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RESEARCH ARTICLE

SYNTHESIS OF THE X-RAY DIFFRACTION AND SCANNING ELECTRON MICROSCOPE TO STUDY POWDER METALLURGY PREPARED BY 0.6(ZrO_2) 0.4(ZnO)

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ABSTRACT

The aim of this research prepared and studying some structure properties of $(ZnO)_{0.4}$ $(ZrO_2)_{0.6}$ nanocomposite Prepared by Powder metallurgy Technique. The structural and surface morphology of the nanocomposite were examined by means of X-Ray Diffraction and scanning electron microscopic (SEM). The particle size calculations were done using XRD Scherer's formula (15nm),and sample contain a mixture of the monoclinic, tetragonal hexagonal structure.

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INTRODUCTION

Zirconia is an important material because of its use in different fields of chemistry such as ceramics and catalysis (Inoue *et al.,* 2000). Zirconia (ZrO2) is one of the important materials used in the industry because of its high melting point, high mechanical properties, (Yue-Feng Zhu *et al.,* 2008). Zirconia is a wide band gap (5.0–5.5 eV) transition metal oxide with excellent mechanical, thermal, optical, and electrical properties. Pure crystalline zirconia exhibits three crystallographic phases:monoclinic (m-ZrO2), tetragonal (t-ZrO2), and cubic (c-ZrO2) phases. Monoclinic zirconia is the stable phase in pure state at room temperature (Dezhi Tan *et al.,* 2011) Zirconium oxide is one of the most intensively studied materials owing to its technologically important applications in oxygen sensors, fuel cell electrolytes, catalysts and catalytic supports, metal oxides emiconductor devices, superior thermal and chemical stability etc. (Liu *et al.,* 2008). Zirconia is an oxide of zirconium element, a transition metal in the periodic table that has relative mass 91.224 grams per mole. The zirconia polymorphs have different densities. The monoclinic phase has the smallest density which is 5.6 g/cm3, while tetragonal and cubic phase have density around 6.10 g/cm3 and 6.27 g/cm3 (Suciu *et al.,* 2006).

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Stabilized zirconia crystals are not easily produced at low temperatures without the application of specific pressure and the addition of stabilizer (Callister, 2007; Milman *et al.,* 2009). Zirconia is well known as a material for structural applications due to its excellent properties, such as high mechanical strength, fracture toughness, and hardness (Bandyopadhyay, 2008). Zirconia is a polymorphic compound (Dercz *et al.,* 2008). The crystal structures of zirconia are monoclinic, tetragonal, and cubic. The unstable crystal structures of zirconia at room temperature and pressure conditions requires the addition of other compounds as stabilizer agents to maintain the zirconia crystal phase that is obtained. The monoclinic structure of zirconia particularly forms at the temperatures around 4000C-11700C. It transforms to a tetragonal structure at 11700C-23700C, while at the higher temperatures, which is 23700C-26000C, a cubic structure of zirconia is formed (Dercz *et al.,* 2008). This polymorphic transformation caused modifications in the density and physical properties of zirconia (Callister, 2007). Zirconia with tetragonal and cubic structures has higher density and higher crystallization temperature than the monoclinic structure (Dercz *et al.,* 2008; Callister, 2007). Among the ceramic semiconductors, zirconia is a special transition-metal oxide that possesses the bifunctional characteristics of weak acid and weak base properties (Su *et al.,* 2000; Wong *et al.,* 1997). The P-type semiconductor exhibits abundant oxygen vacancies on

