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Mechanical characterization of microwave sintered zinc oxide

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Abstract. The mechanical characterization of microwave sintered zinc oxide disks is reported. The microwave sintering was done with a specially designed applicator placed in a domestic microwave oven operating at a frequency of 2·45 GHz to a maximum power output of 800 Watt. These samples with a wide variation of density and hence, of open pore volume percentage, were characterized in terms of its elastic modulus determination by ultrasonic time of flight measurement using a 15 MHz transducer. In addition, the load dependence of the microhardness was examined for the range of loads 0·1–20 N. Finally, the fracture toughness data (K_{IC}) was obtained using the indentation technique.

Keywords. Zinc oxide; microwave sintering; microhardness.

1. Introduction

The application of microwave energy for the processing of ceramics has become an attractive area of research and innovation recently. The major advantages of the microwave processing of ceramic materials are accelerated densification rate as a result of direct, instantaneous, volumetric heating, controlled grain growth and uniform microstructure and achievement of a high density without the use of sintering aids (Lee and Case 1999; Goldstein 1999). Several studies have been reported on the sintering of ZTA (Lee and Case 1999), Y-PSZ (Goldstein *et al* 1999), $MgAl_2O_4$ (Goldstein *et al* 1998), alumina (Xie *et al* 1999), joining of ceramic composites (Aravindan and Krishnamurthy 1999), SiC/Al_2O_3 cement composite (Leiser and Clark 1998) etc by the use of microwave energy. The microwave sintering of very high purity ZnO powder has been reported by Birnboim *et al* (1998). The purpose of the present work was to evaluate the mechanical properties e.g. Young's modulus, hardness (H) and indentation fracture toughness (K_{IC}), of the microwave sintered zinc oxide disks prepared from a low purity (99·9%) commercial powder.

2. Experimental

The sintering of the commercial zinc oxide (ZnO) powder (99·9%, Loba-Chemie, India) was done using a specially designed applicator (figure 1) placed in a domestic microwave oven having a output power level tunable up to a maximum of 800 W and operating frequency of 2·45 GHz. The samples were sintered as a function of the exposure time and power level. The sintering temperature was measured using a chromel-alumel thermocouple for temperatures below 1000°C and by a Pt–13% Pt–Rh thermocouple for temperatures above 1000°C. The measurement accuracy was ± 5°C and ± 10°C for the chromel-alumel and the Pt–13% Pt–Rh thermocouple, respectively.

The phase purity of the sintered product was checked by the X-ray diffraction (XRD) technique with a CuK_a target. The density (r) of the sintered samples (diameter, 9·5 mm; thickness, 1·4 mm) was measured by Archimedes' principle. The volume % open porosity (P) was calculated as

$$P\ (\%) = [\{1 - (r/r_{th})\} \times 100], \quad (1)$$

assuming a theoretical density (r_{th}) value of 5·61 g/cc (Birnboim *et al* 1998). Typical polished sample had a surface roughness (CLA) of ≈ 0·5 μm. The fractured surface obtained at room temperature was used to study the microstructure of the sintered product by the scanning electron microscope (SEM).

The Young's moduli of the sintered samples were calculated from the ultrasonic wave velocity data obtained using the conventional, ultrasonic time-of-flight technique (Martin *et al* 1996) and appropriate couplant (ULTRAGEL II, Echo Ultrasound, USA). A 15 MHz transducer was used for this purpose. For each sintering condition, at least three to five samples were used to obtain the average data. The hardness data was obtained for the load range (0·1–20 N) using a microhardness tester fitted with a Vicker's square pyramidal indenter. Similarly, the fracture toughness data was obtained for the load range (5–20 N) using the same indenter, following the procedure of Anstis *et al* (1981).

3. Results

The XRD data (figure 2a) show that the microwave sintering process retained a pure phase of the ZnO samples. Typical fracture surface observation of a dense product in

