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Nanoindentation Measurements for B_2O_3-Y-TZP Electrolyte Used in HT-SOFC

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Abstract. Nanoindentation analysis used to evaluate mechanical characterizations including elastic modulus, yield stress, hardness, and fracture strength of doping B_2O_3 in yttria stabilized tetragonal zirconia (B_2O_3-Y-TZP) to use as electrolyte in solid oxide fuel cell worked at high temperature (HT-SOFC). X-ray diffraction (XRD) one of rapid analytical technique primarily used for phase identification and microstructure of sintered electrolyte. Scanning electron microscopy (SEM) have been utilized for morphology studies of the sintered specimens. B_2O_3 doped Y-TZP specimens obtained elastic modulus(E) 6.42E+10 Pa with yield stress σ (N/m2) as much as 1.63E+07, hardness as much as 465kg/mm^2, and fracture strength as much as 3.1393E+17 MPa.M1/2. While crystallite size of B_2O_3 doped Y-TZP estimated to be 137 nm, and the grain size of B_2O_3 doped Y-TZP was estimated to be 426 nm.

1.Introduction

Nanoindentation is recognized as a technique to characterize the mechanical properties of materials at very small scales or ultra low applied load indentation [1]. Fuel cells is one of advanced urgent need technologies it is a power sources that convert the chemical energy of the inlet fuel electrochemically to electricity and heat. And considered one of promising energy technology that could reduce global warming due to significantly increasing the efficiency of electrical power production [2].

The SOFCs provided greater fuel flexibility this referred to convert conventional hydro- carbon fuels to syngas containing hydrogen by using fossil or renewable hydrocarbon fuels, like natural gas. Thus, SOFC displayed the simplest and inexpensive requirement of fuel processing equipment than most other FCs types [2,3].

Y_2O_3 stabilized zirconia is well-known for its high mechanical properties encompassed of hardness, toughness, and strength [4]. Previous experimental results

obtained that B_2O_3 facilitated phase transformation from cubic-to monoclinic phase [5]. In addition, B_2O_3 is knows as a glass former [6].

Therefore, in the current work sintered 2 mol% B_2O_3 doped Y-TZP pellets have been manufactured and its mechanical properties have been evaluated by nanoindentation exami- nation with a diamond cube corner indenter.

2.Materials and methods

2.1 Materials used

The starting materials were used in experimental work of this paper including nano powder 3mol% yttria-stabilized zirconia (Y-TZP) was supplied by Hongwu International Group Ltd,China, Stearic acid and B_2O_3 powders were sup- plied by Merck, Germany.

2.2 Instruments

All experimental work was carried out at Materials and Energy Research Center (MERC) in Iran. The phases of starting and sintered specimens analyzed via Philips analytical X-ray (XRD) type PW1930 with cobalt $\lambda k\alpha 1$=1.78901 radiation tube operating at 40 kV and 30 mA. The particle size analyzed via scanning electron microscope (SEM) TESCAN Vega III Czech Repuplic used 5 KV to get the image. While crystallite size analyzed with Williamson-hall analysis. The mechanical properties of sintered specimens estimated by nanoindentation tester type Triboscope model Hysitron at room temperature system carried out with International Standards Organization (ISO14577) with a Cube corner indenter with average radius of curvature less than 50 nm. The tip of the testing instrument was calibrated by Oliver-Pharr method (ISO 14577) [7,8].

2.3 Mixing Method

Mixing method was done via Spex 6000 Mixer/Miller with zirconia jars and zirconia balls in different sizes with diameter range from 10 to18 mm for 60 min to preparation doped specimens with 2mol% B_2O_3 with Y-TZP.

3.Experimental procedure

The prepared mixtures were compacted by cold pressing method at 27.50 MPa in a die 20 mm in diameter and 0.6 mm thick. After this step all green specimens were sintered in air with sintering program including raising temperature to 673°K with 1 hr as holding time to make sure of removed binder, then kept for 1 hr at 1100°K and finally sintered at 1923°K for 180 min this step done using a Retsch box furnace with heating rate 2°K/min to avoid thermal stress of specimens during sintering.