its surface. The high ionexchange capacity and redox activities make it useful in many catalytic processes as catalyst, support (Li *et al.,* 2001) and promoter or in solid fuel cells (Egger *et al.,* 2004). Zirconia-based ceramics are found in a remarkable variety of technological and commercial applications such as catalysts, oxygen sensors (Subbarao, 1988), high dielectric constant materials for very large scale integrated circuits, and as gate dielectrics in metal oxide-semiconductor devices (Wilk and Wallace, 2000). zinc oxide has attracted considerable attension over the last years, it is one of promising materials which usually appears as a white powder or yellow wish white powder changing from white to yellow when heated in air and reverting to white on cooling (Harloff, 1995). It occurs in nature with the mineral name ″zincite″ and crystallizes preferentially in the hexagonal wurtzite-type structurel which is most stable at ambient conditions (Prince, 2006). ZnO is relating soft material with approximate hardness of (4.5) on Mohs scale, has low thermal expansion and high temperature, also has high heat capacity, becides that, has large exciton binding energy of60 MeV at room temperature, with good piezoelectric characteristics (Ozgür, 2005). The ZnO powder is widely used as additives into numerous materials and products including plastics, ceramic, glass, rubber (car tyres) besides that it is used as catalyst (in oil and petrochemical industry) and as sunscreens (ointments, creams and lotions to protect against sunbeam and other damages to the skin (clinical pharmacology (it is ability to neutralize acid and for its mild bactericidal properties). Also it is used as a sintered parts in varistors which is known as Voltage Dependent Resistor (VDR) for protection against inductives surges or power surges. Besides that it is used in Ferrites which as in television radio and tele -communication applications (Somiya *et al.,* 2003)

As a wide band gap II–VI compound semiconductor, zinc oxide (ZnO) (Eg 3.3 eV at 300 K) has attracted a great deal of interest because of its prospects in visible and ultraviolet optoelectronic devices. Compared with another extensively used wide band gap semiconductor GaN, ZnO has a larger exciton binding energy ((60) meV). As-grown ZnO almost always exhibits strong n-type conductivity, which is attributed to low energy native point defects, such as zinc interstitials (Zni) and oxygen vacancies (VO) (Fenggong Wang *et al.,* 2008). There are many methods for fabrication of metal matrix composites (MMCs) such as powder metallurgy, squeeze casting, and compocasting. For the metal matrix composites, molten metal mixing is a cost effective method while powder metallurgy is costly, and squeeze casting provides good infiltration quality of chopped performs (Khorramie *et al.,* 2012)

MATERIALS AND METHODS

Raw materials of high purity are used, because the presence of impurities affects the properties of prepared materials. All the chemical materials used are commercial one and are of analytical grade.

Stoichiometric calculations

The appropriate weight percentage of each oxide to be mixed is calculated by the following formula;

Weight % of oxide =

A: molecular weight of oxide.
B: required weight of the sample.
C: sum of the molecular weight of the given composition.

$$(ZnO)_{0.4}\,(ZrO_2)_{0.6}$$

Mixing

The powders (ZnO and ZrO_2) are mixed to obtain a uniform distribution of the components. These are mixed using a variable speed electric mixer for two hours for the purpose of obtaining a homogeneous mixture and the non-agglomerated mixtures are then dried in an oven at 80 °C for 2 hours.

Pellet formation

A mold is designed for the manufacture of samples in the form of pellet in diameter (9mm) and thickness (5mm) and the weight of the sample is (1.3 g). It uses hydraulic press with a pressure of (500-700 psi), and the diameter of the mold used (1cm) with a hight of (3cm).

Sintering

The green Pellets are loaded on refractory plates (pure alumina container) and sintered at temperature of (800, 1000 °C) for four hours, and then cooled in the furnace to room temperature.

Apparatus

The X-ray diffraction pattern were recorded using XRD-6000 with CuKα (λ=1.5406A°) that have an accelerating voltage of 220/50HZ which is produced by SHIMADZU company, and the scanning electron microscope used in imaging the nanoparticles was a VEGA//EasyProbe which is a favorable combination of a scanning electron microscope and a fully integrated energy dispersive X- ray microanalyser produced by TESCAN, s.r.o., Libušina trída.

