Study Some Physical and Mechanical Properties of Ceramic – Ceramic Fibers Composite

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Abstract

Zirconia fibers have been prepared by convertional method using cotton threads impregrated in zirconium oxychloride solution ($ZrOCl_2$. $8H_2O$). X-ray diffraction shows the crystallinity of zirconia and optical microscopy shows the fibers fabrication. Differnet percentage (2 ,4 ,8 , 10 and 12) of prepared zirconia fibers mixed with ZnO powder.

All the specmen sintered at 1250 ˚C for 2hrs. Physical properties (density and volume shrinkage) were measured and Mechanicl properties (Vicker΄s hardness,fracture strength and fracture toughness by indentation method) were calculated, 10% of fiber content has the maximum values for these properties for the composite.

Keywords: Zirconia fibers, composite, fracture toughness, fracture strength.

دراسة بعض الخواص الفيزيائية والميكانيكية لمتراكب سيراميك - الياف سيراميك

الخلاصة

تم تحضير الياف الزركونيا بالطريقة التقليدية وذلك بغمر الياف القطن بمحلول من اوكسي كلوريد الزركونيوم. استخدمت حيود الاشعة السينية لغرض التأكد من تبلور الزركونيا. وكذلك استخدم المجهر الضوئي للتأكد من الحصول على الالياف.

تم اضافة نسب من الياف الزركونيا (2,4,8,10 and 12) الى مسحوق اوكسيد الخارصين. حيث لبدت كل النماذج بدرجة حرارة 1250 ˚C لمدة ساعتين. تم قياس الخواص الفيزيائية (الكثافة,التقلص الحجمي). وكذلك تم حساب بعض الخواص الميكانيكية (صلادة فيكرز ، متانة الكسر والقساوة) حيث اتضح ان اعظم قيمة لهذه الخواص كانت عند نسبة 10% من ألياف الزركونيا.

Introduction

Zinc oxide has attracted considerable attension over the last years, it is one of promising materials which usually appears as a white powder or yellowwish white powder changing from white to yellow when heated in air and reverting to white on cooling [1].

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 [2]. 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 of 60 MeV at room temperature, with good piezoelectric characteristics [3]. The ZnO powder is widely used as additives into numerous materials and products including plastics, ceramic, glass, rubber (car tyres)

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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 [4].

To enhance the mechanical properties of such ceramic, Yittria stabilized Zirconia (Ysz) used as an additive to toughen ceramic [5].

Because zirconia is very useful in its stabilized state, in some cases, the tetragonal phase can be metastable, if sufficient quantities of metastable tetragonal phase is present, then an appliect stress magnified by the stress concentration at a crack tip, can cause the tetragonal phase to convert to monoclinic, with the associated volume expansion.

This phase transformation can then enhance the fracture toughness and other mechanical properties [6-7].

Selim et.al have studied the low voltage of ZnO varistor.The device showed a nonlinear current voltage characteristic when annealed at 800-900 ˚C in air and N_2 gas [8].

Eda et.al have studied the grain growth control in ZnO body using seed grains by sintering a mixture of ZnO fine powder. They found that the anomalous grain growth is caused by difference between the radii of ZnO fine powder and ZnO seed grain [9].

Haen et.al studied the synthesis of ZnO nanoparticles via aqueous carboxylate gelation route and investigated on the thermal decomposition of the gel [10].

Hawang et.al prepared ZnO as nanocrystalline using glecine as fuel and ZnO nitrate as oxidant, the result powders show high specific surface area and possess small primary crystallite size after sintering in air at 1050 ˚C for 1.5 h, where, the sinterd density was 92% of theoretical density of ZnO [11].

Zhou et.al also studied the preparation of nanoparticles of ZnO,the powders appeared do be reqularly spherical or elliptical and their sizes range from 20 to 40 nm.

Miguel et.al have studied the mechanical properties of ZnO doped by SnO_2. (12) They found that the elastic modulus (static and dynamic) were two times higher for SnO_2 and ZnO alone. Also the similar behavior was found for the bending strength. [13].

