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Mechanical stresses in lead zirconate titanate thin films formed on substrates differing in temperature coefficients of linear expansion

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Abstract. The article considers the influence of linear and bending stresses on the magnitude of the internal field and self-polarisation in thin lead zirconate titanate (PZT) films formed on silicon and glass-ceramic ST-50 substrates by radio-frequency magnetron sputtering. PZT composition corresponded to the region of the morphotropic phase boundary. It is assumed that bending stresses in "thin PZT film-substrate" bimorph structures lead to the appearance of an internal electric field caused by the diffusion of charged oxygen vacancies (Gorsky effect in ferroelectrics).

Keywords: thin ferroelectric films, lead zirconate titanate, morphotropic phase boundary, mechanical stresses, internal electric field, self-polarisation, Gorsky effect

Introduction

In the last decade, thin ferroelectric films have been increasingly used in various microelectromechanical systems (MEMS), not only as sensors, actuators, acoustic devices, infrared (IR) sensors (Izyumskaya et al. 2007; Muralt 2001; Polla 1995), but also in magnetoelectric devices, harvesters (energy microstorage), microwave converters, etc. (Bukharaev et al. 2018; Eerenstein et al. 2006; Kang et al. 2016; Ma et al. 2021; Muralt 2008). The vast majority of MEMS application of ferroelectric films (about 95%) is associated with the use of lead zirconate titanate (PZT) thin films, the composition of which corresponds to the morphotropic phase boundary (MPB) region, where the electromechanical, pyroelectric, and other important physical parameters of the films reach extremely high values (Song et al. 2021).

From a technological point of view, it is most efficient to use the so-called self-polarised films, which, as a result of synthesis, are characterised by macroscopic polarisation comparable in magnitude to spontaneous polarisation (Afanasjev et al. 2001; Bruchhaus et al. 1999; Kholkin et al. 1998; Sviridov et al. 1994). However, it is by no means always possible to obtain such films, and the reason for this is the effect of mechanical stresses on them from the side of the substrate. Since, in practice, the majority of used polycrystalline PZT films are formed on platinised silicon substrates and characterised by one or another growth texture, the appearance of mechanical stresses is usually associated with the difference in the temperature coefficients of linear expansion of the film and the substrate (Bruchhaus et al. 1999; Ogawa et al. 1991; Pronin et al. 2003). In this case, the question of the discrepancy between the lattice parameters of the film and the substrate (or the conducting sublayer) can be excluded from consideration.

Moreover, in a number of works it is assumed that mechanical stresses are the main reasons leading to the formation of microscopic polarisation (Bursian et al. 1969; Delimova et al. 2021; Garten, Trolier-McKinstry 2015; Gruverman et al. 2003; Sviridov et al. 1994; Yudin, Tagantsev 2013). An analysis of the literature discussing the role and influence of mechanical stresses on the properties of thin PZT films showed that, firstly, it is necessary to separate the effect of linear and bending mechanical stresses on the orientation of the ferroelectric polarisation, and, secondly, the degree of their influence can vary depending on the number of factors, such as film deposition temperature, annealing temperature (synthesis temperature of the perovskite phase), degree of film porosity, one or the other growth texture, presence of sublayers used and their thickness, etc. The lack of unambiguity and clarity in assessing the contribution of mechanical stresses to the occurrence and magnitude of self-polarisation in thin PZT films determined the aim of this study.

Objects and methods of study

The comparison of physical properties was carried out on polycrystalline PZT films formed on substrates with different temperature coefficients of linear expansion. Their composition corresponded to the MPB region. Thin films were prepared using a two-stage method. At the first stage, they were deposited on substrates using the method of radio-frequency (RF) magnetron sputtering of a PZT ceramic target; at the second stage, they were annealed in air at temperatures of 550–600 °C (Pronin et al. 2010). Thin wafers of silicon and glass-ceramic ST-50 were used as substrates. At room temperature, the temperature coefficients of linear expansion of these substrates and the investigated PZT solid solution are 2.8×10^{-6} K^{-1}, 5.0×10^{-6} K^{-1} and 3.0×10^{-6} K^{-1} respectively.

