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Mechanical properties of microwave sintered 8 mol% yttria stabilized zirconia

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This paper reports the synthesis and characteristics of nano sized 8 mol% yttria stabilized zirconia (8YSZ). The nano sized 8YSZ powder (15 nm) has been prepared by the sol-gel technique. The phase formation was confirmed by the X-Ray Diffraction (XRD) analysis and the particle size verified by transmission electron microscope (TEM). The prepared 8YSZ green samples were sintered in a microwave furnace at 1500°C for three different holding times, such as 5, 10 and 15 min. The density of the sintered samples was measured with the Archimedes method. This method revealed 98% of theoretical density of the 8YSZ sample. The surface morphology of the sintered samples was examined by the scanning electron microscope (SEM). The mechanical properties, including nanohardness, elastic modulus and coefficient of friction of the high density sample have been evaluated.

Key words: Sol-gel, nano 8YSZ, microwave sintering, nano hardness, elastic modulus, coefficient of friction.

INTRODUCTION

8 mol% yttria stabilized zirconia (8YSZ) has found many engineering applications, because of its high strength, fracture toughness, and high thermal stability (Lange et al., 1986). It is used as an electrolyte material in oxygen pumps, and in oxide fuel cells, owing to its oxide ion conductivity at high temperatures and relatively low cost (Minh, 1993; Steele, 2001; Fukul et al., 2004). Although 8YSZ possesses high ionic conductivity, its low mechanical properties limit its applications. In order to increase the mechanical properties of 8YSZ, some important factors should be considered, that is, particle size, grain size, and different sintering methods. Nanopowder offers the possibility of manufacturing dense ceramics at a low sintering temperature, and sintering techniques can have considerable influence on the grain growth of the samples (Mazaheri et al., 2009). So the synthesis of nano 8YSZ powder is essential in research fields. Until recently, a wide variety of chemical methods have been used to prepare 8YSZ powder, such as co-precipitation, solution combustion, hydro thermal treatment, hydrolysis of alhoxides and sol-gel synthesis (Kumar and Manohar, 2007). Usually, 8YSZ is synthesized by a mixed-oxide reaction method. However, the powders prepared by this method usually exhibit high degree of agglomeration, and an inhomogeneous particle size, thus requiring a subsequent high-temperature treatment. The sol-gel synthesis is a chemical solution process. In comparison with other techniques, the sol–gel process has shown significant advantages, including excellent chemical stoichiometric, compositional homogeneity and lower crystallization/sintering (processing) temperature, due to the mixing of the liquid precursors on the molecular level (Liu et al., 2007; Surowiak et al., 2001; Fan and Kim, 2002). Nowadays, we need minimum processing time, high density, uniform sized grains and good mechanical properties. Many