Khor, etal have studied the effect of ZnO on dielectric properties and electrical conductivity of ternary zinc magnesium phosphate glasses,the dielectric properties increased with ZnO content. [14]

Adawiya et.al have studied the effect of alumina doping on structural and optical properties ZnO thin film by pulsed lazer deposition. They founed that increasing Al_2O_3 content increasing the roughness of the film.[15]

Kumar et.al that synthesized ZnO powder through combustion route without any calcinations step. Nanoparticles of ZnO obtained and when sintered at 1200˚C a 97% of theoretical density obtained [16].

The aim of this work is to fabricate zirconia fibers and used to study physical and mechanical properties of the composite ceramic-ceramic fibers.

Experimental procedures

1- Materials

A- High purity of ZnO has been used as a starting materials (matrix) which was supplied by Zinc company, New Jersey. USA with average particle size of 0.5 μm.

B -Zirconium oxychloride ($ZrOCl_2.\ 8H_2O$) which was supplied by Zirconium oxide 14603-Germany; used to fabricate a zirconia fibers.

2- Fibers fabrication

The process of fibers manufacturing involves the preparation of 1M of $ZrOCl_2.\ 8H_2O$ solution with which to impregrate a conventional cotton threads chosen for its wicking rate.

The fabrication process was achieved by the following steps :

1- preparation the solution of zirconium oxychloride in concentration of 1M solution.
2- Selection acotton as a substance fibers.
3- Impregnation of the cotton in the solution for three days and extraction the solution and dried at 100˚C in an oven for 24 hrs.
4- Heat treatment was achieved slowly at two stages, first,heat to 350 ˚C with very low heating rate (2˚C/min), that needs 3 hrs to reach this temperature then raised the temperature to 600˚C for 1 hrs and slow cooling at the furnace.
5- X-ray diffraction used to postulate the phase transformation of zirconia after sintering. The intensities and d-spacing of ZnO_2 phases have been identified by using ASTM No.36-1551

Quantity of tetragonal and monoclinic phase of zirconia determined by using equation : (1) [17]

$$X_m = [I_m(11\bar{1}) + I_m(111)] / [I_m(1111) + I_m(11\bar{1}) + I_t(111)] \quad …..(1)$$

Where X_m is the percentage of monoclinic phase $I_m(111)$,I_t (111) are the intensity of monoclinic and tetragonal respectively of (111) plane and ($11\bar{1}$) plan.

The XRD aualysis done by Twin-X (Oxford instruments) with Nickl filter cukα radiation (λ = 1.54056 ˚A).

3- Composite preparation five percentages of zirconia fibers (2,4,8,10,12) have been mixed thoroughly with ZnO powder for two hrs which resulting in producing uniform distribution which give consistent performance during pressing and sintering.
4- Compaction has been done in stainless steel die with (10mm) diameter. The compaction load used was 30KN, 1.5 wt.% of polyvenal alcohol used as a binder.
5- Sintering the prepared green samples at 1250 ˚C for 2 hrs with 5˚C/min as a heating and cooling rate.
6- Measurments

A - Apparent density

According to ASTM-C.20 (Archimedean method) density was calculated by the equation (2) [18].

$$P = W_d / (W_w - W_s) \quad …..(2)$$

Where P = apparent density
W_d = Dry weight in g
W_w = saturated weight in g
W_s = suspended weight (after boiling in water) in g.

B- Volume shrinkage

To calculate volume shrinkage equation(3) was used :

$$\text{Volume shrinkage \%} = (V_1 – V_2) / V_2 \times 100\ \% \quad …..(3)$$

Where V_1 and V_2 are the volume befor and after sintering respectively.

C- Vickers Hardness.
Equation(4) was used to calculate the Vickers microhardness using(Digital microhardness tester,Time group Inc.924) machine.

