

# THERMAL SHOCKING OF A THIN LEAD ZIRCONATE TITANATE PIEZOELECTRIC CERAMICS DISC

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**Abstract:** Fatigue studies showed that a material degrades due to change in temperature. Thermal shocking is one of the most severe phenomenons in degradation of piezoelectric ceramics. A lead zirconate titanate thin piezoelectric ceramics disc has been experimentally analyzed to observe its thermal shocking effects. Disc was shocked from 100°C from a thermal chamber environment to de-ionized water at 20°C. Dielectric constant and coupling factors at frequency of maximum and minimum impedance have been measured for thirty five shocks. A noticeable change in values has been observed by using reliable instrumentations and appropriate equations. The study will be useful in modeling, development and the reliability of sensitive piezoelectric instruments facing such conditions.

**Keywords:** Piezoelectric, Lead zirconate titanate, thermal shock, dielectric constant, impedance, frequency

## Introduction

Lead Zirconate Titanate (PZT), Barium Titanate ( $\text{BaTiO}_3$ ), and Lead Metaniobate ( $\text{PbNb}_2\text{O}_6$ ) are generally favored as smart sensing materials. These materials are being used in critical engineering systems and smart structures. Fatigue failure due to electrical and thermal shocking is a major cause in degradation of these materials. Piezoelectric material after poling and due to cyclic stress undergoes degradation of its properties [1]. Sensors and accelerometers in aerospace industry are designed to withstand the high pressure and temperature changes during take-off and flight. Temperature changes in underwater applications as sonar may attain upto 100 °C and high thermal shock resistance is required in this case. Heat shocks cause the resonant frequency to change, thus a degradation of the piezoelectric properties may occur [2].

Fracture studies have been conducted for degradation of permittivity with time at room

and elevated temperatures. Degradation of piezoelectric properties with cyclic loading after poling and its fatigue behavior with or without poling has been studied [3]. Heat transfer effect in ferroelectric materials, electric impact loading, thermal effects of piezoelectric sensors and heat generation rate in piezoelectric materials have also been investigated [4]. Thermal shocks in a plate of finite thickness have been attempted. Thermal shock and thermal fatigue of ferroelectric thin film were investigated by the pulsed laser tests by Zheng *et al* [5]. Fatigue studies show that material degradation of PZT ceramics is strongly influenced by temperature and by the electromechanical fatigue. Lead zirconate titanate ceramics shows a decrease in dielectric constant and the resonance frequency when subjected to thermal shock. Importance of temperature stability for dielectric constants and resonance frequencies have been discussed by Lee and Kim [6]. Thermal shock resistance of the materials was evaluated by water quenching and a subsequent three point bending test to determine flexure strength degradation. Degradation of various properties of the piezo devices in the presence of water and AC voltage

has been investigated by Xiang *et al* [7] with the conclusion that water is an important cause for the degradation of PZT piezoelectric ceramics.

Dielectric constant is an important parameter, especially in the piezoelectric devices such as resonators and filters used in the electronic circuits. Impedance is also dependent on the dielectric constant of the piezoelectric. Currently, there are limited data available on the thermal shocking and quenching effect of a thin PZT disc. In the present research work, the focus was to investigate the degradation of thin PZT disc due to thermal shocking and its quenching effect in deionized water. A noticeable degradation in capacitance and dielectric constant has been observed which may further change other piezoelectric properties.

## Materials and Methods

### *Piezoelectric specimen*

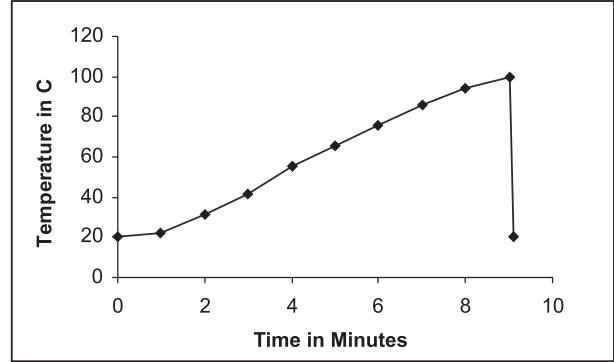
A Lead Zirconate Titanate piezoelectric disc 0.191mm thick and 12.7 mm diameter supplied by Piezo System Inc. was used for the experimentation. The specimen was nickel electroded on major faces, and its dielectric constant was about 1850 @1KHz, Curie temperature 350 °C, and density 7800 Kg/m<sup>3</sup>.

