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Improvement of c-Axis Parallel Orientaition of ZnO film on Silica Glass Pipes with Various Diameters for SH-SAW Pipe Sensor

Takuya Wakabayashi
Doshisha University
Kyotanabe, Japan
ctud1040@mail4.doshisha.ac.jp

Shinji Takayanagi
Doshisha University
Kyotanabe, Japan
stakayan@mail.doshisha.ac.jp

Mami Matukawa
Doshisha University
Kyotanabe, Japan
mmatsuka@mail.doshisha.ac.jp

Yuta Takamura
Doshisha University
Kyotanabe, Japan
bmp1112@mail4.doshisha.ac.jp

Takahiko Yanagitani
Waseda University
Shinjuku, Japan
yanagitani@waseda.jp

***Abstract*— Shear horizontal surface acoustic wave (SH-SAW) sensors have been studied for measurements of liquid properties. The long-distance propagation of SH-SAW with multiple wave roundtrips on a pipe surface improve the measurement sensitivity. c-Axis parallel oriented ZnO films can excite the SH-SAW and be grown on a curved surface by a sputtering deposition. In this study, the ZnO film growths on pipe surfaces with various pipe diameters were demonstrated. The c-axis parallel oriented ZnO film with high orientation was only obtained on the pipe surface facing the sputtering target plane. A slit which limits the deposition area and an automatic rotation system of the pipe were effective for the uniform film growth on the entire pipe surface. This film/pipe substrate structure is a good candidate for the SH-SAW sensor with high sensitivity.**

Keywords*— *ZnO; c-Axis parallel oriented film; Pipe structure; Sputtering deposition; Surface acoustic wave

I. Introduction

Conductivity and viscosity sensors are widely researched in medical and environmental fields [1-2]. Shear horizontal surface acoustic wave (SH-SAW) sensors are suitable for measurements of conductivity and viscosity because it can propagate with low leakage energy into liquid [2]. Piezoelectric single crystals such as 36º-rotated Y-cut X-propagating $LiTaO_3$ are generally used for the sensors [3]. On the other hand, we have demonstrated the film growth of c-axis parallel oriented ZnO on a glass plate [4] and the SH-SAW excitation [5]. ZnO films can be grown on curved surface by a sputtering deposition.

The velocity and amplitude of the SH-SAW are affected by the viscosity and conductivity of the liquid on the propagation path. The long-distance propagation is necessary in order to detect the small changes of the velocity and amplitude. Multiple wave roundtrips on a ball surface or a pipe surface leads to the long-distance propagation [6]. Therefore, c-axis parallel oriented ZnO film on the pipe makes it possible to obtain a high-sensitive liquid sensor.

Irradiation of highly energetic negative ions from the ZnO sputtering target induced the growth of the c-axis parallel orientation [7]. The highly oriented film was obtained above the target erosion area. In previous study, the c-axis parallel oriented ZnO film was grown on a part of a glass pipe surface [8]. The Shear mode bulk acoustic wave (BAW) roundtrips were observed with the pipe structure sensor. However, the SH-SAW roundtrips were not observed because the ZnO film was not grown on the entire surface of the pipe. Therefore, it is necessary to rotate the pipe substrate during the ZnO deposition.

In this study, we demonstrated the pipe-surface deposition of the c-axis parallel oriented ZnO film by the split of the deposition area and the automatic system of the substrate rotation. The crystalline orientations of the ZnO films were evaluated by X-ray diffraction (XRD) analyses. In addition, we also demonstrated the ZnO deposition on a smaller pipe with 6 mm diameter for the practical SH-SAW pipe sensor.

