Advances in Materials
2014; 3(3): 11-15
Published online August 20, 2014 (http://www.sciencepublishinggroup.com/j/am)
doi: 10.11648/j.am.20140303.11
ISSN: 2327-2503 (Print); ISSN: 2327-252X (Online)

Synthesis and electrical characterization of a PLZT piezoelectric-ceramic

José Guadalupe Miranda-Hernández[1], Ernesto Suaste-Gómez[2], Carlos Omar González-Morán[1], Héctor Herrera-Hernández[1], Enrique Rocha-Rangel[3, *]

[1]Universidad Autónoma del Estado de México (CU-UAEM-Valle de México), Industrial Engineering Department, Atizapán de Zaragoza, México
[2]Centro de Investigación y de Estudios Avanzados (CINVESTAV-IPN), Electrical Engineering Department, México, D. F., México
[3]Universidad Politécnica de Victoria (UPV), Research Department, Cd. Victoria, Tamaulipas, México

Email address:
erochar@upv.edu.mx (E. R. Rangel)

To cite this article:
José Guadalupe Miranda-Hernández, Ernesto Suaste-Gómez, Carlos Omar González-Morán, Héctor Herrera-Hernández, Enrique Rocha-Rangel. Synthesis and Electrical Characterization of a PLZT Piezoelectric-Ceramic. *Advances in Materials.* Vol. 3, No. 3, 2014, pp. 11-15. doi: 10.11648/j.am.20140303.11

Abstract: This research describes the synthesis of an outstanding ceramic-composite piezoelectric CCP (lead-lanthanum-zirconate-titanate, PLZT) by means of powders technique procedures. Full dense CCP compacts were obtained with a platinum wire implanted in the center of the piezoelectric, which were used to investigate the microstructural and opto-thermal properties. The microstructural details of this ceramic were investigated by optical microscopy; whereas the opto-thermal characterization was performed by measuring the electrical signal in a bidimensional setup under four different temperatures: 20°C, 35°C, 50°C and 75°C. A 160mW/cm2 LASER beam was used in order to produce the optical energy which is detected by the CCP. A total of one hundred of measurements were registered. Measurements showed that, in the explored thermal range, the CCP signal magnitude increased from 87.2 to 147.2 pA. About the microstructural analysis, the microstructure obtained show different phases as characteristic of the processing method, including porosity. The peculiar optical and thermal properties observed in the piezoelectric ceramic are promising for possible applications in temperature-controlled optical devices that require electrical outputs.

Keywords: Piezoelectric-Ceramic, PLZT, Opto-Thermal, Pt Wire Implanted

1. Introduction

Today, there is a growing demand in the manufacture of structural materials, in which these properties become very important for applications in the aerospace, medical and mechanical industries [1,2], but the importance is not only in structural conditions, but also in their electrical response. Research of electric phenomena in materials is because it has been found that the microstructure affects electrical resistance and electrical conductivity [3]. In this sense there are several kinds of ferroelectric materials that exhibit photovoltaic effects under near-ultraviolet light and can be observed in all pyroelectric crystals or in polarized ferroelectrics ceramics under uniform illumination in the absence of an external field. When the material is illuminated after poling, voltage and current can begenerated due to the separation of photo induced electron and holes. It means that the photovoltaic effect is characterized by the production of steady-state short-circuit current and open-circuit voltage, whose values can exceed the band gap of the material by several orders of magnitude. This is considered an optical property of the material itself which has potential applications for supplying energy transfer in micro electromechanical systems and optoelectronic devices [4-6]. Therefore, photocurrent is a very important parameter for optical detection [7]. The steady current in the absence of applied voltage, called photocurrent, is considered the result of photo carriers and the asymmetric electromotive force induced by near-ultraviolet radiation [8,9]. However, this photo-ferroelectric effect spans to a large wavelength range which overlaps the visible spectrum. The high chemical stability of many ferroelectric oxides coupled with a photo-response in the visible range increased interest either in biological and space environment application [6]. The behavior of the photovoltaic effect in ferroelectrics is similar to the one in semiconductor p-n junctions. There are three types of photovoltaic samples, namely bulk single plate, bulk bimorph and film.

