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In situ X-ray diffraction of lead zirconate titanate piezoMEMS cantilever during actuation

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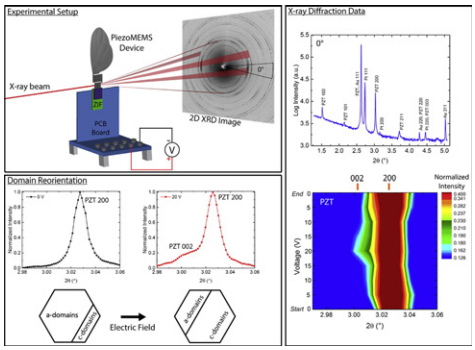
HIGHLIGHTS

- Synchrotron X-ray diffraction successfully probed the electric-field-induced response of a functional piezoMEMS device.
- 90° domain reorientation was observed in the PZT morphotropic phase boundary film used in the piezoMEMS device.
- Results show that X-ray diffraction can be used to detect 90° domain reorientation in small length scale piezoMEMS devices.

GRAPHICAL ABSTRACT



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ABSTRACT

Synchrotron X-ray diffraction (XRD) was used to probe the electric-field-induced response of a 500 nm lead zirconate titanate (52/48, Zr/Ti) (PZT) based piezoelectric microelectromechanical system (piezoMEMS) device. 90° ferroelectric/ferroelastic domain reorientation was observed in a cantilever comprised of a 500 nm thick PZT film on a 3 μm thick elastic layer composite of SiO_2 and Si_3N_4. Diffraction data from sectors both parallel- and perpendicular-to-field showed the presence of ferroelastic texture, which is typically seen in *in situ* electric field diffraction studies of bulk tetragonal perovskite ferroelectrics. The fraction of domains reoriented into the field direction was quantified through the intensity changes of the 002 and 200 diffraction profiles. The maximum induced volume fraction calculated from the results was 20%, which is comparable to values seen in previous bulk and thin film ferroelectric diffraction studies. The novelty of the present work is that a fully released ferroelectric thin film device of micron scale dimensions (down to 60,000 μm^3) was interrogated *in situ* with an applied electric field using synchrotron XRD. Furthermore, the experiment demonstrates that 90° ferroelectric/ferroelastic domain reorientation can be characterized in samples of such small dimensions.

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1. Introduction

Piezoelectric microelectromechanical systems (piezoMEMS) are MEMS devices which utilize piezoelectric films to convert between electrical and mechanical energies. Lead zirconate titanate ($PbZr_xTi_{1-x}O_3$) (PZT (Zr/Ti)) thin films with a composition close to the morphotropic phase boundary (MPB), $x = 0.52$, are widely used in piezoMEMS due to their high piezoelectric response [1]. The high piezoelectric constants of high performance PZT thin films enable low voltage mm-scale robotics [2–4]. Miniaturizing robotics could provide benefits in defense applications, including higher levels of stealth entry and access to locations that would otherwise be un-achievable. Depending on the configuration of the PZT film, piezoMEMS actuators are capable of generating lateral, vertical, and torsional motion. This allows PZT films to be utilized as motion generators for insect-like microrobotics.

The electromechanical response of ferroelectric materials is influenced by two main contributions; (1) the intrinsic piezoelectric effect, and (2) extrinsic contributions, predominantly from domain wall motion. In bulk PZT ceramics, domain wall motion contributes to a large fraction of the overall piezoelectric response of the material [5]. In general, there are two types of domain wall motion, 180° and non-180°. The domain wall contributions to the piezoelectric response is typically dominated by non-180° domain wall motion [5–7], due to the field-induced ferroelectric/ferroelastic distortions that accompany the motion of these walls. 180° domain walls can also add to the piezoelectric response, as seen in work done on PZT thin films under high ac fields [8]. Ultimately, non-180° domain reorientation can be tracked as a function of the electric field by diffraction methods, including XRD.

