



# Creep behavior of soft and hard PZT ceramics during mechanical loading and unloading



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## ABSTRACT

The creep behavior of strain and electrical polarization in two poled PZT with different compositions was investigated during stress loading and unloading. Being different from the standard testing methods, the creep behaviors at all stress levels were characterized using a single sample, rather than a fresh sample at each load level. During loading, the creep of polarization and strain are observed at higher stress than that in soft PZT due to the effect of internal bias field. During unloading, significant recovery of strain and polarization was observed in the hard PZT, demonstrating that a compressive stress of 380 MPa cannot reorient the ordered defect dipoles in Fe doped hard PZT. Two equations  $P_{<MML:MROW/>0.5} = \alpha_1 \log(t + t_0) + \beta_1$  and  $S_{\text{creep}}^{0.5} = a_2 \log(t + t_0) + \beta_2$  were summarized to describe the creep polarization and the creep strain, respectively. Compared with the conventional power law creep equations, the two equations can fit the experimental results well in the whole range of stress holding.

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## 1. Introduction

Ferroelectric actuators have been widely used in modern industries due to their peculiar electromechanical coupling properties, compact size, and fast response [1]. For instance, they can serve as an active component to move the probe in scanning probe microscopy to a designated position [2]. However, in practice, accuracy and stability of ferroelectric actuators are limited by their creep behaviors. That is, both the polarization and the strain vary slowly with time when ferroelectric actuators are subjected to a constant stress or an electric field for a period. During the past decades, many methods have been developed to compensate for the creep behavior of ferroelectric ceramics [3]. A typical method is using a displacement sensor combined with a feedback control technique [2,3]. However, this method is limited in application because of its high cost and complicated design. Thus, developing open-loop control methods is very important [4,5]. As the accuracy of open-loop control methods depends on the mathematical models for creep, the creep behavior of ferroelectrics should be well understood first.

The reports concerning the creep behavior of ferroelectrics can be traced back to the early work by Subbarao et al. in 1957 [6]. They observed the strain creep in barium titanate ceramics

during stress holding and after unloading. Later, Berlincourt and Krueger [7] reported the evolution of strain with time during electric field holding and after unloading. However, being limited by the understanding of domain switching in ferroelectrics, no further discussions were presented in these two works.

With the development of ferroelectric actuators and their wide applications as precision positioners, there have been works conducted subsequently concerning the creep behavior of PZT ceramics under compression, tension, and electric field loading since 1998 [8–15,19]. Heilig and Härdtl investigated the creep polarization of soft (Nd doped) and hard (Nd and Fe doped) PZT during mechanical depolarization [8]. It was showed that soft PZT showed primary creep behavior when the holding time was larger than 40 ms; however, for hard PZT, a strange creep behavior of polarization was observed. Heilig and Härdtl argued that the creep behavior differences in soft and hard PZT were caused by the effect of dopants. Additionally, they found that the amplitude of the creep polarization was dependent on the applied stress levels. The creep behavior of soft PZT during compressive loading was also investigated by Forrester and Kisi [9], Guillon et al. [10], and Zhou and Kamlah [11]. Forrester and Kisi [9], Zhou and Kamlah [15] reported that the polarization creep and strain creep could be fitted well by the Andrade power law creep equation. Guillon et al. [10] proposed a viscoplastic model to simulate the creep behavior of ferroelectrics. The tensile creep behavior of ferroelectrics was firstly investigated by Fett and Thun [12]. They found that for both poled and unpoled

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PZT, the creep strain could be modeled by the power law creep equation. Later, the tensile creep was also investigated by Kim and Lee [13]. At work, ferroelectric actuators were normally subjected to large electric fields to get required actuations, thus some works concerning the creep behavior of PZT under electric field loading were also reported [14,15]. Being similar to that under mechanical loading, results showed that the creep of ferroelectrics under electric field loading could also be modeled by the power law creep equation. Moreover, the temperature depended creep of PZT under compression was also investigated [16], as ferroelectric actuators might work in high temperature environment [17–19] in practical applications.

Though great progresses have been achieved in understanding and modeling the creep of ferroelectrics, problems still exist. For example, the power law creep equation was verified could model the creep behavior of ferroelectrics well in a wide range of time, but at the beginning of load holding, significant discrepancy existed between the predicted and the measured results. Such discrepancy has been reported by Heilig and Härdtl [8], and was also observed in our results in this work (Fig. 10). Actually, this discrepancy can also be extracted from the results by Zhou and Kamlah [10], and that by Fett and Thun [12]. In some situations, e.g., injectors for automobile engines, high speed actuation with high accuracy is demanded. Therefore, an accurate model which can describe the creep of ferroelectric in the whole range of load holding is required. In addition, some efforts were also conducted to reduce the creep by replacing soft PZT with hard PZT [20], since hard PZT has larger coercive stresses and coercive electric fields [21,22]. However, investigations concerning the creep behavior in hard PZT are very rare. Thus in this work, we investigated the creep behaviors in both soft and hard PZT. The polarization and longitudinal strain were measured to characterize their creep behavior. Based on the experimental results, two equations were proposed to describe the creep polarization and the creep strain in ferroelectrics. Compared with the power law creep equation, these two equations are superior because they can fit the experimental results well in the whole range of stress holding.