RESULTS AND DISCUSSION

x-ray diffraction

X-Ray diffraction (XRD) has been performed for the identification of the crystal structure and growth orientation of the nanostructures. The crystal structure obtained was investigated by XRD.

All diffraction data are in good agreement with JCPDS files No.36-0420, No.17-0923,No.37-1484, No.13-0307, No.03-0515, No.36-1451,and No.05-0664.

Figure (1) shows the XRD pattern of $(ZnO)_{0.4}(ZrO_2)_{0.6}$ composite nanostructure. Both ZnO and ZrO_2 peaks appears in the pattern which confirms the presence of both materials. The XRD pattern of the synthesized is shown in Figure (1).

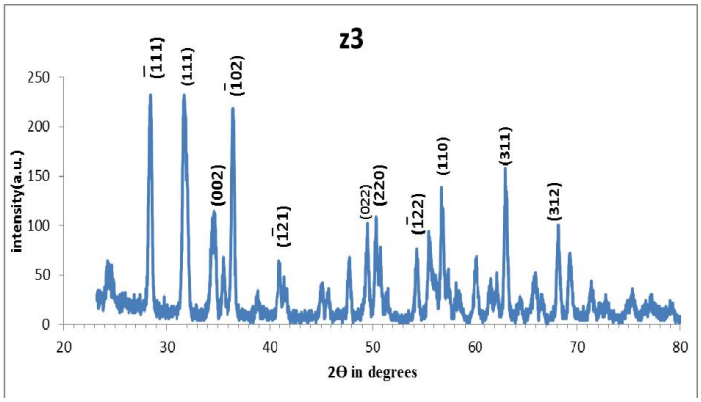


Figure 1. XRD pattern of $(ZnO)_{0.4}(ZrO_2)_{0.6}$ sample sintering at 1000 °C

The information about crystal structure can be obtained from the diffraction peaks occurring at a particular angle and intensity. The purity and crystallinity of the obtained(ZnO-ZrO_2)nanoparticles were examined by using XRD analysis. Fig. 1. show the XRD pattern of (ZnO-ZrO_2) nanoparticles sintered at 1000°C. The XRD pattern of (ZnO-ZrO_2) in Fig1. sample contain a mixture of the monoclinic and tetragonal phases , whereas the XRD pattern of ZrO_2 shows only broad reflections originating from monoclinic ZrO_2 nanoparticles, XRD diffraction has exhibited a mixed tetragonal and monoclinic long-range-order structures in the 1000 °C. Also it is clear that all the peaks were matched well with the hexagonal structure of ZnO but with asmall peak very low intensity at 2θ = 34.54˚, 2θ = 56.78˚. This is shown by the small diffraction main peak intensity of t-ZrO_2 at a 2θ angle of 41.37˚,54.27˚, The main phase of ZrO_2 is shown by two main peaks at diffraction angles of 28.4 and 31.82, from crystals planes of (ˉ111) and (111) respectively.

The detailed analysis of the XRD and the assignments of various reflections are given in Table (1).

Table 1. Strongest three peaks of Fig.1

No.	Peak No.	2Theta (deg)	d(Aº)	FWHM (deg)
1	17	31.8268	2.80943	0.55360
2	16	28.4470	3.13506	0.38330
3	20	36.4843	2.46076	0.35550

Particle Size Calculation from x-ray diffraction.Considering the peak at degrees, average particle size has been estimated by using Debye-Scherrer formula (Nath *et al.,* 2007; Hall *et al.,* 2000).