II. ZnO Film Growth on the Entire Surface of the Pipe by the Split of the Deposition Area.

A. Sample preparations

The c-axis parallel oriented ZnO film was grown on the entire pipe surface of the substrate by RF magnetron sputtering system, as shown in Fig. 1. A glass pipe (20 mm diameter, 50 mm long, 1.5 mm thickness) was used as a substrate. Fig. 2 shows substrate settings of the split of the deposition area. In our previous study, the c-axis parallel oriented ZnO film with highly crystallization was obtained above the erosion area. Therefore, a slit was set under the pipe substrate in order to prevent the ZnO film being grown at the side surface of the pipe. ZnO films were deposited on the glass pipe at every 180º in sample I and at every 60º in sample II. Therefore, the deposition process was repeated two and six times in sample I and II, respectively. Table I shows deposition conditions in one deposition process. The thickness of ZnO film was estimated to 4.2 μm in this conditions.

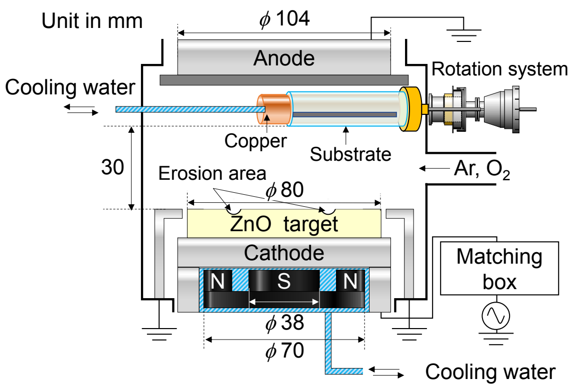

Fig. 1. Schematic image of RF magnetron sputtering system.

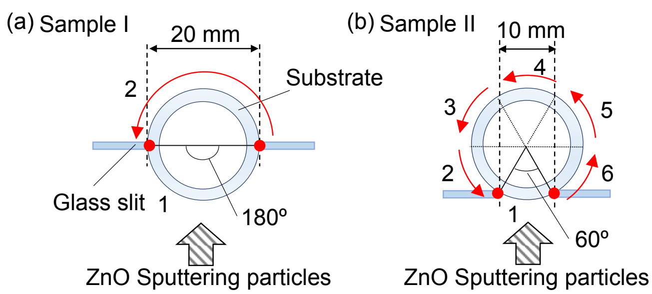

Fig.2. Substrate settings of the split of the deposition area in (a) Sample I and (b) Sample II.

TABLE I. Deposition Conditions in One Deposition Process

Deposition time (hour)	6
RF power supply (W)	100
Ar / O_2	1 / 3
Gas pressure (Pa)	0.1

B. *Crystalline orientations of the ZnO film*

The crystalline orientation of the ZnO film was measured by an x-ray diffractometer (X' Pert Pro MRD, PANalytical). Fig. 3 shows the XRD measurement points of the ZnO film samples on the glass pipe. The measurements were carried out at every 45° in the circumferential direction of the pipe.

Fig. 4(a) shows 2θ-ω XRD patterns of the sample I. Strong $(11\bar{2}0)$ peak was observed at the measurement point A and E. Highly crystallization films were obtained at the substrate surface facing the target plane, as expected. Compared with these peaks, the peak intensities at the point B, D, F, and H were weak. The degrees of the crystallization were degraded with distance from the substrate surface facing the target plane. There is no peak in the point C and G because they were covered with the glass slit.

Fig. 4(b) shows 2θ-ω XRD patterns of the sample II. $(11\bar{2}0)$ peak was observed at the all measurement points. In addition, ω-scan rocking curves of each $(11\bar{2}0)$ peaks were measured. FWHM value of the curve at the point B in the sample II was 8.5° whereas that in the sample I was 11.1°. The narrow width of the glass slit prevented the ZnO film being grown except on the substrate surface facing the target plane. The split of the deposition area and the multiple deposition are effective for the highly-oriented-film growth on the entire pipe surface.

