

# Characterization of smart-materials based on lead lanthanum zirconate titanate

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This paper deals with the characterization of smart-material combinations based on the principle of domain polarization; that provides change in voltage with pressure. Different binary, ternary and quaternary material combinations have been studied from the point of view of phases present, Curie temperature and their ferroelectric nature. Lead lanthanum zirconate titanate (PLZT) material based devices were fabricated and characterized. A smooth change in output voltage with respect to small change in pressure has been observed and discussed. The reliability tests on the devices have been performed to examine their long-term stability.

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## 1. Introduction

This paper presents the efforts made in the development of smart materials based on domain polarization that may provide sensing action, i.e. a change in physical parameter like pressure may offer a variation in electrical parameter like voltage. This type of sensing action may find wide applications in different types of transducers and actuators.

Different material combinations have been studied. Some of the materials may exhibit good piezoelectric response after domain polarization and may be useful for above stated applications, while few of them may provide phase transition with change in pressure and may find applications in pressure indicators, pressure monitors etc. Doping of minor quantities of impurities may improve the properties of these materials; make variation in phase transition and thus make them suitable for typical applications [1-2]. These materials include the following:

barium titanate ( $\text{BaTiO}_3$ ), lead titanate ( $\text{PbTiO}_3$ ), lead zirconate ( $\text{PbZrO}_3$ ), PZT [ $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ ], PZT–PMS–PZN [ $0.9\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3-(0.1-x)\text{Pb}(\text{Mn}_{1/3}\text{Sb}_{2/3})\text{O}_3-x\text{Pb}(\text{Zr}_{1/3}\text{Nb}_{2/3})\text{O}_3$ ], PZT–PFW–PMN [ $0.9\text{Pb}(\text{ZrTi})\text{O}_3-0.03\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.07\text{Pb}(\text{Mn}_{1/3}\text{Nb}_{2/3})\text{O}_3$ ], PMN–PZN–PT–PZ [ $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3-\text{Pb}(\text{Zr}_{1/3}\text{Nb}_{2/3})\text{O}_3-\text{PbTiO}_3-\text{PbZrO}_3$ ], sodium niobate ( $\text{NaNbO}_3$ ), potassium niobate ( $\text{KNbO}_3$ ), lithium niobate ( $\text{LiNbO}_3$ ), potassium sodium niobate ( $\text{K}_x\text{Na}_{1-x}\text{NbO}_3$ ), lithium sodium niobate ( $\text{Li}_x\text{Na}_{1-x}\text{NbO}_3$ ), lead free piezoelectrics like KNN–LT–LS [ $(\text{K}_{0.5}\text{Na}_{0.5})\text{NbO}_3-\text{LiTaO}_3-\text{LiSbO}_3$ ]. Some properties of these binary, ternary and quaternary material combinations [3-13] are summarized in the Table 1. Most of these materials can be used for sensing purpose after domain polarization. In addition to above, the materials in which more than one phase is present can be used for switching by phase transformation.

Table 1. Summary of some properties of piezoelectric materials.

Material combination	Curie Temperature ( $^{\circ}\text{C}$ ) $\approx$	Phases present	Nature
$\text{BaTiO}_3$	130	Tetragonal	Ferroelectric
$\text{PbTiO}_3$	500	Tetragonal	Ferroelectric
$\text{PbZrO}_3$	230	Orthorhombic	Antiferroelectric
PZT	360	Rhombohedral/ Tetragonal	Ferroelectric
PZT–PMS–PZN	275	Rhombohedral/ Tetragonal	Ferroelectric
PZT–PFW–PMN	273	*	Ferroelectric
PMN–PZN–PT–PZ	*	Tetragonal/ Pseudocubic	Ferroelectric
$\text{NaNbO}_3$	641	Orthorhombic	Antiferroelectric
$\text{KNbO}_3$	435	Tetragonal/ Orthorhombic/ Rhombohedral	Ferroelectric
$\text{LiNbO}_3$	1157	Rhombohedral	Ferroelectric
$(\text{K}_x\text{Na}_{1-x})\text{NbO}_3$	420	Tetragonal/ Orthorhombic	Ferroelectric
$\text{Li}_x\text{Na}_{1-x}\text{NbO}_3$	640	Rhombohedral/ Orthorhombic	Ferroelectric
KNN–LT–LS	253	Tetragonal/ Orthorhombic/ Rhombohedral	Ferroelectric