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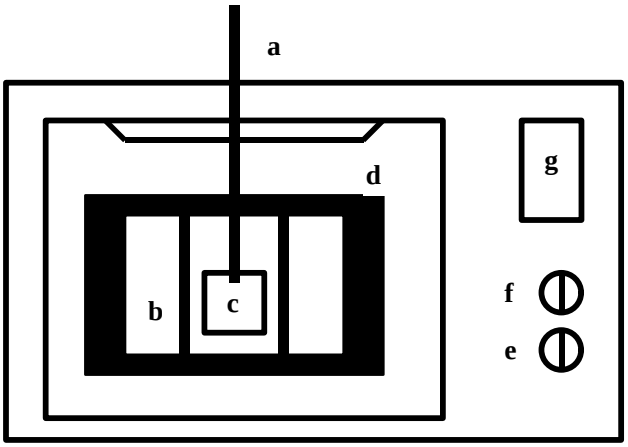
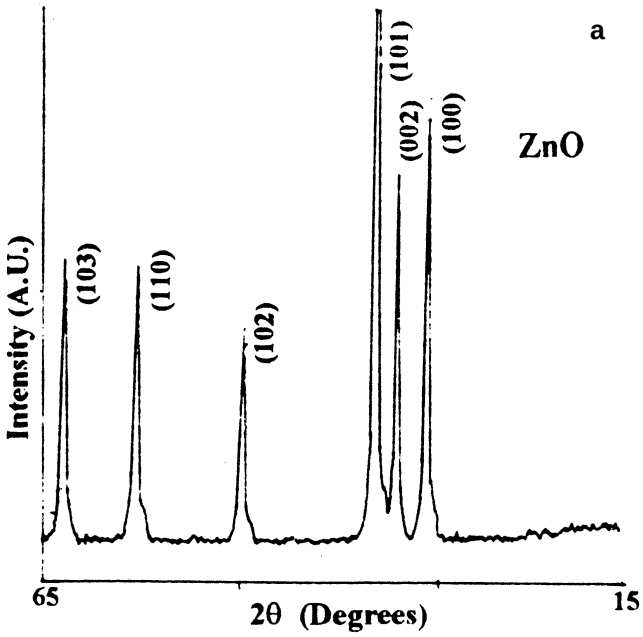


Figure 1. Microwave heating system (a. thermocouple, b. porous refractory box, c. sample, d. fibrous insulation material, e. power control, f. timer and g. display panel).



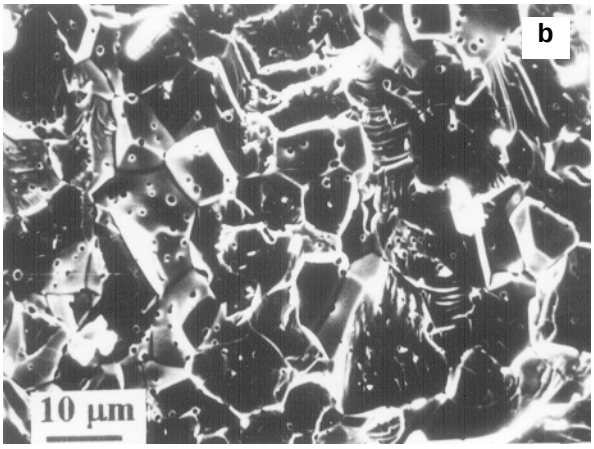


Figure 2. a. Typical X-ray diffraction pattern of the microwave sintered ZnO and **b.** typical microstructure on the fracture surface of microwave sintered ZnO.

SEM indicated a grain size of about 8·2 μm (figure 2b). The microstructure was nearly uniform with negligible open porosity. However, the presence of some residual, intragranular porosity were seen.

The data on variation of density and volume % open porosity (*P*) for the microwave sintered products as a function of sintering temperature, are shown in figures 3a, b, for the cases of constant exposure time and constant power level, respectively. The synthesis of the ZnO samples under microwave sintering at a power output in the range of 40–100% of the maximum power input of 800 W for an exposure time of 60 min resulted in having a relative density of about 60–90% of the theoretical values. Whereas, the density was 70–99·9% when the samples were microwave sintered at the maximum output

