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# An orientation-controlled $KNbO_3$ thick film transducer for high resolution ultrasonic imaging

## VHF 帯超音波イメージングを目的とした $KNbO_3$ 配向制御膜トランスデューサ

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### 1. Introduction

Piezoelectric films are expected to be used in highly sensitive ultrasonic transducers. The performance of transcuers is directly related to the properties of the piezoelectric film materials.

$KNbO_3$ is a candidate piezoelectric material for each application owing to its excellent piezoelectric properties. However, the films of $KNbO_3$ and $KNbO_3$-based materials have not been widely utilized as transducer materials despite a number of reports describing various deposition techniques. Because it is difficult to deposit $KNbO_3$ films with superior electrical properties. This is related to a potassium deficiency in the films due to the high volatility of potassium during the deposition and/or annealing processes at a high temperature and/or under vacuum conditions. Hence, $KNbO_3$ films with ferroelectric and piezoelectric properties have hardly been reported, especially thicker films above 10 μm have not been reported. It has not yet been possible to obtain thick films, even though the piezoelectric properties and thicker films are important for the design of ultrasonic transducers.

Therefore, we tried to fabricate thick films of $KNbO_3$ using hydrothermal method[1,2] which is escapable for the potassium deficiency. In addition prototype ultrasonic transducer using hydrothermal $KNbO_3$ was fabricated and evaluated as the ultrasonic transmitting and receiving devices.

### 2. Experimental Procedure

The $KNbO_3$ thick films were grown at 240 ºC on $(100)_c$ $SrRrO_3$ // $SrTiO_3$ substrates by the hydrothermal method. The $(100)_c$-oriented $SrRrO_3$ layers used for bottom electrodes were epitaxially grown on the (100) $SrTiO_3$ substrates by a sputtering method[3]. 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 m*l* solution of 10 mol/*l* KOH (Kantokagaku) and 1.0 g of niobium oxide powder ($Nb_2O_5$, purity 99.95%, Kantokagaku) were used as source materials of K and Nb, respectively. The $(100)_c$ $SrRrO_3$ // $SrTiO_3$ 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 ºC for a hydrothermal chemical reaction.

The thickness of the obtained films grown on $(100)_c$ $SrRrO_3$ // $SrTiO_3$ substrates was determined by a scanning electron microscopy (SEM; Hitachi S-4800) 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 $CuK\alpha_1$ radiation. The dielectric and piezoelectric properties were measured using $Pt/KNbO_3/SrRuO_3$ capacitors at room temperature; after Au or Pt deposition by evaporation method. The needle-type electrode was connected to the top electrode and the $SrRuO_3$ bottom electrode was grounded through the Ag paste. The dielectric properties and the piezoelectric properties were measured with an impedance analyzer (HP HP4194A) and a laser Doppler velocimeter (Polytec OFV-3001). The ultrasonic transmitting and receiving properties of the prototype ultrasonic transducer for 20μm-thick hydrothermal $KNbO_3$ film were measured in degassed water with an ultrasonic Pulser Receiver (Olympus 5910PR).

### 3. Results and Discussion

Figure 1 shows a logarithmic scale XRD pattern for a 16μm-thick $KNbO_3$ films. Only $\{100\}_{pc}$ peaks of the $KNbO_3$ were observed, with the exception of the coexistence of small intensity peaks (less than 1% ) of $\{110\}_{pc}$ located around 32º. In plane orientation was ascertained by X-ray pole figure measurement, meaning the epitaxial groeth of this film.

Figure 2 shows the relationship between the piezoelectric strain and the driving electric field versus strain measured at 100 kHz. The effective longitudinal piezoelectric constant, $d_{33}^{\,eff}$, calculated from the linear region indicated in Fig. 2 from 0 to 100 V in the butterfly loops was estimated to be 86 pm/V. Additionally, $\varepsilon_r$ and dielectric loss at 100 kHz were 415 and 0.08, respectively. Our present results indicate that the hydrothermal method enables the excellent $KNbO_3$ thick film without any doping or solid solution, which might be related to the low process temperature of the hydrothermal method.

