THE GROWTH OF LOW DISLOCATION DENSITY $\text{Sr}_{1-x}\text{Ba}_x\text{Nb}_2\text{O}_6$ CRYSTALS

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The dislocation structures of both pure and Nd doped strontium barium niobate crystals, grown by the Czochralski method, were studied using an etch pit technique. It was determined that dislocations in the boule were being propagated from the seed and were confined to the center of the crystal. Typical dislocation density was $5 \times 10^4 \text{ cm}^{-2}$. Through the careful control of growth parameters and use of seed material cut from the dislocation free outer portion of a crystal, it was possible to grow crystals with very low dislocation densities, $1 \times 10^2 \text{ cm}^{-2}$, and on occasion dislocation free crystals.

Key words: dislocation, electro-optic, etch pit, pyroelectric, single crystal, $\text{Sr}_{1-x}\text{Ba}_x\text{Nb}_2\text{O}_6$.

Introduction

Single crystal $\text{Sr}_{1-x}\text{Ba}_x\text{Nb}_2\text{O}_6$ (SBN) has shown promise as an electro-optic material [1], and both pure and rare-earth doped SBN have been shown to be suitable pyroelectric detector materials [2,3]. Such applications place demanding requirements on material quality. Defects in these materials, either structural or stoichiometric, can affect device performance.
The effect of structural defects on the properties of single crystal Si and Ge and the influence of growth conditions on such defects in these materials is well known (4,5). Therefore, in an effort to gain insight into how the properties and quality of SBN crystals are influenced by growth conditions, a study of their dislocation structure was made using an etch pit technique.

Experimental Procedure

Both pure and Nd doped SBN crystals were grown by the Czochralski method which was described in an earlier paper (6). All crystals were grown in the [001] direction, with a planar liquid-solid interface, in air, and had a Sr:Ba ratio of 1. The Nd modification was made by adding 1 weight percent of Nd$_2$O$_3$ to the melt.

After growth the crystals were prepared for etching. Using a wire saw, 1mm thick slices were cut from the crystals both perpendicular and parallel to the [001] growth axis. The slices were then polished. One micron diamond paste was used to achieve the final surface finish. In addition to the polished surfaces a pair of mating fracture surfaces was made by breaking a crystal perpendicular to the growth axis. Mirror image etch pit patterns on the fracture surfaces would then establish the validity of the etch. The etching reagent was an aqueous solution of 49% hydrofluoric acid used at room temperature as reported by Ito and Furuhat (7).

Results and Discussion

Figure 1 shows two typical crystals of the sort that were grown for these experiments. The crystals are 4-5mm in diameter. The upper crystal contained Nd and appeared pale lavender. The crystals were grown under identical, and as can be seen from their morphology, well regulated conditions.