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# Scaling and stress dependence of sub-coercive field dynamic hysteresis in $0.6\text{Pb}(\text{Zr}_{1/2}\text{Ti}_{1/2})\text{O}_3\text{-}0.4\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3$ ceramic

R Yimmirun<sup>1</sup>, N Wongdamnern, N Triamnak, M Unruan, A Ngamjarrojana, S Ananta and Y Laosiritaworn

Department of Physics, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand

E-mail: rattikornyimmirun@yahoo.com

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## Abstract

The scaling behavior of sub-coercive field dynamic ferroelectric hysteresis under the influence of stress was investigated in rhombohedral  $0.6\text{Pb}(\text{Zr}_{0.5}\text{Ti}_{0.5})\text{O}_3\text{-}0.4\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3$  (0.6PZT–0.4PZN) bulk ceramic. The scaling relation of hysteresis area  $\langle A \rangle$  against frequency  $f$ , field amplitude  $E_0$ , and stress  $\sigma$  for the minor loops takes the form of  $\langle A - A_{\sigma=0} \rangle \propto f^{-0.36} E_0^{4.03} \sigma^{0.19}$ , indicating the difference in the energy dissipation between the stressed and stress-free conditions. While the scaling obtained is very similar to that of soft and hard PZT ceramics, slightly faster responses to  $f$  and  $E_0$  indicate the ease of polarization orientation in this ceramic with a simpler domain structure compared to commercial PZT ceramics. However, the difference in mechanical properties of these materials could contribute to a variation in the response to stress. While the  $E_0$  exponent obtained in this study agreed well with that derived from the Monte Carlo simulation based on the Q-state planar Potts model, the difference in the  $f$  exponent obtained experimentally and theoretically was attributed to the depolarizing effects presented in the bulk ceramics.

(Some figures in this article are in colour only in the electronic version)

## 1. Introduction

Dynamic hysteresis characteristics have become an important consideration because the hysteresis area  $\langle A \rangle$  as a function of the field amplitude  $E_0$  and frequency  $f$  presents much information that is critical for many ferroelectric applications [1, 2]. Theoretical studies have been carried out to understand the dynamic response of hysteresis curves in spin and polarization systems [3–7]. In particular, attention is focused on power-law scaling

$$\langle A \rangle \propto f^m E_0^n \quad (1)$$

(where  $m$  and  $n$  are exponents that depend on the dimensionality and symmetry of the system). Based on three-

dimensional models, it has been suggested that  $m = 1/3$  and  $n = 2/3$  as  $f \rightarrow 0$ , whereas  $m = -1$  and  $n = 2$  as  $f \rightarrow \infty$  [3, 5, 8]. Experimental investigations on a few thin-film systems have also been reported with variation in the scaling relations obtained [9–12]. On the contrary, there are only a few reports on the hysteresis scaling behavior of bulk ferroelectric ceramics [13–16].

Our previous investigations have already shown stress-dependent scaling behaviors in commercial soft and hard lead zirconate titanate ( $\text{Pb}(\text{Zr}, \text{Ti})\text{O}_3$ ; PZT) ceramics [14, 15]. The results suggested that the scaling behavior depended strongly on the dimensionality of the system, and also suggested that the different states of domains existing in the ceramics may play a key role in controlling the scaling behavior. Unfortunately, it was too difficult to retrieve such

<sup>1</sup> Author to whom any correspondence should be addressed.

information for the domain structures of commercial ceramics. Therefore, this study was aimed at a ferroelectric material with a distinct domain configuration. Previous works on  $0.6\text{Pb}(\text{Zr}_{0.5}\text{Ti}_{0.5})\text{O}_3$ – $0.4\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3$  (0.6PZT–0.4PZT) ceramics with rhombohedral structure have reported excellent electrical properties suitable for various applications [17–20]. Due to its many potential applications and relatively simple domain configuration, the 0.6PZT–0.4PZN ceramic was thus chosen for this study. Practically, ferroelectric ceramics are normally used under sub-coercive field conditions in most applications; it is therefore of interest to obtain scaling relations for such conditions. Interestingly, earlier works showed that the scaling behavior of the minor and saturated loops was significantly different [11, 12].

