

## Development of small ultrasonic probe using KNbO<sub>3</sub> piezoelectric thin films

KNbO<sub>3</sub> 圧電結晶薄膜を用いた高周波超音波プローブの開発

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### 1. Introduction

Hydrothermal method has a number of advantages as piezoelectric material for ultrasonic transducers; low deposition temperature, high-purity, deposition on a three-dimensional structure, and a large thickness. Therefore, hydrothermally deposited Lead Titanate Zirconate (PZT) polycrystalline films were reported for ultrasonic transducers material. However, breakdown field strength of the hydrothermally PZT polycrystalline films was poor, hence, for high frequency operation such as 20MHz or over, it is difficult to make a generation of ultrasound using the hydrothermally PZT polycrystalline films<sup>1)</sup>.

On the other hand, for a measurement of remanent polarization, a hydrothermally epitaxial KNbO<sub>3</sub> thin-film deposition has been reported<sup>2)</sup>. As the reported result, the epitaxial KNbO<sub>3</sub> thin-film shown promising for high threshold electric field intensity. Therefore, in this study, we tried fabrication of an high frequency ultrasonic probe using the KNbO<sub>3</sub> epitaxial film by hydrothermal method and investigated ultrasonic transmitting and receiving properties which are yet to be revealed.

### 2. Experimental Procedure

The KNbO<sub>3</sub> thick films were grown at 240 °C on (100)<sub>c</sub> SrRrO<sub>3</sub> // SrTiO<sub>3</sub> substrates by the hydrothermal method. The (100)<sub>c</sub>-oriented SrRrO<sub>3</sub> layers used for bottom electrodes were epitaxially grown on the (100) SrTiO<sub>3</sub> substrates by a sputtering method<sup>3)</sup>. An autoclave (PARR, 4748) that contained an inner vessel made of Teflon to resist high alkali solutions was utilized for the hydrothermal growth. A 20 ml solution of 10 mol// KOH (Kantokagaku) and 1.0 g of niobium oxide powder (Nb<sub>2</sub>O<sub>5</sub>, purity 99.95%, Kantokagaku) were used as source materials of K and Nb, respectively. The (100)<sub>c</sub> SrRrO<sub>3</sub> // SrTiO<sub>3</sub> substrate was kept facing down with a Teflon folder in the inner vessel, and the above-mentioned source materials were mixed and placed in the autoclave. The autoclave was shut tight and placed in a constant-temperature

oven (Yamato DS-400) maintained at 240 °C for a hydrothermal chemical reaction.

The thickness of the obtained films grown on (100)<sub>c</sub> SrRrO<sub>3</sub> // SrTiO<sub>3</sub> substrates was determined by a scanning electron microscopy (SEM) and a surface profilometer (Veeco DEKTAK 3ST). The crystal structure and the orientation of the films were characterized by X-ray diffraction analysis using a four-axis diffractometer (HRXRD; Philips X'Pert MRD system) with CuKα<sub>1</sub> radiation. The dielectric and piezoelectric properties were measured using Pt/KNbO<sub>3</sub>/SrRuO<sub>3</sub> capacitors at room temperature; after Au or Pt deposition by evaporation method. The needle-type electrode was connected to the top electrode and the SrRuO<sub>3</sub> that was peeled from SrTiO<sub>3</sub> substrate was grounded through the Ag paste. The dielectric properties and the piezoelectric properties were measured with an impedance analyzer (HP HP4194A). As the transducer of the small ultrasound probe, we used a SUS pipe of 40 mm in length, with a 50 μm - KNbO<sub>3</sub> film set on its top face. The ultrasonic transmitting and receiving properties of the prototype ultrasonic transducer for 20μm-thick hydrothermal KNbO<sub>3</sub> film were measured in degassed water with an ultrasonic Pulser Receiver (Olympus 5910PR). Figure 1 shows the system used to measure transmitting and receiving characteristics of ultrasound.

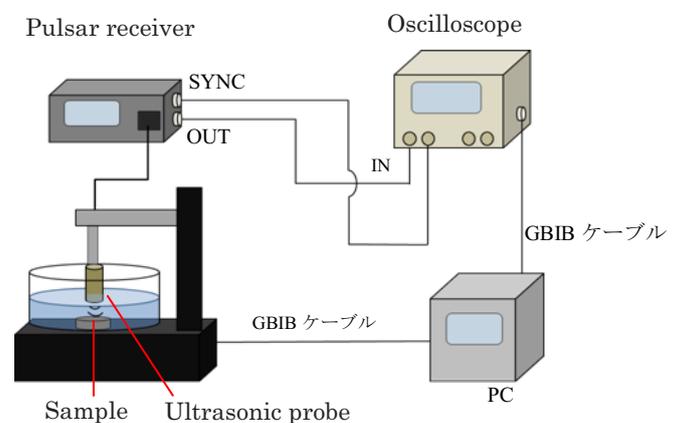


Fig. 1 Measurement system for received ultrasound waveform.