### *Test Setup and Variables*

There is no single technique for measuring piezoelectric material characteristics. However, some standards allow measuring some parameters and then using the appropriate relationships, other piezoelectric parameters can be found. A piezoelectric thin PZT disc was investigated under thermal shocking from 100 °C to deionized water at 20 °C in a thermal chamber to observe the degradation in its properties. Initially the capacitance and dissipation factor, of the as received specimen (without shocking) was measured. For reliability of the results, two discs were used at the same time for the shocking and measurements in the same environ-

mental condition. The temperature of the disc was measured by using a spring loaded thermocouple and digital data acquisition system directly attached to the specimen. The rate of change in temperature and shocking is shown in Fig.1.

The specimen was placed in the environmental chamber and heated to 100 °C and then



**Figure 1.** Rate of Change in Temperature and Shocking in De-ionized Water.

quenched in de-ionized water at about 20 °C for a specific time and then dried. The specimen was again placed in the thermal chamber for the same temperature range and quenched again. Five such shocks were introduced. The values of capacitance after these five shocks were measured at a frequency of 1KHz. Six series each with five shocks were performed and the respective capacitance values were measured. The data collected for 35 such shocks were analyzed for the degradation in dielectric constant and coupling factor. The frequencies of maximum and minimum impedance were observed between 100 KHz and 200 KHz. Capacitance at these particular frequencies was recorded (Table 1). The dielectric constant and coupling factors were measured by using the following equations [8,9].

$$K_3^T = \frac{t_a \times C_p}{A \times \epsilon_0} \quad (1)$$

$$K_{eff} = \text{SQRT} (f_n^2 - f_m^2) / f_n^2 \quad (2)$$

$$K_{31} = \text{SQRT} (\psi / (1 + \psi)) \quad (3)$$

**Table1.** Values of Capacitance ,Coupling Factors and Dielectric Constant at Frequency of Maximum ( $f_m$ ) and at Frequency of Minimum ( $f_n$ ) Impedance.

Shock#	$f_m$ Hz	$f_n$ Hz	$C_p@1KHz$ pF	$C_p$ at $f_m$ pF	$C_p$ at $f_n$ pF	$K_{eff}$	$K_{31}$	$K_3^T$ at $f_m$
0	160000	165500	10880	58040	-52967	0.255	0.279	9888
5	156785	165106	11100	55650	-53045	0.313	0.34	9481
10	151570	160270	11220	45350	-36430	0.324	0.352	7726
15	147075	154530	11330	37520	-22150	0.306	0.333	6392
20	142000	152500	11260	31060	-16741	0.364	0.393	5291
25	141000	156000	11280	28120	-11570	0.427	0.457	4790
30	123000	157000	11320	20250	-1140	0.621	0.644	3450
35	116500	153500	11320	17230	250.35	0.651	0.671	2935

where  $\Psi = \pi/2(f_n/f_m) \times \tan |\pi/2 \times (f_n - f_m)/f_m|$

$f_m$  = Frequency of maximum impedance [Hz]

$f_n$  = Frequency of minimum impedance [Hz]

$C_p$  = Equivalent parallel capacitance [F]

$t_a$  = Average thickness of testing material [m]

$A$  = Area of guarded electrode [m<sup>2</sup>]

$K_3^T$  = Dielectric constant

$K_{eff}$  = Effective coupling factor

$K_{31}$  = Coupling factor with transverse excitation

$\epsilon_0$  =  $8.854 \times 10^{-12}$

$\Psi$  = Phase Angle

The impedance and admittance of a piezoelectric ceramic element varies as a function of frequency. Impedance analysis allows various piezoelectric constants to be measured. The effective piezoelectric coupling coefficient,  $K_{eff}$ , is a comprehensive coefficient relating the dielectric, mechanical and piezoelectric constants of the material, and represents the effectiveness of the energy transfer. Higher the  $K_{eff}$  value, the higher is the effectiveness of converting mechanical energy into electrical energy [10].