After polishing and 15 minutes thermal etching at 1673°K to get morphology of specimens by SEM Cambridge with 10.0X resolution and sputter coating with Au. XRD used to identify the phase, crystal structure and crystallite size of sintered specimen. The mechanical properties of sintered specimens estimated by nanoindentation tester done with applied indenter tip with increasing normal load on the surface of the tester

specimen. When the penetration depth of tip indenter reached a preset maximum value, the normal load will be gradually, reduce until partial or complete relaxation occurs [9]. In current work, the P*max* load ranges from908.8 to 984.5 μN, which applied at fixed rate of about 196.9 μN/S. The nanomechanical proper- ties of the B_2O_3 doped Y-TPZ such as reduced elastic modulus and hardness, evaluated from the load–displacement nanoindentation data using the widely accepted Oliver and Pharr method [10, 11]. The young modulus calculated using equation (1).

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{1}$$

Where: Er is reduced elastic modulus, v is Poisson ratio for the endurance material, Ei and vi are the elastic modulus and Poisson ratio of the indenter, respectively. The hardness was evaluated from equation (2):

$$H = \frac{P_{\max}}{A} \tag{2}$$

Where A: contact area at that load, Pmax: maximum load, and the stiffness (S) was estimated from equation (3) [12]:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \tag{3}$$

4.Results and Discussion

In Figure(1) can be shown the particle size of B_2O_3, while in Figure(2), the particle size of Y-TZP can be seen. The XRD pattern of Y-TZP powders in Figure (3) obtained phases of Y-TZP with a small amount of ZrO_2 impurity phases.

In Figure (4) can be seen the XRD pattern of the sintered powder of Y-TZP. While Figure (5) shown small peaks of B_2O_3 and substantial peaks of Y- TZP doped B_2O_3 in the sintered specimen.

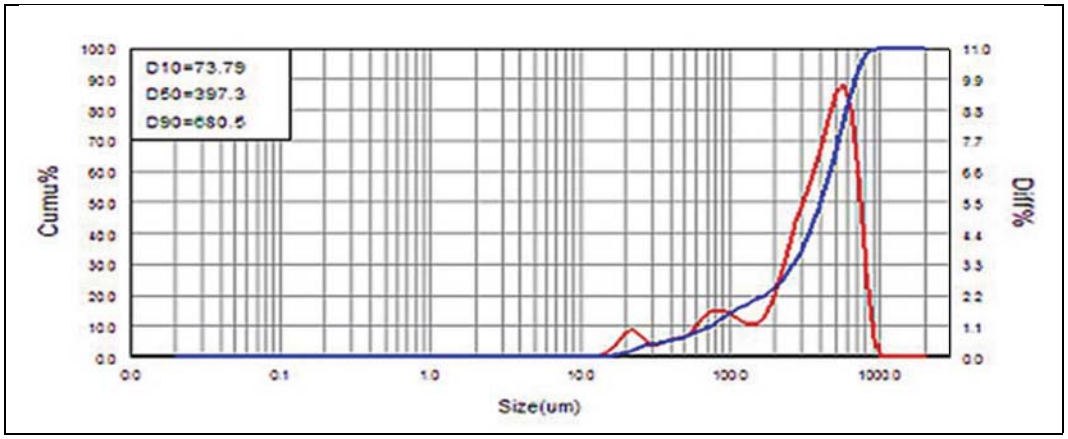

Figure 1. Particle size analysis of B_2O_3

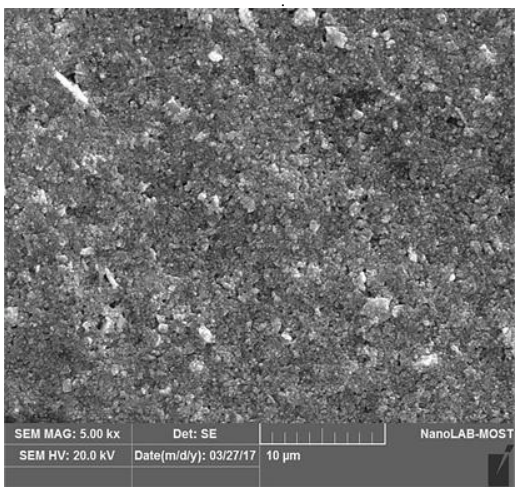

Figure 2. SEM of Y-TZP powder.

