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LIGHT INFLUENCE ON NANOMECHANICAL CHARACTERISATION OF PLZT CERAMICS

WPŁYW ŚWIATŁA NA CHARAKTERYSTYKI NANOMECHANICZNE CERAMIKI PLZT

The relaxor ferroelectrics materials are continuously attracting considerable attention due to interesting combination of their mechanical, electrical and chemical properties and their application in microelectromechanical devices (MEMS and HOEMS). To be more specific, their excellent electrostrictive and photostrictive properties coupled with domain mobility and good stability made these "new materials" important component of electroceramics MEMS platforms. Concerning fabrication of these systems the real dilemma for industry stems from mechanical properties of the component in ultra-high stresses that arise in microelectromechanical applications and high residual stress that is created during production process.

It is already known that mechanical behaviour of these ferroelectric materials largely depends on random elasto-electric fields connected with the defects distribution and diffusion of defect charges driven by applied external stress. The aim of this paper is an investigation of light induced mechanical anisotropy for polycrystalline and highly photostrictive PLZT ceramics.

We demonstrate that mechanical testing can be effectively used for drawing decisive applications conclusions from the hardness and elastic modulus distribution. Technological modification of ferroelectric materials significantly affect mechanical properties. Particularly in PLZT ceramics we observed light influenced significant changes in values of hardness and Young Modulus as well as a friction coefficient.

Keywords: relaxor ferroelectrics; MEMS; mechanical properties; nanoindentation

Relaksory ferroelektryczne są wciąż przedmiotem intensywnych badań naukowych ze względu na interesującą kombinację ich mechanicznych, elektrycznych i chemicznych własności znajdujących zastosowanie w mikromechanicznych urządzeniach (MEMS i HOEMS). W szczególności ich wysokie parametry elektrostrykcyjne i fotostrykcyjne w połączeniu z dużą ruchliwością domen ferroelektrycznych i wysoką odpornością na zmęczenie ferroelektryczne stanowią o ich wysokiej pozycji w aplikacjach MEMS. Przy długoczasowych aplikacjach niezwykle istotnym stają się dobre własności mechaniczne.
Powszechnie wiadomo, że mechaniczne charakterystyki materiałów ferroelektrycznych w dużej mierze zależą od przypadkowych pół elektrycznych i naprężeń mechanicznych powiązanych z rozkładem defektów i dyfuzją ładunków defektowych spowodowanych przez naprężenia mechaniczne. Celem tego artykułu jest wyjaśnienie wpływu warunków technologicznych na twardość mechaniczną ceramiki PLZT.

1. Introduction

The improvement of diverse physical properties has already been demonstrated for variety of nanophase and nanocomposite ceramics obtained in the past decade. Consequently, the development of advanced ceramics encountered nowadays is frequently limited to a proper combination of a specific electrical but also mechanical properties, that should provide a required complex characteristics of final material. The PLZT based ceramics is exhibiting excellent transducers and actuator properties but very interesting fact is that, values of these coefficients are significantly dependant on the light intensity. Additionally very promising is that, in actuator applications, the wireless light induced actuating is achievable (photostrictive effect) [1-5]. Nevertheless, the mechanical properties of above mentioned materials appear essential for its long term reliability in micromechanical applications [6].

In effective actuators structure the PLZT is used mainly in form of disks or thin fibres, which radius is of several hundreds micron. Considering the optimal efficiency, there is a great need of precise characterization of these PLZT elements given light induced anisotropy with reference to size effect caused by photostrictive material expansion. Consequently, there were several effort

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to determine theoretically light induced change in properties [7 - 8] but little experimental work has been done on the mechanical testing or investigations. Particularly light induced change of hardness and elastic modulus under light illumination remains still not investigated and these mechanical properties have to be clarified.

The photoconductivity of ferroelectrics is the basis of number of phenomena involving the effect of nonequilibrium carriers on the ferroelectric properties and the phase transitions. It is necessary to know the influence of photoconductivity on electrical parameters connected with filing of local energy levels but also on the material and mechanical properties. From the thermodynamical considerations the photodeformation effect follows as a result of the effect of electrons on the spontaneous polarisation, that is meant the influence of nonequilibrium electrons on deformations of the ferroelectrics. In these materials photodeformation has positive sign, goes to zero near the Curie point and is not observed in paraelectric region.

The aim of this paper is to present the results of the complex nanoindentation measurements of the PLZT material that enable us to determine the hardness, Young's modulus and light induced anisotropy in this interesting structure.

In order to investigate this interesting material and avoid brittle ceramics drawbacks we focused on nanoindentation measurements which is capable to record the complex surface-interior response in nanoscale. The latter method due to the tips of nanometric range, allows us to measure hardness, elastic modulus and detect defects in the central areas of the grains.