The application of an electric field during diffraction measurements can provide useful information regarding the electromechanical response of the ferroelectric sample [9–12]. The electric field induced lattice strain of a ferroelectric material can be calculated from peak shifts. In diffraction measurements, intensity exchanges between degenerate reflections, *e.g.* 002 and 200 for tetragonal ferroelectrics, can yield quantitative domain reorientation information [13–18]. Meanwhile, the disappearance and appearance of peaks for on and off-field measurements can help identify electric-field-induced phase changes [14,19].

In thin films, the extrinsic contribution can be affected by crystallographic texture, grain size, and residual stress [3,20–24]. The crystallographic texture of the ferroelectric film controls the amount of domain reorientation, with {001} films showing the highest amount of non-180° domain reorientation in tetragonal perovskites [25,26]. Small grain sizes are known to lead to increased pinning of domain walls [24,27], and residual stresses from the growth process within the ferroelectric film can reduce domain wall mobility [25,28]. It is worth noting that these residual stresses are a product of all of the previously mentioned factors and can also change as a function of film thickness, ultimately affecting the dielectric response of the film [29]. For example, after processing ferroelectric films grown on silicon substrates, the film is subjected to tensile stress, after cooling, due to the thermal expansion mismatch between the film and substrate [30]. The tensile stress yields a preferred polarization in the plane of the film, and increases the energy barrier to reorient the polarization of domains out-of-plane [31].

Altering the mechanical boundary constraints, by substrate removal and/or introduction of porosity to the ferroelectric film influences the film's domain reorientation response. For example, Wallace et al. [32], used synchrotron X-ray diffraction (XRD) on {100} textured PZT films to show that the removal of ~75% of the underlying substrate led to a six times increase in the amount of domain reorientation. The partial removal of the substrate allowed for the in-plane stress to be partially relieved, thus, lowering the energy barrier for inducing domain reorientation. A similar study comparing porous and dense as-processed {100} PZT films found that porous films achieved higher domain reorientation values [33]. This increase in domain reorientation was attributed to a reduction in domain wall pinning as a result of lowering the film stiffness due to porosity within the film, lowering the average stress state, and/or changing field concentrations. Thus, the mechanical boundary constraints can modulate the domain reorientation response in ferroelectric thin films.

Micro-fabricated PZT cantilevers have dramatically different boundary constraints than as-processed films. PZT cantilevers have been studied both through electrical property measurements and with XRD. Morioka et al. [34], has studied the electric-field structural response of 1.1 μm thick PZT (44/56) using a lab source diffractometer and was able to confirm 90° domain reorientation. The composition of this PZT film lies on the tetragonal side of the MPB for the PZT binary system, which in combination with a thick film allows a lab source diffractometer to adequately probe the material's response. Similarly, Morioka et al. [35], conducted a similar study on a microcantilever utilizing a 1.1 μm thick PZT (52/48) in which no 90° domain reorientation was observed. Materials using PZT compositions close to the MPB become more challenging to probe with XRD, especially when utilizing a lab source. Many advances have been made in the field of ferroelectrics due to the availability of synchrotron sources, which can enable improved crystal structure determination and give insight into the average response of the material.

In the present work, the electromechanical response of a functional piezoMEMS device utilizing 500 nm thick PZT (52/48), was probed *in situ* using synchrotron XRD. The film measured in the present study is thinner than those investigated by Morioka et al. [34,35], and the use of a synchrotron source offers enhanced resolution compared to a lab source diffractometer. Earlier measurements done on ferroelectric thin film capacitors have been conducted on electrode areas of 15 mm^2 [32,33,36], but this study explores increasingly lower limits by studying an electrode area of ~0.118 mm^2 on a piezoMEMS device. From the XRD data the domain reorientation response was calculated through the integrated intensity of degenerate reflections, *i.e.* 002 and 200 for tetragonal ferroelectrics. Quantified domain reorientation values were on the same order of domain reorientation values calculated for previous bulk ferroelectric studies. This study highlights the capability of using XRD to characterize ferroelectric films with small area capacitors.

