

## Mechanical and Physical Properties of PZT/ $x$ Al $_2$ O $_3$ Ceramics Systems

Abdulkarim Ziedan Khalf

School of Applied Sciences, University of Technology, Baghdad, Iraq.

### Abstract

The compound of Ferroelectric nanopowder Lead Zirconate Titanate (PZT) Pb(Zr $_x$ Ti $_{1-x}$ )O $_3$  was prepared using Oxalate method. Al $_2$ O $_3$  was doped using solid state sintering method as general system PZT/ $x$ Al $_2$ O $_3$  at ( $x= 0.5, 1, 1.5$  vol. %). The effects of Al $_2$ O $_3$  addition on the mechanical and physical properties of PZT ceramics were investigated. Increased Vickers Hardness with addition Al $_2$ O $_3$  to PZT which its maximum hardness value was found for the samples containing 1vol. %Al $_2$ O $_3$ . Also, the maximum value of dielectric strength of containing 1vol.%Al $_2$ O $_3$ . This is indicating that the addition Al $_2$ O $_3$  into PZT leads to improve dielectric properties of PZT ceramics. It is observed that the mechanical properties of the ceramics systems PZT/ $x$ Al $_2$ O $_3$  are larger than of PZT ceramics.

**Keywords:** PZT ceramics, Oxalate method, Dielectric and Mechanical Properties, Ferroelectric.

الخصائص الميكانيكية والفيزيائية لنظم (PZT/ $x$ Al $_2$ O $_3$ ) السيراميكية

عبد الكريم زيدان خلف

قسم العلوم التطبيقية, الجامعة التكنولوجية, بغداد, العراق.

### الخلاصة

تم إعداد مركبات فيروكهربائية ذي الصيغة (Pb(Zr $_x$ Ti $_{1-x}$ )O $_3$ ) باستخدام طريقة الاوكزالات. ثم إضافة (Al $_2$ O $_3$ ) الى (PZT) بطريقة تفاعل الحالة الصلبة وحسب الصيغة (PZT/ $x$ Al $_2$ O $_3$ ) وعند النسب ( $x= 0.5, 1, 1.5$  vol. %). وقد تم التحقق من تأثير إضافة (Al $_2$ O $_3$ ) الى (PZT) على الخصائص الميكانيكية والفيزيائية. زيادة الصلادة مع إضافة (Al $_2$ O $_3$ ) الى (PZT) والذي عثر على أعلى قيمة للصلادة للعينات التي تحتوي (1vol.% من (Al $_2$ O $_3$ ). أيضا أعلى

قيمة لمتانة العزل الكهربائي عند النسبة (1 vol.%) من (Al<sub>2</sub>O<sub>3</sub>)، وهذا يدل على أن إضافة (Al<sub>2</sub>O<sub>3</sub>) الى (PZT) يؤدي الى تحسين خواص العزل الكهربائي لل (PZT). لوحظ أن الخواص الميكانيكية للنظام (PZT/ $x$ Al<sub>2</sub>O<sub>3</sub>) أكبر من (PZT).

الكلمات المفتاحية: سيراميك PZT ، طريقة الاوكزالات ، الخواص العزلية والميكانيكية ، الفيروكهربائية.

## Introduction

Lead Zirconate Titanate (PZT) Pb(Zr<sub>x</sub>Ti<sub>1-x</sub>)O<sub>3</sub>; and its related materials exhibit excellent piezoelectric properties. PZT has a perovskite structure ABO<sub>3</sub> which solid solution of ferroelectric PbTiO<sub>3</sub> and ant ferroelectric PbZrO<sub>3</sub> at different Zr/Ti ratios. Extraordinary high piezoelectric activities are found at the morphotropic phase boundary (MPB) composition. This MPB is located around PbTiO<sub>3</sub>:PbZrO<sub>3</sub> ≈1:1(1,2,3). The excellent piezoelectric properties of PZT ceramic can be applied to many areas, thus it's used in the applications such as sensors, transducers, multilayer ceramic capacitors, actuators and RAM (4,5).