Capacitance of the tested specimen was measured by using impedance analyzer and dielectric test fixture. Dielectric test fixture model

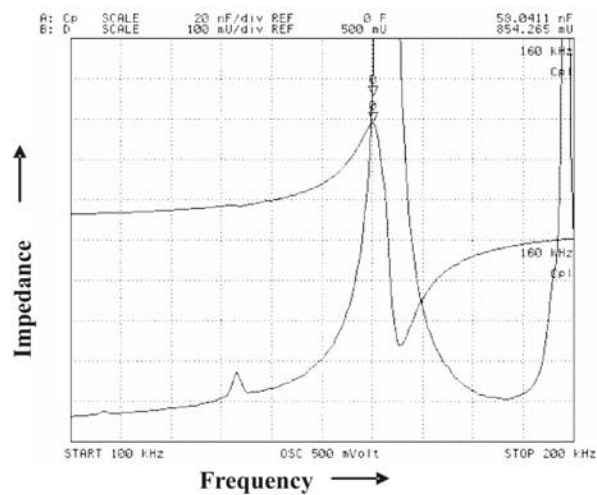
16451B was selected as fixture for the testing of the material. The fixture was attached with LCR meter and impedance analyzer 4294A, which uses the 4-terminal pair measurement configuration. Electrode-D of the test fixture was selected for measurements. This electrode is appropriate for measuring those materials which already have thin film electrodes. The values obtained were then used for the calculation of dielectric constant and coupling factors ( $K_{eff}$ , and  $K_{31}$ ).

## Results and Discussion

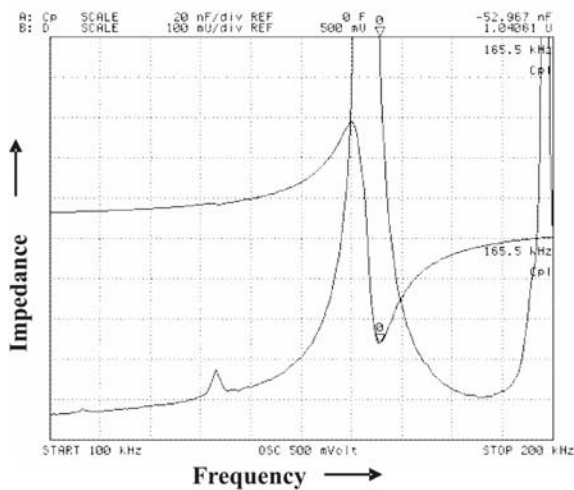
The measurement of capacitance and dielectric constant during thermal shocking was a challenging job. However, the latest impedance analyzers and compatible test fixtures have the capability to measure these parameters accurately. Thin disc shocked in de-ionized water showed a noticeable difference between shocked and unshocked conditions in its frequency of maximum impedance and frequency of minimum impedance. This difference has been attributed to change in dipole movement and direction. This change may definitely affect the grain size and the density of the specimen, but this aspect was not tested in this study.

The heating rate was observed to be approximately linear up to 100 °C as shown in Fig.1. The shocked material's capacitance values were

measured before testing and after every five shocks. Fig. 2 (a,b) indicates the capacitance values at frequency of maximum and minimum impedance before conduction of the test for which only one peak was observed, whereas a number of peaks were observed for the shocked material at the frequency of maximum and minimum impedance (Fig. 3a,b). The number of peaks shows the degradation and reorientation of dipole arrangement. The dissipation factor was also greatly affected by thermal shocking