2. Experimental procedure

The fabricated piezoMEMS device utilized $Pb(Zr_{0.52}Ti_{0.48})O_3$ thin films synthesized using chemical solution deposition at the US Army Research Laboratory. Details of the sample synthesis, including the layer stacking is described in Ref. [37]. In short, the composite actuators are comprised of a 3 μm thick elastic layer comprised of a multilayer of SiO_2 and Si_3N_4 thin film, a 35 nm thick TiO_2 layer, a 100 nm Pt bottom electrode, a 500 nm PZT (52/48) actuator layer, and a 100 nm Pt top electrode. The PZT film was targeted for a thickness of 500 nm with a {100} crystallographic texture. Since the piezoMEMS mm-scale wing closely resembles a cantilever geometry, the term PZT cantilever will be used interchangeably to refer to the piezoMEMS device. Fig. 1 (a) shows an SEM image of the fully fabricated piezoMEMS device. The actuation motion comes from two PZT films oriented orthogonal to one another, producing two types of motions that can be individually actuated, stroke and pitch, shown on Fig. 1 (b).

In this experiment, only the stroke portion of the cantilever was actuated, irradiated, measured, and quantified in order to reduce the signal of gold, platinum, and chromium on the pitch portion of the cantilever. The volume irradiated of the electrically-active PZT was ~60,000 μm^3, which is approximately equivalent to irradiating a strand of human hair that is 60 μm in diameter and 21 μm in length. SEM images of the plan view of the stroke and the pitch are shown in Fig. 1.

An *in situ* electric field dependent diffraction experiment was carried out at beamline 11-ID-C of the Advanced Photon Source (APS) at Argonne National Laboratory using a Perkin Elmer detector placed approximately 2 m away from the sample. This is a similar setup which has been reported previously in references [32,33,36]. Using a constant

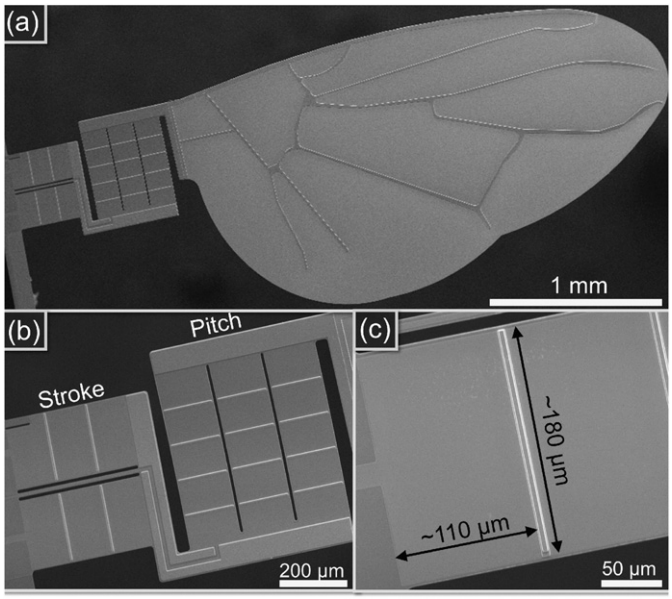

Fig. 1. SEM images of (a) a fabricated piezoMEMS device, (b) the stroke and pitch component that utilizes PZT, and (c) the zoomed in portion of the stroke displaying approximate dimensions of the electrode area.

wavelength of $\lambda = 0.10801$ Å, a beam 350 μm tall by 400 μm wide was used to optimize the diffraction signal of the stroke portion of the PZT cantilever. This beam remained on the probed actuator during electric-field-driven-motion of the cantilever. A standard sample, cerium dioxide (CeO_2), was used to calibrate the sample-to-detector distance, beam center, and detector orthogonality. Voltage was applied using a Keithley 2410C 1100 V source meter. The applied voltage was incrementally increased to a maximum of 20 V in 5 V increments, then decreased down to 0 V. The XRD patterns were measured for 5 min at each voltage step to obtain data with reasonable statistics because the irradiated volume was small.

A special stage was designed for this experiment to apply electric fields to the PZT cantilever. A 24-pin zero insertion force (ZIF) socket was mounted on a printed circuit board (PCB) with wires soldered from the ZIF socket to another PCB board that allowed application of an electric field *via* alligator clips. To attach the special PCB stage onto the stage at 11-ID-C, an additional attachment stage was fabricated. This attachment stage allowed for minimal movement of the PCB stage when removing and attaching the alligator clips. A full setup is shown in Fig. 2 (a). The PZT cantilever was mounted with the stroke portion of the cantilever aligned parallel to the X-ray beam; this allowed the crystallographic texture of the PZT thin film to be measured during acquisition.