Since piezoelectric ceramic based on PZT have poor mechanical properties such as low bending strength and low fracture toughness. Thus in some applications at high power and high stress; mechanical properties of this material become critically important (6,9).

There are many methods for preparation of PZT ceramic represented solid state reaction and wet chemical methods. Conventional ceramic method is most widely used process for the preparation of multicomponent solid. While wet chemical synthesis is increasingly utilized for the synthesis of at various ceramic powders due to merits of fine particle size of the products, better homogeneity and lower processing temperatures in comparison to the conventional ceramic route. Several of wet chemical methods for the preparation of perovskite titanate ceramics including the hydrothermal synthesis, sol gel processing and oxalate method (6,7).

The improving to the mechanical properties of PZT ceramic by adding Al<sub>2</sub>O<sub>3</sub> become appropriate to the applications with high power and high stress. The improved mechanical

properties are proposed to be due to Al $_2$ O $_3$  nanoparticles reinforcing the grain boundaries and acting as effective pins against microcrack propagation (8,9).

In the present study the fabricated of PZT/ $x$ Al $_2$ O $_3$  composite using Al $_2$ O $_3$  doped PZT powder is prepared and the effects of Al $_2$ O $_3$  addition on the mechanical and physical properties of the PZT/ $x$ Al $_2$ O $_3$  composites have been investigated.

### **Experimental procedure**

PZT/ $x$ Al $_2$ O $_3$  composites were fabricated from Al $_2$ O $_3$  which was blended with the calcined PZT powder. The starting PZT powder had a composition of Pb(Zr $_{0.5}$ Ti $_{0.5}$ )O $_3$  and the average particle size was (36nm). The starting materials used in this study were PbCl $_2$  (99.9% Merck), ZrO $_2$  (99.9% Merck), TiO $_2$  99.8% Riedel) and oxalic acid (C $_2$ H $_2$ O $_4$ ·2H $_2$ O) (B.D.H).

PZT powder which are prepared using Oxalate method which was represented by preparing aqueous solution of (PbCl $_2$ ). Then adding (ZrO $_2$  and TiO $_2$ ) powders to aqueous solution (PbCl $_2$ ), after this step has been added aqueous solution of oxalic acid to the mixture solution of oxides. Mixture above is known as precursor which was stirred and heated at 80°C till a clear viscous resin is obtained.

The precipitates are separated from the mother liquor by a filter paper for twice with deionized water; the washed precipitates were oven dried at 100 °C for 2h then calcined at 1000 °C for 2h.

The Al $_2$ O $_3$  powder was mixed with the calcined PZT powder in ratios of (0.5, 1.0, 1.5 vol. %). This mixture was grinding and calcined at 1000 °C for 2h. Then, the powder was pressed at 2500Kg/cm $_2$  into disks. These Disks were then sintered at 1100 °C for 2h. XRD was used to investigate any phase differences between the different sample types. A Vickers hardness (HV) value was measurement by using (XCHL-11A model multi-function, Hardness Tester).

Density of the sintered samples was measured by Archimedes method with distilled water as the fluid medium. The Dielectric strength for samples measured by using device (type of RSG, Germany) with applied voltages which rise to 2kv/sec.

### **Results and Discussion**

Fig. (1) show the X-Ray Diffraction Patterns to PZT sample which is prepared by Oxalate Method at sintered temperature 1100°C for 2h. The sample prepared exhibits a pure phase perovskite having tetragonal symmetry and the tetragonality (c/a) ratio is (1.029).

At sintered temperature 1100 °C for 2h, the pure phase and lattice parameter of PZT which agree well with Joint Committee on Powder Diffraction Standard data (JCPDS)(pdf#33-0784). The grain size PZT ceramics is of 36nm.

A mechanical property of PZT/ $x$ Al $_2$ O $_3$  ceramics in terms as Vickers Hardness (HV) was investigated. Vickers Hardness as a function of Al $_2$ O $_3$  content of PZT/ $x$ Al $_2$ O $_3$  ceramics is shown in Fig.(2). It can be seen from Fig.2 that the PZT/ $x$ Al $_2$ O $_3$  samples displayed high hardness than the pure PZT. So that suitable content of Al $_2$ O $_3$  could effectively improve mechanical properties of PZT ceramics. Therefore doped Al $_2$ O $_3$  into PZT; slightly increased the Vickers Hardness value. The maximum hardness value was found for the samples containing 1 vol. %Al $_2$ O $_3$  while this percentage is higher than the hardness suffer a decrease.

