Simultaneous viscosity evaluation in the MHz to GHz range with low TCF resonators consisting of shear mode piezoelectric thin films on AT-cut quartz crystal

横波励振圧電薄膜/低 TCF 基板構造共振子による MHz 帯-GHz 帯にわたる液体粘性の一括評価

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1. Introduction

In order to study relaxation characteristics of liquid, a wide frequency sweep in the range of MHz to GHz is desired. Conventional sensors such as QCM (Quartz Crystal Microbalance) [1] and FBAR (Film Bulk Acoustic Resonator) [2], however, basically operate only at the MHz range or the GHz range, respectively. Wide frequency operation from MHz to GHz by using a single device is required for more accurate and real-time measurement.

We have focused on HBAR (High overtone Bulk Acoustic Resonator), which consists of piezoelectric thin films on the substrate. Unlike other usual sensors, quite a number of resonant frequency peaks can be observed in the HBAR as shown in **Fig. 1**.

In this study, resonant frequency shifts due to change in viscosity of liquid (where glycerin concentration was varied) were experimentally measured and simulated with Mason's equivalent circuit model.



peaks in (a)QCM and (b)HBAR sensor

2. Method

2.1 Fabrication of HBAR

It has been reported that the piezoelectricity is strongly enhanced when 43% Sc is doped to AlN films [3]. In addition, we have recently reported the quasi-shear mode electromechanical coupling k'_{15} of ScAlN reaches maximum at c-axis tilt angle of approximately 30° [4]. The shear mode ScAlN HBAR was fabricated as shown in **Fig. 2** (top electrode: Au, piezoelectric film; approximately 30° c-axis tilted ScAlN, bottom electrode: Al, substrate: AT-cut quartz crystal). Experimental k'_{15} of the ScAlN thin film is estimated to be 0.395 ($k'_{15}^2 = 15.6\%$) by using a conversion loss method [5].



Fig. 2 HBAR sensor consisting of c-axis tilted ScAlN on temperature stable AT-cut quartz crystal

2.2 Experiment of HBAR Viscosity Sensor

The real part of the impedance Z_{real} of the HBAR in contact with the glycerin solutions was measured by a network analyzer (E5071C, Agilent Technologies). The anti-resonant frequencies were determined from the peaks of the Z_{real} . Six glycerin solutions with different concentrations (0 wt.%, 20 wt.%, 40 wt.%, 60 wt.%, 80 wt.%, 97 wt.%) were measured in the range of 30 MHz-1.5 GHz. These experiments were performed at 25°C in a thermostatic chamber (MC-100, YONEZAWA INC.), and the temperature was monitored using a platinum resistance thermometer.

2.3 Analysis of HBAR Viscosity Sensor by Mason's Equivalent Circuit Model

In this study, the resonant frequency shifts of the HBAR were simulated by using Mason's equivalent circuit model including liquid loading. Assuming the glycerin solutions as Newtonian fluids, **Eq. (1)** was employed for representing the liquid loading impedance Z_L (ω : angular frequency, η : viscosity, ρ : density, subscript L: liquid).

$$Z_{\rm L} \approx \sqrt{\rho_{\rm L} \cdot (j\omega\eta_{\rm L})} = \sqrt{\frac{\omega\eta_{\rm L}\rho_{\rm L}}{2}} + j\sqrt{\frac{\omega\eta_{\rm L}\rho_{\rm L}}{2}} \qquad (1)$$

3. Results and Discussions

3.1 Frequency Characteristics of Real Part of Impedance

Fig. 3 shows the real part of the impedance of the HBAR in contact with glycerin solutions with different concentrations. Anti-resonant frequency decreasing with increasing liquid viscosity can be observed in MHz and GHz range.



Fig. 3 Experimental anti-resonant frequency shift due to the change in liquid viscosity

3.2 Anti-resonant Frequency Shift

Fig. 4 shows the experimental plots and the theoretical curves of anti-resonant frequency shifts. In Fig. 4(a), there is considerable variation in the experimental plots. This is probably because of the peak deformation due to the existence of longitudinal waves or the wave diffraction. To eliminate such negative effects, a HBAR consisting of a pure shear mode c-axis parallel ZnO film was newly fabricated. The same experiments were performed using the new HBAR. As shown in Fig. 5, the discrepancy of the experimental plots was suppressed. Frequency peaks in GHz range, however, could not be observed because the piezoelectricity of c-axis parallel ZnO films is smaller than that of 30° c-axis tilted ScAIN films.

On the other hand, in Fig. 4(b), the experimental shifts are found to be smaller than the theoretical curve. This is because the viscous model of Eq. (1) cannot represent the viscoelasticity of 97 wt.% glycerin solutions.

4. Conclusion

Liquid viscosity could be evaluated simultaneously in the range of 30 MHz-1.5 GHz with HBARs consisting of shear mode ScAlN film and ZnO film. We intend to establish a new viscous model representing liquid viscoelasticity and viscous relaxation.



Fig. 4 Experimental and theoretical anti-resonant frequency shifts from 0 wt.% glycerin solution in the HBAR consisting of c-axis tilted ScAlN in the case of (a)20 wt.% and (b)97 wt.% glycerin solution



Fig. 5 Experimental and theoretical anti-resonant frequency shifts from 0 wt.% glycerin solution in the HBAR consisting of c-axis parallel ZnO in the case of 20 wt.% glycerin solution

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