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c-Axis tilted ScAlN film shear mode resonators for detection of biomolecular interactions

c 軸傾斜配向すべりモード ScAlN 薄膜共振子を用いた生体反応検出

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1. Introduction

In order to measure the biomolecular interactions, the resonators operating in liquids are required. Therefore, the shear mode resonators are necessary instead of normal thickness extensional mode resonators. QCM (quartz crystal microbalance) is suitable for sensing biomolecules since QCM operates in shear mode [1]. The resonant frequency changes due to the mass loading caused by the molecules binding on the resonator surface. Thin resonators are needed for accurate measurement because the sensitivity is inversely proportional to the mass of the entire resonator as shown in Eq. (1).

$$\frac{\Delta f}{f_0} = -\frac{\Delta m_{load}}{m_{resonator}} \quad (1)$$

QCM, however, has limitation in its thickness because of difficulty in the mechanical polishing. In contrast, the use of FBAR (film bulk acoustic resonator) allows high sensitivity because of their small mass of entire resonator. c-Axis tilted ZnO or AlN-based shear mode FBARs for biosensing applications were reported by Weber and Wingqvist [2,3]. However, shear mode electromechanical coupling coefficient (k'^2_{15}) values in these reports were low (which are 1.7 % (ZnO) and 2 % (AlN)). Higher k'^2_{15} value makes it possible to improve signal-to-noise ratio. We fabricated the quasi shear mode resonator using ScAlN films which possess high k'^2_{15} [4]. Fig. 1 shows the calculated electromechanical coupling coefficient k'^2_{15} and k'^2_{33} of ScAlN. The elastic and piezoelectric constants used in this calculation were obtained from first principles calculations [5].

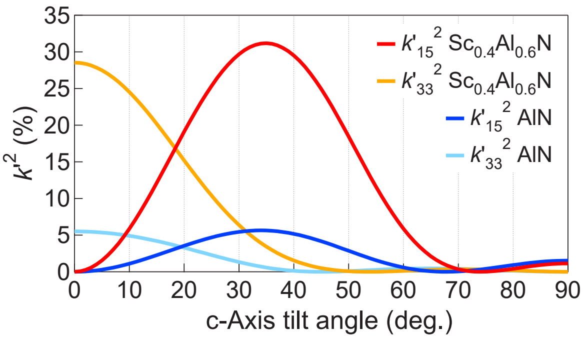

Fig. 1 Theoretical electromechanical coupling coefficient as a function of c-axis tilt angle to the substrate normal in AlN and ScAlN

Both k'^2_{15} and k'^2_{33} values of ScAlN films exceed those of non-doped AlN. k'^2_{15} value of the ScAlN increases with increasing of c-axis tilt angle and reaches maximum value at 35°. Furthermore, k'^2_{15} and k'^2_{33} of ScAlN show that the resonator operates in only shear mode at the angle exceeding 50°.

In this study, the c-axis 50° tilted ScAlN film resonators which possess high k'^2_{15} with no thickness extensional mode operation were fabricated. The streptavidin-biotin interactions were monitored by using the resonators.

2. Fabrication of the resonator

Fig. 2 shows the structure of fabricated resonator (Top electrode: Au, piezoelectric layer: c-axis tilted ScAlN, Bottom electrode: Ti). c-Axis tilted ScAlN films were grown by glancing angle sputtering deposition.

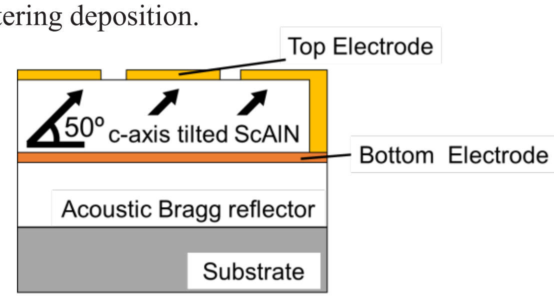

Fig. 2 c-Axis tilted ScAlN film resonator structure

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Fig. 3 is the XRD (0002) pole figure for ScAlN film. The pole concentration at 45° indicates that the c-axis tilt angle of the film is 45°. k_{15} of the c-axis tilted ScAlN films was determined to be 16.3 % by using a resonance-antiresonance method [6]. TCF value of ScAlN film resonator in this study was determined to be -40 ppm/°C.