There are many types of ferroelectric materials that exhibit photovoltaic effects when they are stimulated with light at near ultraviolet. When a material is illuminated after its polarization, it can generate voltage and current due to separation of electrons and holes by a photo-induced internal electric field. This is how the photovoltaic phenomenon in ferroelectrics exhibits several potential applications for transfer energy micro electromechanical systems and optoelectronic devices [10]. Therefore, the photocurrent is an important parameter for optical detection [11]. The steady current in the absence of an applied voltage is considered as a result of the photo-induced carriers and asymmetrical electromotive force due to radiation from the near ultraviolet [12, 13]. The increase photovoltaic current measured in a sample with poor composition originating from the basic mechanism of the photovoltaic effect [14]

Recently, a new lead-lanthanum-zirconate-titanate (PLZT) bulk single plate, called ceramic-controlled piezoelectric with one Pt wire (CCP) has been produced; this CCP has a Pt-wire implant (300 µm of diameter and 1 cm long) in order to form free upper and lower faces which measure electric events as shown in Fig. 1.

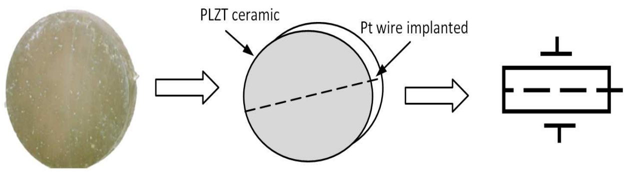

Figure 1. *CCP and its electrical symbol.*

The sample preparation of this CCP is described in the following section. The Pt-wire was chosen as an implant because it possesses high resistance to chemical attack, it has excellent high-temperature characteristics (melting point 1,768.3 °C), and it has stable electrical properties and thermal conductivity with small variations. When CCP is polarized, the mobile charges in the Pt-wire accumulate on its surface until the resulting field completely cancels the external field applied to the conductor. This results in electrical equilibrium within the Pt-wire; thereby, the internal electrical field is zero, $\varepsilon = 0$ [8].

Until now, there are no reports about the CCP reaction to combined stimuli of light and temperature. In this work, it was found that CCP reacts efficiently to this kind of stimuli.

2. Experimental Procedure

Piezoelectric-Ceramic (ferroelectric): The bases of these materials are; Lead oxides and Zirconate (> 99 % purity, Sigma-Aldrich) and Titanate (PZT), (> 99.9 % purity, Johnson Matthey), when the material's matrix is modified with Lanthanum (> 99 %, Rare earth metals and compounds) it is known as PLZT.

Ferroelectric: Lead Lanthanum Zirconate Titanate (PLZT) is a $Pb_{1-x}La_x(Zr_{1-y}Ti_y)_{1-x/4}O_3$ ceramic with x = 0.09 and y = 0.35, generally denoted as (9/65/35) with a finally composition of PbO 62.7 wt %, La_2O_3 4.5 wt%, ZrO_2 24.2 wt % and TiO_2 8.6 wt % obtained by chemical equilibrium. This ceramic was produced by powders technique through an oxide-mixing: raw materials were mixed by mill of high energy type planetary (Pulverisette 2, Fritsch) for 20 min in polyvinyl container, alcohol drops were added with a rate of 1.5 drops per gram of mixture. 1.5 grams of the milled powder mixture was pressed at 3,500 Kg/cm^2 by uniaxial pressing forming discs of 10 mm diameter and 2 mm of thickness. During this process, a Pt-wire of 0.3 mm diameter and 1 cm length was implanted longitudinally in the middle of each ceramic. These pieces were sintered in air, in a platinum crucible, with a heating rate of 5 °C/min from room temperature to 600 °C and a second heating rate of 10 °C/min from 600 °C to 1,200 °C. Then, the ceramic was kept at 1,200 °C for one hour. After sintered, silver electrodes on the lower face and the Pt-wire of the CCP were placed. After powder processing technique, the ceramic was electrically poled, at 1.5 kV/mm for one hour at 60 °C in a silicone oil bath in order to be used as light sensors [15]. The Archimedes' principle was used to measure density of the sintered samples. Finally, the fracture toughness was estimated by the indentation fracture method, using a Vickers microhardness tester (Wilson Instruments, S400) and applying Evans and Charles equation [16].