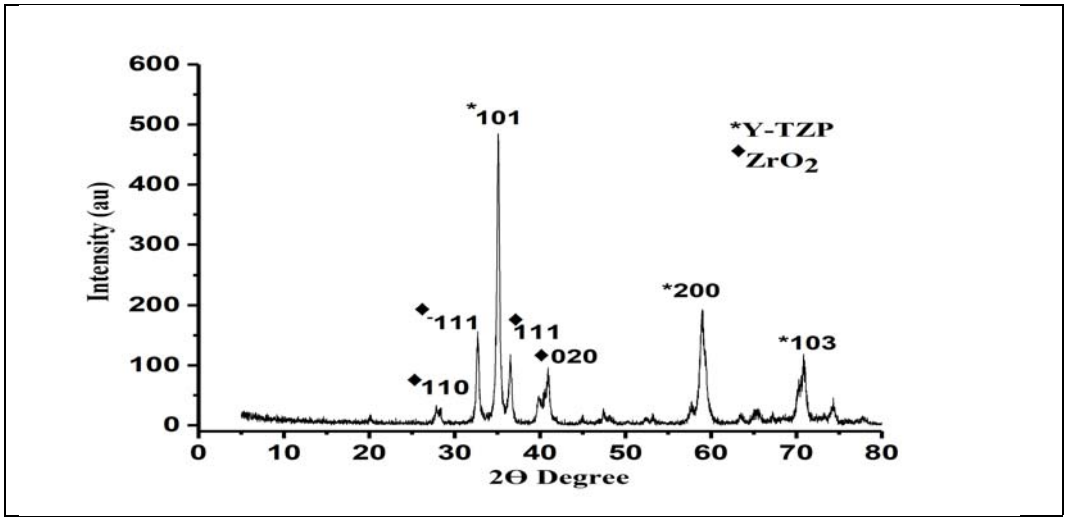

Figure 3. XRD pattern of Y-TZP powders.

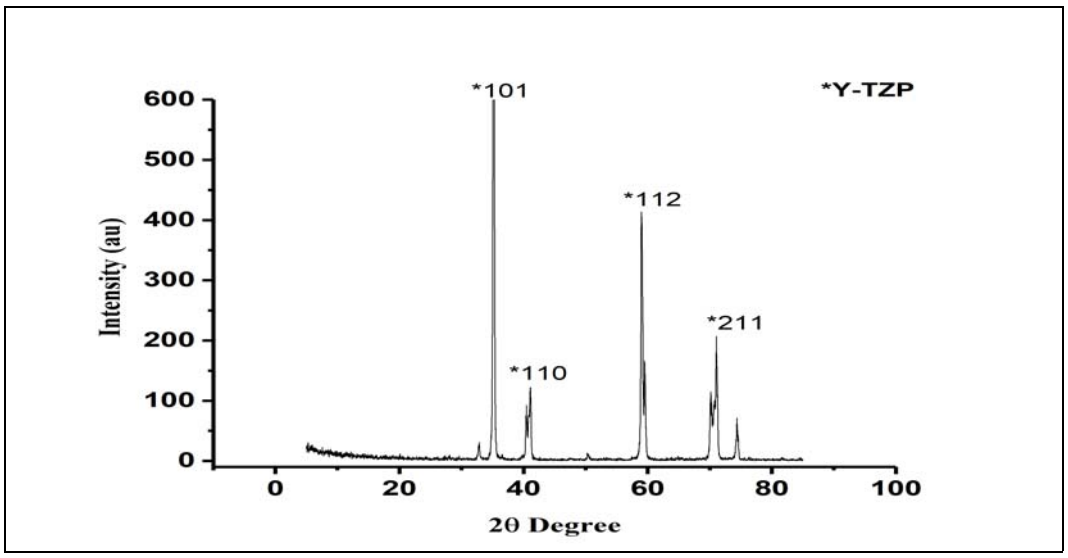

Figure 4. XRD date for sintered Y-TPZ.