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researchers have suggested different types of sintering methods, to achieve the maximum density of the sintered 8YSZ sample and improve its mechanical properties. By using the hot press, and spark plasma sintering methods, Dhal et al. (2007) achieved the 98% density of the 8YSZ sample and measured its mechanical properties. Mazaheri et al. (2008) used microwave sintering, and achieved 96% density for 8YSZ sample. Microwave sintering has been performed by many researchers for different 8YSZ applications (Ciacchi et al., 1996; Upadhyaya et al., 2001; Thridandapani et al., 2009; Janney et al., 1992). Microwave sintering is a more advanced sintering technique than conventional sintering. In the conventional sintering method, an external heating element is used to generate the heat, and it is transferred to the sample via, conduction, convection, and radiation, which produces a high temperature gradient and internal stresses (Coasta et al., 2003). But, in microwave sintering, the heat is generated internally, within the test sample, by the rapid oscillations of the dipole at microwave frequency (Phani and Santucci, 2006). Since electromagnetic waves are used to generate the heat in microwave sintering, a large amount of heat can be transferred to the interior of the ceramic sample. The salient features of microwave sintering are it's volumetric and uniform heating, and short processing time. In general, nano indentation has been performed for thin films and crystals. Soyez et al. (2000) found the mechanical properties of polycrystalline YSZ, using the Nano indentation technique. Further, Voevodin et al. (2001) fabricated YSZ/Au nano composite films and evaluated their mechanical behaviour by the nano indentation technique. Gaillard et al. (2009) applied the nano indentation technique on the yttria stabilized zirconia crystal and analysed the phase transformation of the single and polycrystal. However, Mukhopadhyay et al. (2009) applied the nano indention technique and calculated the nano hardness of pressure less sintered alumina samples. Many investigators applied the microwave sintering on the commercially available 8YSZ powder pellets and studied the mechanical properties by using the micro indentation technique, but the nano indentation studies of microwave sintered 8 mol% yttria stabilized zirconia bulk sample have not been reported by the previous researchers. 3YSZ -TZP ceramics was one of the promising materials for tribological applications, because of its high wear resistance, bending strength, fracture toughness and mechanical strength and low friction coefficient than cubic zirconia (Ran et al., 2007). Many investigators found the tribological characteristics of 3 mol% of yttria stabilized zirconia (3YSZ) sintered samples with and without metal oxide additives of CuO, MnO_2, MgO, B_2O_3 and reduced the coefficient of friction (Bas et al., 2004). It is known that 8YSZ materials are mostly used as solid oxide fuel cell (SOFC) applications, during the operation solid oxide fuel tend to bend at higher temperatures so it is necessary to improve their mechanical strength, and tribological characteristics, and hence it is necessary to give importance to the coefficient of friction of 8YSZ.

In this present investigation, the nano 8YSZ powder was synthesised by the sol-gel wet chemical route, followed by microwave sintering. The nano mechanical properties, including nano hardness, elastic modulus and one of the important tribological properties coefficient of friction were evaluated. And the results are compared with the mechanical properties reported by the micro indentation technique of previous research.

MATERIALS AND METHODS

In the sol-gel process, zirconium oxychloride ($ZrOCl_28H_2O$), yttrium nitrate hexa hydrate ($Y(NO_3)_3.6H_2O$), and oxalic acid ($C_2H_2O_4$) were used as the starting materials (Kumar and Manohar, 2007). These respective salts were dissolved in 200 ml of millipore water in the stoichiometric ratio of 1 M. The initial solutions of the precursors were mixed under constant stirring by a magnetic stirrer, until a transparent viscous gel was formed. The obtained gel was dried in a hot air oven at 45°C for five days. The dried gel was calcined at 600°C for 3 h and well ground in a planetary mill for 5 h in an ethanol medium at a rotational speed of 300 rpm. The planetary jar mill and grinding balls were made of tungsten carbide material. The milled powder was dried in air at 60°C for 24 h and the powder was characterized by the X-ray diffractometer (XRD, Seifert 3000P) with Cu - Kα radiation (λ = 1.5406 Å). The XRD patterns were recorded in the 2θ scanning range of 10° to 80°. The phase formation of the nano 8YSZ was analysed by the XRD and the particle morphology was analysed by the transmission electron microscope (TEM) [JEOL-1200 EX II operated at 120 kV (max)]. The milled powder was mixed with the polyvinyl alcohol (PVA) binder and pressed in to pellets (10 mm in diameter) at a pressure of 40 MPa using a uni axial press. The 8YSZ green samples were sintered in a microwave furnace (V. B. C. C India) at 1500°C at a 1.1 kW 2.45 GHz frequency, at a heating rate of 100°C / min for three different holding times that is, 5, 10, and 15 min. In the microwave sintering furnace, a special susceptor is designed to generate heat against the microwave at a heating rate of 100°C/min. A non-contact optical sensor (RAYTEK, USA) was used to measure the temperature in the range of 600 to 1600°C; the time temperature profile is programmed by the Eurotherm temperature indicator cum programmer. The density of the samples was measured by the Archimedes method. The microstructure of the sintered and polished samples was analysed by the scanning electron microscope (SEM) (HITACHI Model S-3400 JAPAN). The average grain size of the sintered samples was measured by the linear intercept length method (Mendelson, 1969).