$H_v = 1.8544\ P / d^2$ …..(4)
Where
H_v = Vickers microhardness in kg/mm^2
P = The load in kg.
d=The indentor diagounal length (mm)

D- Dimetrical compression strength (Fracture strength).
Equation (5) was used to calculate the fracture strength using controlled electronic Universal testing machine (WDW-50E) (Time group Inc).
$\sigma = 2P / \pi dt$ ….(5)
where
σ = fracture strength in MPa.
P = applied load in kg.
d = diameter of the specimen in mm.
t = thickness of the specimen in mm.
E- Fractur toughness calculated by using indentation methode by measuring Vickers hardness and direct measuring of the crack length that occurred when applying the load. The applied load in this test was 1kg,crack length was measured by using optical microscope in the hardness machine. The fracture toughness (k_{1c}) was calculated using equation (6) [19].
$K_{1c}=0.0319P/a\sqrt{l}$ ….(6)
Where
K_{1c} = is the fracture toughness is MPa $m^{1/2}$
P = is the load in kg.
a = is the dimeter of the indent and .
l = is the crack lenth.

Results and Discussion

Room temperature X-ray diffraction (XRD) was carried out on the synthesized zirconia fibers for phase indentification.Fig (1) shows the spectrum of (XRD) for the zirconia fibers which indicate the crystallinity of zirconia.It consists of two phases ; first is monoclinic and the other is tetragonal phase. The relative tetragonal and monoclinic values were calculated using equation (1), it was found that tetragonal phase is 17% and 83% for monoclinic phase. This spectrum was compared to the ASTM cards (N 1997 JCPDS) No. 37-1484 for monoclinic and No-02-0733 for tetragonal phase. Fig(2) shows the optical photograph of prepared zirconia fibers.which are used in the toughening of ZnO ceramic. It ic clear from the photograph the forming of fibers shape.

Final product density is one of the main factors that influence mechanical and physical properties of ceramic materials. Theoretical density of the composite can be calculated from the densities of their components by rules of mixture. The density of the prepared composite was calculated using equation (2). The effect of the content of zirconia fibers on the density of ZnO is shown in Fig(3) which shows that an improvement has been occured in densities with increasing fibers content.

Using equation (3) volume shrinkage was calculated. the effect of Volume shrinkage is represented is fig(4) which shows an increase in volume shrinkage as the zirconia fibers content increasing which that has a relation with the density of the comosite,where at increasing the volume shrinkage means that porosity decreased and density is increasing By using equation (4)

Vickers microhardness of prepared samples was calculated,the variation in hardness with zirconia fibers contents was presented in fig(5). It is clear that at increasing the zirconia fibers the hardness increased,and the maximum hardness was at 10% and then decreased. This behavior is simlar to the work done by Bengisu et.al [17]. who obtain the highest hardness at 10% of ZrO_2 doping Al_2O_3.

Fig (6) shows the effect of fibers content on the fracture strength of the composite.

It is clear that increasing the fiber content the strength is increased until 10% and decreased slightly this may be due to the flaw size, where the average flaw size in the ceramics increased by the addition of fibers, that may be due to the reduction in consequence of volume expansion associated with the transformation of tetragonal zirconia to monoclinic phase [18].

Fig (7) shows the effect of fibers content on the fracture toughness of ZnO ceramic, which also cleard that an improvement shown on the fracture toughness of ZnO by increasing the fibers contents, again 10% has the maximum value of the ZnO toughness. This improvement may be due to fiber pull-out or crack deflection as a toughening mechanisms [20].

Conclusions

The following remarks are concluded from the preparing of fibers and toughening ZnO ceramic which are.

1- The fabricated fiber consists of two phases,which appeared highly monoclinic and tetragonal.
2- Density of the composite increased as fibers content increased.
3- Microhardness,fracture strength are increased for their maxmim values of concentration of 10% zirconia fibers.
4- Improvement in fracture toughness is achieved in this composite.