2. Experimental

The mixed oxide method was employed for the of PLZT ceramics fabrication. Commercially available PbO, ZrO_2, TiO_2, La_2O_3 powders were used as starting materials for ball mill grinding process. The weighted powders for PLZT (3/52/48) composition were mixed for 16h with zirconia balls as the grinding media and ethanol as the solvent. After milling, the resultant powders were dried in an oven and then calcined in a high-temperature furnace at 900 °C for 1 h. A custom-designed stainless steel die was used to obtain 10-mm in diameter and 1 mm in height disks and finally sintered in the uniaxial hot pressing method (HP) in the ambient atmosphere [10-11].

The nanoindentation tests were performed at constant room temperature with the TriboScope® nanoindenter (Hysitron Inc., Minneapolis, Minnesota), a complete system package includes hardware, electronics and software. The nanomechanical properties of the PLZT disks, namely, the reduced elastic modulus and hardness, were evaluated from the load-displacement nanoindentation data using the widely accepted Oliver and Pharr method [11]. The Triboscope AFM interface enable us to image the sample, choose the indent location and reflect tests surfaces giving quantitative, reliable and reproducible results. The disks were wet-grounded on a sandpaper (grade of 1200#) and subsequently polished on soft cloth with diamond paste with particle size of 1μm.

The well calibrated Berkovich tip with 150 nm radius, was used to analyse the mechanical properties of PLZT ceramics. The test were performed in darkness and in 720 nm laser diode (PMMF-201P-RD PHOTONIC SYSTEMS) illumination for comparison. For nanometric photostriction measurement the nanoindenter set up was impemented (resolution of 0,5 nm). The indentation load Pmax ranged from 100 to 1000 μN, while the applied loading/unloading rates was equal 10 μN/s and there were performed at least 100 indents for one experimental diameter.

Tribological characteristics of friction coefficients for PLZT ceramics were obtained from10μm-long scratches using a spherical diamond indenter at loads in the range of 50 to 600?N which are controlled by the vertical force transducer. A second transducer connected to the "floating" plate of the vertical force transducer records lateral or frictional forces through the entire length of the scratch. Friction coefficients (μ) were calculated from the quotient of lateral to normal forces for each tissue and plotted versus lateral displacement.

3. Results and discussion

The example of indent pattern and indent depth profile is shown in Fig. 1. Before starting and after finishing the projected contact area of the tip was calibrated using indentation into the standard specimen of fused quartz. The perfectiveness of the tip shape was verified before and after measurements of each specimen and the appointed difference before and after analyses on fused quartz was under the value of standard division, i.e. error bar. The nanomechanical properties, viz. the reduced modulus and nanohardness, were directly evaluated from the load-displacement curve obtained by the nanoindentation.

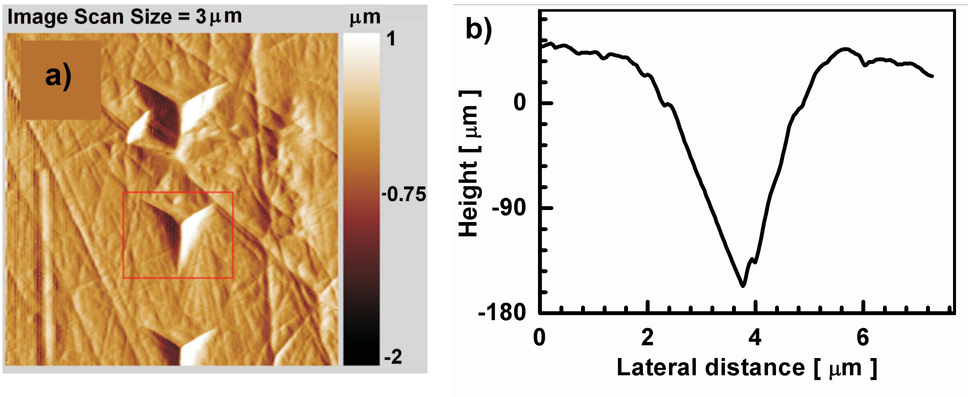

Fig. 1. The indent pattern image (a) and depth profile (b) of PLZT ceramics

The dynamic of propagation of the surface movement under light illumination connected with anisotropic domain mobility in rhombohedral twinning system was shown in the Figure 2. The measured shift distance of the polycrystal surface in the direction [001] was of the range of 15 nm. Surprisingly, the time constant distribution of rhombohedral twinning and polycrystal slip is rather slow and the whole relaxations process takes about 25 s.

