INVESTIGATION OF DOMAIN STRUCTURES IN $LiNbO_3$ SINGLE CRYSTALS GROWN BY CZOCHRALSKI METHOD

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Abstract

Lithium Niobate ($LiNbO_3$) single crystals, grown by Czochralski method at the congruent composition, have ferroelectric microdomains. These microdomains were investigated by chemical etching with hydrofluoric acid (HF) and nitric acid (HNO_3), and by using optical microscopy, scanning electron microscopy and atomic force microscopy.

Introduction

$LiNbO_3$ single crystal, obtained by the Czochralski method as a ferroelectric material, is applied to nonlinear optical, piezoelectric and surface acoustic wave (SAW) device. It has an acentric, uniaxial crystal structure, and ferroelectric domains reflecting inversion of the crystal structure occur in opposite directions along the c-axis. These domain forms possess unusual pyroelectric, piezoelectric, nonlinear optical, etc. properties.

Experimental Procedure

The starting materials were high purity (99.99 wt%) Li_2CO_3, Nb_2O_5. These powders were mixed at congruent composition (48.6 mol% Li_2O), and were re-

acted at 950℃ for 12hrs to remove CO_2 gas and to get $LiNbO_3$ phase. The $LiNbO_3$ mixture was melted in a Pt crucible and single crystal was grown by Czochralski method at 3.5mm/hr in air. After grown, $LiNbO_3$ crystal cut in slice form at Z-axis and optical-quality polished z-plate. To investigate multidomain of $LiNbO_3$ single crystal, chemical etching was done with hydrofluoric acid (HF) and nitric acid (HNO_3) at 110℃ for 10, 20, 30sec, 1, 2, 3min

The mulidomain structures (etch pit and hillock) were observed by optical microscopy, scanning electron microscopy. Atomic force microscopy was used to investigate reverse-polarized domain.

Result and Discussion

Fig.1 shows etch pit for various etching times. After 2min etching, many etch pits appear on negative surface. Fig.2 are scanning electron microscopy micrographs of etch pits, hillocks.

Fig.3 are AFM images of hillocks and etch pits on reverse-polarized domain and estimated cores of hillocks. $LiNbO_3$ single crystal grown by Czochralski method, mixed negative and(or) positive domain before poling. It is considered that multidomains are origined by thermal distribution, local stress, water vapor and atmosphere gases. Fig.4 shows micrograph of cross-sectional view. It appears reverse-polarized domains.

Conclusion

Etching of $LiNbO_3$ (0001) crystal surface by etchant much more rapidly than of the oppositely polarized (0001) crystal surface, etch pits and hillocks corresponding to the location of microdomain form on the +Z and -Z surface.

Initial shape of hillocks cores was observed to core-like, but with increased etching, it should needle-like shapes before disappearing.

From SEM and AFM image, the diameters of microdomain cores can be measured from 200 to 250 ㎚.

Reference

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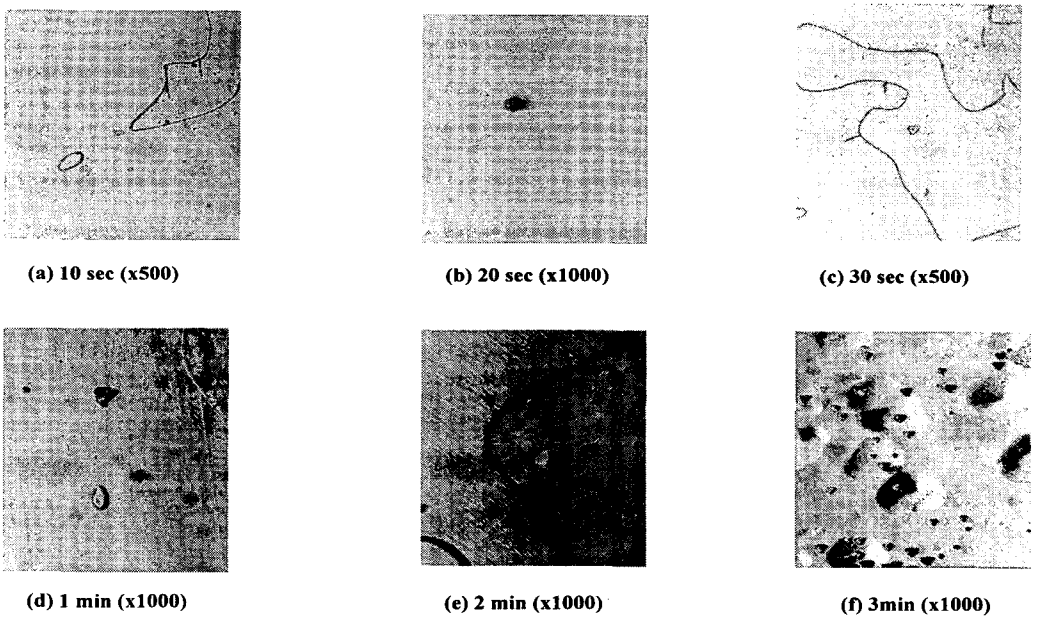
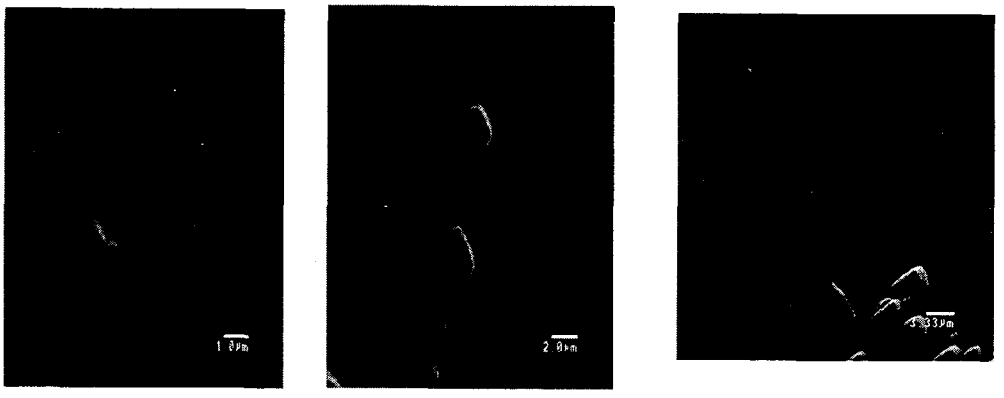