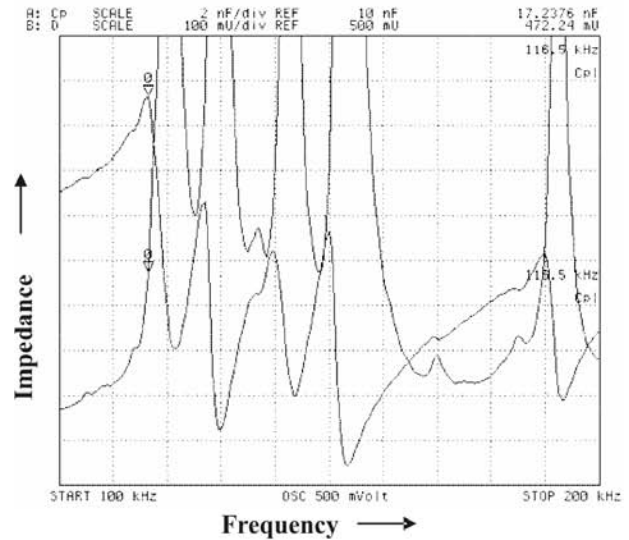
(a)



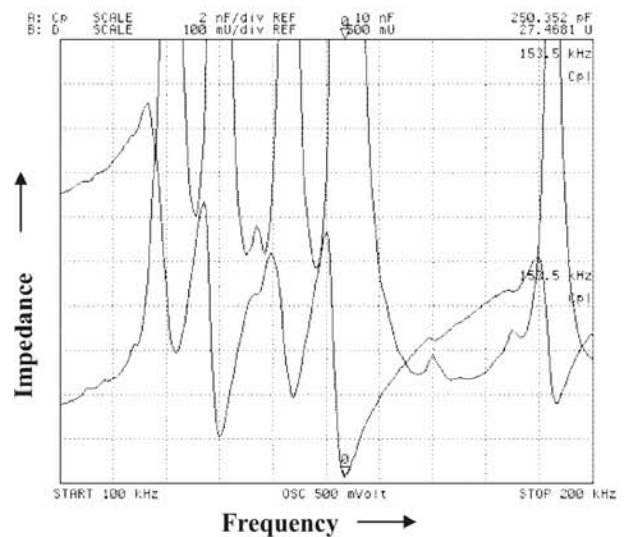
(b)

**Figure 2.** Value of Capacitance for Un-shocked Disc w.r.t Frequency (100KHz - 200KHz). (a) Capacitance in nF at Frequency of Maximum Impedance, (b) Capacitance in nF at Frequency of Minimum Impedance

conditions as indicated in Figs. 2 and 3. The value of capacitance at the frequency of maximum and minimum impedance showed interesting results. The frequency of maximum impedance observed for the unshocked specimen was 160 KHz, whereas after thirty five shocks it was about 116.5 KHz. This is approximately 28% less than the value obtained for the unshocked specimen.



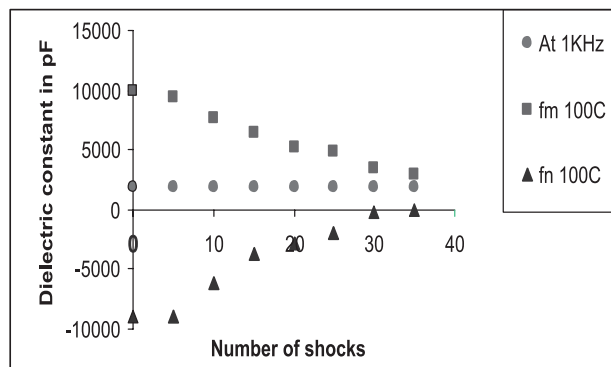
(a)



(b)

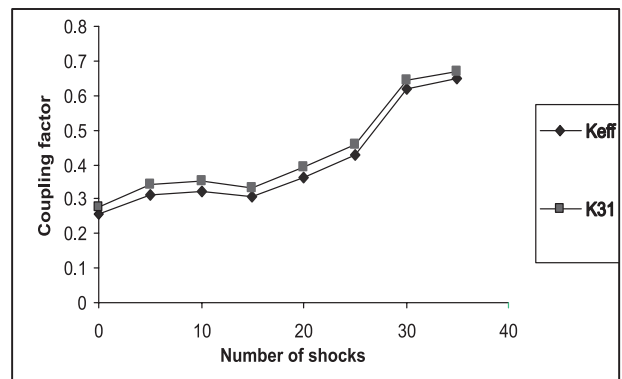
**Figure 3.** Value of Capacitance After Thirty Five Shocks w.r.t Frequency (100KHz - 200KHz). (a) Capacitance in nF at Frequency of Maximum Impedance, (b) Capacitance in pF at Frequency of Minimum Impedance

