

# The Influence of Oxygen on Czochralski Growth of Oxide Single Crystals

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## ABSTRACT

When growing the oxide single crystal including Li-ion, optimum oxygen condition is needed. Color and crack are caused in single crystal according to the change in the condition of the oxygen.  $\text{LiTaO}_3$  crystals grown from off-composition of congruent melt composition under oxygen deficiency condition generated dark color and crack.  $\text{LiNbO}_3$  crystals grown from congruent melt composition under oxygen deficiency condition didn't generate any crack.  $\text{LiNbO}_3$ ,  $\text{LiTaO}_3$  crystals grown from congruent melt composition under optimum oxygen condition caused pale yellow color or colorless with no crack. Color gradually became colorless and generated cracks according to Oxygen excess.

## 1. INTRODUCTION

One of the difficulties in growing the oxide single crystals is a defect formation in the crystal related to the growth atmosphere. Generally speaking, oxide crystals are well grown in air condition. But this say is not the correct concept because reduced atmosphere should be done as crucible material like iridium and a crystal is often degraded by color center defects. The scope of this report is to study the influence of Oxygen on Czochralski Growth of Oxide Single Crystals, especially on well known  $\text{LiNbO}_3$ ,  $\text{LiTaO}_3$ .

## 2. EXPERIMENTS

### 2.1 $\text{LiNbO}_3$ single crystal growth

To grow  $\text{LiNbO}_3$  single crystals, temperature gradient from above the melt surface was lowered by installing the after heater. For the diameter control, load cell installed to the pulling head senses the instant weight and the personal computer controls all the crystal growth parameters. For raw material, 48.38mole% and 48.52mole<sup>(1)</sup>%  $\text{Li}_2\text{CO}_3$  using  $\text{Li}_2\text{CO}_3(99.99\%)$ ,  $\text{Nb}_2\text{O}_5(99.99\%)$  were chosen. oxygen conditions to grow crystals were dry air, oxygen deficiency(dry air+N<sub>2</sub>), oxygen excess(dry air+O<sub>2</sub>). Mixed material were proceeded through weighing, mixing, calcinating process and melted in the Pt-crucible(8 OD x 8H, cm) by rf-heating. And the crystal growth was done from the 128<sup>o</sup>Y-axis seed by

rotating and pulling process.

## 2.2 LiTaO<sub>3</sub> single crystal growth

For growing LiTaO<sub>3</sub> single crystals, 48.58mole%, 48.65mole<sup>(2)</sup>%Li<sub>2</sub>CO<sub>3</sub> using Li<sub>2</sub>CO<sub>3</sub>(99.99%), Ta<sub>2</sub>O<sub>5</sub>(99.99%) were chosen as the raw material. Growth oxygen conditions were N<sub>2</sub>+O<sub>2</sub>(2%below), N<sub>2</sub>+O<sub>2</sub>(2%–5%;vol.), O<sub>2</sub>+N<sub>2</sub>(5%over). Mixed material were proceeded through weighing, mixing, calcinating process and melted in the Ir-crucible(8 OD x 8H,cm) by rf-heating. And the crystal growth was done from the Y-axis seed by rotating and pulling process as that of LiNbO<sub>3</sub>.

## 3.RESULTS AND DISCUSSIONS

Fig.1 shows the full grown LiNbO<sub>3</sub> single crystals. The color was caused dark yellow color and the crack was not generated in case of the oxygen deficiency condition, yellow color and clear in case of the air condition and pale yellow color and crack was generated in case of the oxygen excess condition. fig.2 shows the full grown LiTaO<sub>3</sub> single crystals. The color was caused dark yellow color, the crack was generated due to oxygen deficiency and off-congruent composition in case of the N<sub>2</sub>+2%below oxygen condition, pale yellow color and clear in case of the N<sub>2</sub>+(2%–5%)oxygen condition and colorless and crack in case of the N<sub>2</sub>+5%above oxygen excess condition.

## 4.CONCLUSIONS

When growing the oxide single crystal including Li-ion, optimum oxygen condition is needed. Color and crack are caused in single crystal according to the change in the condition of the oxygen. LiTaO<sub>3</sub> crystals grown from off-composition of congruent melt composition under oxygen deficiency condition generated dark color and crack. LiNbO<sub>3</sub> crystals grown from congruent melt composition under oxygen deficiency condition didn't generate any crack. LiNbO<sub>3</sub>, LiTaO<sub>3</sub> crystals grown from congruent melt composition under optimum oxygen condition caused pale yellow color or colorless with no crack. Color gradually became colorless and generated cracks according to Oxygen excess.