Fig. (3) Show that incorporating values of the bulk density of PZT/ $x$ Al $_2$ O $_3$ , that the density was lowered by doped Al $_2$ O $_3$  into PZT. The decrease of density with of Al $_2$ O $_3$  content indicates that, first; the sintering temperature was not enough. To obtain higher density the sample which include higher content of Al $_2$ O $_3$  required higher sintering temperatures. Second; it may be consider that the higher content of Al $_2$ O $_3$  in PZT samples causes the higher porosity; therefore the bulk density is decreased.

Apparent Porosity was measurement by Archamidic method. Fig.(4) shows the apparent porosity as a function of content Al $_2$ O $_3$  in PZT. The apparent porosity is increased with addition of Al $_2$ O $_3$  into PZT as show in Fig.4.

This increases due to addition of Al $_2$ O $_3$  which indicating that the Al $_2$ O $_3$  particles inhibited grain growth. Also indicates that the sintering temperature of the samples was not complete. Fig.(5), represents the behavior of apparent density with the addition of Al $_2$ O $_3$  to PZT

Fig. (6) The value of dielectric strength which was measurement at 2Kv/sec a raise in the applied voltage. The dielectric strength of PZT/ $x$ Al $_2$ O $_3$  increased with increased of Al $_2$ O $_3$  in PZT. Decreased in dielectric strength at sample containing 1.5vol%Al $_2$ O $_3$ , and the maximum value of dielectric strength of containing 1 vol.%Al $_2$ O $_3$ . This is indicating that the addition Al $_2$ O $_3$  into PZT leads to improve dielectric properties of PZT ceramics.

### Conclusions

The present work has shown that high mechanical properties PZT ceramic containing Al $_2$ O $_3$  Nano particle can be obtained by Al $_2$ O $_3$  doped PZT powders. The results produced the improvement in the mechanical properties. The maximum hardness value was obtained PZT with 1Vol. % Al $_2$ O $_3$ . The reduction of density for samples could be related to the lower sintering temperature. Finally, it is clearly shown that the mechanical properties of the ceramics PZT/ $x$ Al $_2$ O $_3$  are generally better than of pure PZT according Vickers Harness results.