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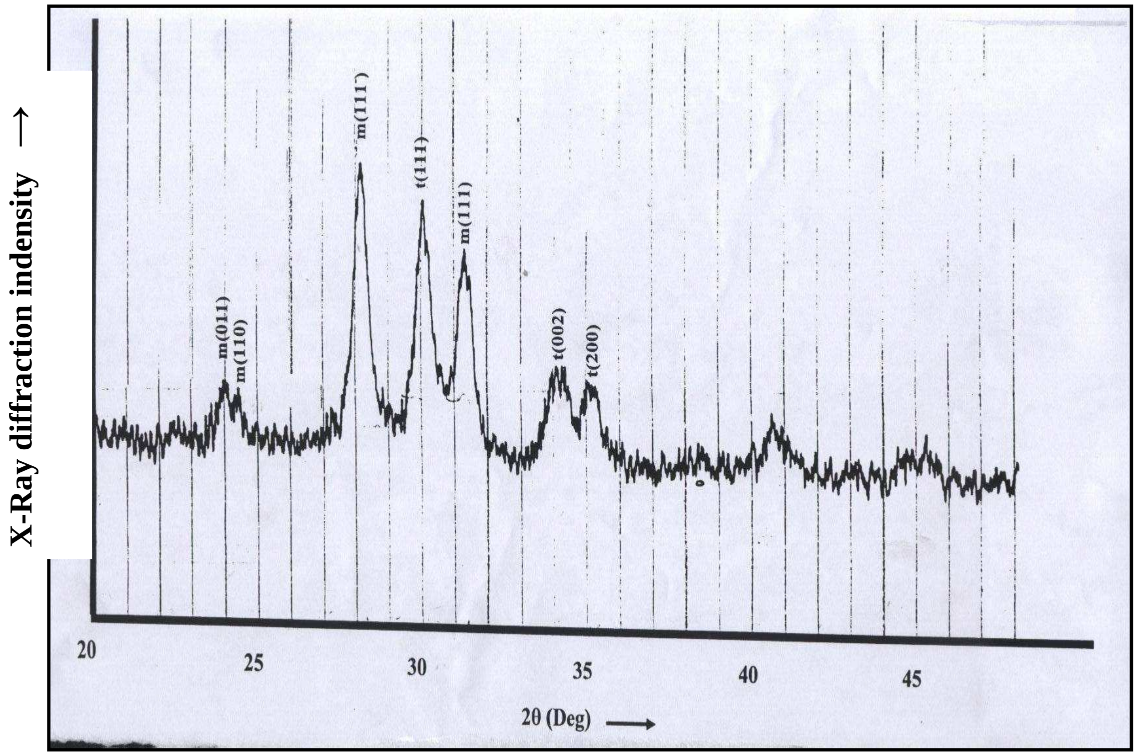