Similarly, the frequency of minimum impedance observed at 165.5 KHz for the unshocked and 153.5 KHz for the shocked specimen showed a difference of only 7.5 %. This difference shows that the maximum and minimum resonance is dependent on the respective frequencies. It was also observed that the value of capacitance continuously decreases from the initial to the shocked condition at the frequency of maximum impedance, and vice versa at the frequency of minimum impedance. By using Equation 1, the dielectric constant calculated with respect to the number of shocks is shown in Fig. 4. This constant is a very important property, and depends upon the physical condition of the material. The change in the dielectric constant in this work indicates that the physical condition of the material was changed due to change in dipole lengths and directions. The change in capacitance and the relative dielectric constant is due to dipole moments inside the material. Due to thermal shocks, the displacement of electrons may cause re-orientation of these dipoles. This misalignment of polarization and displacement of charge may result in random orientation of the dipoles, which further changes its capacitance and dielectric values [11]. The description of dielectric constant is very difficult in thermal cycling problems because the orientations of molecular size dipoles changes frequently in such shocking conditions. Theory becomes more difficult because of electrostatic interaction



**Figure 4.** Change in Dielectric Constant w.r.t. Number of Shocks (From 100°C-20°C).

between dipoles. However, the measurements  $f_m$  and  $f_n$  is one of the reliable method to determine the capacitance value at a particular frequency. The relative difference in the frequencies of maximum and minimum impedance depends on both the material coupling factor and resonator geometry. For this reason a quantity called the effective coupling factor has been used. In an earlier study Viehland [12], the stress dependence of electromechanical properties of various piezoelectric ceramics has been reported. Viehland found that coupling coefficients and piezoelectric coefficients are relatively high under stress. He determined the effect of uniaxial stress upon the electromechanical properties of various piezoelectric ceramics. The change in values was observed with the change in stress and electric field. In the present work, effective and transverse excitation coupling factors were determined by resonance method and indicated in Table 1. By using Equations 2 and 3, the values obtained were plotted as indicated in Fig. 5. After thermal shocking, the transverse and effective coupling factors increased and became very close to each other. Table 1 reveals that the decrease in  $f_m$  was accompanied by decrease in  $f_n$  and their difference was continuously increasing with thermal shocking. This difference resulted in the increase in coupling factor. A thermal stress developed in the thin PZT due the thermal shock that may have caused an increase in the coupling factor. It was observed during the



**Figure 5.** Effective and Transverse Coupling Factors w.r.t. Number of Shocks (From 100°C-20°C).

experimentation that wrong selection of guarded electrode and the distance of PZT disc from the edges under the guarded electrode may affect the results. Therefore, for the reliability of the results, the values were taken with absolute care in placing the specimen under the electrode, and were repeated with discs for shocking and measurements.

### Conclusion

Thermal shocking is one of the major causes of degradation of piezoelectric materials. A thin lead zirconate titanate disc was analyzed during thermal shocking from 100 °C to 20 °C in de-ionized water. Dielectric constant and coupling factor were measured by using the capacitance and frequency of maximum and minimum impedance values obtained by precise instrumentation. Dielectric constant at  $f_m$  decreases by increasing the number of shocks, and is an expected normal behavior. Thermal shocking changes dipole length and causes reorientations of these dipoles which attributes to the changes in capacitance and dielectric constant of the the material. Effective and transverse coupling factors continuously increased. The difference in frequency of maximum and minimum impedance widened throughout, which resulted in an increase of the coupling factor. This increase in coupling factor is thought to be due to low shocking temperature difference. Further experimentation is recommended with high temperature difference in thermal shocking.

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