According to our calculations (Fig. 1), at room temperature, in the "PZT-silicon" bimorph structure tensile stresses act on the thin film from the side of the substrate, while in the "PZT-glass-ceramic" bimorph structure compressive stresses act. The thickness of the studied films was 500–1000 nm. A continuous layer of the lower platinum electrode 100–200 nm thick was formed on the surface of the substrates. To study the ferroelectric properties, contact pads of platinum 200 × 200 μm in size were deposited on the surface of the formed films.

To study the dielectric polarisation (P-V curves), a modified Sawyer-Tower measuring circuit was used. An automated setup based on an E7-20 immittance meter made it possible to measure the reversible dependences of sample capacitances (C-V curves) at bias voltages in the range of ± 40 V.

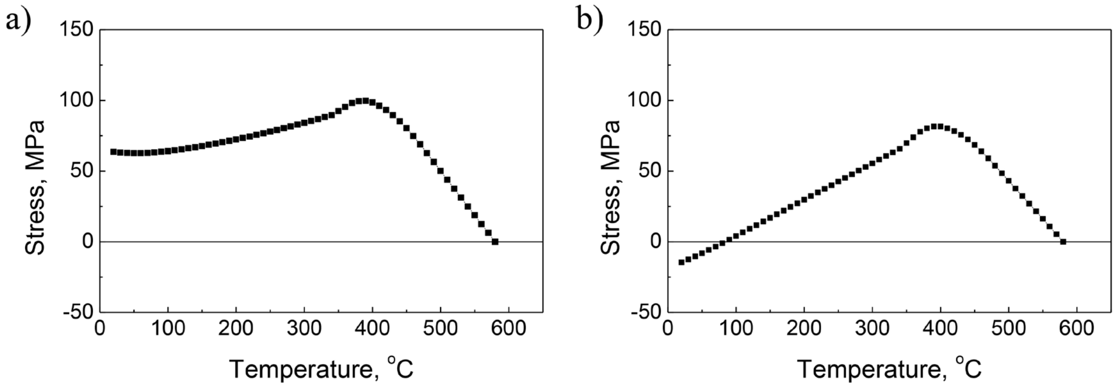

Fig. 1. Calculated temperature dependences of mechanical stresses arising in $PbZr_{0.5}Ti_{0.5}O_3$ (PZT) films deposited on (a) silicon and (b) glass-ceramic substrates (negative stress values correspond to film compression)

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Influence of linear mechanical stresses

The nature of mechanical stresses (compressive or tensile) arising in thin polycrystalline (ceramic) films depends on the difference in the temperature coefficients of the thin film (α_f) and the substrate (α_{sub}). In the "PZT-silicon" structure, where the composition of the film corresponds to the MPB region (i. e., with the ratio $\alpha_{PZT} > \alpha_{Si}$), two-dimensional tensile stresses, if they are sufficiently high, orient the spontaneous polarisation vector in the direction as close as possible to the substrate plane (Fig. 1a, Fig. 2a). In the "PZT-glass-ceramic" structure (i. e., at $\alpha_{PZT} < \alpha_{glass}$), compressive stresses reorient the ferroelectric polarisation in a direction close to the normal to the substrate surface (Fig. 1b, Fig. 2b).

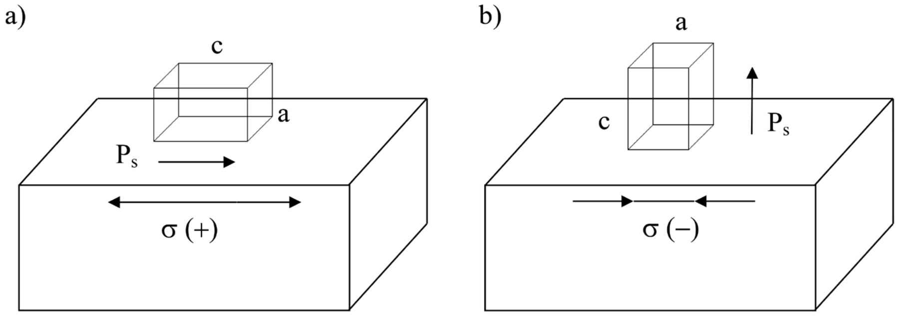

Fig. 2. Diagram illustrating the effects of mechanical tension (σ+) (a) and compression (σ-) (b) with sides of the substrate on deformation of the unit cell and the orientation of the polar axis in <100>-textured tetragonal PZT film

Calculations of the magnitude and sign of mechanical stresses (Pronin et al. 2003) showed that tensile stresses of ~60 MPa act on the film from the side of the silicon substrate (Fig. 1a), while small compressive stresses act on the side of the glass-ceramic substrate.