3. Result and Discussion

3.1. Microstructure

Through powders technique they were fabricated piezo electric-ceramics, this technique allows to obtain a homogeneous powder mixture and involves a process of consolidation in solid state that depends of the load of the compaction and densification of sintered powder; it is, by the importance in the control of the microstructure. This material are summited a taste of hardness, the hardness determinate is 592 HV. The high value of the hardness is due to the good sintered process, because final density of the material was > 98%. The Fig. 2 parts a), b) and c), show a characteristic microstructure of this materials taken by optival microscopy (OM), in these pictures it is notable the phases presents in the microstructure considering that the raw material are only single metal oxides, therefore there are not chemical reaction between these materials. The PLZT ferroelectric microstructure show a homogeneous distribution of different materials where the white phase is present in higher proportion corresponding to the matrix material, in this case to PbO and maybe the gray phase corresponds to ZrO_2, this is not confirm by optics microscopy, but started of the condition that the material with major composition are PbO and ZrO_2, and that all materials used in the manufacture of these materials are oxides. In the figure 2 a) is appreciated the white phase and porosity but in the figure 2 b) is more notable this phase; it is, by the pictures was taken a bigger magnifications. It is well known, that the presence of pores affects the microstructure and consequently the electrical properties. Particularly the porosity directly affect in the electric response due to pores are obstacles in the electrical continuity [2].

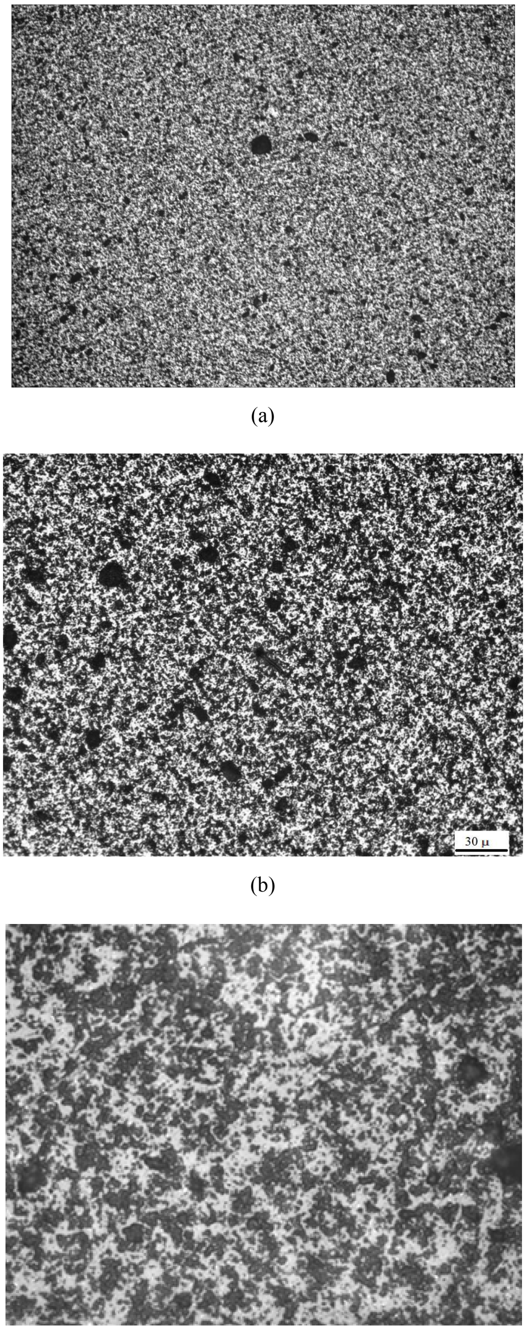

Figure 2. *Optical microstructure of PLZT part a) 10X, part b) 20X and part c) 100X.*

3.2. Opto-Thermal Characterization

The experimental Bi-dimensional setup used to obtain graphics from CCP is shown in Fig. 3. It was placed on a vibration isolation optic table in order to prevent voltage variations; the system was placed into a closed dark enclosure in order to avoid air currents and undesirable light.

