National Conference on "Recent Trends in Mathematical Modeling, Simulation Methods, Computations, and Physical Sciences" (MMSCP-2019), Organised by Hislop College, Nagpur, Maharashtra, India
International Journal of Scientific Research in Computer Science, Engineering and Information Technology
© 2019 IJSRCSEIT | Volume 4 | Issue 4 | ISSN : 2456-3307

Treatment of Ferroelectric Fatigue by Removal of Localized Impurities Structures in Lead Meta Niobate Single Crystal

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ABSTRACT

The present work provides an available way of enhancing optical quality of surface and trim down the fatigue properties of the ferroelectric lead meta niobate ($PbNb_2O_6$) single crystal, which will be helpful to use this material as ferroelectric storage device. This study provides a quantitative basis for imaging the local polarization dipoles at microscopic resolution, which are helpful for the investigation about domain wall motion under the application of electric field.

I. INTRODUCTION

Bulk ferroelectric domain inversion and differential etching processes have been used for fabricating the surface microstructures due to dependence of the etch rate and three fold y-direction symmetry in various ferroelectric materials [1]. The performance of many electro-optical devices is strongly based on stability of switchable ferroelectric polarization modes which involves the nucleation and growth of domain structures on crystal-surface under an external electric field, along with better optical quality of topography of crystal [2, 3]. The present article is an attempt to address such type of mechanism concerning domain wall nucleation and impurity-ion dipole interactions in achieving the preferred optical-quality at the surface of ferroelectric lead meta niobate ($PbNb_2O_6$) single crystal. The main objective of this study is to explore the crystal's surface with good optical ability, which will be helpful to eliminate the ferroelectric fatigue and localized behavior of impurities in $PbNb_2O_6$ single crystal at microscopic level. For improvisation of the optical quality of surface of $PbNb_2O_6$ single crystal, the domain wall nucleation and etching process is applied with external electric field, and underlying principle and mechanism of this technique is discussed with the evidences of surface micrographs of crystal.

II. MATERIALS AND METHODS

The single crystals of lead meta niobate ($PbNb_2O_6$ or PN) grown from melt by employing Goodman's technique in a slightly modified way [4]. The dried constituent oxides in molar composition 1:1 (22.3190gm of PbO and 26.5810gm of Nb_2O_5, analytical grade with 99.4% purity purchased from MERCK) grounded together and packed in to a 50cc platinum crucible. Programmable Gallenkamp furnace is set to melt the materials in crucible at temperature 1623ºK for sufficient soaking time of about twelve hours. To avoid stray nucleation in crystal-growth process, a cooling and reheating process was performed in a suitable rate manner, through a program set in this furnace. It is observed that the obtained $PbNb_2O_6$ crystals are of pale yellow in color, oxygen deficient and containing platinum ions as main impurity due to use of platinum crucible. By XRD-pattern of this grown material, the single orthorhombic phase at room temperature and lattice parameters with point group are calculated, while its ferroelectric nature and phase transition temperature (570ºC) is confirmed through hysteresis study [5].

The micro-structure surfaces of grown lead meta niobate ($PbNb_2O_6$ or PN) single crystal studied under reflection by microscope of METZER Company. The domain structure and its evolution during nucleation and polarization reversal processes has been investigated which offer full and direct information on the static and dynamic properties of this grown ferroelectric PN single crystal. The improvised optical quality of surface of PN crystal is observed through surface micrographs after the successive application of external electric field in forward/reverse direction followed by etching with dilute ammonium nitrate [$(NH_4)_2NO_3$], and microscopic changes in domain structures are discussed in the next section.

III. RESULTS AND DISCUSSION

Generally, the stabilized impurities in the form of dipole, cracks as the dislocation type of defects, roughness exponent of charges, space charges, grain boundaries, oxygen vacancies etc. are the realistic issues that always hindered the visibility of movements of domain walls, and consequently, are believed to be the causes of fatigue properties in ferroelectric materials. Domain wall dynamics, grain size effect on domain-transition, predominance of crystal-defects under external electric field have already been reported by us through the study of surface-micrographs for this ferroelectric $PbNb_2O_6$ single crystal [6, 7]. Less twinned and thin flake of about 0.1mm $PbNb_2O_6$ crystal is chosen for improvement of optical quality of its surface. The viewing direction is along the crystallographic [110] direction in Figure 1, where several impurity-segregation can be easily seen in the form of clusters with 90º and 180º domain walls, marked by arrows. Here, the impurities exist in the form of dipoles (made with nearby vacancies) stabilized themselves by forming micro-domains around the dipole in the crystal structure. The task to remove such type of impurities found difficult as they form dipole-clusters by interacting with electrons freed from the vacancies. Moreover, their behavior can be considered as relaxed dipoles shifting from their ionic states, getting more stabilized by accommodated themselves in various octahedral-voids in the structure when crystal was cooled in the growth process. Although, the impurity-ions (relaxed dipoles) are not visible in Figure 1, the impurity-aggregates and dislocation in the form of cracks present good picture of impurity-content on the surface of the $PbNb_2O_6$ crystal under study.