A similar approach can be extended to films that differ in growth texture. Fig. 3 shows the changes in the value of the macroscopic polarisation relative to spontaneous polarisation in PZT films with a change in the lattice symmetry in the MPB region from the tetragonal to the rhombohedral modification under conditions of tension (for the case of a silicon substrate, Fig. 3a) or compression (for the case of a glass-ceramic substrate, Fig. 3b). It is clear that the effect of two-dimensional stresses on the change in macroscopic polarisation differs radically with a change in the growth orientation of a thin film.

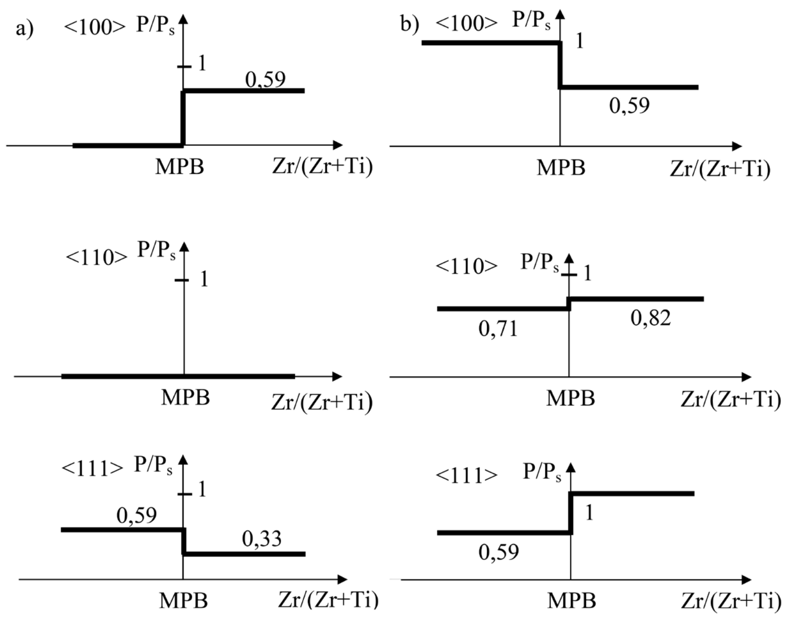

Fig. 3. Diagram of the self-polarised state (in relative units) in the MPB region for <100>, <110> and <111>—oriented thin PZT films formed on silicon (under tension) (a) and glass-ceramic (under compression) (b) substrates

In the "PZT-glass-ceramic" structure, a thin film is in a state of compression, and the macroscopic polarisation can reach the value of spontaneous polarisation depending on the growth orientation and the elemental ratio (Zr/Ti), corresponding to the tetragonal (T) or rhombohedral (Rh) modification of the ferroelectric phase. In the "PZT-silicon" structure, tensile mechanical stresses of the order of 60 MPa are quite enough to reorient the polarisation in the direction as close as possible to the plane of the substrate, and a jump corresponds to the transition from the tetragonal to the rhombohedral phase at MPB region, leading to a decrease in the self-polarisation value to unacceptably low values for practical applications in (111)-oriented films (Fig. 3a) (Pronin et al. 2003).

In a real situation, the magnitude and orientation of macroscopic polarisation is determined by the ratio of mechanical and electrical forces acting on a thin film. Fig. 4 shows the hysteresis loops of thin PZT films of composition corresponding to the MPB region formed on silicon and glass-ceramic (Fig. 4b) substrates. It is clear that the asymmetry of the hysteresis loop is more pronounced in the film formed on the glass-ceramic substrate than on the silicon one. While in the second case the value of the internal field was 48 kV/cm and the value of self-polarisation was 0.7 of the value of the spontaneous polarisation, in the first case the internal field reached only 28 kV/cm and the value of self-polarisation did not exceed 0.4 of the spontaneous polarisation. The difference in self-polarisation values reflects the effect of tensile stresses on the film from the side of silicon.