Figure (1) XRD spectrum of zirconia fibers

Fig(2) Optical photograph of zirconia fibres

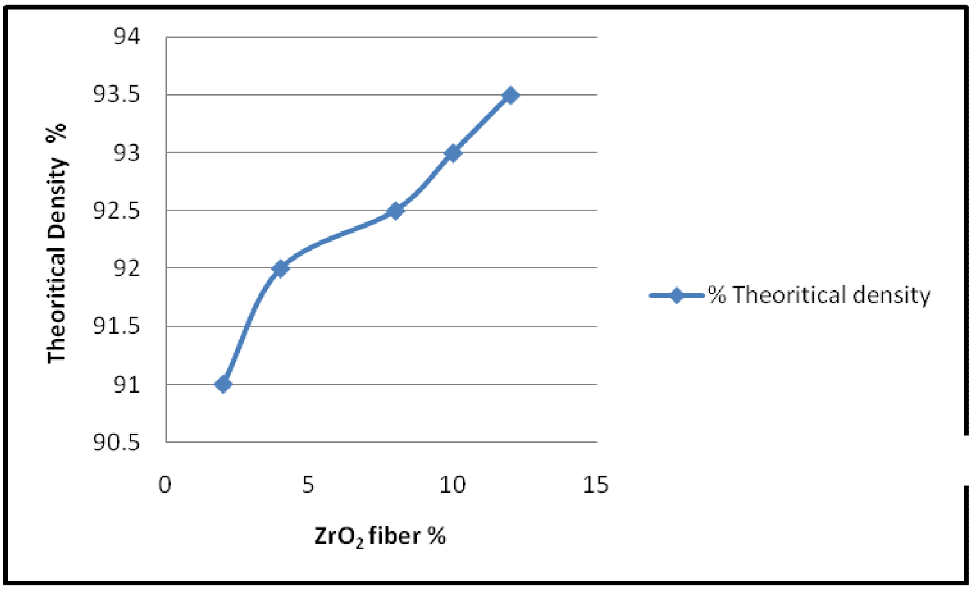

Figure (3) The effect of ZrO_2 fiber on the density of ZnO

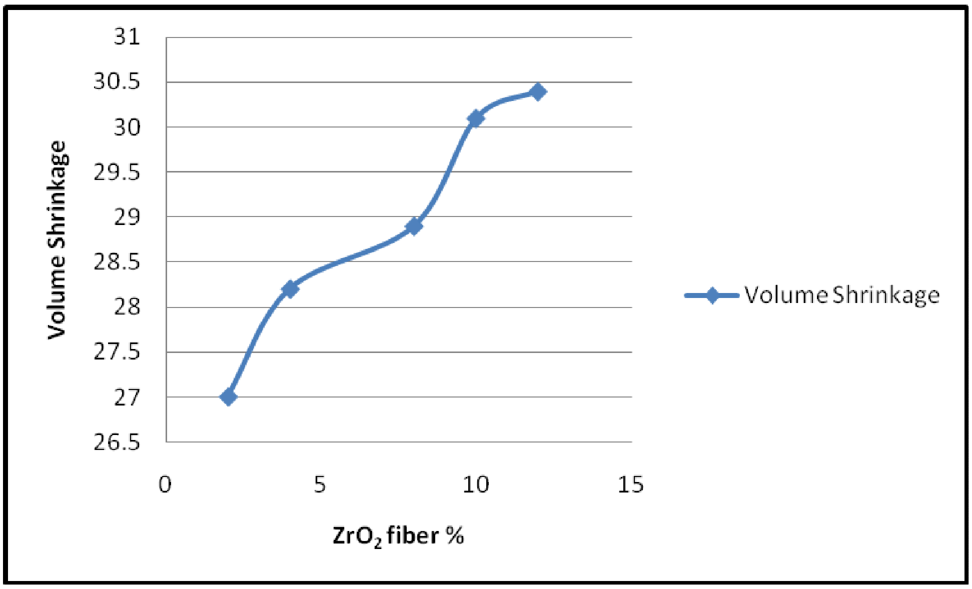

Figure (4) Volume Shrinkage as a function of ZrO_2 for ZnO –ZrO_2 Composite.