More importantly, in many applications the ceramic specimens are often subjected to mechanical loading, either from the design of the device itself or from operating conditions which induce stresses [1, 21]. Therefore, prior knowledge of how the material properties change under different load conditions in materials is inevitably crucial for the proper design of a device and for suitable selection of materials for a specific application. In many previous investigations the electrical properties of ceramics were found to strongly depend on stresses ( $\sigma$ ) [22–26]. Since  $f$ ,  $E_0$ , and  $\sigma$  have been reported to impose significant influence on the dynamic hysteresis responses of ferroelectric ceramics, it is therefore the major aim of this study to obtain the stress-dependent scaling behavior of sub-coercive field dynamic hysteresis in 0.6PZT–0.4PZN ceramic.

## 2. Experimental procedures

Disc-shaped samples of tetragonal structure 0.6PZT–0.4PZN ceramic with a diameter of 10 mm and thickness of 1 mm were used in this study. Details on the fabrication and characterization of this material can be found elsewhere [19, 20]. Its basic properties are: dielectric constant (1 kHz)  $\epsilon_r = 1616$ ; Curie temperature  $T_C = 284^\circ\text{C}$ ; piezoelectric strain constants  $d_{33} = 457 \text{ pm V}^{-1}$ ; planar coupling factor  $k_p = 0.59$ ; mechanical quality factor  $Q_m = 62$ ; coercive field (10 Hz)  $E_c = 12 \text{ kV cm}^{-1}$ . The dynamic hysteresis ( $P$ – $E$ ) loops were characterized at room temperature ( $25^\circ\text{C}$ ) by a modified Sawyer–Tower circuit with  $f$  ranging from 2 to 100 Hz and  $E_0$  from 0 to  $10.5 \text{ kV cm}^{-1}$ . The field was applied to a sample by a high voltage AC amplifier (Trek 610D) with the input sinusoidal signal from a function generator (HP 3310A). The  $P$ – $E$  loops were recorded by a digital oscilloscope (HP 54645A, 100 MHz). A detailed description of this system is given elsewhere [25]. Effects of the external stress on the dynamic hysteresis were investigated with the compressometer developed for simultaneous applications of the mechanical stress and the electric field [26]. The compressive stress, applied parallel to the electric field direction, was supplied by the servohydraulic load frame and monitored with the pressure gauge. The  $P$ – $E$  loops were recorded as a function of mechanical stress applied discretely between 0 and 163 MPa for each applied field and frequency. At each constant stress, the loop was obtained after 20 sampling cycles to average out the noise deformation.