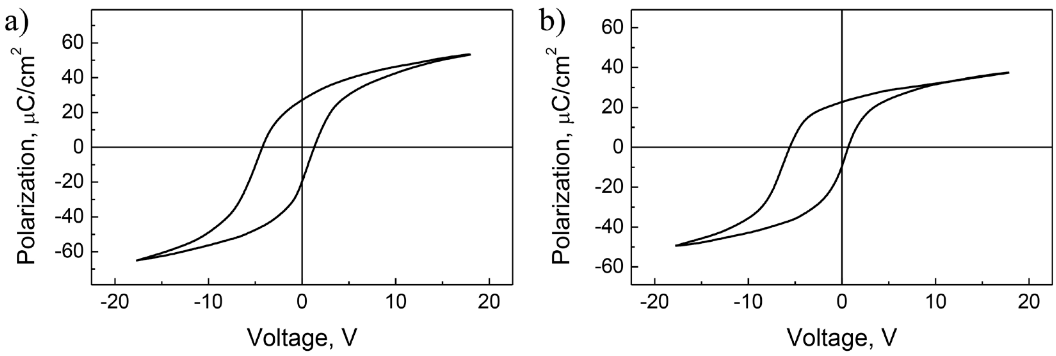

Fig. 4. P-V loops of thin PZT films deposited on (a) silicon and (b) glass-ceramic substrates.
Films thickness—500 nm, sintering temperature—550 °C

Influence of bending mechanical stresses

Despite the fact that the main reason for the appearance of internal field and self-polarisation in thin PZT films is considered to be the charging of the lower interface of the thin-film structure (Afanasjev et al. 2001; Kholkin et al. 1998), the effect of bending mechanical stresses cannot be ruled out. It is well known that, under the action of a gradient of mechanical stresses in the crystal lattice, atoms shift relative to each other, where larger atoms are displaced in the direction of extension and small atoms—in the opposite direction, causing the appearance of an internal field and polar dipoles (Bursian et al. 1969). Subsequently, the effect was called the flexoelectric effect (Gruverman et al. 2003; Yudin, Tagantsev 2013).

Experiments revealed that in the case of strong bending stresses (at a curvature radius of ~30 cm) a change in the sign of the mechanical stress gradient leads to a reorientation of the macroscopic polarisation. Such effects were observed on thin plates of barium titanate ($BaTiO_3$) (Bursian et al. 1969) (Fig. 5) and in thin PZT films (Gruverman et al. 2003). Moreover, a polar state can also arise in thin nonpolar layers (Ehre et al. 2007). In this regard, the flexoelectric effect is still considered one of the probable causes of an internal electric field and the self-polarisation associated with it (Delimova et al. 2021). Estimates of the polarising electric field performed for thin PZT films showed that the magnitude of such fields is several orders of magnitude lower than the coercive field. Therefore, the authors conclude that there is another, stronger contribution to the flexoelectric effect (Gruverman et al. 2003).

One of such possible contributions can be the effect associated with the directed diffusion of charged vacancies, better known in relation to the multidirectional diffusion of atoms in metal alloys as the Gorsky effect (or the upward diffusion effect) (Gorsky 1935). The essence of the effect is that under action

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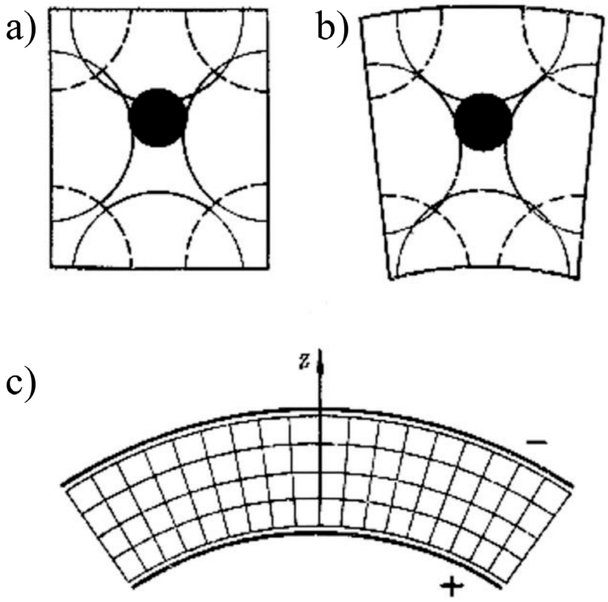