Fig.1 patterns of etch pits



(a) Etch pit (b) hillocks (c) etch pit & hillocks (45 ° Tilt)

Fig. 2 SEM micrographs after etching

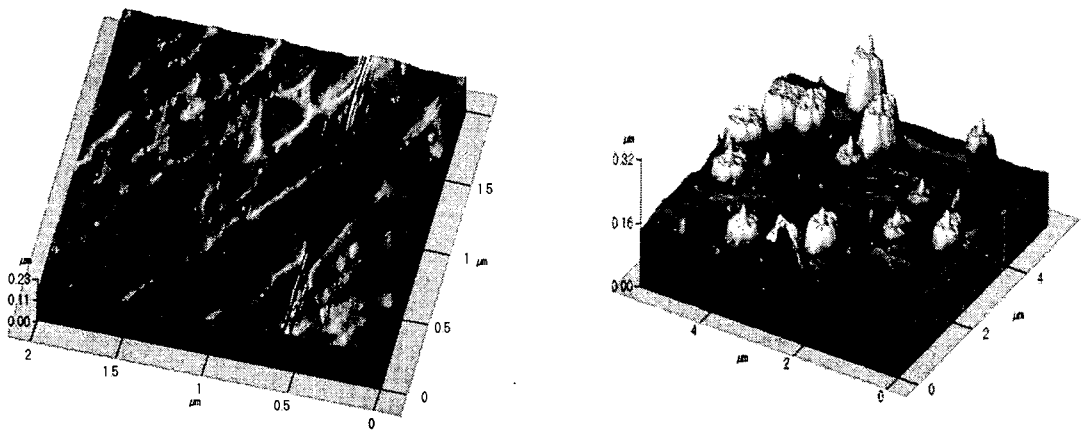


Fig.3 AFM image

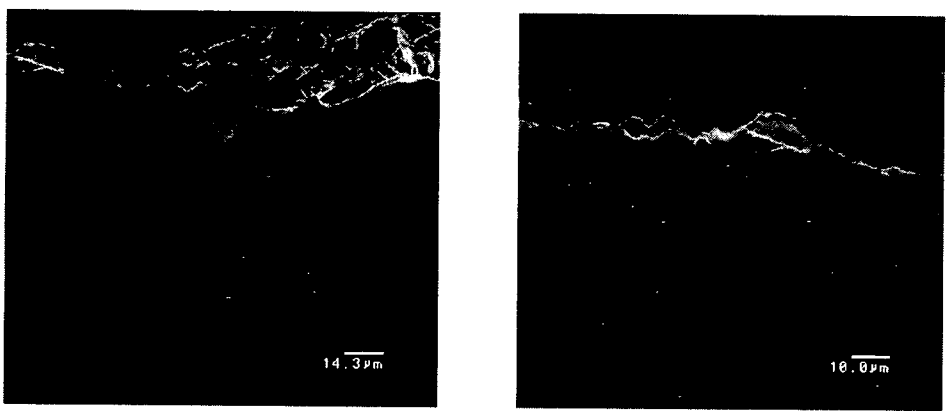


Fig.4. SEM micrographs (cross-sectional view)