## 3. Results and discussion

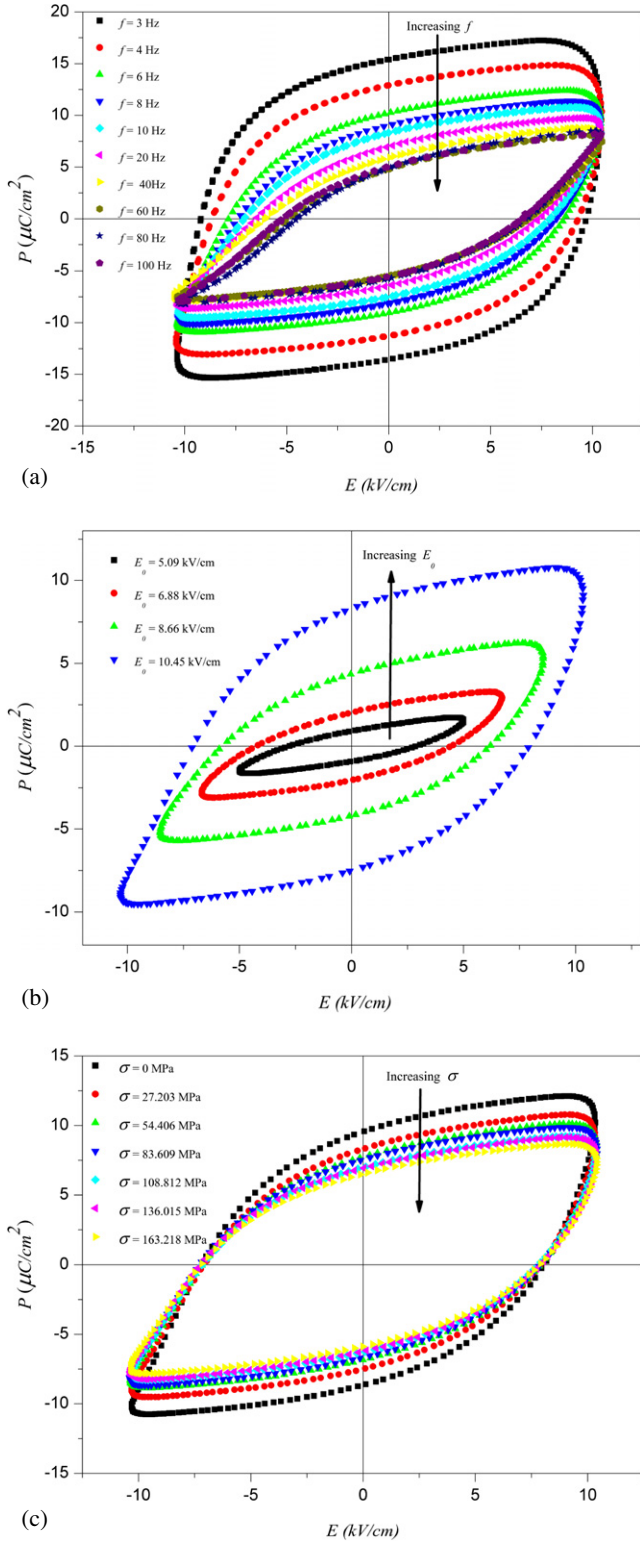
The hysteresis loops at different  $f$  but fixed  $E_0$  ( $10.5 \text{ kV cm}^{-1}$ ) and  $\sigma$  (27 MPa), at different  $E_0$  but fixed  $f$  (10 Hz) and  $\sigma$  (27 MPa), and at different  $\sigma$  but fixed  $E_0$  ( $10.5 \text{ kV cm}^{-1}$ ) and  $f$  (10 Hz) are shown in figures 1(a), (b), and (c), respectively. For a particular applied stress, as expected, the dependence of the loop pattern and area  $\langle A \rangle$  on  $f$  and  $E$  is remarkable. At fixed  $E_0$ , the loop area  $\langle A \rangle$  and remnant polarization ( $P_r$ ) decrease with an increase of frequency, as shown in figure 1(a). The dependence of the hysteresis loop on  $E_0$  is depicted in figure 1(b). Similar observations have also been reported in soft and hard PZT ceramics [27–29]. In addition, the  $P$ – $E$  loop area  $\langle A \rangle$  is found to decrease steadily with increasing stress, as reported in previous investigations [24–26].

To obtain suitable scaling relations, one can first follow the suggested scaling law given in equation (1) to determine exponents  $m$  and  $n$  directly from the experimental data. By plotting  $\langle A \rangle$  against  $f$  at fixed  $E_0$ , one obtains the exponent  $m$ . On the other hand, the exponent  $n$  can be obtained by plotting  $\langle A \rangle$  against  $E_0$  at fixed  $f$ . In conjunction with the least-square-fitting method, the exponents  $m = -0.36 \pm 0.03$  and  $n = 4.03 \pm 0.06$  were obtained. As plotted in figure 2, it is revealed that the experimental data can be fitted (with  $R^2 = 0.99$ ), within the measured uncertainty, by