Fig. 5. Distortion of elementary cells of barium titanate under external electric field application (a) in an infinite crystal; (b) and (c) in a thin plate, leading to a flexoelectric effect

of mechanical stresses gradient, atoms with a larger ionic radius move to the region of sample tension, while atoms with a smaller radius move to the region of compression. This effect is reversible: when the external load is removed, the concentration of point defects is equalised over the sample. Subsequently, A. M. Kosevich extended the effect to directed diffusion of vacancies (Kosevich 1975). As applied to thin oxide ferroelectrics, we can talk about the diffusion of charged oxygen vacancies (Barbashov, Komysa 2005; Pronin et al. 2017).

In this connection, a comparative study of the aging of thin PZT films formed on silicon and glass-ceramic substrates was carried out in this work. The studied samples were initially characterised by asymmetry of C-V characteristics and P-V loops, that is, they were characterised by the presence of an internal field and self-polarisation. Then the capacitor thin-film PZT structures were subjected to high-temperature annealing above the Curie temperature (at ~ 400 °C) and then aging within 10^6 s at room temperature.

High-temperature annealing of samples usually leads to symmetrisation of hysteresis loops (P-V), which is associated with a symmetrical redistribution of mobile charge carriers (electrons) between the upper and lower interfaces of the structure (Okamura et al. 1999; Pronin et al. 2002a). The experiments showed that the films formed on the glass-ceramic substrate had practically symmetrical P-V characteristics even after aging (Fig. 6b). The situation was different in a film formed on a silicon substrate. In that case aging led to the appearance of an internal field, the averaged over the film thickness magnitude reached 5–7 kV/cm (Fig. 6a). It corresponded to the appearance of a surface charge of ~0.4 μC/cm^2.

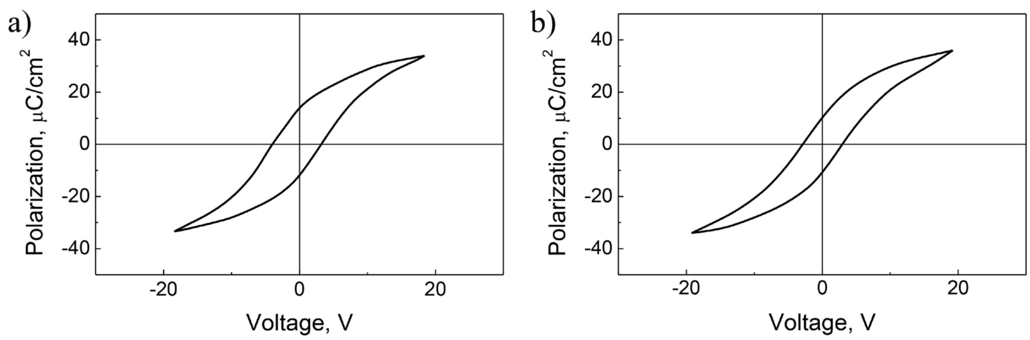

Fig. 6. P-V loops of thin PZT films formed on (a) silicon and (b) glass-ceramic substrates after annealing and subsequent aging

We assumed that the reason for the appearance of the field is the directed diffusion of charged oxygen vacancies towards the upper interface of the structure, which is caused by bending of the "PZT-silicon" bimorph structure due to the difference in the temperature coefficients of linear expansion of the thin film and the silicon substrate (Fig. 7), in contrast to the "PZT-glass-ceramic" structure, in which the action of two-dimensional mechanical stresses on a thin film is insignificant at room temperature. The calculation of the radius curvature of the structure using the Stoney formula (Bruchhaus et al. 1999) showed that its value is several orders of magnitude larger (~130 m) than in the case of the flexoelectric effect. With such a bending, the diffusion of oxygen vacancies occurs in the direction of structure compression, i. e., in the direction of the free surface of the film, and the orientation of the internal field occurs in the direction of the substrate.