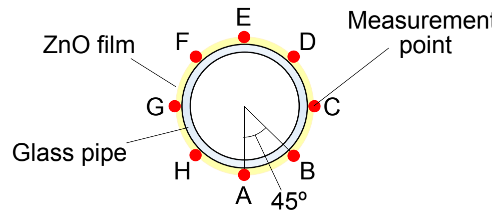

Fig. 3. Measurement point of the ZnO film in XRD analyses.

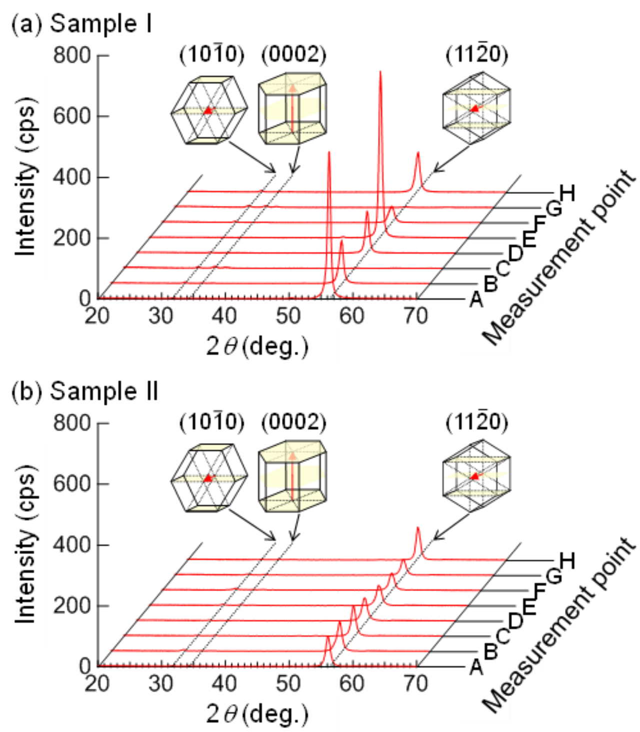

Fig. 4. 2θ-ω XRD patterns of the ZnO films of (a) sample I and (b) sample II. The measurement points were shown in Fig. 3.

III. ZnO Film Growth on the Entire Surface of the Pipe with the Automatically Rotation

A. *Sample preparations*

The non-uniform film growth near the glass slit in the multiple deposition is problem for the SH-SAW pipe sensor. Therefore, it is necessary to rotate the pipe substrate during the deposition. In this chapter, the glass pipe was automatically rotated by connecting a motor to the substrate rotation system in Fig. 1 for the uniform film growth on the entire surface of the glass pipe. Fig. 5 shows substrate settings in the film growth with the automatic rotation of the substrate. Deposition time was set to 36 hours in order to deposit with the same thickness of the sample II. A stainless steel slit was used instead of the glass slit because the glass is broken by the stress of thick ZnO film. The glass slit was set in the same manner as the deposition of the sample II. The total gas pressure and the argon-oxygen ratio were same in Table I. Deposition conditions were set to the RF power of 100 W and the rotational speed of 2.4 rpm.

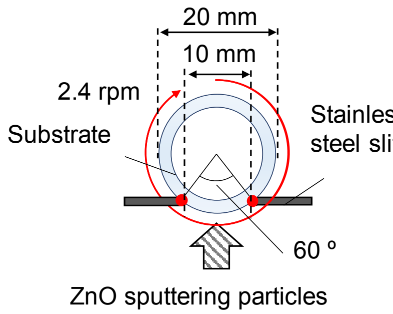

Fig. 5. Substrate settings in the film growth with the automatic rotation of the substrate.