$$\langle A \rangle \propto f^{-0.36} E_0^{4.03}. \quad (2)$$

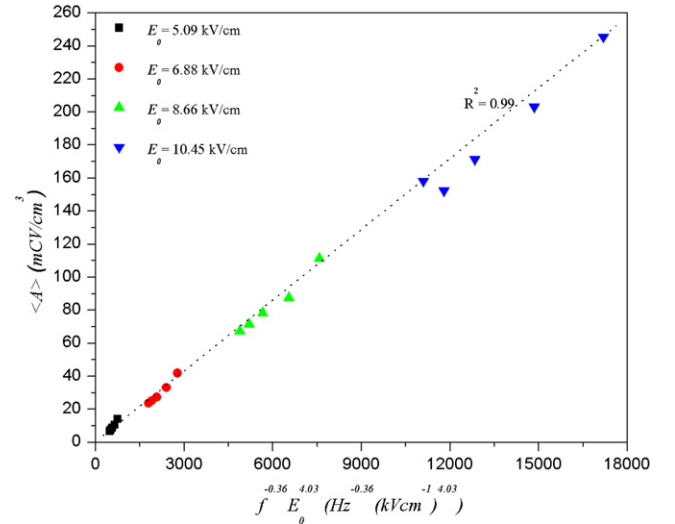
Here, the first interesting observation is that the minor-loop scaling in PZN-modified PZT bulk ceramics is generally similar to that of PZT thin film [5, 9], as well as that of the minor loop of PZT bulk ceramics [13, 15]. This is attributed to the fact that the main polarization orientation mechanism in thin-films and in sub-coercive field conditions for bulk ceramics is likely from the same reversible  $180^\circ$  domain rotation (which also occurs at a much faster rate than the irreversible process), as the irreversible non- $180^\circ$  domain switching normally accompanied by mechanical strain occurs at a higher  $E$ -field [13, 15, 30–34]. More importantly, the scaling relation obtained in equation (2) indicates that  $\langle A \rangle$  decays slightly more quickly with  $f$  and grows faster with  $E_0$  than in commercial soft PZT bulk ceramic with  $\langle A \rangle \propto f^{-0.33} E_0^3$  [13]. This indicates the ease of the polarization orientation process in rhombohedral 0.6PZT–0.4PZN ceramics that leads to faster polarization orientation kinetics, as compared to complex domain structures of commercial PZT bulk ceramics which require a higher energy barrier for the polarization orientation process.

Considering the  $E_0$  exponent of 4.03, which is approximately of the same order as that in PZT thin film [5, 9], as well as those in minor loops of PZT bulk ceramics [13, 15], the reversible domain wall vibration is also believed to play an additional role in the dependence of hysteresis area on  $E_0$  at a sub-coercive field. In the nucleation and growth concept [5, 9, 35] domain reversal can be characterized by simultaneous contributions from both the new domain nucleation rate and the domain boundary motion velocity, with the latter responding faster to the electric field [36, 37]. Since the reversible domain wall vibration makes a greater



**Figure 1.** Hysteresis loops under sub-coercive field conditions for 0.6PZT–0.4PZN ceramic (a) at various  $f$ ,  $E_0 = 10.5 \text{ kV cm}^{-1}$ , and  $\sigma = 27 \text{ MPa}$ ; (b) at various  $E_0$ ,  $f = 10 \text{ Hz}$ , and  $\sigma = 27 \text{ MPa}$ ; and (c) at various  $\sigma$ ,  $f = 10 \text{ Hz}$ , and  $E_0 = 10.5 \text{ kV cm}^{-1}$ .

contribution to the dependence of hysteresis area with  $E_0$  in sub-coercive conditions, the response to  $E_0$  occurs at much faster rate than at a higher field amplitude. It should also be



**Figure 2.** Scaling of hysteresis area  $\langle A \rangle$  against  $f^{-0.36} E_0^{4.03}$  for 0.6PZT–0.4PZN ceramic under sub-coercive field conditions.

noted that the  $E_0$  exponent  $n = 4.03$  agrees very well with the  $n$  value of 4 obtained from the previous simulation study using the Monte Carlo method based on the Q-state planar Potts model [38].

In addition, the  $f$  exponent of  $-0.36$  is smaller in absolute value than that obtained theoretically from  $(\Phi^2)^2$  and  $(\Phi^2)^3$  models for non-saturated loops (exponent  $-1$ ) [3]. The difference could be attributed to depolarizing effects from space charges on grain boundaries, induced electric field from interface layers, immobile defects etc [39], acting as a buffer to the polarization-reversal mechanism in bulk ceramics. As a result, the hysteresis area must show a relatively weaker dependence on  $f$  than that of the theoretical models. Furthermore, the value of the  $f$  exponent is believed to be related to available domain states in the material, previously proposed in our investigations [13, 14], or growth dimension in the growth kinetics model proposed by Ishibashi and Orihara based on the Kolmogorov–Avrami model [40, 41].