Nanohardness and coefficient of friction

The nano indentation test was applied on a fully dense microwave sintered 8ysz sample using a nano-indenter (CSM open platform Switzerland) with Berkovich indenter calibrated with a standard silica specimen, by running a standard continuous stiffness measurement (CSM). The CSM continuously measures the stiffness, and allows the hardness and elastic modulus to be determined as a continuous function of the penetration depth. The nano hardness was found to be a strongly sensitive function of load and depth (Mukhopadhyay et al., 2009). In this regard the unique advantage of the nano indentation technique is that, it can measure the mechanical properties at the micro structural length scale. The

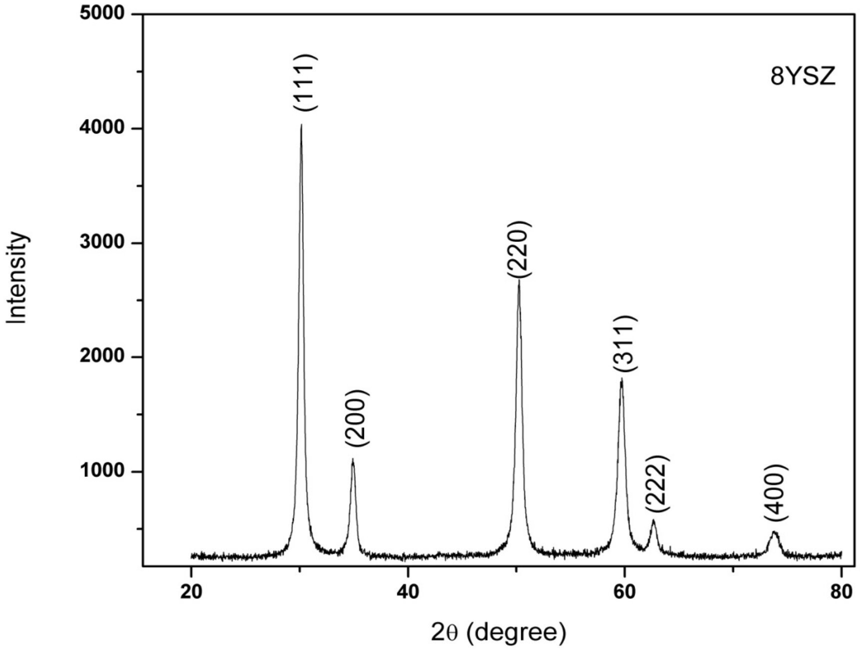

Figure 1. XRD pattern of 8YSZ Nano powder calcined at 600℃.

load is defined as the total force on the indenter and the depth is measured from the displacement of the indenter's starting position. The load and the displacement into the surface are continuously measured during loading and unloading. The load applied was 200 mN and the loading and unloading rate was 400 mN/min. The hardness and elastic modulus were calculated from the load-displacement curve using the Oliver and Pharr (1992) method. The important tribological property, coefficient of friction, was evaluated by a micro tribo meter for the fully dense 8YSZ sample. The coefficient of friction was found to be 0.2 against Si_3N_4 under the following conditions; the load applied was 2N and the reciprocal scratching was 2 mm.

RESULTS AND DISCUSSION

X-ray diffraction studies

The XRD pattern of the 8YSZ nano powder calcined at 600℃ for 3 h is as shown in Figure 1. The corresponding diffraction peaks coincide with the JCPDS file number: 82-1246 (Kumar and Manohar, 2007). The average crystalline size of the 8YSZ sample has been calculated, using the Debye–Scherrer formula given in Equation (1)

$$D=0.98\lambda / \beta \cos\theta \qquad ---- \qquad (1)$$

where D is the average particle size in nm, β is the full width at half the maximum (FWHM) of the X – ray reflection expressed in radians, and θ is the position of the diffraction peaks in the diffractogram. The average crystalline size of the nano 8YSZ was 20 nm (Li et al., 2007).