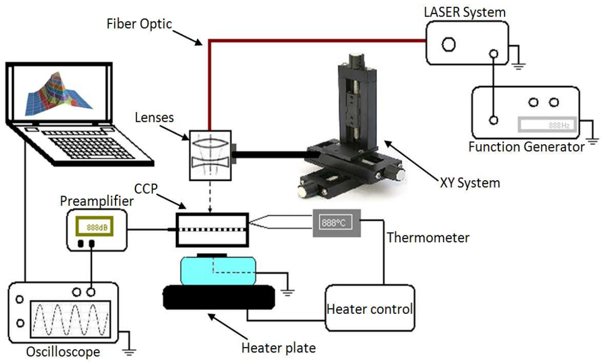

Figure 3. *Bi-dimensional setup to obtain an opto-thermal characterization of CCP varying its temperature.*

The LASER used was a 650 nm (light system BWF1) which has a coupled fiber optic and was modulated through a function generator with an output of 700 mHz of frequency and a sinus waveform. The objective lenses reduced the LASER beam spot to a 1 mm of diameter size. The objective lenses and the fiber optic were coupled with an X-Y translation microscope stage in order to move the LASER beam and to get different excitation dots on the CCP. Radiation of the light was 160 mW/cm^2.

The CCP was placed on a controlled heater plate, and a digital thermometer was added in order to measure the CCP temperature. The electrical contacts from CCP were connected to a preamplifier (SRS-Low Current Noise Preamplifier SR570) in order to scan it; these signals records were registered by an oscilloscope (Agilent DSO3062A) which measured its peak to peak voltage. At the same time the oscilloscope was interfaced to a Lap Top (Sony VAIO) computer in order to construct a 3-D graphic.

With this system, it is possible to get three axes, X and Y, which are the surface of the ceramic, and axis Z which represents the peak to peak voltage. Due to the beam of light thickness, the analysis of CCP was done in steps of 1 mm precise per axis; it means 10 mm in X axis and 10 mm in Y, with a total of 100 points recording.

In the bidimensional setup, a 2-D scan response was obtained; the records were from 0 to 9 in axis “x” and axis “y”. This graphic shows a great increment in the Pt-wire zone because the ferroelectric domains are very close to each other. The maximum photovoltaic current response was 147.2 pA at 160 mW/cm^2 of illumination. Fig. 4 to 7 show the magnitude of the photovoltaic current of the CCP registered when temperature changed from 20°C to 75°C. These results demonstrate that this CCP allowed to detect low temperature changes thus generated 3-D graphics. Quantitative sensitivity comparison with other types of opto-thermal devices would be relative due to the chemical compositions of each sensor; finally whatever the type of sensor, they only give a percentage measure parameter.