The *Fit2D* program was used to reduce the 2D diffraction images, obtained from synchrotron measurements, in order to perform further analysis [38]. The 2D diffraction patterns were reduced to 1D plots of 2θ *versus* intensity for sectors that represent scattering vectors that are parallel (0°) and perpendicular (90°) to the applied electric field direction. The integration range used in *Fit2D* for each sector was 20° in the azimuthal direction of the 2D image. Fig. 2 (b) illustrates the sections integrated on the 2D image that were used for data analysis and discussion. The importance of both parallel- and perpendicular-to-field sectors is the orientation of the lattice planes probed relative to the applied field. For example, the parallel-to-field sector probes lattice planes that are oriented parallel to the surface of the film, *i.e.* oriented perpendicular-to-electric-field direction; this yields information about the out-of-plane response of the film. The perpendicular-to-field sector probes lattice planes that are perpendicular to the surface of the film, *i.e.* oriented parallel to the electric field direction, and yields information about the in-plane response of the film.

3. Discussion

Fig. 3 (a) shows evidence of domain reorientation between the 0 V and 20 V diffraction patterns of the 002 and 200 diffraction peaks. The development of the 002 reflection upon application of an electric field shows direct evidence of *a*-domains reorienting to *c*-domains. When an electric field is applied, the fraction of *c*-domains increases due to 90° ferroelectric/ferroelastic domain reorientation. The behavior is seen in diffraction patterns by an increase in intensity of the 002 reflection in parallel-to-field direction. The absences of the 002 reflection, for the initial diffraction pattern at 0 V, can be explained as the initial volume of *c*-domains being too small to be detected by X-rays, as shown in Fig. 3 (a). When an electric field is applied, some *a*-domains switch to *c*-domains, thus, increasing the volume of *c*-domains. The resulting increase in volume of *c*-domains is significant enough to be distinguished by X-rays, leading to the development the 002 peak. These results indicate that the amount of domain reorientation is substantial, which has been seen in bulk PZT MPB compositions, [39] but not on PZT films that are less than 1 μm due to the available XRD instrumental resolution [35].

In Fig. 3 (b) the absence of a PZT 002 peak at perpendicular-to-field direction provides further evidence of the existence of 90° domain reorientation. When domain reorientation occurs within a ferroelectric material, its effect can be seen in the diffraction pattern as a preferred distribution of intensities, referred to as ferroelastic texture. This ferroelastic texture is most prominent in the parallel- and perpendicular-to-field directions. Since intensity is conserved when comparing a diffraction signal from various scattering vectors, an increase in the 002 reflection in the parallel-to-field direction leads to a decrease in the perpendicular direction, as seen in many bulk ferroelectric studies [14,40]. Based on the present results, this suggests that the majority of the *c*-oriented domains have switched to the out-of-plane direction, and thus, are measured in the parallel-to-field sector at 20 V.

MPB bulk PZT has been known to undergo electric-field-induced phase transitions [19,41–43]. However, the films explored in the present work are assumed to be single phase tetragonal and do not exhibit an electric-field-induced phase transition. Due to the low diffraction signal from the PZT cantilever and small irradiated volume, it is difficult to obtain intensity from 00*l* reflections at 0 V, which would help in further distinguishing the initial phase of the film. If a phase transition was occurring this means that the structure of the PZT film, at 0 V, starts as a rhombohedral structure and upon electric field application a phase transition occurs from rhombohedral to tetragonal. This scenario seems less likely, because previous studies done on bulk and thin film MPB PZT have suggested that the tetragonal phase fraction decreases with the applied electric field [19,41–43]. Additionally, if a phase transition occurred, the transition would still exhibit some domain texturing in the tetragonal phase, because there is an absence of the 002 at the perpendicular-to-field direction, Fig. 3 (b). This absence of the 002 peak at the perpendicular-to-field direction is typically associated with 90° domain reorientation. However, the possibility of a phase transition cannot be fully ruled out since the phase identification of this sample is limited by the XRD signal.