Morphological studies of 8YSZ using TEM and SEM

The particle morphology of the 8YSZ nano powder was analysed by the TEM, as shown in Figure 2. The TEM study confirms that the average particle size was 15 nm. The sol-gel derived nano 8YSZ particle size was very small, compared to the same particles prepared by the Co-precipitation technique (Keshmiri and Kesler, 2006). From the microwave sintering results, three types of sinterd samples of different densities were obtained that is, A (Density 92%)' B (Density 95%), C (Density 98%) corresponding to 5, 10 and 15 min holding time respectively. The surface morphology of the microwave-sintered 8YSZ samples A, B, C has been depicted in Figure 3(a), (b) and (c).

The density of the microwave sintered samples were increased due to the increase in the sintering temperature (or) holding time. Because of the holding time during the microwave sintering process, the sintered sample (C) shows higher density (98%) with an average grain size of (<900 nm) than the other two samples 'A' and "B'. The SEM image of the sample 'A' shows the maximum pores and agglomerates. Sample' B' appeared with minimum pores, from this it can be concluded that the minimum holding time during sintering is reponsible for the poor densificaton and pores appearing in the microstructure of the samples A and B. Mazaheri et al. (2008) prepared 8YSZ nano powder with average

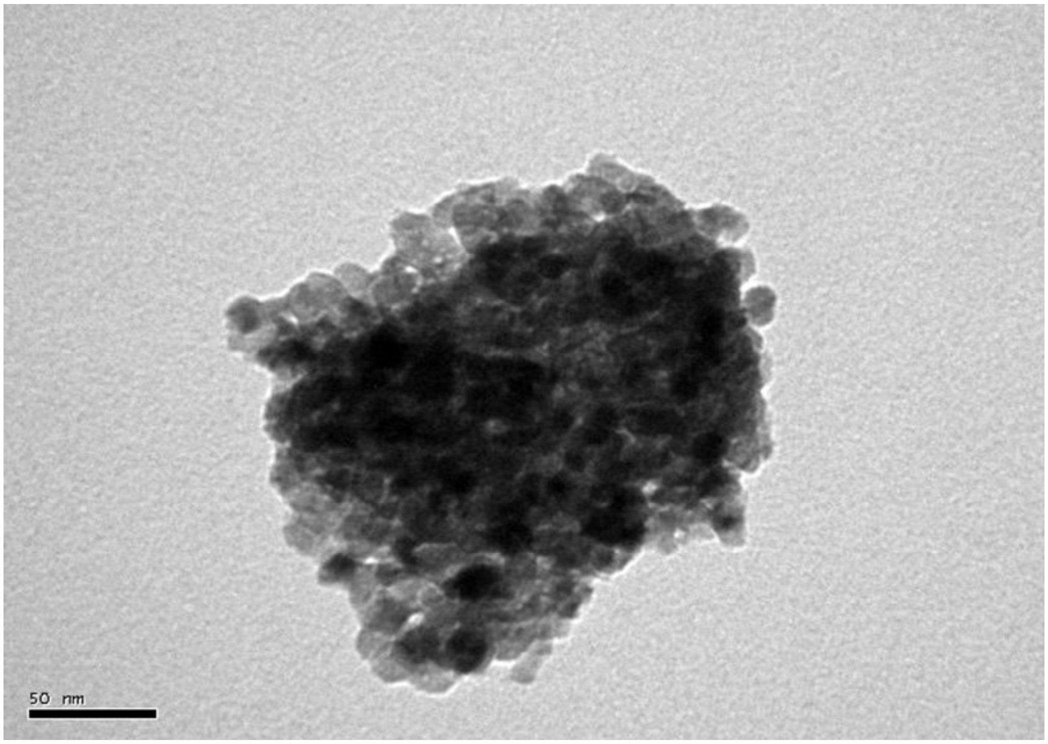

Figure 2. TEM image of 8YSZ NANO powder.