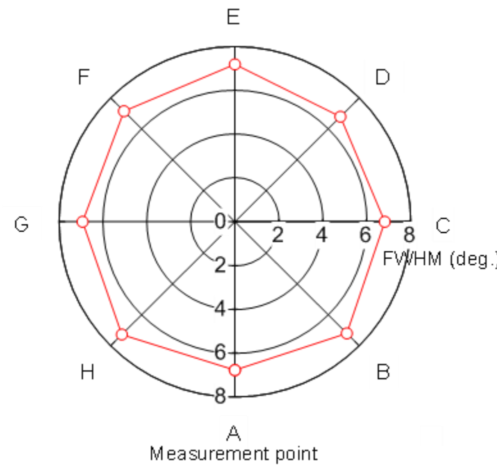

Fig. 7. FWHM of $(1\bar{1}20)$ ω-rocking curves in the ZnO sample deposited with the automatic rotation of the pipe substrate. The measurement points were shown in Fig. 3.

B. *Crystalline orientations of the ZnO film*

Fig. 6 shows 2θ-ω XRD patterns of the ZnO sample deposited with the automatic rotation of the pipe substrate. They were preferentially oriented in $(11\bar{2}0)$ plane at all measurement points. The film growth with the automatic rotation of the pipe substrate make it possible to obtain the c-axis parallel oriented ZnO film on the entire surface of the pipe. FWHM values of $(11\bar{2}0)$ ω-scan rocking curves were also calculated, as shown in Fig. 7. The FWHM values of the sample were smaller than sample II. Therefore, the oriented film was obtained on the entire surface of the pipe. The automatic rotation system of the pipe was effective for the uniform film growth on the entire pipe surface.

Fig. 8 shows a $(11\bar{2}2)$ XRD pole figure in the sample at the point A. The angle between $(11\bar{2}2)$ and $(11\bar{2}0)$ plane in ZnO crystal is 32º. Therefore, $(11\bar{2}2)$ po1e figure of $(11\bar{2}0)$ oriented ZnO film indicates poles at ψ = 32º. Two concentrated $(11\bar{2}2)$ poles were observed near ψ = 32º, ϕ = 350º and ψ = 32º, ϕ = 170º in Fig. 8. The c-axis direction almost corresponded to the pipe axis direction. SH-SAW excitations are expected using the c-axis parallel oriented film by the deposition with the automatic rotation of the substrate.

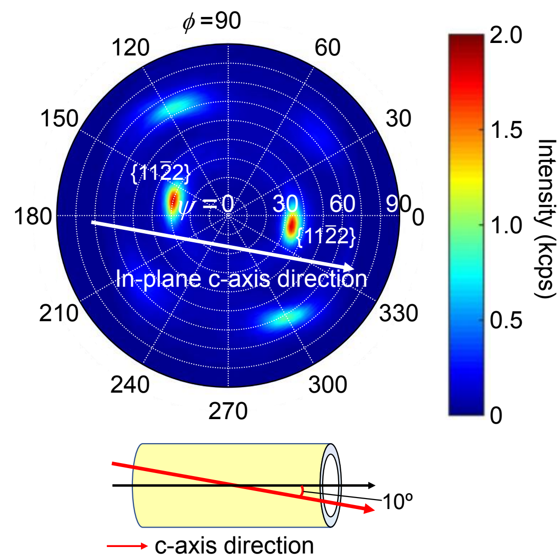

Fig. 8. $(11\bar{2}2)$ pole figure of the ZnO sample deposited with the automatic rotation of the pipe substrate. The measurement point was the point A in Fig. 3.

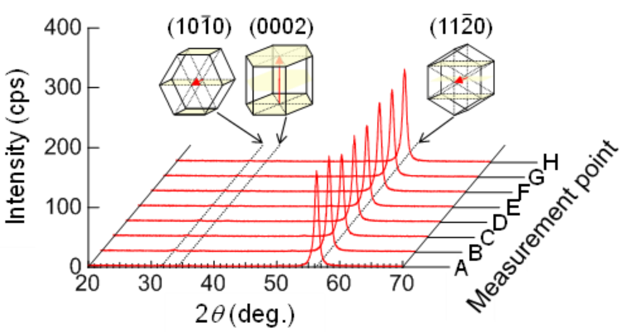

Fig. 6. 2θ -ω XRD patterns of the ZnO sample deposited with the automatic rotation of the pipe substrate. The measurement points were shown in Fig. 3.