In order to quantify the change in the volume fraction of *c*-oriented domains, integrated intensity values for the 002 and 200 reflections were obtained *via* line-profile analysis. Line-profile analysis was carried out using two Pearson-VII functions. An example of the peak fit conducted on the 002 and 200 reflection at 20 V for the parallel-to-field data is shown in Fig. 4. The integrated intensity values for the 002 peak are shown on Fig. 5 (a) and illustrates the development of the 002 peak during the experiment. For the 0 V diffraction pattern only, the integrated intensity value for the 002 peak was assigned to be zero since only the 200 reflection is quantifiable. With increasing voltage, the integrated intensity value for the 002 peak increases as well. From Fig. 5 (a) the integrated intensity values for the 002 peak show that there is a slight hysteresis present in the domain structure after

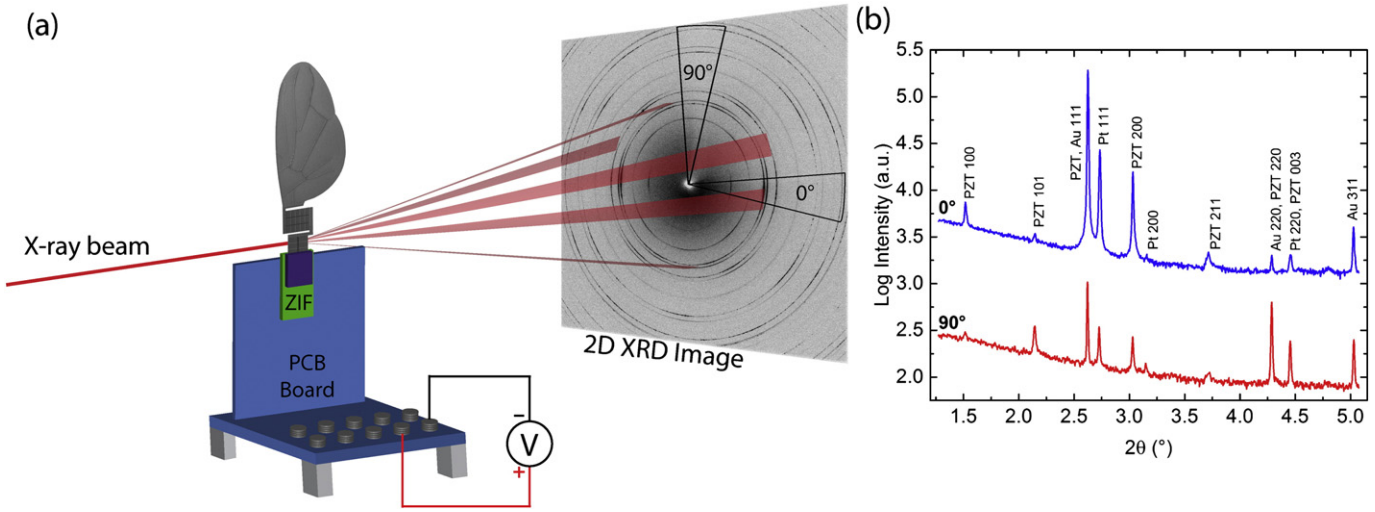

Fig. 2. a) Experimental setup used for the *in situ* diffraction experiment at 11-ID-C, and b) sectors chosen for integration on a 2D diffraction image; 0° sector describes the parallel-to-field direction and 90° sector describes the perpendicular-to-field direction.

the experiment, so that the integrated intensity values do not fully return to zero after the experiment.

