Journal of Alloys and Compounds 696 (2017) 166–170

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Nanoscale insight into the domain structures of high Curie point $Pb(In_{1/2}Nb_{1/2})O_3$-$PbTiO_3$ single crystal

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A R T I C L E I N F O

Article history:
Received 6 September 2016
Received in revised form
17 November 2016
Accepted 19 November 2016
Available online 19 November 2016

Keywords:
Ferroelectric domains
Piezoresponse force microscopy
High *Tc*
Relaxor single crystal

A B S T R A C T

Recently high-Curie point relaxor ferroelectric single crystals represented by the binary (1-x)$Pb(In_{1/2}Nb_{1/2})O_3$-$x$$PbTiO_3$ (PINT, PINTx) and ternary $Pb(Mg_{1/3}Nb_{2/3})O_3$–$Pb(In_{1/2}Nb_{1/2})O_3$-$PbTiO_3$ (PMN-PIN-PT) attracted much attention. In order to given an insight into the domain structures for these high Curie temperature T_c single crystals, in this work, PINT single crystal, grown by a modified Bridgeman method, was studied by piezoresponse force microscopy (PFM). Results indicated that the PINTx single crystal exhibited strip-like long-range-order ferroelectric domain structures at room temperature with x across the morphotropic phase boundary ranging from 0.28 to 0.42. The electric-field induced domain evolution was performed to give an insight into the local poling behavior for rhombohedral compositions PINT0.28 and PINT0.34 (with giant piezoelectric strain response). The temperature-dependent PFM results indicated that the PINT single crystal exhibited good temperature stability in nanoscale and clear domain patterns could still be observed above the T_c.

1. Introduction

Relaxor based ferroelectric single crystals, represented by $Pb(Mg_{1/3}Nb_{2/3})O_3$–$PbTiO_3$ (PMNT) and $Pb(Zn_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ (PZNT), have attracted much attention due to their superior piezoelectric and electromechanical properties and they are being actively considered for next-generation high-performance sensors and transducers [1–4]. However, several disadvantages of this family of piezoelectric crystals notably limited their application to date. During applications the binary PMNT single crystals exposed the disadvantages of relatively low coercive field (2–3 kV/cm) and depolarization temperature (usually less than 80 °C for PMNT with the composition at the morphotropic phase boundary (MPB)) [5]. The temperature rise may easily lead to the deterioration of the piezoelectric sensors and transducers which is undesirable.

In order to further improve the global performance of PMNT single crystals, high Curie point binary crystal (1-x)$Pb(In_{1/2}Nb_{1/2})O_3$-$x$$PbTiO_3$ (PINT, PINTx) and ternary relaxor $Pb(Mg_{1/3}Nb_{2/3})O_3$-$Pb(In_{1/2}Nb_{1/2})O_3$-$PbTiO_3$ (PMN-PIN-PT) single crystals were developed [6–8]. These crystals exhibited substantial improvements in the temperature stability and coercive fields while sustaining superior piezoelectric and electromechanical performances. High performance piezoelectric devices were also demonstrated recently based on these high Curie temperature ferroelectric crystals [9,10]. In order to further extend their applications, more fundamental research is quite necessary to give an insight into the structure and domain evolution.

It is well known that the domain structure and its evolution under external conditions play an important role as an extrinsic contribution for the piezoelectric response. Studies on the domain evolution could provide an important insight into the macroscopic response. In previous work, the domain study on PMNT crystals has been well investigated with hierarchical domains on various length scales [11–15]. In this study, we aim to study the composition and temperature dependence of the nanoscale domain structures of high Curie point PINT single crystals. Piezoresponse force

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http://dx.doi.org/10.1016/j.jallcom.2016.11.247

microscopy (PFM) was utilized here to characterize the nanoscale electromechanical properties and local polarization switching behavior. The PINT*x* crystal with *x* ranging from 0.28 to 0.42 were studied. The electric-field induced domain evolution was performed to give an insight into the local poling behavior in the rhombohedral PINT*x* compositions. Temperature-dependent PFM results indicated the PINT single crystal exhibited good temperature stability in nanoscale slightly above the Curie temperature.

2. Experimental

The high-quality PINT*x* single crystals were grown directly from the melt by the Bridgman technique [6]. According to the phase diagram of PINT*x* proposed in previous works, here four representative compositions *x* of 0.28, 0.30, 0.34, 0.42 were chosen, which was rhombohedral phase for PINT*x* with $x < 0.37$ and tetragonal structure for PINT0.42 [6]. The as-grown ingot was oriented along <001> direction using an X-ray diffractometer and then cut into plates with typical size of $5 \times 5 \times 0.5$ mm^3. The room-temperature ferroelectric hysteresis loops and strain curves were measured using a ferroelectric analyzer along with a laser interferometer (TF2000, Aixacct, Aachen, Germany). The ferroelectric domain was characterized by PFM (MFP-3D, Asylum Research, USA). For PFM characterization, the samples were carefully polished using polycrystalline diamond with different sizes. The root mean square (RMS) roughness of all the samples was less than 3 nm. The PFM measurements were performed in the dual AC resonance tracking (DART) PFM modes. This technique could enhance contrast in the amplitude and phase image and reduce the topographical crosstalk while mapping the local electromechanical properties [16]. Conductive cantilevers ASYELE-01 were used with tip coating of Ti/Ir (5/20). The nominal spring constants were 2 N m^{-1} with a fundamental resonance frequency of the free tip-vibration (non-contact resonance) of approximately 70 kHz. All the PFM images were taken at an ac voltage of 3 V.