IV. ZnO Film Growth on the Small-Pipe Surface

A. *Sample preparations*

c-Axis parallel oriented ZnO films were grown on the smaller pipe substrate for the practical SH-SAW pipe sensor. Fig. 9 shows the pipe settings in the ZnO deposition. The diameter of the glass pipe was 6.0 mm. ZnO film depositions were performed on the pipe surface facing the target plane with a slit width of 6 mm or 10 mm. The deposition conditions were same in Table I.

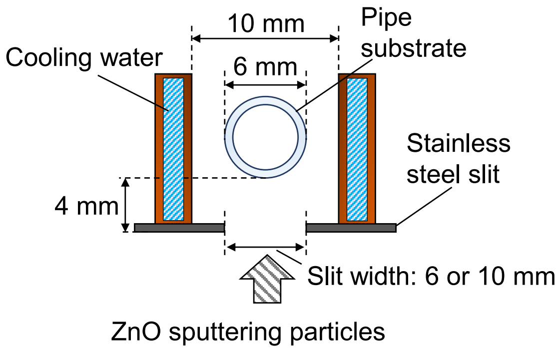

Fig. 9. Schematic image of the substrate settings for the smaller pipe.

B. Crystalline orientations of the ZnO film

Fig. 10 shows 2θ-ω XRD patterns of the ZnO samples on the 6-mm-diameter pipes. A $(11\bar{2}0)$ peak was observed in the both samples. On the other hand, other peaks were also observed. Then, $(11\bar{2}0)$ ω-scan rocking curves were measured. FWHM values in the sample deposited with the slit width of 6 mm and 10 mm were 6.7º and 5.3º, respectively. As a result, although the FWHM values were relatively small, a preferentially $(11\bar{2}0)$ orientation was not obtained. Bombardment of highly energetic negative ions from the ZnO sputtering target is necessary to obtain the $(11\bar{2}0)$ orientation [7]. Because the pipe surface facing the target plane was 4 mm distant from the slit surface, as shown in Fig. 9, the energetic-negative-ion bombardment to the substrate from the target may decrease. Substrate settings which increase the ion bombardment are required.

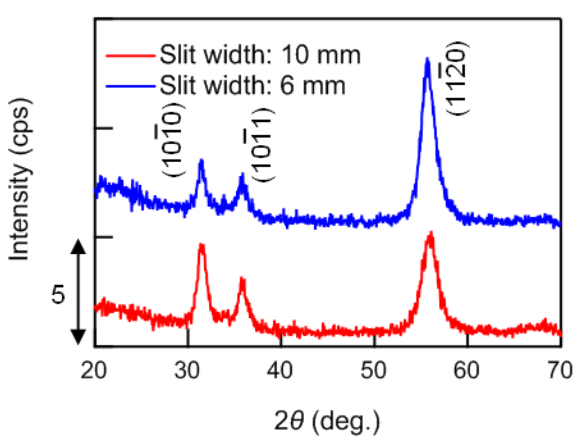

Fig. 10. 2θ-ω XRD patterns of the ZnO samples on the 6-mm-diameter pipes with the slit width of 6 mm and 10 mm.

V. CONCLUSION

Deposition methods of the c-axis parallel oriented ZnO film on the silica glass pipe surface were investigated. Highly crystallized and oriented film was only grown on the substrate surface facing the sputtering target plane. Therefore, the film deposition with the slit which prevent the ZnO film being grown at the side surface of the pipe was one effective method. Furthermore, the automatic rotation system of the pipe substrate leads to reduce the non-uniform film growth near the slit. In ZnO film growths on small pipes with 6 mm diameter, a preferentially c-axis parallel orientation was not obtained. Further investigations of substrate settings for the small pipe substrate were required. In addition, the SAW propagation properties of the pipe structure sensor with the c-axis parallel oriented ZnO film were expected.

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