The volume fraction of domains aligned in a given sector (v_{002}) can be quantified by using Eq. (1),

$$v_{002} = \frac{\frac{I_{002}}{I'_{002}}}{\frac{I_{002}}{I'_{002}} + 2\frac{I_{200}}{I'_{200}}} \tag{1}$$

where I_{002} is the integrated intensity of the 002 reflection and I_{002}' is the reference intensity from a Powder Diffraction File™ for the 002 reflection ($I_{002}'=107, I_{200}'=260$) [44]. The ratio of the reference intensities of a Powder Diffraction File™, I_{002}', and the intensities of the sample, I_{002}, are used to account for the difference in structure factors for 002 and 200 reflections. From the obtained volume fraction of 002 domains, the fraction of domain reorientation (η_{002}) can be calculated using Eq. (2). In Eq. (2), $v_{002}^{0\ field}$ corresponds to the initial un-poled state of the film. By comparing the induced volume fraction relative to the volume fraction at zero field ($v_{002}^{0\ field}$), Eq. (2) would account for an initial domain orientation, if present.

$$\eta_{002} = v_{002} - v_{002}^{0\ field} \tag{2}$$

The methodology described above has been utilized widely in various polycrystalline ferroelectrics, including both bulk and thin films [14,20,32,33]. Typical η_{002} values for electrically poled bulk ferroelectric ceramics can range anywhere from 0.13–0.46 in the direction parallel-to-the electric field [40], to 0.02–0.05 for as-processed PZT films on silicon substrates that have been electrically poled [32,33].

Due to the measureable signal of the PZT 002 reflection, as a result of using a synchrotron source, Eq. (1) can be used to further quantify the measured diffraction signal. Fig. 5 (b) shows the volume fraction of 002 oriented domains as a function of voltage, which was quantified using the XRD data. The maximum volume fraction of 002 oriented domains, seen at 20 V, was 20%. This value of v_{002} is on the order of domain volume fraction changes seen in bulk ferroelectric PZT ceramics and those seen in the study domain by Wallace et al. [32] on released

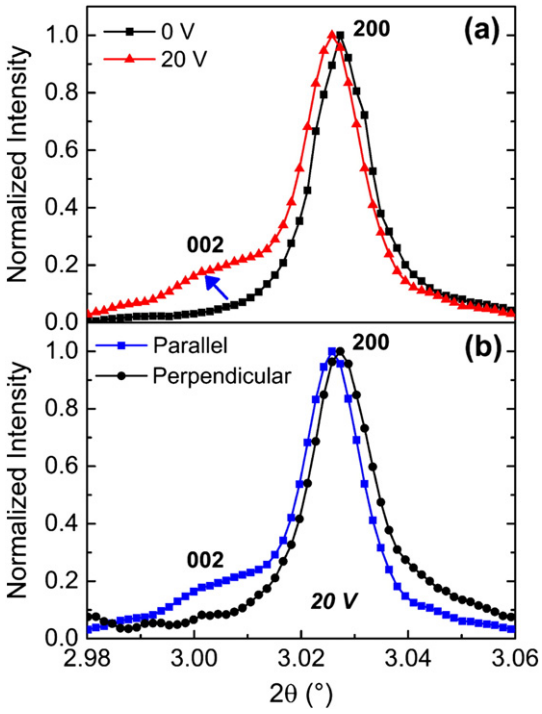

Fig. 3. 1D diffraction patterns of 002/200 reflections, (a) at 0 V and 20 V, and (b) comparing both diffraction signals from scattering vectors that are parallel and perpendicular to the electric field.

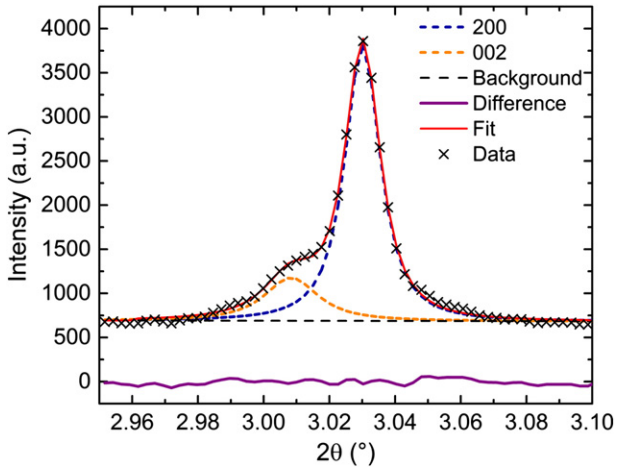

Fig. 4. Peak fit of 002 and 200 reflections at 20 V using two Pearson-VII functions.