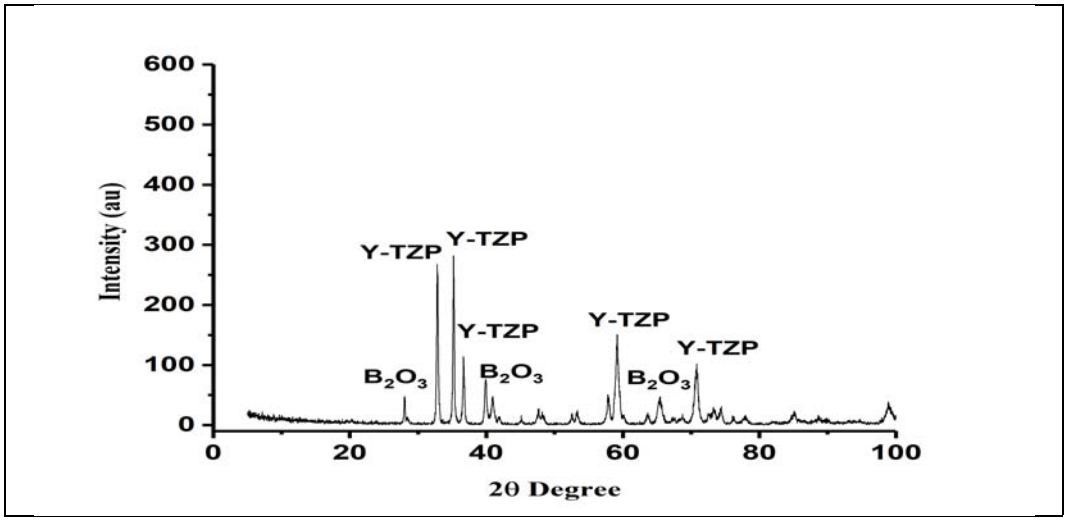

Figure 5. XRD date for sintered B_2O_3 doped Y-TPZ.

Williamson-hall analysis obtained that the peak broadening is mostly due to the crystallite size [13]. Based on this analysis, the crystallite size for Y-TZP was estimated to be around 137 nm while, B_2O_3-Y-TZP around 895nm.

$$B\cos(\theta) = \frac{K\lambda}{D} + 4Strain\sin(\theta) \tag{4}$$

Whereβ=instrumental broadening, D=crystallite size, K=shape factor (0.9), and λ= wavelength of Cokα radiation. SEM micrographs of sintered Y-TPZ and B_2O_3-Y-TZP were shown in Figure (6) and Figure (7). It can be noticed that the doped Y-TZP displayed grain growth in comparison to pure Y-TPZ due to effect of 2%mol B_2O_3. Also, the average grain size of sintered Y- TPZ is about 426 nm, while the B_2O_3 doped Y- TZP is about 1225 nm.

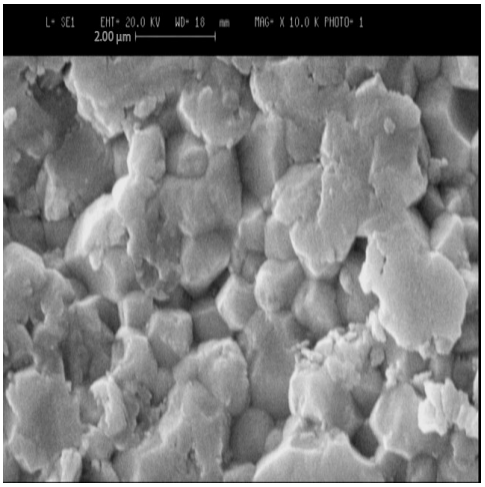

Figure 6. SEM micrographs of sintered Y-TPZ

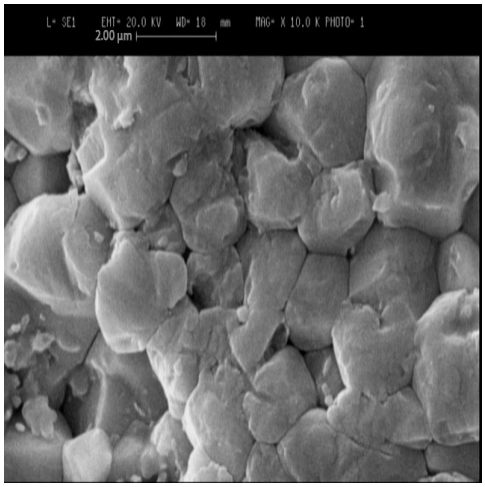

Figure 7. SEM B_2O_3-Y-TZP

SEM micrographs of sintered Y-TPZ and B_2O_3-Y-TZP were shown in Figure (6) and Figure (7).

The results of mechanical properties of elasticity modulus for both Y-TPZ and B_2O_3-Y-TPZ can be calculated using equation(1). In load-displacement curve of load-displacement curve in Figure (8), and Figure (9).