To investigate the scaling behavior under the effect of applied stresses, the stress ( $\sigma$ ) term should be included, i.e.

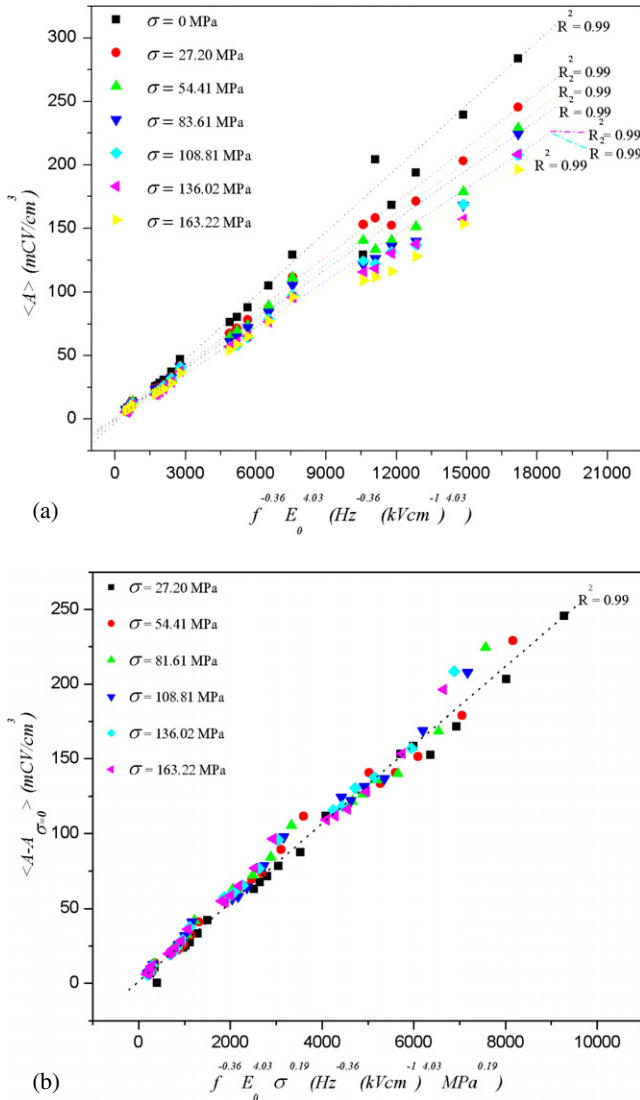
$$\langle A \rangle \propto f^m E_0^n \sigma^p. \quad (3)$$

However, due to the increasing number of exponents, to simplify the problem the validity of the scaling law  $\langle A \rangle(\sigma) \propto f^{-0.36} E_0^{4.03}$  is assumed for all applied stresses. Consequently, the area  $\langle A \rangle$  for each stress is plotted against  $f^{-0.36} E_0^{4.03}$ , as shown in figure 3(a), and it can be seen from the least-square linear fits that reasonably good linear relations can be found. As a result, the condition of universality having  $m = -0.36$  and  $n = 4.03$  in 0.6PZT–0.4PZN bulk ceramic is confirmed. Hence,  $\langle A \rangle \propto (a + b\sigma^c) f^{-0.36} E_0^{4.03}$  may be written and by substituting the fitted parameters, it is found that

$$\langle A \rangle - \langle A_{\sigma=0} \rangle = \langle A - A_{\sigma=0} \rangle \propto f^{-0.36} E_0^{4.03} \sigma^{0.19}, \quad (4)$$

where  $\langle A_{\sigma=0} \rangle$  refers to the stress-free hysteresis area which will be a dominant term for zero stress. The data-collapsing





**Figure 3.** (a) Scaling of hysteresis area  $\langle A \rangle$  against  $f^{-0.36} E_0^{4.03}$  at various  $\sigma$ . (b) The collapse of the scaling area  $\langle A - A_{\sigma=0} \rangle$  against  $f^{-0.36} E_0^{4.03} \sigma^{0.19}$  on the same linear-line (with small fluctuation) for 0.6PZT-0.4PZN ceramic under sub-coercive field conditions.