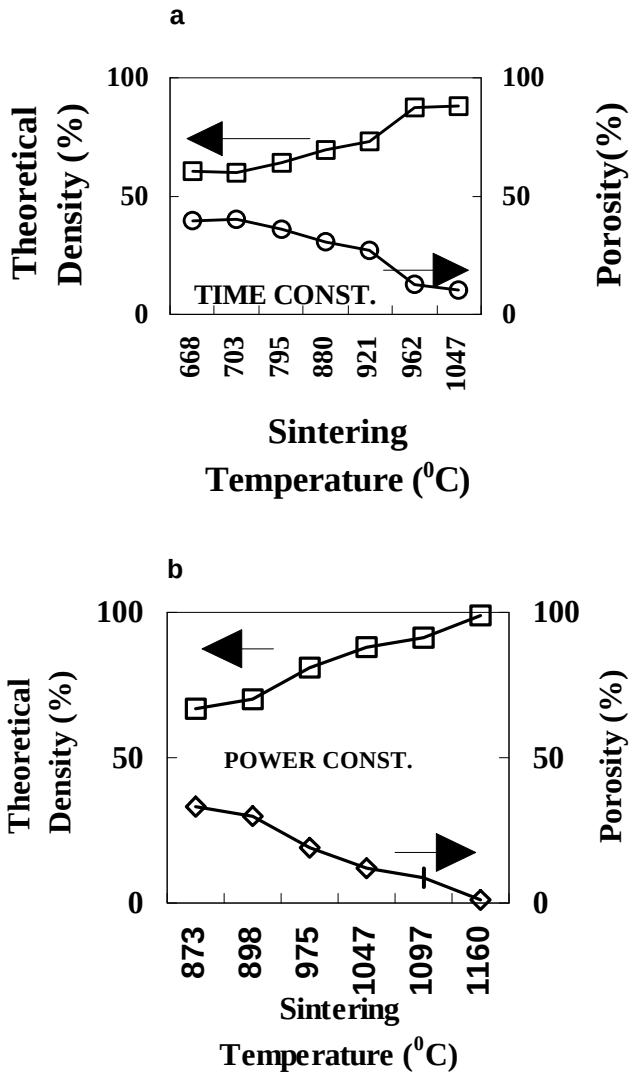
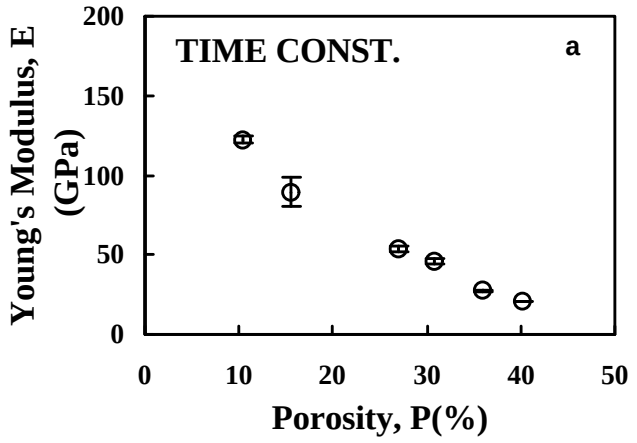


Figure 3. a. Relationship between relative density, porosity (*P*) and sintering temperature of ZnO for a constant microwave exposure time of 60 min and **b.** relationship between relative density, porosity (*P*) and sintering temperature of ZnO for a constant microwave power exposure of 800 W.



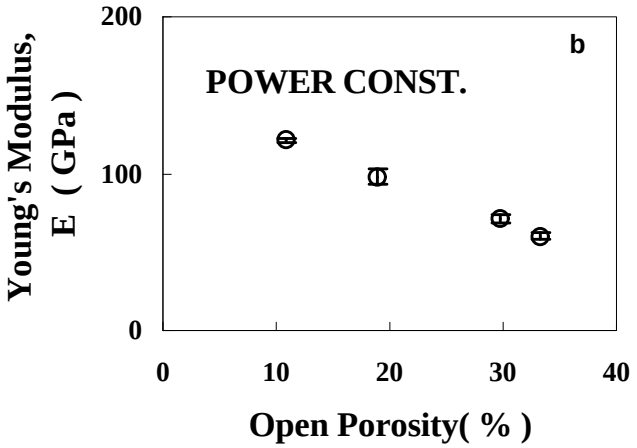


Figure 4. **a.** Dependence of Young's modulus (E) on porosity (P) of ZnO sintered with the constant microwave exposure time and **b.** dependence of Young's modulus (E) on porosity (P) of ZnO sintered with the constant microwave power level.

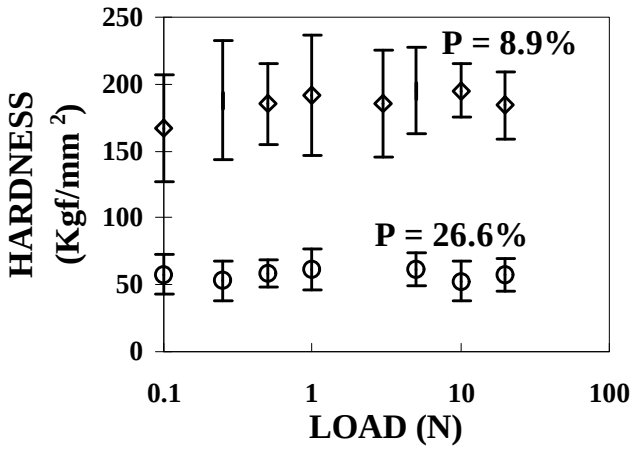


Figure 5. Vicker's microhardness data of ZnO.