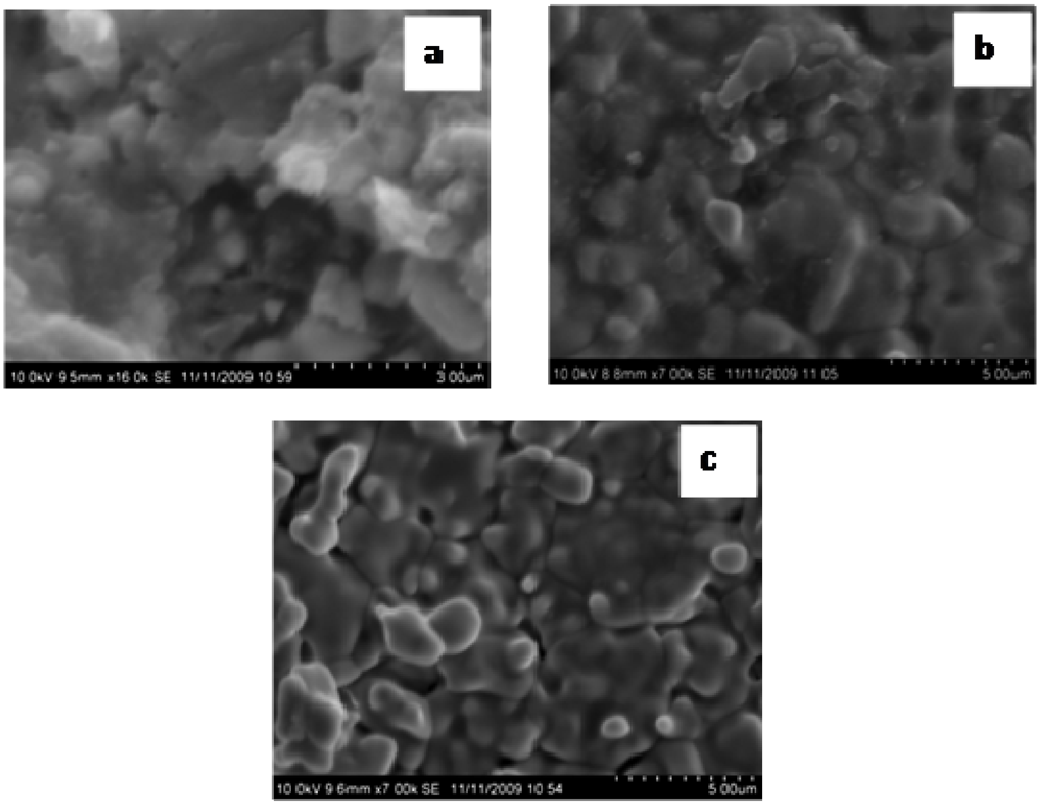

Figure 3. (a),(b) and (c) SEM image of microwave sintered samples at1500℃.

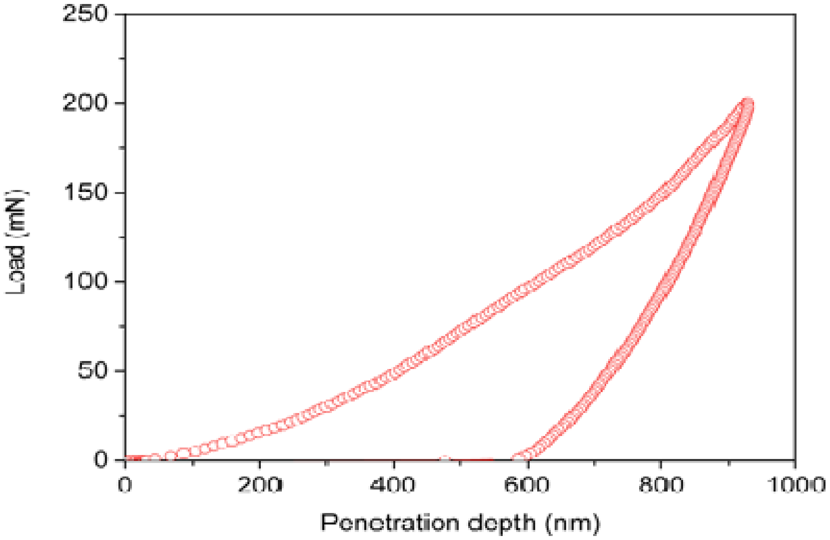

Figure 4. Load versus penetration depth curve of 8YSZ sample.