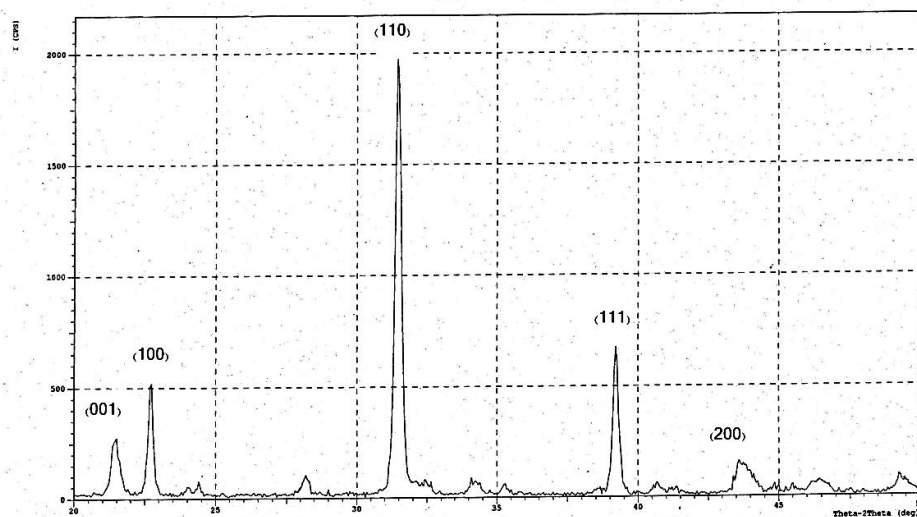
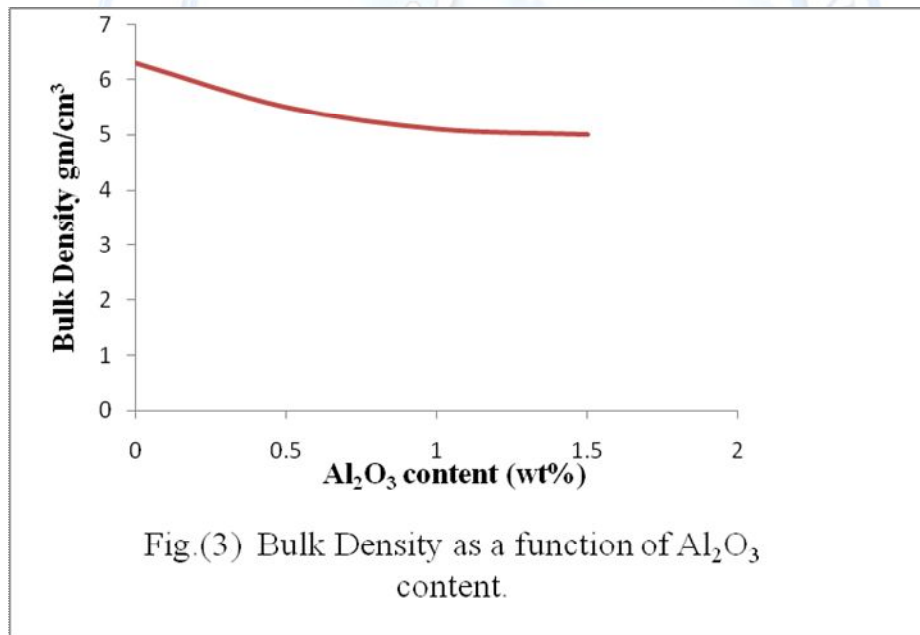
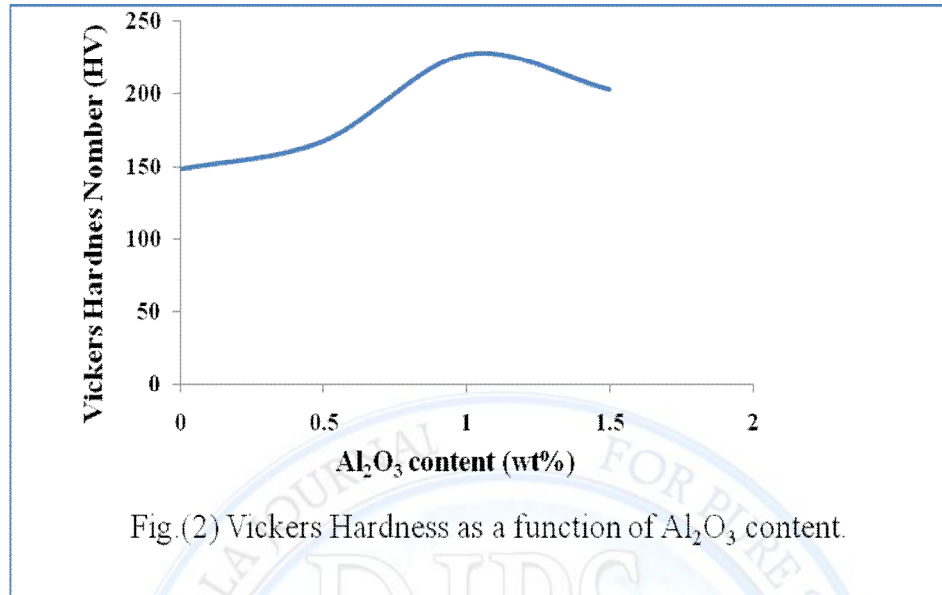


Fig. (1) XRD patterns of PZ $_{0.5}$ T $_{0.5}$  prepared at 1100°C for 2h.