3. Results and discussion

Fig. 1 shows the room-temperature composition dependence of the topography, out-of-plane (OP) PFM amplitude and phase images of four PINT*x* samples with *x* of 0.28, 0.30, 0.34, 0.42. In the PFM phase images, the dark contrast areas represented domains with downward polarization orientation, while bright contrast represented upward direction. From the phase images shown in Fig. 1, obvious domain patterns characterized by the clear contrast could be observed in all the compositions. The local piezoelectric hysteresis also exhibited 180° phase change, confirming the ferroelectric characteristics of the compositions. From Fig. 1, regular strip-liked domain patterns could be observed in the PINT0.28, PINT0.30, and PINT0.34 single crystals, which all located in the rhombohedral region at room temperature from the phase diagram [6]. The three compositions exhibited similar domain widths of about 0.5 μm and domain lengths of tens of microns. Nonsmooth domain boundaries and partial polar nanodomains (PND) could also be detected. These results were quite different from the PMNT single crystals with *R* phase (*x* from 0.20 to 0.30, located in the rhombohedral region) which exhibited wave-like self-assembled domain patterns and irregularity by PFM [11,12]. Also the composition *x* increasing in PMNT single crystal could induce the substantial increasing of the domain size and domain regularity while the composition *x* increasing from 0.28 to 0.34 in PINT single crystals had little effect on the domain patterns. This should be

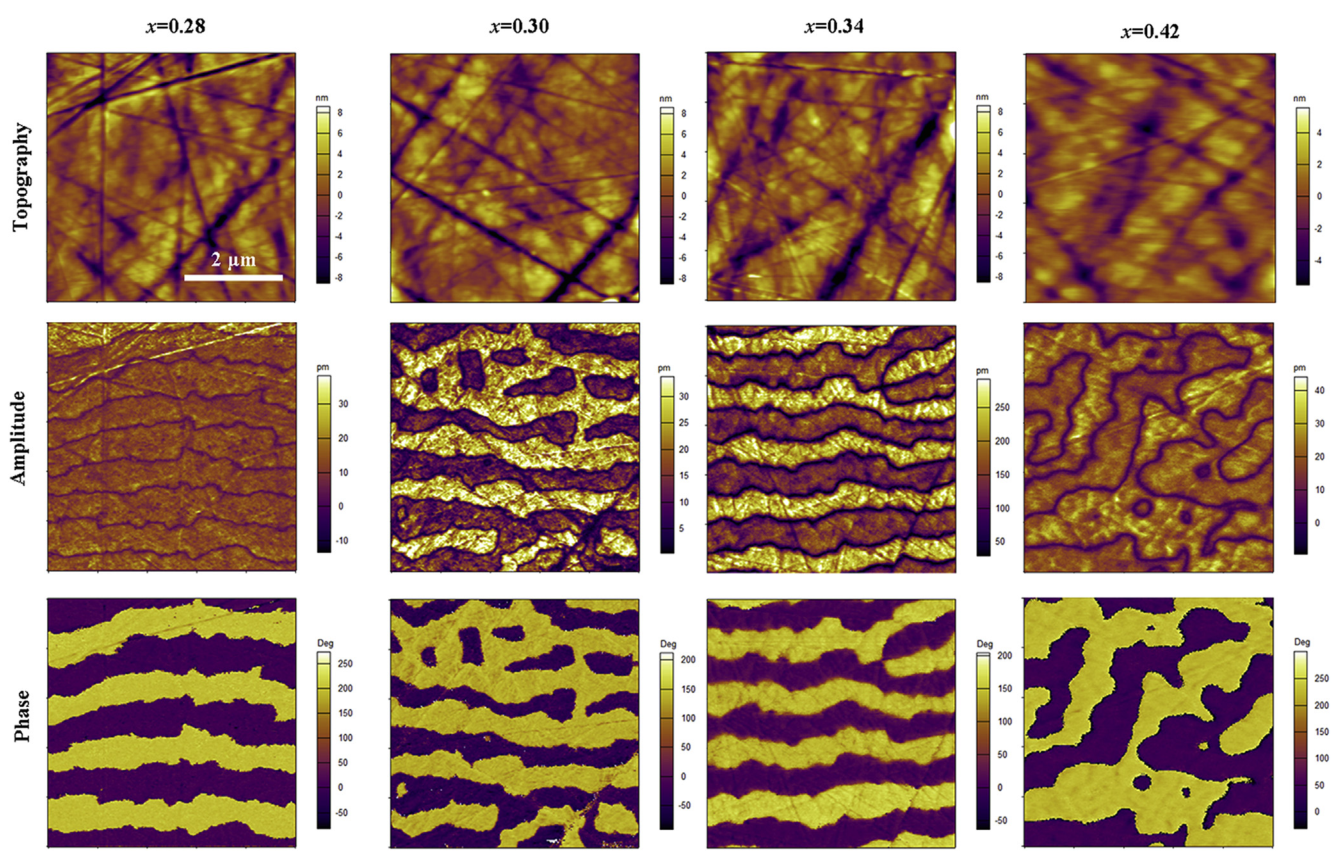

Fig. 1. The composition dependence of the topography and domain structures of PINT*x* with *x* from 0.28 to 0.42 (the OP PFM images were shown here).