Particle size of 30 nm by smouldering combustion technique and applied microwave sintering on 8YSZ green samples and achieved 98% density with uniform grain size of 2.35 µm. Rajeswari et al. (2010) used commercially available 8YSZ powder (TZ-8Y Tosh.Tokyo,Japan) with an average particle size of 205 nm then performed microwave sintering method, got high density sample (98%) with an average grain size of 2.77 µm. Here the microwave sintering study results revealed the obtained grain size was (<900 nm) small in size comparared to the previous researcher's reports (Mazaheri et al. 2008; Rajeswari et al., 2010). The main reson for this minimum size grain (<900 nm) were present in the microstructure of the sample was due to the nano sized particle (15 nm) which confirmed the particle size influences on the grain size, and the microstructure of the sample. Figure 4 presents typical load-depth curves obtained by the nano indentation technique for the microwave sintered 8YSZ sample. This curve is fairly smooth and kink-free which implies that sample C' was fully sintered with minimum pores (Lu et al., 2006). The measured nano hardness, elastic modulus and coefficient of friction of the three microwave sintered samples A, B, C were shown in Table 1. Among the three samples highly densified sample, C has better enhanced mechanical properties and lower coefficient of friction than the other two sintered samples A, B. The hardness and elastic modulus were found to be 13.2Gpa and 210 Gpa, respectively for the sample 'C' these measured nano mechanical properties hardness and elastic modulus of microwave sintered 8YSZ sample agreed well with mechanical properties found by the micro indentation technique of the previous research reports. Dhal et al. (2007), Gogotsi et al. (1995) and Donzel and Roberts (2000) proved the mechanical properties of 8YSZ sample measured by microhardness not depend on the various grain size of the samples prepared by different sintering techniques. Mazaheri et al. (2009), performed two-step sintering method to sinter 8YSZ sample and measured the, hardness value13.51 G Pa by applying the micro indentation technique. Mazaheri et al. (2008) employed the microwave sintering technique to sinter the fully stabilized cubic zirconia samples and applying the micro indentation technique, reported two types of hardness values 12.97 Gpa (for a lower heating rate of 5°C /min) and13.72 G Pa (for a higher heating rate of 50°C/min). In the present study, 100°C/min heating rate was used in the microwave furnace during the sintering of the specimen, and the obtained nano hardness and elastic modulus were 13.2 and 210 GPa.

Wellman et al. (2004), applied the nano indentation test on the bulk zirconia sample without yttria addition and reported that the hardness and elastic modulus values are 11.32 and 168 GPa. Menvie et al. (2009) conducted the Berkovitch nano-indentation tests on pristine and irradiated YSZ polycrystals, and measured the hardness values as 12 to 15 GPa. The value of the Young's modulus at the maximum indentation depth was 200 GPa. The present investigation concludes that the nano hardness results were close to the micro hardness results of previous study reports.

The nano indentations were performed mostly at various positions of sintered ceramic sample viz well inside the grain and far away from the grain boundary, close to the grain boundary and on the grain boundary interface. In this investigation the nano-indentation test was conducted inside the grain surface of 8YSZ sample (Mukhopadhyay et al., 2009). Which leads to the