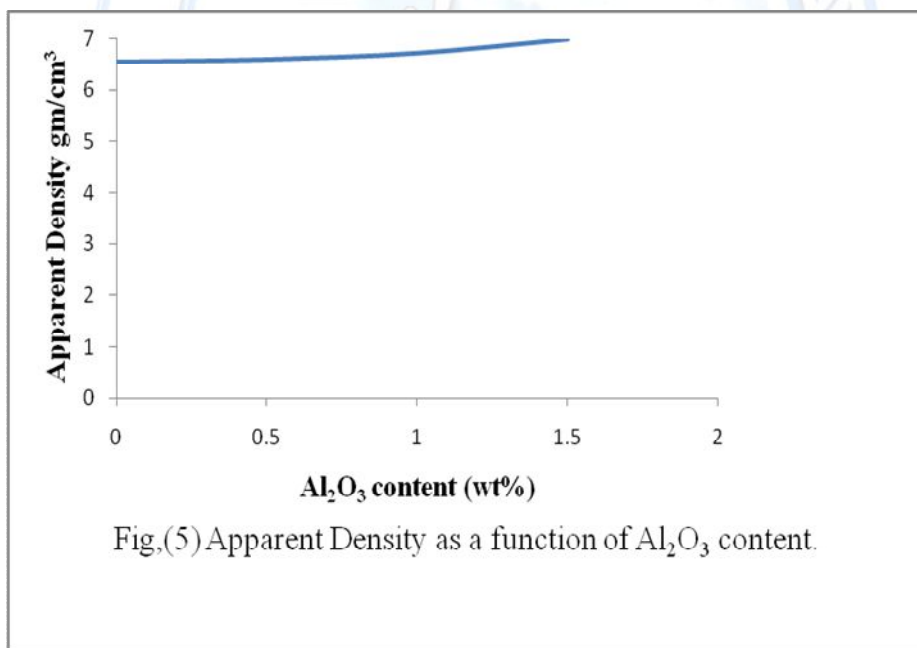
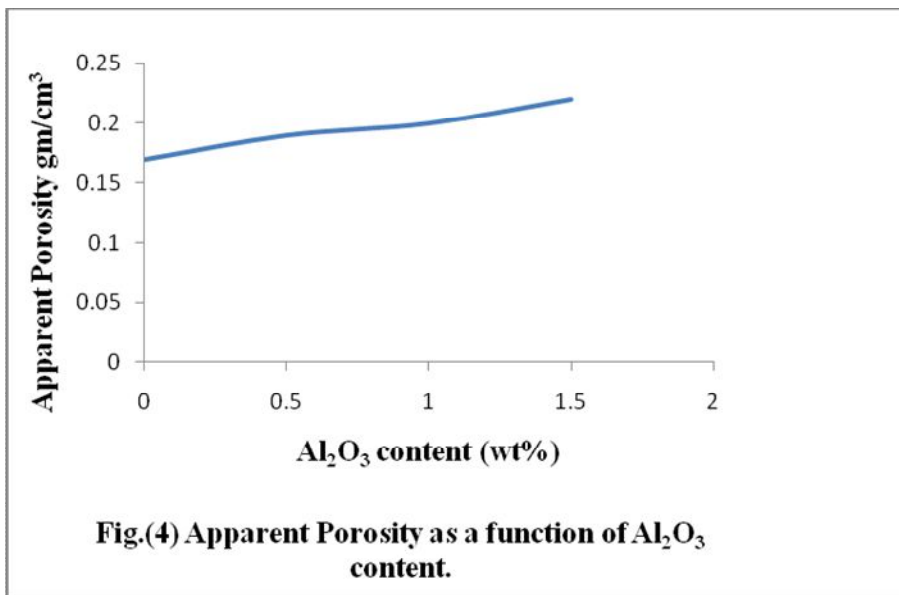
Mechanical and Physical Properties of PZT/xAl<sub>2</sub>O<sub>3</sub> Ceramics Systems