### 3. Results and Discussion

Figure 2 shows a cross-sectional SEM image of 50  $\mu\text{m}$ -thick  $\text{KNbO}_3$  films. Figure 3 shows a logarithmic scale XRD pattern for a 50 $\mu\text{m}$ -thick  $\text{KNbO}_3$  films. Only  $\{100\}_{\text{pc}}$  peaks of the  $\text{KNbO}_3$  were observed, with the exception of the coexistence of small intensity peaks (less than 1% ) of  $\{110\}_{\text{pc}}$  located around  $32^\circ$ . In plane orientation was ascertained by X-ray pole figure measurement, meaning the epitaxial growth of this film. Dielectric constant  $\epsilon_r$  and dielectric loss at 100 kHz were 415 and 0.08, respectively. Our present results indicate that the hydrothermal method enables the excellent  $\text{KNbO}_3$  thick film without any doping or solid solution, which might be related to the low process temperature of the hydrothermal method.

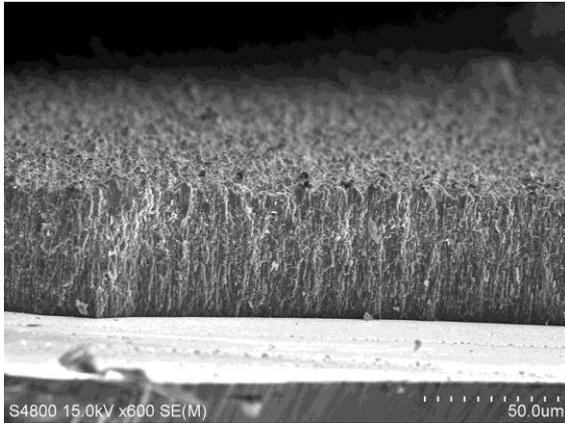


Fig.2 Cross-sectional SEM image of 50  $\mu\text{m}$ -thick epitaxial  $\text{KNbO}_3$  film deposited on  $(100)_c\text{SrRuO}_3//\text{SrTiO}_3$  substrate.

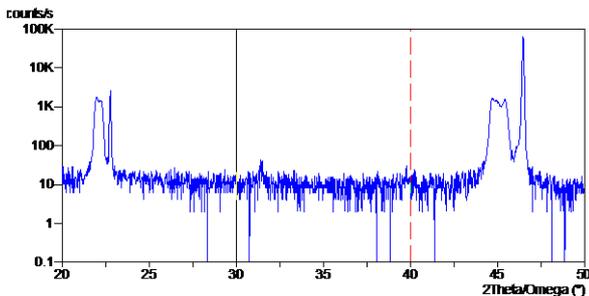


Fig. 3 XRD pattern of  $\text{KNbO}_3$  thick film grown on  $(100)_c\text{SrRuO}_3//\text{SrTiO}_3$  substrate.

Figure 4 shows the receiving voltage waveform resulting from ultrasound transmitting and receiving experiments using the small ultrasound probe in the water. A receiving waveform of I was confirmed 1.3  $\mu\text{s}$  after the transmission. Receiving waveforms of II and III were multiple reflection echo. Moreover two peaks were observed at approximately 40 MHz and 120 MHz in Fig.4 (b). The frequency response was analyzed using Mason's equivalent circuit. According to the result, the 40 MHz peak is excited by thickness mode of piezoelectric effect. This result indicated that the prototype ultrasonic transducer realized the ultrasonic transmitting and receiving at high frequency in the degassed water.

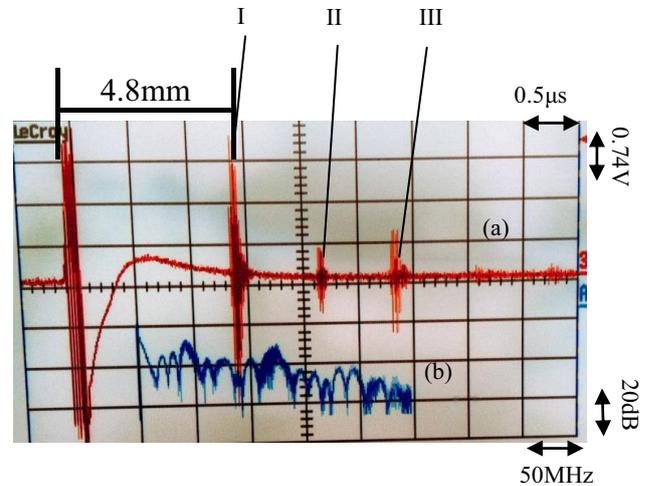


Fig.4 Amplitudes of the received waveform (a) and the power spectrum of the ultrasonic wave form (b).

### 4. Conclusions

The epitaxially-grown 50  $\mu\text{m}$ - $\text{KNbO}_3$  thick films were successfully obtained and the small ultrasonic probe using the hydrothermal  $\text{KNbO}_3$  thick film was able to transmit and receive of the ultrasonic waves over 40 MHz.

### References

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