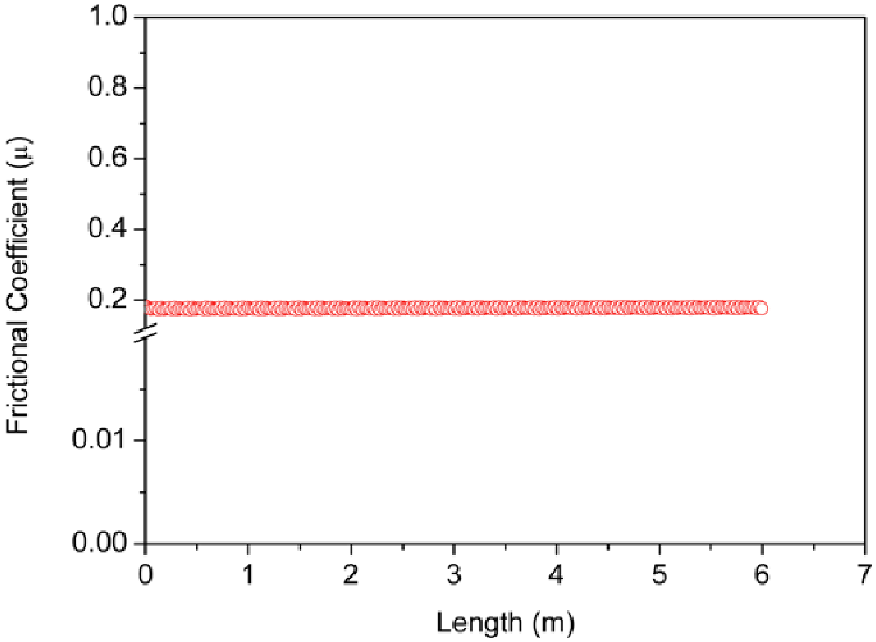

Figure 5. Length versus coefficient of friction diagram.

Table 1. Mechanical properties of Microwave sintered 8 mol% YSZ samples for different densities

S/N	Density of the sample (%)	Hardness (G.Pa)	Elastic modulus (G.Pa)	Coefficient of friction
1	92	10. 12	185	0.4
2	95	11.5	198	0.35
3	98	13.2	210	0.2

maximum hardness, and young modulus and these results were close to the mechanical properties found by the micro indentation technique.

The nano hardness and elastic modulus results obtained by the nano indentation technique were comparable with the microhardness values reported by previous investigators. The reason for the similarity between the nano indentation and micro indentation mechanical properties of the 8YSZ sintered sample was the minimum grain size and higher micro structural homogeneity in the sample.

The coefficient of friction is an important factor in understanding the tribological behaviour. The frictional behaviour is described by the coefficient of friction (COF) which is defined as the ratio of friction force and normal force. Pasaribu et al. (2003) reported that the coefficient of friction of 1% CuO doped with alumina against the silicon nitride was 0.6. Min-Soo et al. (2008) conducted a wear test on a, ZrO_2 disk with three structural ceramic ball materials including ZrO_2, Al_2O_3, and SiC and reported the coefficient of friction was relatively lower in ZrO_2 disk and SiC ball viz 0.4 to 0.5. Shen et al. (2009) reported that the coefficient of friction of the undoped Yttria stabilized tetragonal Zirconia poly crystals sample remains steady. They measured the coefficient of friction of pure 3YSZ-TZP, was 0.6 to 0.7, and that of dense 8 mol% CuO doped 3Y-TZP ceramics was 0.2 to 0.3 when sliding against an alumina ball under unlubricated conditions.

Winnubst et al. (2004) reported that the coefficient of friction of the 3YSZ- CuO doped sintered sample was (μ= 0.2-0.3) against an alumina ball. Here in our investigation the friction coefficients of the microwave sintered 8 mol% YSZ sample measured against silicon nitrate at room temperature and under dry sliding condition was 0.2 (Figure 5). From the result it is seen that the curve is almost stable, in this investigation the obtained coefficient of friction was 0.2 which is quite low. The main reason for the low coefficient of friction obtained for the 8YSZ microwave sintered sample was the fine grains were present in the microstructure of the sintered sample. This study concludes that the decrease in the grain size of 8YSZ by the microwave sintering reduces the coefficient of friction.

Conclusion

In this study, 8 YSZ nano powder was prepared by the sol-gel method. The cubic phase of the nano 8YSZ particle was confirmed by the X-ray diffraction studies. The particle size was about 15 nm. The SEM image of the sintered sample confirms the presence of sub-micron grains <900 nm. The achieved density of the sintered 8YSZ sample was 98%. The nano mechanical properties hardness, elastic modulus and coefficient of friction of the microwave sintered 8YSZ samples were studied by the Berkovich nano-indenter and micro tribometer and the obtained results were compared with the previous researcher's reports.The hardness and elastic modulus measured by the nano indentation technique agreed well with that of the mechanical properties reported by the micro indentation technique. The coefficient of friction of the microwave sintered 8YSZ sample value agreed well with that of the 3YSZ-CuO doped sample. This study confirmed the fine grains size in the microstructure which enhanced the mechanical properties of 8YSZ.

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