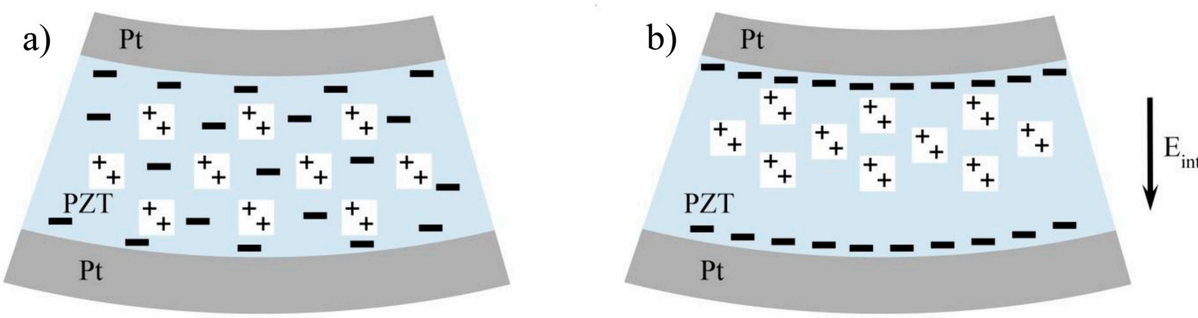

Fig. 7. Schematic representation of the space charges distribution in a thin PZT film after annealing above the Curie temperature (at ~ 400 °C) (a) and aging (b). The arrow shows the orientation of the internal electrical field vector (E_{int})

Assuming the charge is accumulated due to the diffusion of charged oxygen vacancies, the minimum required volume concentration of charged oxygen vacancies in a thin PZT film with a thickness of 1 μm should be no less than ~ 10^{17}/cm^3 under the assumption that all the vacancies present in the bulk of the film are concentrated near the upper interface of the structure. Taking into account that the activation energy of oxygen vacancies is ~ 0.22 eV (Pronin et al. 2002b), it is necessary to make a correction for the fraction of charged vacancies, which at room temperature are approximately two orders of magnitude smaller than uncharged vacancies. Taking this into account, the required concentration of vacancies (N^v) must be no less than ~ 10^{19}/cm^3, i. e., a value quite admissible for thin PZT films. Taking into account that the value of the diffusion coefficient of oxygen vacancies in a thin PZT film at room temperature is not so high (its estimates in the literature give the value $D_v \sim 5 \times 10^{-16}$ cm^2/s) (Holzlechner et al. 2014), and not all vacancies have time to diffuse during the experiment towards the upper interface, the real value of N^v should be increased by another order of magnitude.

Conclusion

The paper compares the action of linear biaxial mechanical stresses acting on a thin PZT ferroelectric film from the side of substrates, in which the integral temperature coefficient of linear expansion is either higher (glass-ceramic substrate ST-50) or lower than that of thin PZT films (silicon substrate), whose composition corresponds to the MPB region.

It is shown that the use of a silicon wafer as a substrate leads to stretching of the PZT film and partial reorientation of the ferroelectric polarisation vector in the direction as close as possible to the plane of the substrate. At the same time, this leads to bending of the "PZT-silicon" bimorphic structure and creates conditions for the directed diffusion of charged oxygen vacancies and the formation of an internal electric field, i.e., to the Gorsky effect in ferroelectric structures.

It is shown that, at a sufficient concentration of oxygen vacancies in a thin PZT film, bending stresses can lead to formation of an internal field and the resulting self-polarised state.

For a more thorough study of the Gorsky effect and a quantitative assessment of its contribution to the formation of an internal field and self-polarisation in thin-film ferroelectrics, it is necessary to further study the diffusion of oxygen vacancies at temperatures above room temperature and take into account

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the contribution to the bending of the “PZT-silicon substrate” structure from such sublayers as silicon dioxide, an adhesive layer of titanium (or titanium oxide), as well as platinum electrodes (lower and upper).

Conflict of interest

The authors declare that there is no conflict of interest, either existing or potential.

Author contributions

The authors have made an equal contribution to the preparation of the text.

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