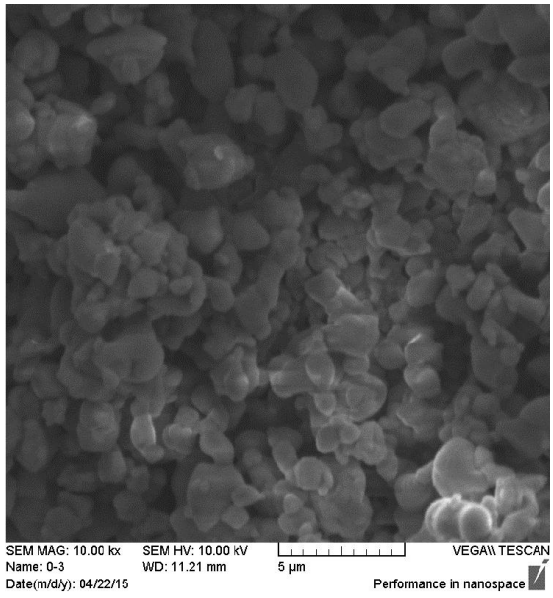
$D = 0.9\lambda / \beta \cos \theta$ …… (1)

Where λ is the wavelength of X-ray (0.15406 nm). β is FWHM (full width at half maximum).

θ is the diffraction angle. For sample sintering at 1000°C:
D = 15 nm

Scanning electron microscope

The SEM images of prepared (ZnO-ZrO_2) shown in Figure (2)



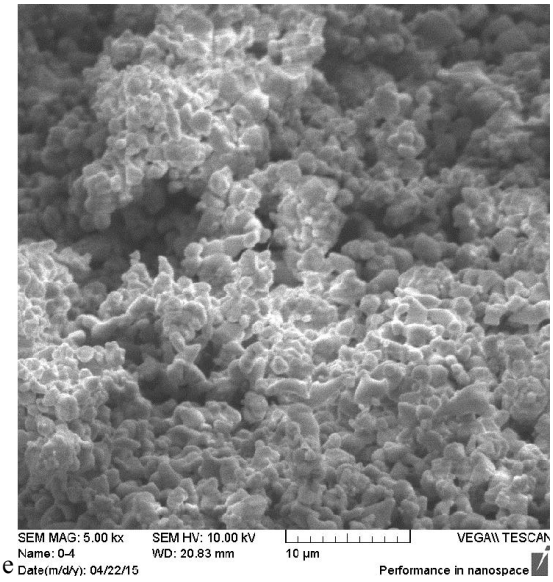


Figure 2. SEM images of $(ZnO)_{0.4}(ZrO_2)_{0.6}$ sample sintering at 1000 °C

SEM images are shown in Fig. 2. We can observe round or oval structures with average size of 20 nm, it can be concluded that these structures are agglomerates of individual grains of zirconium dioxide and zinc oxide. The agglomerates are bound to each other creating large aggregated structures. The binding between agglomerates in large aggregate is rather weaker than between individual grains of zirconium dioxide and zinc oxide. Grain boundaries between agglomerates are well seen in the images. According to the SEM micrographs given in Fig. 2 It appears to be similar to coral shape.

Conclusion

Were successfully prepared $(ZnO)_{0.4}$ $(ZrO_2)_{0.6}$ nanocomposite using Powder metallurgy technic. The phase of the zirconium dioxide and zinc oxide formed was a sample contain a mixture of the monoclinic, tetragonal of ZrO2 and hexagonal structure of ZnO, average particle size has been estimated by using

Debye-Scherrer formula (15 nm), by SEM The binding between agglomerates in large aggregate is rather weaker than between individual grains of zirconium dioxide and zinc oxide.

REFERENCES

Bandyopadhyay A.K. 2008. Nano Materials, New Age International, New Delhi.

Callister W. 2007 Materials Science and Engineering An Introduction, 7th ed. New York: John Wiley & Sons, Inc.

Callister W. 2007. Materials Science and Engineering An Introduction, 7th ed. New York: John Wiley & Sons, Inc, 2007.

Dercz G., Prusik, and L. Pajak, 2008. "X-Ray and SEM Studies on Zirconia Powder," *Journal of Achievement in Materials and Manufacturing Engineering*, Vol. (31), 408-414.