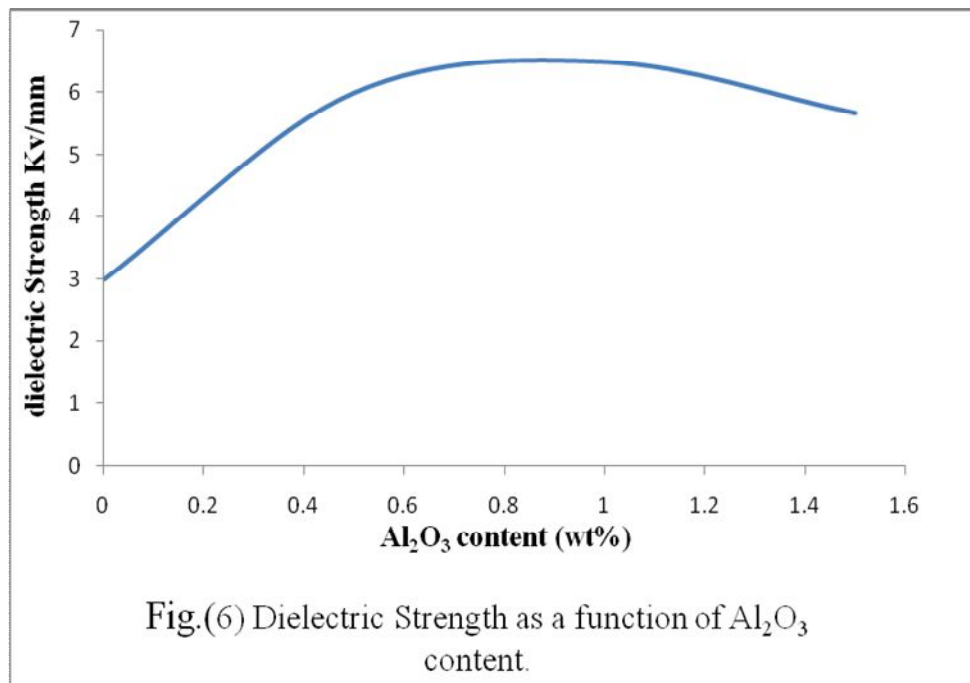
Abdulkarim Ziedan Khalf



Mechanical and Physical Properties of PZT/xAl<sub>2</sub>O<sub>3</sub> Ceramics Systems

Abdulkarim Ziedan Khalf





### References

1. B. Jaffe, W. R. Cook, H. Jaffe, Piezoelectric Ceramic Press, London, 1971.
2. P. Jaita, A. Watcharapasorn and S. Jiansirisomboon "Effect of BNT on Microstructure and Mechanical Properties of PZT Ceramics" Journal of the Microscopy Society of Thailand 24(1), 21-24(2010).
3. P. Xiang, X. Dong, C. Feng, N. Zhong and J. Guo " Sintering behavior, mechanical and electrical properties of lead zirconate titanate/Nio composites from coated powders" ceramics International 30 (2004) 765-772.
4. C. Puchmark and G. Rujijanagul "Microstructure and Mechanical properties of PZT Adding with Nano-particle Impurity" proceeding of the 27<sup>th</sup> MST Annual Conference 20-22 January 2010Samui, Thailand.
5. A. Chin, S. Member, M. Yang, C. L. Sun and S. Y. Chen "Stack Gate PZT/Al $_2$ O $_3$  one Transistor Ferroelectric Memory" IEEE Electron Device Letters, Vol. 22, No.7, JULY 2001.



6. S. Kumar "Chemical Characterizations of the surfaces and Interfaces of Barium Titanate and related electronic Ceramics" PH.D. Thesis, Materials research Center Indian institute of science Bangalore, India, January, 2006.
7. S. phanichphant and R. Heimann " Hydrothermal synthesis of submicron to Nano sized ferroelectric powders, properties and characterization" CMU-Journal, vol. 3(2), pp. 113, 2004.
8. U. Taffner, V. Carle and U. Schafer "Preparation and Microstructural Analysis of High-Performance Ceramics" ASM Handbook Volume 9: Metallography and Microstructures 2004 ASM International.
9. M. Sternmitzke "Structural Ceramic Nanocomposites" J. Eur. Ceram. Soc., 17, 1061-1182(1997).

