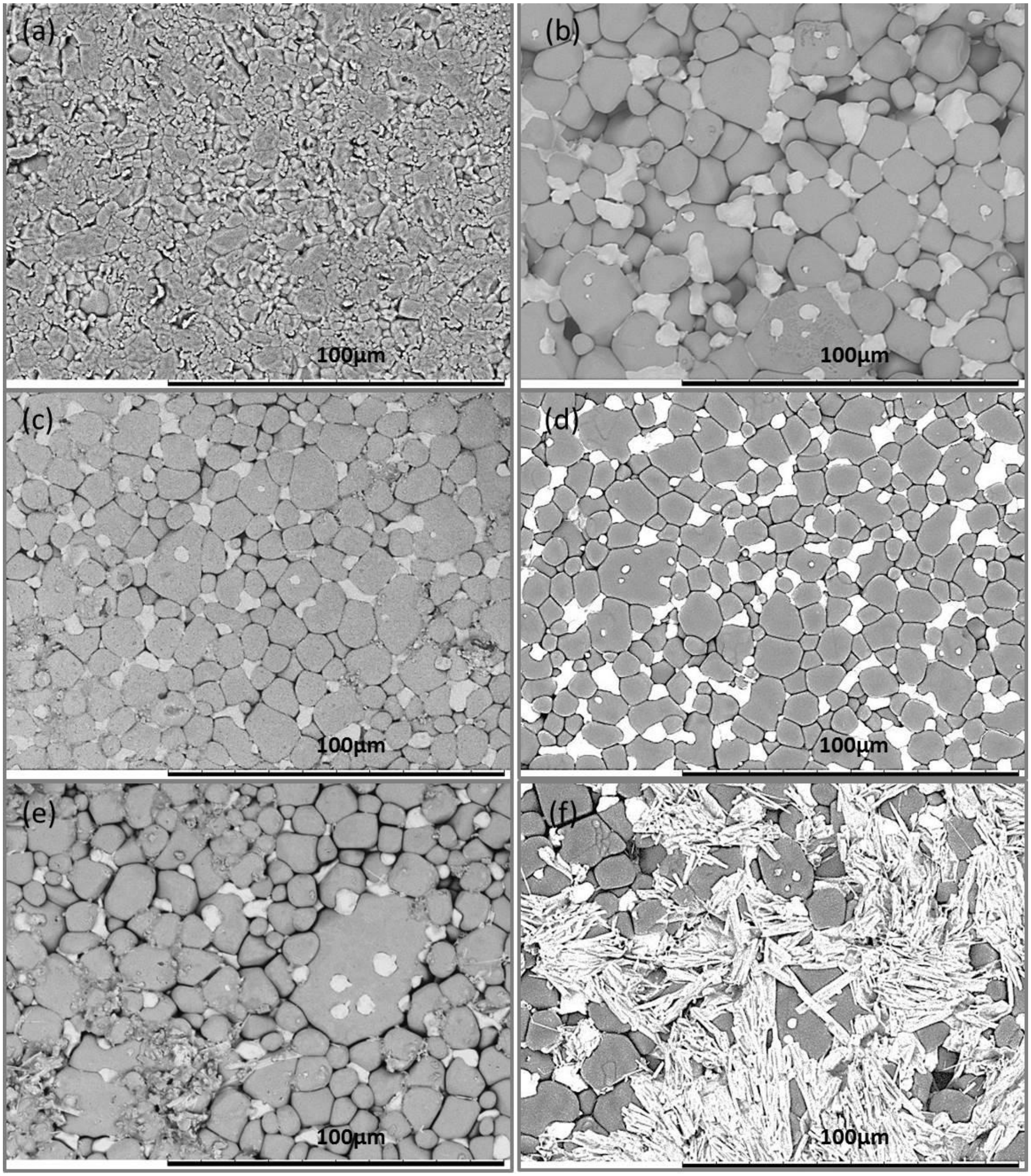

Fig. 3 Scanning electron micrographs of Al_2O_3 ceramics with (a) 0 wt.% of 8YSZ, (b) 5 wt.% of 8YSZ, (c) 10 wt.% of 8YSZ, (d) 15 wt.% of 8YSZ, (e) 20 wt.% of 8YSZ and (f) 25 wt.% of 8YSZ, at 1K magnification. Light grains: zirconia; dark grains: alumina