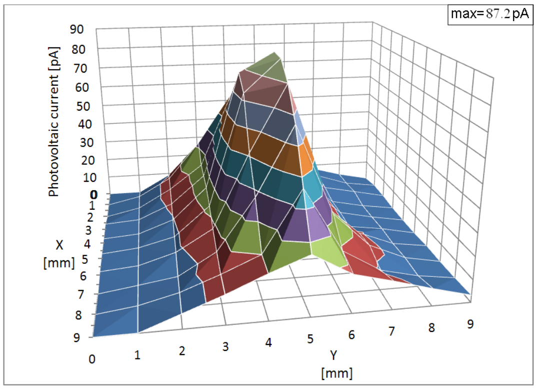

Figure 4. *CCP scan at 20°C and 160 mW/cm² of illumination.*

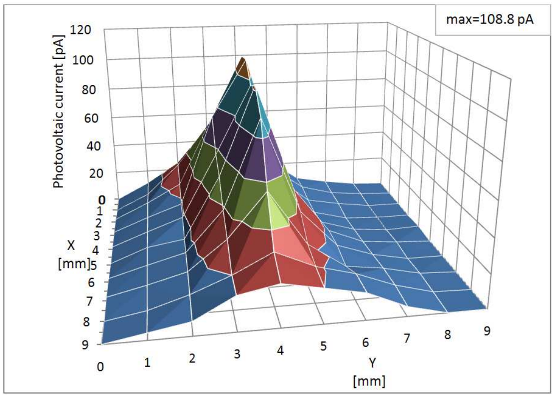

Figure 5. *CCP scan at 35°C and 160 mW/cm² of illumination.*

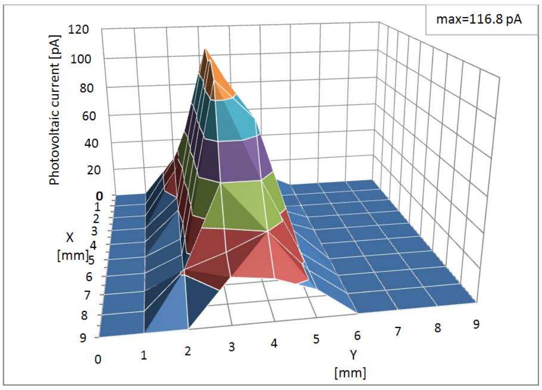

Figure 6. *CCP scan at 50°C and 160 mW/cm² of illumination.*

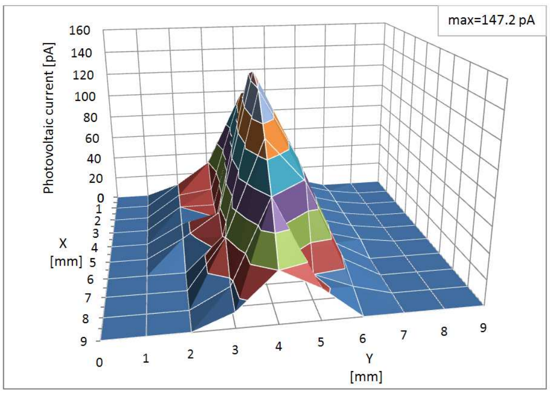

Figure 7. *CCP scan at 75°C and 160 mW/cm² of illumination.*

3.3. Fracture Toughness

The Fig. 8. Presents an indentation mark used for determining the fracture toughness of the piezoelectric through cracks growing in the edges of the mark and using the equation proposes by Evans [11]. In fig. 8 it is observed that the growth of the crack follows a linear path which apparently spreads easily based on its size. The fracture toughness of the piezoelectric was of 2.33 $MPam^{0.5}$, value that corresponds to fragile material, typical of ceramic.

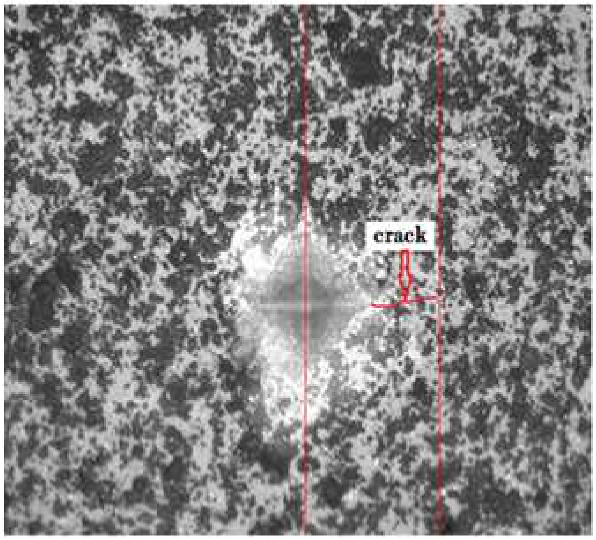

Figure 8. *Crack growing used for determining the fracture toughness of the piezoelectric ceramic.*

4. Conclusion

Ceramic-piezoelectric was fabricated satisfactory by powders technique.

The microstructure of this material show the phases present. The refined and homogeneous incorporation of Lanthanum in a matrix (PZT) improves in electrical properties.

Dependence of temperature in CCP was investigated through bi-dimensional experimental setup. In the present work, photovoltaic current signal increased drastically with temperature variation. Due to its pyroelectricity and Pt implant, CCP can be useful in possible applications as temperature-controlled optical devices that require electrical outputs [17].

It was shown that the CCP described in this work has the following advantages: The increase in surface analysis is superior because of the Pt-wire works as a third electrode. Furthermore, this CCP can be used at much higher temperatures and higher illumination intensity as compared to conventional Si-based. In general, this ceramic offer good versatility as a opto-thermal device due to its ferroelectricity and can have novel applications such as opto-thermal switches associated at opacity sensors [18] applied to: gas emission control, polymer manufacture, friction sensors, food control, body sensor and others.

Acknowledgements

Authors wish to thank to the processing laboratories of CINVESTAV-IPN, UAEM-VM and UPV. In addition, JGMH are grateful to PROMEP by the support given to carry out this work through Project 103.5/13/6535.

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