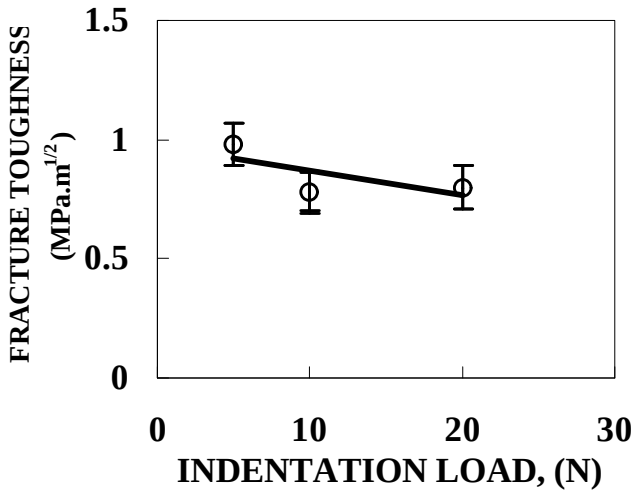


Figure 6. Indentation fracture toughness data of ZnO.

power level of 30–80 min exposure time. The results are shown in figures 3a, b. In both the cases when the samples were sintered at a constant microwave exposure time and microwave power exposure, there was a sharp decrease in the volume % open porosity with an enhanced densification. However, there was a small difference between the cases in terms of the densification rate. In the case of constant exposure time, the rate of densification was faster in the initial stage, but slowed down slightly above 950°C. In the case of constant microwave power exposure however, the densification rate increased linearly throughout the sintering temperature range (873–1160°C).

In both cases of constant exposure time and constant power level, the Young's moduli data decreased with an increase in volume % open porosity, P shown in figures 4a, b. The value predicted at zero porosity level was 148·58 GPa which is comparable to the data of 124 GPa reported by Martin *et al* (1996).

The indentation load (0·1–20 N) versus the Vicker's microhardness of both the porous and dense ZnO samples is shown in figure 5. Both the porous ZnO (P = 26·6%) and the dense ZnO sample (P = 8·9%) had a nearly load-independent microhardness (H) of about 50 kgf/mm^2 and 175 kgf/mm^2 respectively. It is evident from the present data that the porosity had a very significant effect on the microhardness value of ZnO.

Figure 6 shows the indentation fracture toughness (K_{IC}) data of a typical, dense ZnO sample (r/r_{th} = 0·889) measured with indentation loads of 5, 10 and 20 N. A slight decrease in the fracture toughness values from 1·0 to 0·75 MPa$\sqrt{m}$ is evident with an average of 0·85 ± 0·11 MPa$\sqrt{m}$.

4. Conclusions

The major conclusions of the present work are:

(I) Using low purity, commercial ZnO powder, phase pure product could be prepared by microwave sintering at a frequency of 2·45 GHz.
(II) Depending on whether a constant microwave exposure time or a constant microwave exposure power level was employed, sintered ZnO of theoretical density ≈ 60–99·9% could be produced.
(III) The Young's moduli data of the sintered ZnO samples, measured by the ultrasonic time of flight technique, exhibited a sharp dependence on its volume % open porosity.
(IV) For a given load range of 0·1–20 N, the Vicker's microhardness varied in the range of 50–175 kgf/mm^2 depending on the volume % open porosity present in the sintered ZnO samples. Further, the microhardness data appeared to be insensitive to the variation in the load range investigated in the present study.
(V) For a microwave sintered sample with a reasonably high relative density ($r/r_{th} \approx 0{\cdot}89$), the average value of indentation fracture toughness (K_{IC}) was close to about 0·9 MPa·m$^{0·5}$, for an applied load range of 5–20 N.

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References

Anstis G R, Chantikul P, Lawn B R and Marshall D B 1981 *J. Am. Ceram. Soc.* **64** 533
Aravindan S and Krishnamurthy R 1999 *Mater. Lett.* **38** 245
Birnboim A *et al* 1998 *J. Am. Ceram. Soc.* **51** 1493
Goldstein A, Giefman L and Bar Ziv S 1998 *J. Mater. Sci. Lett.* **17** 977
Goldstein A, Travitzky N, Singurindy A and Kravchik M 1999 *J. Eur. Ceram. Soc.* **19** 2067
Leiser K S and Clark D E 1998 *Ceram. Eng. Sci. Proc.* **19** 367
Lee K Y and Case E D 1999 *J. Mater. Sci. Lett.* **18** 201
Martin L P, Dadon D, Rosen M, Gershon D, Birman A, Levush B and Carmel Y 1996 *J. Am. Ceram. Soc.* **79** 2652
Xie Z, Yang J, Huang X and Huang Y 1999 *J. Euro. Ceram. Soc.* **19** 381