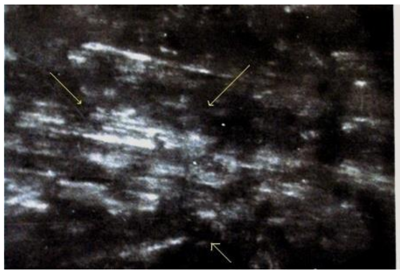

Figure 1. Surface of $PbNb_2O_6$ single crystal without application of electric field and etching.

The same piece of $PbNb_2O_6$ crystal whose surface is depicted in Figure 1, poled with *d.c.*electric field of 5000V/cm for twenty minutes. Figure 2 is the viewed surface of $PbNb_2O_6$ crystal after polling with such electric field of 5000V/cm in which the nucleation of fresh micro-domains of about (4-6) μm around the impurity-content can easily be observed, under the circle. It is pointed out here that the unfavorably oriented relaxed-dipoles in the cluster shown in Figure 1 are diffused in hopping process after the application of electric field, and remaining constituent freed charges are taking part in the nucleation of fresh micro-domains what are appeared in Figure 2. In this way, the relaxed dipoles are reexcited from their ionic state to dipolar state. However, the anti-phase boundaries and the morphology of 90º domain walls, which influence the nucleation of new domains and the mobility of domains, and attributed the physical mechanism for polarization fatigue in ferroelectric $PbNb_2O_6$ crystal.

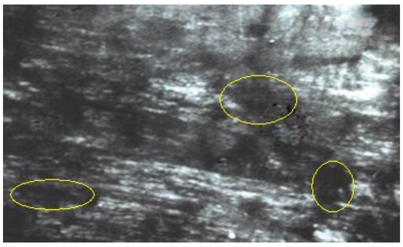

Figure 2. Surface of same piece of $PbNb_2O_6$ crystal after application of electric field 5000V/cm.

Further, the *d.c.*electric field of 15000V/cm in the *reverse direction* is again applied to same piece of $PbNb_2O_6$ crystal for only four minutes and its surface is shown in Figure 3. It can be observed in Figure 3; that the previously nucleated domain walls (Which were present in Figure 2.) are now evaporated from the viewing surface in Figure 3. This has happened because a large number of dipoles are being freed in this evaporation process of domains. Thus, by the application of forward and reverse electric field, the relaxed-dipoles are continuously excited and de-excited from their ionic state to dipolar state and vice-versa; subsequently a sufficient deposition of impurity-content at the surface of PN crystal can be seen in Figure 3.

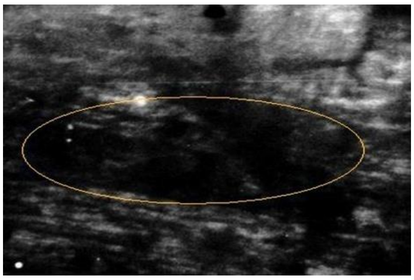

Figure 3. Surface of same piece of $PbNb_2O_6$ crystal after application of electric field 15000V/cm.

This accumulated deposition of impurities is now removed by washing with dilute ammonium-nitrate [$(NH_4)_2NO_3$] and micrograph of washed surface of $PbNb_2O_6$ crystal is shown in Figure 4. Surface residues after etching were removed by post-etching cleaning solution. We can easily see the improvement of optical quality of surface of $PbNb_2O_6$ single crystal by comparing the Figure 1.and Figure 4. This whole process what is performed from Figure 1 to Figure 4 can be further repeated again and again to achieve better surface quality of crystal.

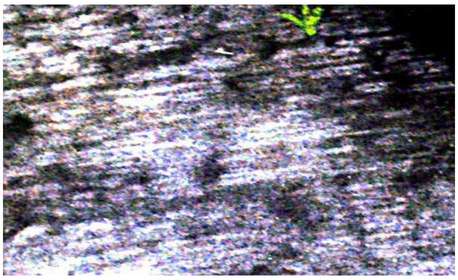

Figure 4. Surface of same piece of $PbNb_2O_6$ crystal after etching by Ammonium Nitrate.

Summarily, the whole process which has been discussed above is the attempt to reduce the ionic displacement consequential from the change of the lattice parameter resulted in octahedral distortion, improving the ferroelectric fatigue of the $PbNb_2O_6$ single crystal.

IV. CONCLUSION

In conclusion, a strong correlation found between the properties of ferroelectric fatigue and the behavior of localized impurity structures in ferroelectric lead meta niobate single crystal. Localized impurity structures are removable by a simple and low priced technique which involved etching and domain wall nucleation under the application of applied electric field to get better optical quality of the surface; it also confirms that lattice-defects can affect the crystal structure and width of domain walls.

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