\* Data was not available

## 2. Experimental

In the experimental part PLZT material consisting of  $\text{PbO}$ ,  $\text{ZrO}_2$ ,  $\text{TiO}_2$  and  $\text{La}_2\text{O}_3$  was prepared and pressure sensitive devices were fabricated broadly by palletising, electroding and electrical polarization. Some of the fabricated samples are shown in Fig. 1. A characterization set-up has been prepared and the devices have been characterised using the same.

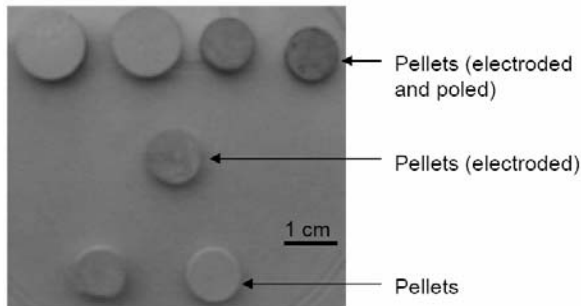


Fig. 1. Pressure-sensitive devices.

In order to perform the characterization of the pressure sensitive devices, two different methods were followed. The first way was to test the devices under dynamic pressure for which an in-house characterization set-up was prepared (see Fig. 2). It essentially consists of a pressure delivery unit with a pressure regulator and a meter, a probing station and a measurement and display part. It also consists of a two-stage control circuitry consisting of an amplifier unit and a set of comparators with LED indication to confirm the change in electrical properties leading to sensing action of the device with change in pressure. The detailed circuitry of this set-up is out of scope of this paper and will be presented separately.

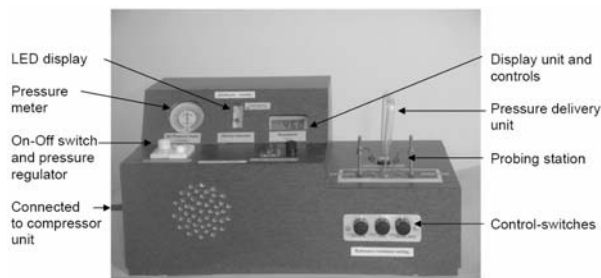


Fig. 2. Characterization set-up for testing the devices under dynamic pressure.

The other way, which was followed to check the device performance, was under static pressure. For this, commercially available disposable plastic injection modules were utilised and after placing the device inside the plastic enclosure, the connections are brought out and the exit points were properly sealed. The devices are then tested with change in pressure.

## 3. Results

### 3.1. Electrical characterization

The samples are subjected to different pressures and the voltage is measured in each case. The initial voltage across the samples is around 2 mV. The devices have been tested both under dynamic and static pressure in the range of 0 to 2 MPa (see Fig. 3 (a) and (b)). In both the cases, sensing action with pressure is observed. In case of dynamic pressure the switching is slow as compared to switching of the samples tested under static pressure. The reason being, that the effective pressure on the device is low during dynamic testing in comparison to static testing due to pressure losses involved. In each graph, one curve indicates the variation in voltage with the increase in pressure while the other is for the decrease in pressure and the variation approximately follows the same path in both the cases.

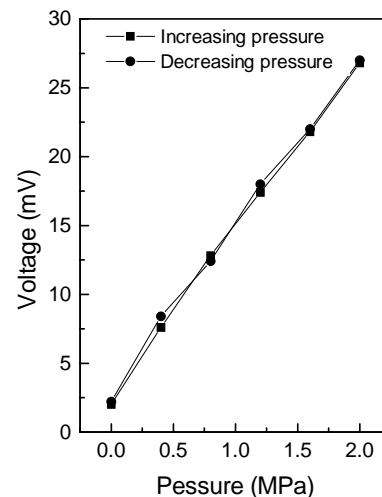
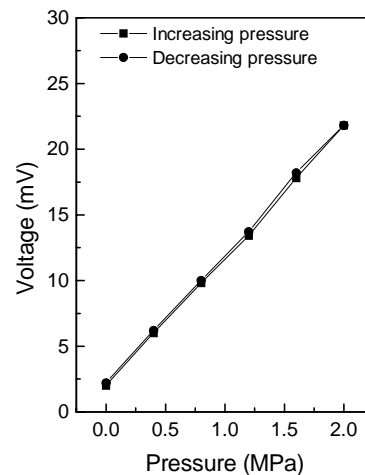