## REFERENCES

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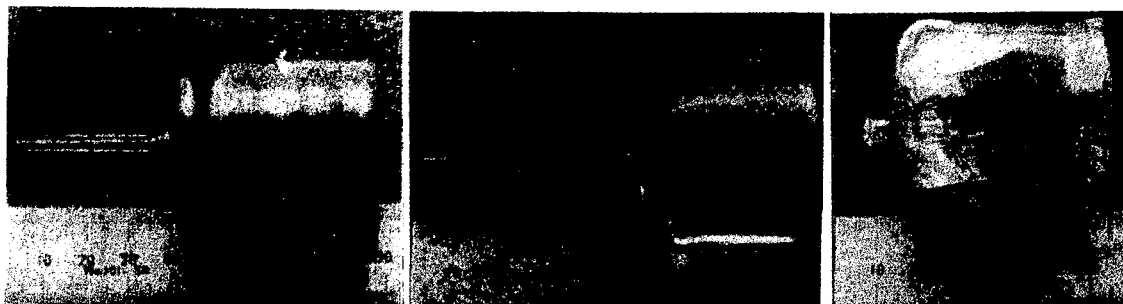


Fig.1 LiNbO<sub>3</sub> single crystal growth

(a) 48.38mole%Li<sub>2</sub>CO<sub>3</sub> oxygen deficiency(dry air+N<sub>2</sub>)

(b) 48.52mole<sup>(1)</sup>%Li<sub>2</sub>CO<sub>3</sub> dry air

(c) 48.52mole%Li<sub>2</sub>CO<sub>3</sub> oxygen excess(dry air+O<sub>2</sub>)

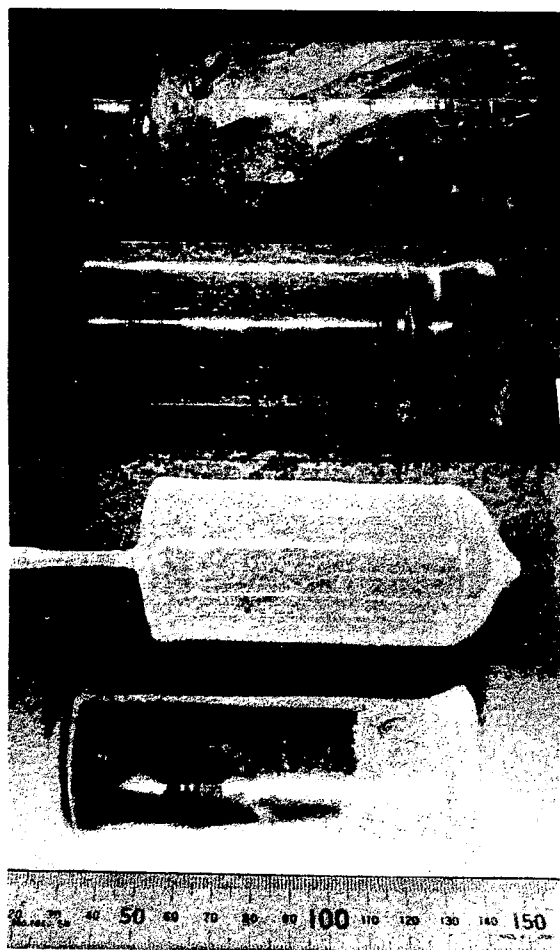


Fig.2 LiTaO<sub>3</sub> single crystal growth

(a) (48.58mole%Li<sub>2</sub>CO<sub>3</sub>N<sub>2</sub>+O<sub>2</sub>(2%below)

(b) (48.65mole<sup>(2)</sup>%Li<sub>2</sub>CO<sub>3</sub>)N<sub>2</sub>+O<sub>2</sub>(2%below)

(c) (48.65mole%Li<sub>2</sub>CO<sub>3</sub>)N<sub>2</sub>+O<sub>2</sub>(2%–5%,vol.)

(d) (48.65mole%Li<sub>2</sub>CO<sub>3</sub>)O<sub>2</sub>+N<sub>2</sub>(5%over)