Dezhi Tan • Geng Lin • Yin Liu • Yu Teng •Yixi Zhuang • Bin Zhu • 2011. Quanzhong Zhao Jianrong Qiu, Synthesis of nanocrystalline cubic zirconia using femtosecond laser ablation, Springer Science, *J Nanopart Res*., 13:1183–1190.

Egger P., G.D. Soraru, S. Dire, 2004. *J. Eur. Ceram. Soc*., 24, 1371.

Fenggong Wang, Maoshui Lv, Zhiyong Pang, Tianlin Yang, Ying Dai, Shenghao Han, 2008. Theoretical study of structural, optical and electrical properties of zirconium-doped zinc oxide, *Applied Surface Science* 254, 6983–6986.

Hall B. D., D. Zanchet and D. Ugarte, 2000. "Estimating nanoparticle size from diffraction measurements, *Journal of Applied Crystallography*", Volume 33, Part 6.

Harloff J. 1995. ″Insitu guantitative measurements of electric fields in ZnO thin film using electrostatic force microscopy″ M.Sc thesis .Univ. of Pennsylvania school of Eng. and applied Sciences.

Inoue M., Sato K., Nakamura T. and Inui T. 2000. Glycothermal synthesis of zirconia-rare earth oxide solid solutions (J). *Catal Lett.,* 65: 79−83.

Khorramie1 S. A., M. A. Baghchesara2 R. Lotfi1 Sh. Moradi Dehagi1, 2012. Synthesis of ZrO_2 nanoparticle by combination of sol-gel auto-combustion method-irradiation technique, and preparation of Al-ZrO_2 metal matrix composites, *Int.J.Nano Dim.* 2(4): 261-267, *Spring*, ISSN: 2008-8868)

Li Y.M., D.H. He, Z.X. Cheng, C.L. Su, J.R. Li, Q.M. Zhu, 2001. *J. Mol. Catal. A: Chem*., 175, 267.

Liu R.J., Y. Li, H. Zhao, F.Y. Zhao and Y.Q. Hu, 2008. Synthesis and characterization of Al2O3 hollow spheres, Mater. Lett., 62, 2593–2595.

Milman V., A. Perlov, K. Refson, S. J. Clark, J. Gavartin, and B. Winkler, 2009. "Structural, Electronic, and Vibrational Properties of Tetragonal Zirconia under Pressure: a Density Functional Theory Study," *Journal of Physics: Condensed Matter*, p. 12.

Nath S., D.Chakdar, and G.Gope; 2007. "Synthesis of CdS and ZnS quantum dots and their applications in electronics, Nanotrends- A journal of nanotechnology and its application", 02(03).

Ozgür U. A 2005. ″Comperhensive review of ZnO materials and devices *J. of Applied Physics.*

Prince E. 2006. "International tables for crystallog raphy. International using of crystallography.

Somiya S., F. Aldinges, N, Claussen and R. M. Springgs Hardbook, Advanced ceramics, Processing and their applications Vol. 2 , p. 32 – 39, 2003.

Su C.L., J.R. Li, D.H. He, 2000. Appl. Catal., A 202, 81.

Subbarao E.C., H.S. Maiti, 1988. *Adv. Ceram*., 24, 731.

Suciu C., A. C. Hoffmann, and L. Gagea, "Sol-Gel Production of ZrO_2 and 8YSZ with New Organic Precursor," *Elsevier Science*, p. 4, Mar. 2006.

Wilk G.D. and R.M. Wallace, 2000. *Appl. Phys. Lett.* 112, 76.

Wong M.S., D.M. Antonelli, J.Y. Ying, 1997. Nanostruct. Mater. 9, 165.

Yue-Feng Zhu a,b,*, Lei Shi a,b, Ji Liang a,b, David Hui c, Kin-tak Lau d, 2008. Synthesis of zirconia nanoparticles on carbon nanotubes and their potential, Composites: Part B 39, 1136–1141.

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