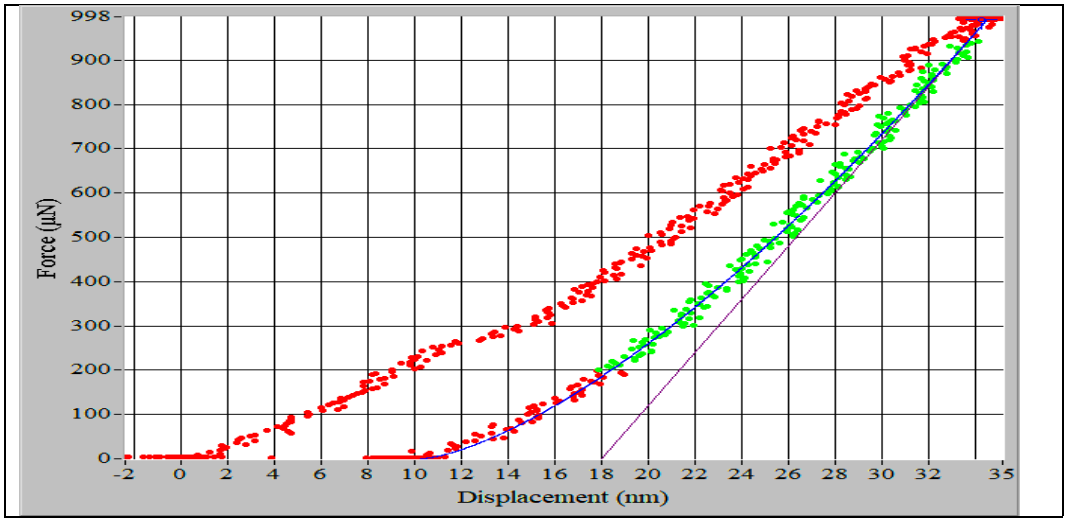

Figure 8. Load-displacement curve (*P–h)* of B_2O_3-Y-TZP.

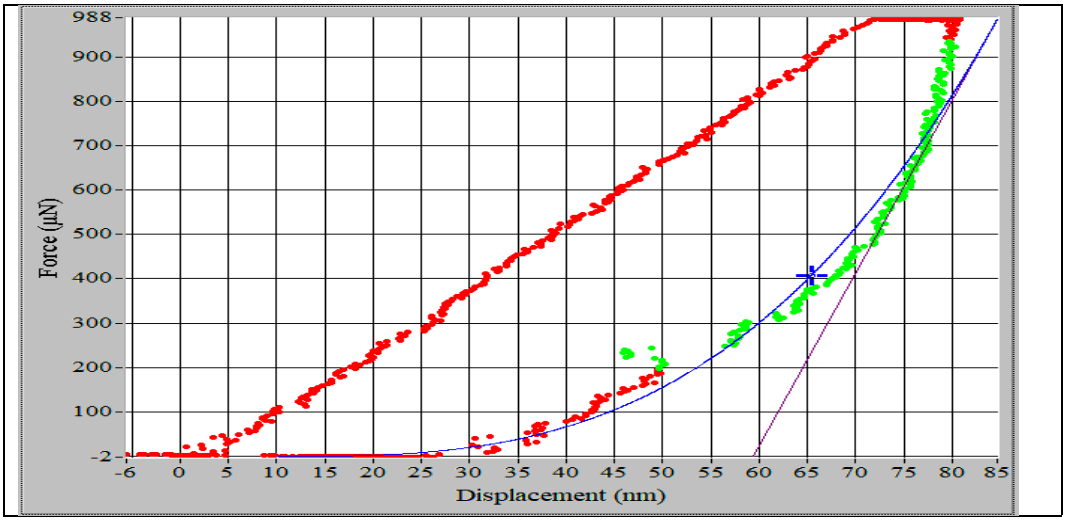

Figure 9. Load-displacement curve (P–h) of B2O3-Y-TZP.

The results obtained in table (1) it can observe from results of mechanical properties the significant effect of doped 2% mol B_2O_3 in values of elastic modulus from 7.595E-7 Pa to 6.42E+10 Pa in Y-TZP, and B_2O_3 -Y-TZP respectively, also improvement in both yield stress, and fracture strength in comparison to values o pure electrolyte, while hardness shown reduction from 8189 Kg/mm^2 to 465 Kg/mm^2 with B_2O_3-Y- TZP.

Table 1. Estimated mechanical properties via nanoindentaion technique for sintered pellets.

Specimen code size	Elastic modulus E (Pa)	Hardness H (kg/mm^2)	Yield stress σ (N/m^2)	Fracture Strength (MPa.M1/2)	Crystal (nm)
Y-TZP	7.595E-7	8189	2.824	3.09E+08	137
B2O3- Y-TZP	6.42E+10	465	1.63E+07	3.1393E+17	895

5. Conclusion

The influence of B_2O_3 content on the densification, tetragonal phase stability, young modulus, hardness and fracture toughness was investigated using nanoindentation technique. Morphology of the sintered specimens was obtained via scanning electron microscopy (SEM), while the phases of both mixtures and sintered doping Y-TZP specimens were characterized by x-ray diffraction (XRD).

6. Acknowledgments

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7. References

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