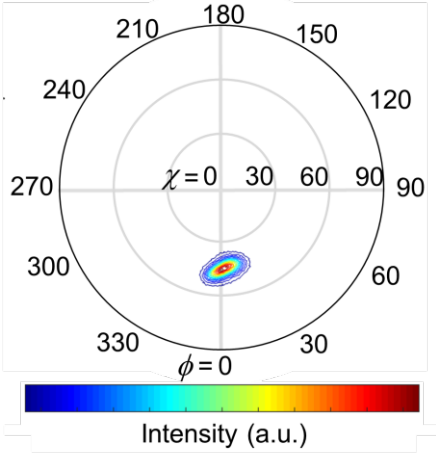

Fig. 3 (0002) pole figure in ScAlN film

3. Detection of biomolecular interactions

3.1 Detection Procedure and system

First, biotin-terminated SAMs (self-assembled monolayers) were formed on the resonator surface. After that, the biotin-coated resonator was immersed in 100 μl of PBS (phosphate-buffered saline, pH 7.2) (Fig. 4(a)). Then, 100 μl of streptavidin solution was introduced into the resonator at 300 seconds (Fig. 4(b)). We measured the quasi-shear mode anti-resonant frequency changes caused by streptavidin binding to biotin immobilized on the resonator surface. The anti-resonant frequencies were measured every 2 seconds by using a network analyzer (Agilent Technologies, E5071C). The obtained data were smoothed by moving averages to reduce short-term variation. The observed linear changes due to sensor drift were eliminated.

3.2 Detection Result

Fig. 4 shows the real-time monitoring of anti-resonant frequency when streptavidin is introduced. By comparing streptavidin concentration of 5 μg/ml with 50 μg/ml, we can see that the amount of frequency change increased with increasing of the concentration.

4. Conclusion

We measured streptavidin-biotin interactions by using c-axis tilted ScAlN film shear mode resonators. The obtained results demonstrate that c-axis tilted ScAlN film shear mode resonators are promising for biosensing application.

Acknowledgment

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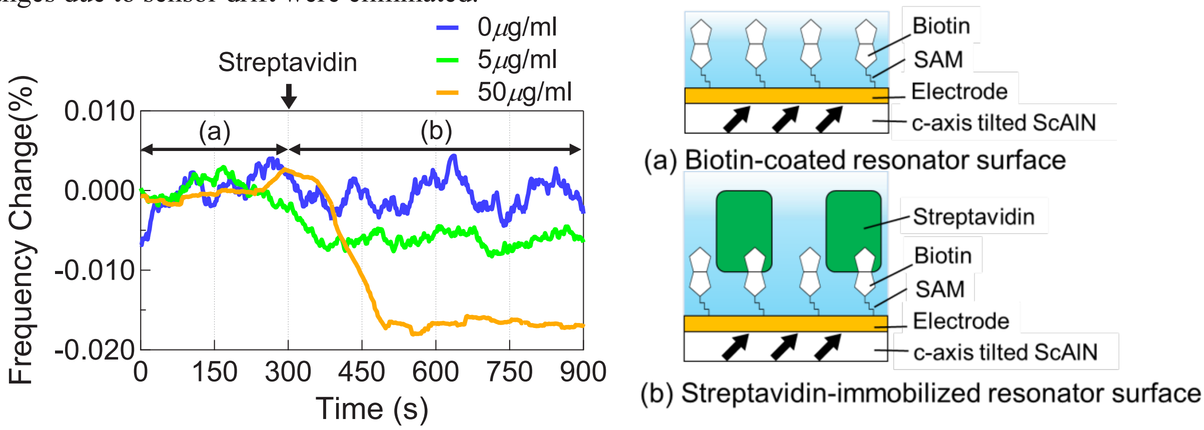

Fig. 4 Real-time monitoring of resonant frequency change