Fig. 3. Variation in voltage with change in pressure for the pressure-sensitive device fabricated on the principle of domain polarization tested under dynamic pressure (a) and static pressure (b).

The voltage varies linearly with pressure and it can be attributed to the piezoelectric nature of the resultant PLZT material formed in the process, which provides voltage output with pressure. The process of electrical poling aligns the dipoles in the Weiss domains (see Fig. 4), thereby increasing the net sensitivity of the material. The linear output voltage with change in pressure can be used for most of the pressure-sensing and control applications. No separate calculation for material constituents for low- and high-pressure applications is required in this case, thus making the device more versatile.

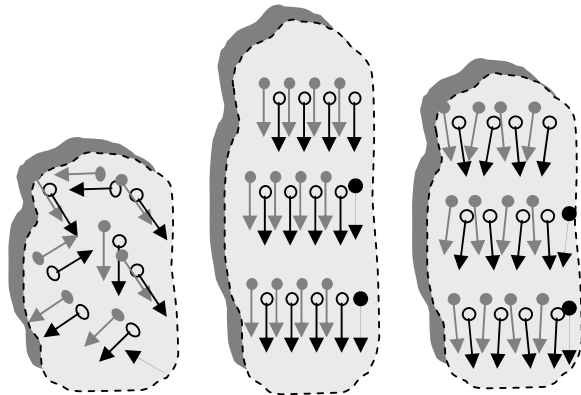


Fig. 4. (a) Alignment of dipoles in Weiss domain before (b), during (c) and after (d) electrical poling in the pressure-sensitive devices based on domain polarization.

### 3.2. Reliability test on pressure-sensitive devices

The reliability of these devices is an important factor to make them usable. The pressure-sensitive devices have been kept in an oven for 1000 h at 100 °C and the values have been recorded at fixed intervals. No significant change in the output voltage of the devices after 1000 h have been observed (see Fig. 5) confirming that the domain polarization is more a temperature-independent technique. These tests establish the long-term stability of these devices and also confirm the temperature insensitiveness of these devices even after long-term exposure at elevated temperatures.

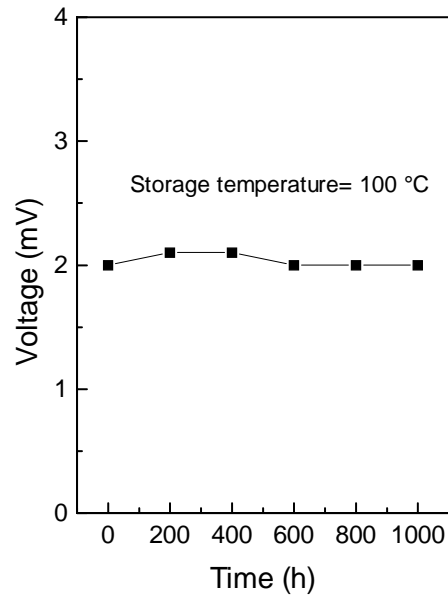


Fig. 5. Variation in initial voltage after storage at 100 °C for 1000 h for pressure-sensitive devices based on domain polarization.

### 4. Conclusions

- Efforts made in the direction of characterization of pressure-sensitive smart-materials based on domain polarization have been presented and discussed.
- A smooth variation in output voltage with respect to small change in pressure has been observed.
- A hysteresis free response is observed.
- Set-ups for testing the devices under dynamic and static pressures were prepared and the results were confirmed on the same.
- A physical explanation about the devices based on domain polarization has been presented.
- The effectiveness of domain polarization for switching action with respect to pressure has been discussed.
- The accelerated life tests on the devices have also been performed to establish their reliability and long-term usage possibility.

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