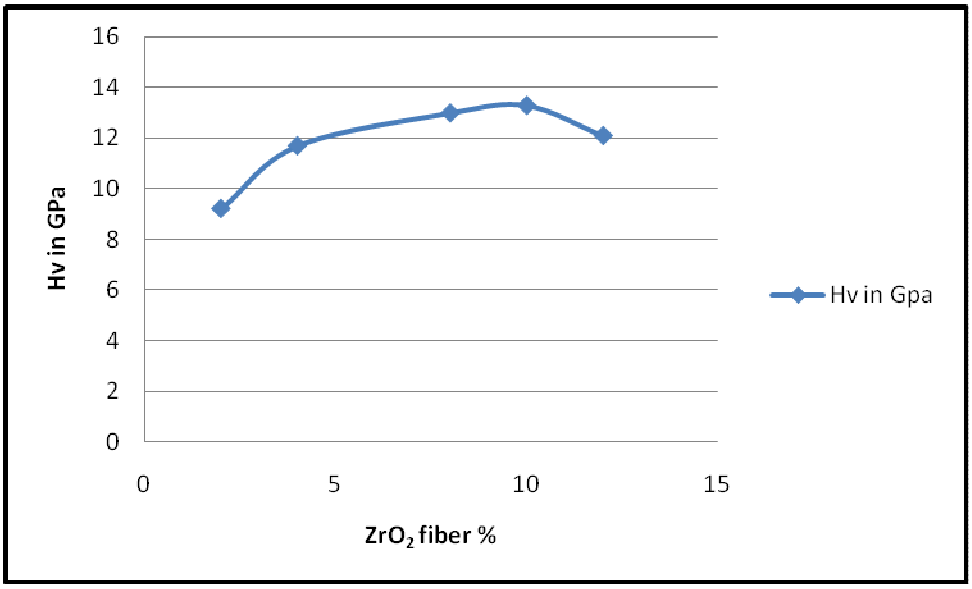

Figure (5) the effect of ZrO_2 fiber on the hardness of ZnO

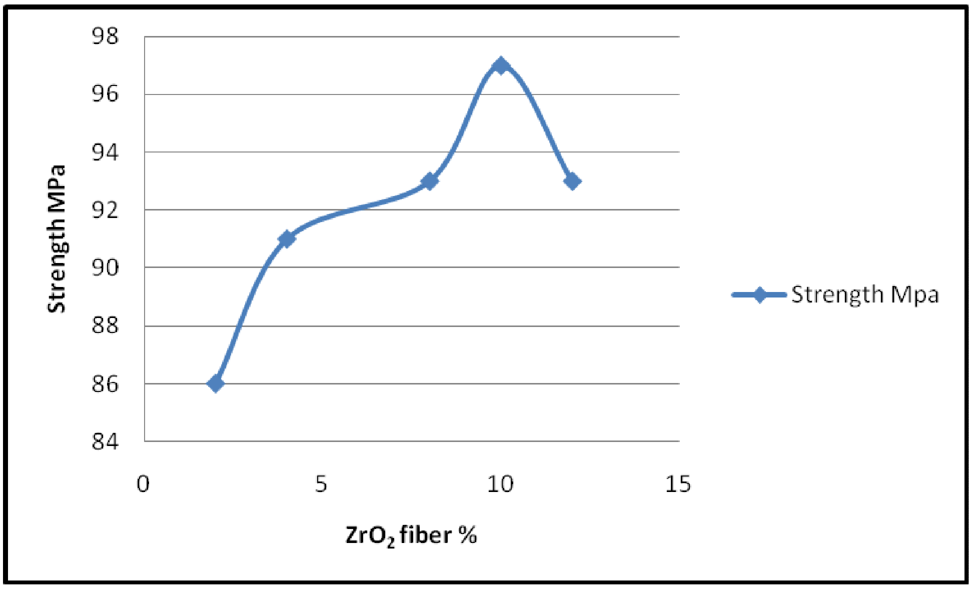

Figure (6) the effect of ZrO_2 fiber on the fracture strength of ZnO.

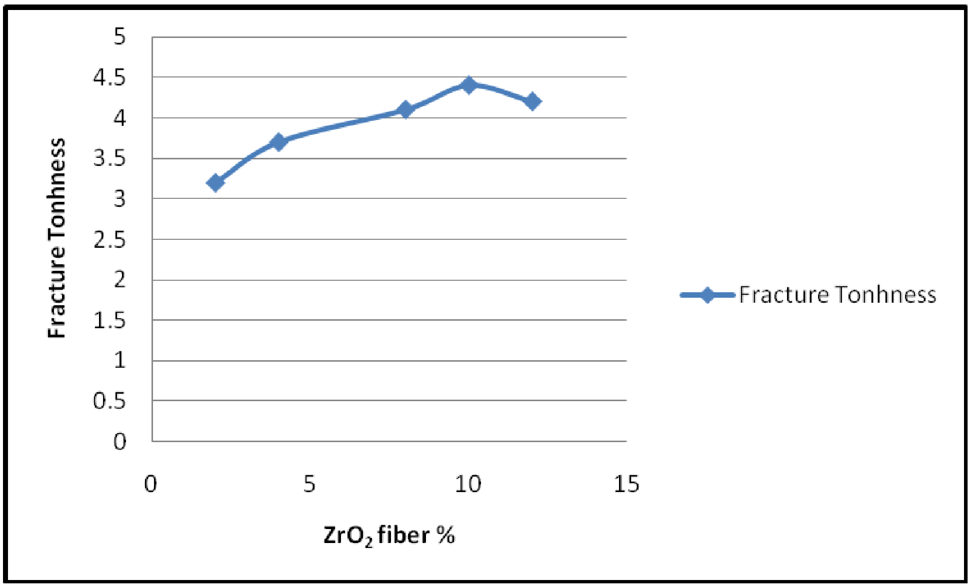

Figure (7) the effect of ZrO_2 fiber on the fracture toughness of ZnO