of the scaling area from all  $f$ ,  $E_0$ , and  $\sigma$  was found to confirm equation (4) as evident in figure 3(b). Note that from the appearance of stress  $\sigma$ ,  $\langle A - A_{\sigma=0} \rangle$ , referring to the difference in energy dissipation between under-stress and stress-free conditions, increases with increasing stress suggesting a decay of  $\langle A \rangle$  with  $\sigma$  at a rate of  $\sigma^{0.19}$  as observed in experiments. Therefore, the difference in the hysteresis area between stressed and stress-free conditions scales with frequency, field amplitude, and stress via the exponents  $m = -0.36 \pm 0.03$ ,  $n = 4.03 \pm 0.06$ , and  $p = 0.19 \pm 0.03$ .

To understand the scaling relation obtained in equation (4), at least qualitatively, one needs to consider the following statements. Since the  $P$ - $E$  loop area indicates the polarization dissipation energy subjected to one full cycle of electric field application, the loop area is therefore directly related to the volume involved in the switching process during the application of electric field [25, 26, 42]. When the

compressive stress is applied in the direction parallel to the direction of the polar axis, the applied stress tends to keep the ferroelectric domains aligned with their polar axes away from the stress direction through the non-180° ferroelastic domain switching processes. Therefore, it takes a larger than usual applied electric field to reorient the domains along the stress direction, resulting in lower values of the saturation polarization. When the electric field is reduced to zero the domains tend to rotate back away from the applied compressive stress direction, resulting in a lower than usual remnant polarization ( $P_r$ ). Therefore, both the saturation and remnant polarizations become lower with increasing compressive stress, hence the coercivity is also lower [24–26]. The polarization dissipation energy is consequently found to decrease with increasing applied stress, indicating that the sample volume contributing to polarization reversal decreases with increasing stress. Similar observations have also been reported in other investigations [22–26, 43, 44].

Furthermore, it is noted that the stress term with exponent  $p = 0.19$  is noticeably different from that for soft and hard PZT bulk ceramics with  $p = 0.44$  and  $0.37$ , respectively [14, 15]. The difference could be attributed to a variation of mechanical properties of these materials. While no other stress-dependent scaling investigation is available for a direct comparison, Viehland *et al* [45] reported a power law-like relation between the hysteresis loss and the applied stress in PMN-PT crystals. In addition, many of the theoretical treatments to explain the stress effect on the hysteresis behavior have been primarily based on constitutive models, micro-mechanical and analytical approaches, thermodynamic phenomenology, and Rayleigh law and Preisach model approaches [46–56]. While these theoretical treatments are useful in understanding hysteresis behavior in ferroelectric materials under the application of stress, none of them derive a direct relation between the hysteresis loss and the applied stress in ferroelectric materials. It is also interesting to point out that recent theoretical and experimental investigations have related the stress dependence of the minor hysteresis loop parameters to the dislocation density in ferromagnetic materials [57–60]. Further investigations, both theoretical and experimental, of the stress-dependent scaling behavior are clearly needed as the stress relations are useful in predicting the practical limitations of devices when used under stress.

#### 4. Conclusions

The scaling behavior of the sub-coercive field dynamic ferroelectric hysteresis of rhombohedral 0.6PZT-0.4PZN bulk ceramics under the effect of mechanical stress has been investigated. The scaling relation for the minor loops takes the form of  $\langle A - A_{\sigma=0} \rangle \propto f^{-0.36} E_0^{4.03} \sigma^{0.19}$ , indicating the difference in the energy dissipation between the stressed and stress-free conditions. While the scaling obtained is very similar to that of soft and hard PZT ceramics, slightly faster responses to  $f$  and  $E_0$  indicate the ease of polarization orientation in this ceramic with a simpler domain structure compared to commercial PZT ceramics. In addition, a difference in the response to stress could be attributed to

a variation in the mechanical properties of these materials. Finally, the  $E_0$  exponent obtained in this study agrees well with that derived from the Monte Carlo simulation based on the Q-state planar Potts model. The difference in the  $f$  exponent obtained experimentally and theoretically can be attributed to the depolarizing effects presented in the bulk ceramics.

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