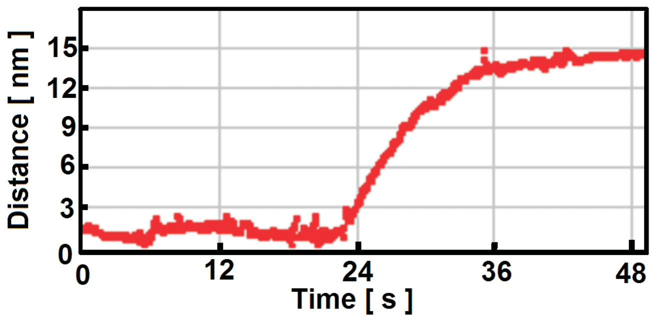

Fig. 2. The dynamics of photostrictive effect of the surface PLZT ceramics

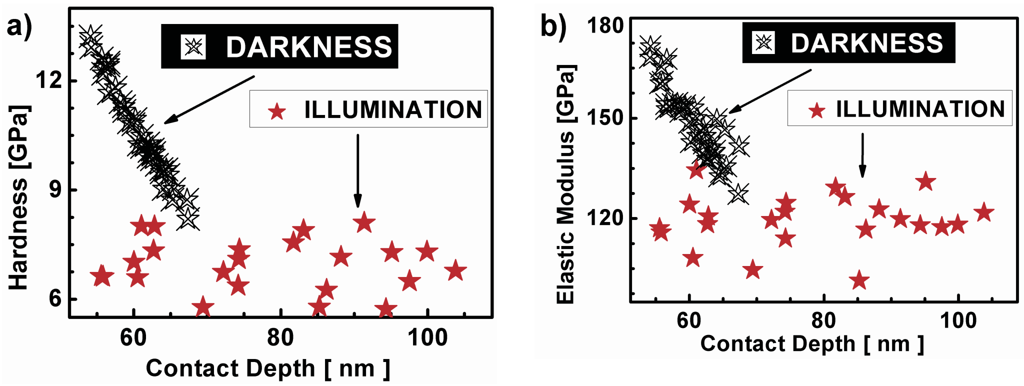

Fig. 3. Hardness(a) and modulus plots(b) of dark (black colour) and illuminated (red colour) PLZT ceramics

The light influence on the mechanical characteristics at PLZT surface, is given by means of nanoindentation method. These experiments confirmed hardness and elastic modulus anisotropy of the samples (Fig. 3). The drop in mechanical parameters is connected with resolved shear stress resulting as a dislocation source denotes increasing distance from the nearest dislocation piled-up to the adjacent grain. The above approach reflects our understanding that the significant drop and scattering of hardness and elastic modulus values, that occurs under light illumination, is associated with the contribution of non-basal slip and grain deformation. The latter is believed to be governed in room-temperature by domain movement and change of the value of polycrystal lattice and orientation factor related to the number of slip surfaces (see Fig. 3 and 4).

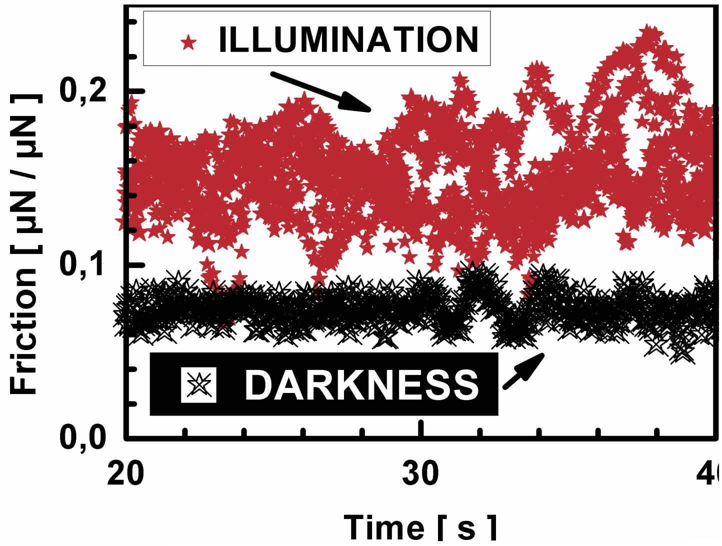

Fig. 4. Friction coefficient of dark and illuminated PLZT ceramics surface

As we expected, after the hardness measurements, the light illumination is influenced the tribological characteristics of PLZT surface given by means of scratch test. These experiments similarly confirmed friction coefficient anisotropy connected with higher domains and grains mobility. In the Figure 4 are presented two well separated friction coefficients distributions: higher and more scattered values of friction coefficient for illuminated sample and the lower level one, more concentrated, from the sample measured in darkness conditions. The statistical average value of friction coefficient changes from 0,11 to 0,21 from dark to illuminated sample respectively. We can interpret resulting difference as a light induced increasing elongation and mechanical strain in PLZT ceramics. The above approach confirmed our understanding that the significant increase in friction coefficient, that occurs under light illumination, is associated with the contribution of non-basal slip and material deformation. The latter is believed to be governed in room-temperature by grains slip, which reduces the value of orientation factor related to the number of slip systems (see Fig. 4).

4. Conclusions

In the present study we successfully confirmed light induced mechanical anisotropy for polycrystalline and highly photostrictive PLZT ceramics.

The values of hardness and elastic modulus decrease significantly under light illumination for polycrystalline PLZT (3/52/48) ceramics and decrease is in proportion to increasing light intensity. We explained resulting difference as a light induced deformation and mechanical strain in PLZT ceramics. The above approach reflects our understanding that the significant drop and scattering of hardness and elastic modulus values, that occurs under light illumination, is associated with increasing distance to the nearest grain and resulted slips. The latter is believed to be governed additionally by domain movement and change of the parameters of polycrystal lattice.

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