## 2. Specimen preparation, testing setup, and experimental procedure

### 2.1. Specimen preparation

Commercially used soft and hard PZT ceramics with grain size of 3–5  $\mu\text{m}$  (Fig. 1) were chosen as model material. The PZT ceramics were provided by Institute of Acoustics, Chinese Academy of Sciences. Soft PZT ceramics was doped with Li and Sb whose composition was  $\text{Pb}_{1.0}[\text{Zr}_{0.49}\text{Ti}_{0.46}(\text{Li}_{0.25}\text{Sb}_{0.75})_{0.05}]_{1.0}\text{O}_3$ ; whereas for hard PZT, Fe and Sb were used as dopants, with a composition of  $\text{Pb}_{1.0}[\text{Zr}_{0.50}\text{Ti}_{0.47}(\text{Fe}_{0.66}\text{Sb}_{0.33})_{0.03}]_{1.0}\text{O}_3$ . The dimension of

the specimens used is  $5 \times 5 \times 10 \text{ mm}^3$ . All their surfaces were firstly polished with  $\text{Al}_2\text{O}_3$  solution. Then two opposite  $5 \times 5 \text{ mm}^2$  surfaces of the specimens were painted with silver pastes as electrodes. Before testing, the specimens were electrically poled at high temperature ( $200^\circ\text{C}$ ). After poling and aging for 24 h, the longitudinal piezoelectric constants ( $d_{33}$ ) of both soft and hard PZT were characterized by a Berlincourt meter, with a value of  $550 \pm 5 \mu\text{C/N}$  and  $278 \pm 4 \mu\text{C/N}$ , respectively.

### 2.2. Testing setup and experimental procedure

The experimental testing setup is shown in Fig. 2. Compressive stress was applied by a Shimadzu testing machine and a spherical hinge was used to avoid any bias compressive stresses. Two alumina blocks were used to insulate the specimen from the loading equipment. Two brass plates with thickness of 0.3 mm were pasted on the alumina blocks working as electrodes. Two strain gauges were glued on two opposite  $5 \times 10 \text{ mm}^2$  faces to measure the longitudinal strain during compressive loading and unloading. A large capacitor with capacitance of 2  $\mu\text{F}$  was connected in series with the testing specimen to collect the charges. Given that the capacitance of the capacitor is about four orders of magnitude larger than the specimen, the charge ( $Q$ ) released from the specimen can be calculated by  $Q = C_0 V$ . Where  $C_0$  means the capacitance of the capacitor;  $V$  is the voltage of the capacitor measured by the charge amplifier. After that, the polarization evolution of the specimen can be calculated by  $P = Q/A$ .  $A$  indicates the area of the electrode, with a value of  $25 \text{ mm}^2$ .

Before testing, the eight corners of the specimen were slightly rounded to prevent possible cracking. The specimen was firstly carefully put on the center of the bottom alumina block. Then a small preload compressive stress of 5 MPa was applied. To minimize the possible bending of the specimen, the strain changes of the two strain gauges pasted on the specimen were checked. Once they were unequal, the position of the specimen was adjusted until they were nearly equal.

During testing, compressive stress with a loading rate of 5 MPa/s was used. When the stress loading or unloading reach to a predetermined level, the stress was kept constant for 300 s. Fig. 1b shows the loading waveform used during testing. A maximum stress of 380 MPa was used. This is because a larger stress is apt to break hard PZT, and 380 MPa is large enough to completely depole soft PZT. After a cycle of compressive loading and unloading, the piezoelectric constant of both soft and hard PZT ceramics was measured, with a value of  $71 \pm 5 \mu\text{C/N}$  and  $182 \pm 4 \mu\text{C/N}$ , respectively.

## 3. Results and discussions

Fig. 3 shows the depolarization curves of soft and hard PZT. As reported by some researchers [21,22], both polarization and strain

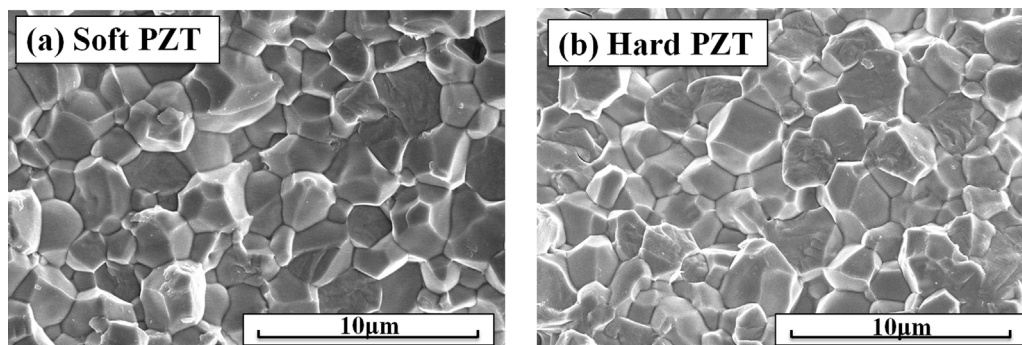


Fig. 1. Scanning electron microscopy (SEM) images of (a) soft and (b) hard PZT ceramics.

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