Based on Fig. 4, the bulk density values gradually increased as the amount of 8YSZ added increase until 15 wt.%. Naga et al. [21] stated that the improved density is very important for close the pore that networks prior to the HIP sintering. Based on the results, the addition of 5 wt.% of 8YSZ increase from 3.20 g/cm^3 to 4.34 g/cm^3 (10 wt.% of 8YSZ) and the density continues to increase until it reaches the value of 7.35 g/cm^3 (15 wt.% of 8YSZ). However, the graph trend starts to decline to 5.04 g/cm^3 (20 wt.% of 8YSZ). The density continues to gradually decrease until 3.29g/cm^3 (25 wt.% of 8YSZ). For the percentage of porosity, in the beginning at 5wt.% of 8YSZ the porosity is 19.48%. The porosity values decrease to 2.24% (10 wt.% of 8YSZ) and 1.94% (15 wt.% of 8YSZ). Then the porosity slightly increased to 4.4% (20 wt.% of 8YSZ) and react maximum to 15% (25 wt.% of 8YSZ). Therefore the effect of HIP sintering on the density of ZTA is proved, with a reduction of porosity. It shows that the optimum addition of 8YSZ is 15 wt.% which has the lowest porosity and highest densification.

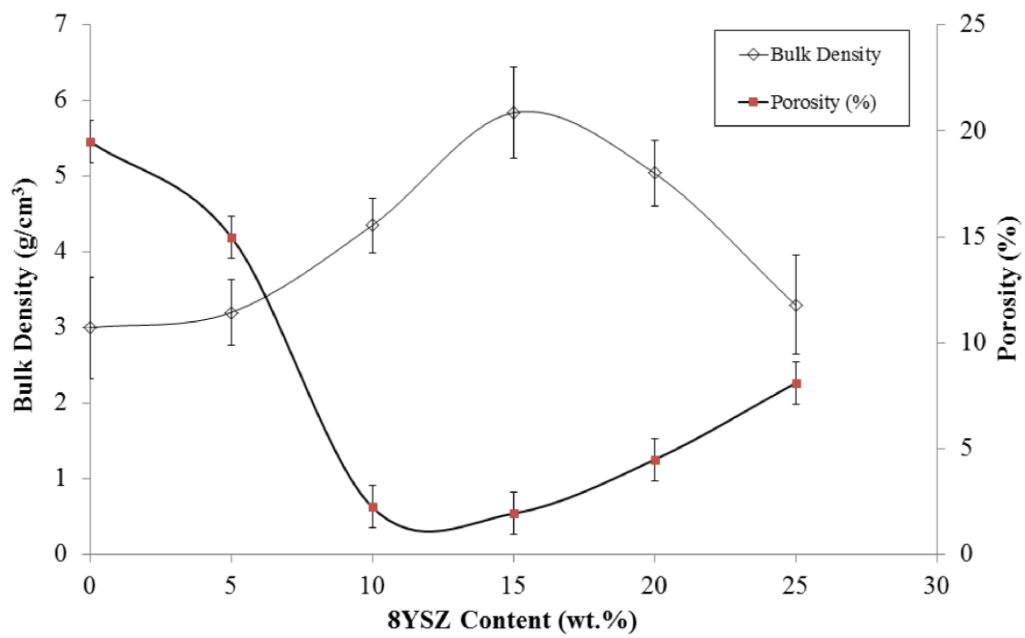

Fig.4 Results of bulk density and percentage of porosity via HIP sintering technique

4. SUMMARY

Microstructure and hardness properties of 8YSZ added Al_2O_3 matrix via HIP have been investigated. An addition of 15 wt.% of 8YSZ provide the optimum properties of Vickers hardness 1706.74 HV. The change in shape of ZrO_2 grains are corresponding to exceeding solubility limits, because of excessive of 8YSZ addition in the Al_2O_3 matrix. Based on the presence work, HIP proved that external pressure sintering technique capable to obtain densified microstructure and improve the hardness of the Al_2O_3 ceramics through the reduction of porosity values.

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