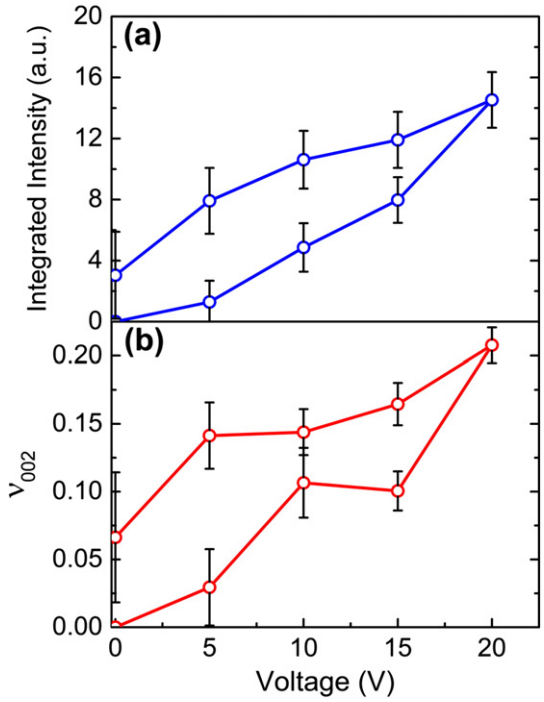

Fig. 5. As a function of voltage, (a) integrated intensity values obtained from peak fitting XRD data, and (b) calculated volume fraction of 002 oriented domains.

{001} tetragonal PZT films. A comparable v_{002} value between the piezoMEMS and bulk is seen as a result of the film's initial preferred orientation, v_{002} 0 V, compared to its induced volume fraction change v_{002}. As-processed PZT films on silicon substrates have a tensile in-plane stress, which leads to a high amount of *a*-oriented domains in the in-plane direction of the film as-prepared. This residual stress is known to increase the energy barrier needed for domains to reorient from *a*-domain to *c*-domain [31], and leads to more *a*-oriented domains in-plane of the film, *i.e.* a higher 200 reflection and a lower 002 reflection at 0 V. This allows for a larger amount of *a*-domains to switch to *c*-domains when an electric field is applied, leading to a large value v_{002} that is comparable to bulk ferroelectrics. However, this large v_{002} is a result of substrate removal, which leads to a relief of in-plane tensile stress and lowers the energy barrier for *a*-domain to *c*-domain reorientation, *i.e.* 90° domain reorientation. Domain reorientation values for as-processed clamped {001} films are typically around 5% [32,33], which is a factor of 4 lower than is reported in this study. Therefore, integrating a fully released PZT film into this piezoMEMS device facilitates 90° domain reorientation due to the absence of the underlying substrate. These results suggest that substrate removal will modulate the functional properties of flexural piezoMEMS actuators.

4. Conclusions

High energy XRD confirmed the first instance of 90° ferroelectric/ferroelastic domain reorientation in a 500 nm thick PZT layer within a piezoMEMS cantilever during actuation. This device utilized an electrode area that is much smaller than previously reported on similar studies (previous dimensions were, 15 mm^2 [32,33,36]), thus, demonstrating the capability of synchrotron X-rays to probe domain reorientation in small volumes. From the quantified XRD data there was a 20% increase in the volume fraction of *c*-domains in the parallel-to-field direction at 20 V, which was attributed to the PZT film being released from its underlying substrate. This change in volume fraction is on the order of volume fraction changes seen in other diffraction based ferroelectric bulk studies and work done on {001} textured partially released tetragonal PZT films. The large amount of domain reorientation seen in this PZT cantilever provides evidence that the removal of the underlying substrate, which is used to grow the film, facilitates domain reorientation response within films incorporated within piezoMEMS devices. This work also highlights that synchrotron XRD is a useful and powerful characterization tool for interrogating physical mechanisms underpinning the electromechanical response of small length scale ferroelectric materials used in piezoMEMS.

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