Figure 3 shows (a) ultrasonic wave form of transmitting and receiving using the prototype ultrasonic probe and (b) its power spectrum. Three peaks were observed at 17 MHz, 95 MHz and 130 MHz. The frequency response was analyzed using Mason's equivalent circuit. According to the result, the 95 MHz peak is exited 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.

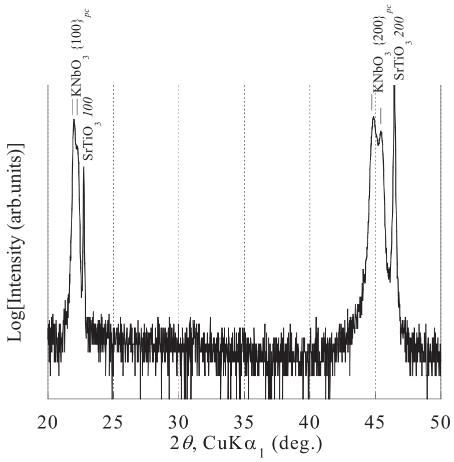


Fig. 1 XRD pattern of $KNbO_3$ thick film grown on (100)c $SrRuO_3$//(100) $SrTiO_3$ substrate.

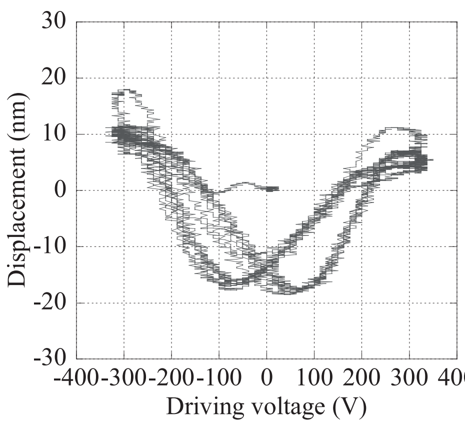


Fig.2 Displacement versus driving voltage butterfly loop of $KNbO_3$ thick film grown on (100)c $SrRuO_3$//(100) $SrTiO_3$ substrate at 100kHz.

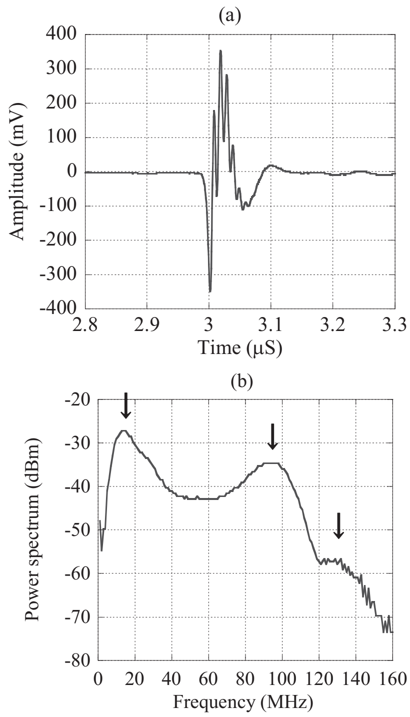


Fig.3 (a) ultrasonic wave form of transmitting and receiving using the prototype ultrasonic probe and (b) its power spectrum.

## 4. Conclusions

$\{100\}_{pc}$-oriented $KNbO_3$ films were successfully grown on $(100)_c$ $SrRuO_3$//$SrTiO_3$ substrates at 240°C by the hydrothermal method. The{100}-oriented $KNbO_3$ thick films showed clear strain butterfly curve originating from the piezoelectricity. $\varepsilon_r$ and dielectric loss at 100 kHz were 415 and 0.08, respectively. The observed piezoelectric constant value, the $d_{33}^{\,eff}$, obtained using a Laser Doppler velocimeter was 86 pm/V. The prototype ultrasonic probe using 20μm-thick hydrothermal $KNbO_3$ film realized the ultrasonic transmitting and receiving at very high frequency bandwidth in degassed water.


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