Liquid loading characteristics of thickness-shear mode resonator consisting of c-axis parallel oriented ZnO film

Rikuya Iwanaga1†, Shinji Takayanagi2, Mami Matsukawa3 and Takahiko Yanagitani4 (1Doshisha Univ.; 2Nagoya Inst. Tech.; 3Waseda Univ.)

Liquid viscosity

1. Introduction

Thickness-shear mode resonators are used for measurements of antigen-antibody reaction and liquid viscosities, because of their small energy leakage of the shear wave into the liquid. The sensitivity of the measurement increase with decreasing the mass of the resonator. AT-cut Quartz Crystal Microbalance (QCM) was widely studied as the viscosity sensor. However, it is difficult to reduce the mass of the single quartz crystal because of the limitation of the mechanical polishing. On the other hand, piezoelectric thin films are expected for the reduction of the resonator mass. In our previous study, a multilayered resonator consisting of c-axis oriented ZnO film was fabricated[1]. The resonant frequency shifts with liquid loading was observed.

Piezoelectric resonances are discriminated mechanical resonances and electric resonances. Viscosity measurements using the thickness-shear mode resonator is generally used series resonant frequency \( f_s \) or parallel resonant frequency \( f_p \), because the \( f_s \) and \( f_p \) shifts due to the mechanical damping by the viscous effect[2]. Frequency of minimal impedance \( f_m \) and frequency of maximal impedance \( f_m \) are not equal to \( f_s \) and \( f_p \) in the piezoelectric resonance. In addition, recently, Itoh and Ichihashi reported that the frequency \( f_s \) of the minimal susceptance of the resonance has the same mass sensitivity as \( f_s \) and is not affected by the viscous load of a Newton liquid[3]. The \( f_s \) and minimal frequency \( f_s \) of the minimal susceptance of the resonance are frequencies which were traditionally used when determining the quality factor for resonator.

In this study, the frequency shifts of \( f_p \), \( f_m \) and \( f_n \) with liquid loadings were measured by using the multilayered resonator consisting of c-axis parallel oriented ZnO film. In addition, they were calculated by one-dimensional transmission line model. The difference of these frequency shifts was then discussed.

2. Experimental method

Figure 1 shows the structure of the shear mode multilayered resonators. They consist of top electrode (0.35 \( \mu m \)) / c-axis parallel oriented ZnO film (2.6 \( \mu m \)) / bottom electrode (0.4 \( \mu m \)) / SOI layer (9.7 \( \mu m \)). Liquid samples were loaded on the sensing area of the resonator. Glycerol solutions (0-45 wt.%) were used as the liquid samples. \( f_p \), \( f_m \) and \( f_n \) of the 1st and 2nd mode were measured by a network analyzer (E5071B, Agilent Technologies). The frequency shifts of the \( f_p \), \( f_m \) and \( f_n \) at each sample loading were calculated by subtracting the \( f_p \), \( f_m \) and \( f_n \) at 0 wt.% sample (pure water) loading. They were theoretically analyzed by a one-dimensional transmission line model using Mason’s equivalent circuit model.

3. Results and discussion

Table I shows \( f_p \), \( f_m \) and \( f_n \) of the multilayered resonator with 0 wt.% sample loading. 1st and 2nd mode resonances were observed around 132 MHz and 262 MHz, respectively. Figure 2 shows the frequency response of the real part of the impedance around 1st mode \( f_p \) with 0-45 wt.% sample loadings. The 1st mode \( f_p \) was decreased as increasing the glycerol concentration. 1st mode \( f_m \) and \( f_n \), with 0-45 wt.% sample loadings were also observed from the frequency response of the absolute value of the impedance. Frequency shifts of the 1st mode \( f_p \), \( f_m \) and \( f_n \) were shown in Fig. 4(a). 1st mode \( f_m \) and \( f_n \) were also decreased as increasing the glycerol concentration. The amount of the
frequency shift of the \( f_m \) was the largest among the 1st mode. Frequency shifts of the 2nd mode \( f_p, f_m \) and \( f_n \) with 0-45 wt.% sample loadings were obtained in the same manner, as shown in Fig. 5(a). The frequency shifts of 2nd mode \( f_p, f_m \) and \( f_n \) show the same tendency. However, the 2nd mode \( f_n \) was increased as increasing the glycerol concentration, because a spurious mode was observed around it. Compared with the 1st mode, the amounts of the 2nd mode frequency shifts were small.

Error bars of the frequency shifts in five times measurement were evaluated. The error bars of the 1st mode and 2nd mode were less than 6 ppm and 3 ppm, respectively. They were very small for the amounts of the frequency shifts. Therefore, the measurement accuracy of the 2nd mode was higher than that of the 1st mode.

The theoretical results of the frequency shifts of \( f_p, f_m \) and \( f_n \) by the one-dimensional transmission line model were shown in Fig. 4(b) and Fig. 5(b). The same tendencies were observed in the experimental results. However, the amounts of the frequency shifts were different. A possible reason is that the film thickness of ZnO set in the simulation is different from the value in the resonator.

### 4. Conclusion

The frequency shifts of \( f_p, f_m \) and \( f_n \) due to the viscous changes were experimentally and theoretically investigated in the multilayered resonator consisting of c-axis parallel oriented ZnO film. Both experimental and theoretical results showed the same tendency. The large amount of the frequency shift was obtained in the 1st mode \( f_p \). Further investigations of the peak attenuation of the impedance at \( f_p, f_m \) and \( f_n \) are expected.

<table>
<thead>
<tr>
<th>Vibration mode</th>
<th>( f_p ) (MHz)</th>
<th>( f_m ) (MHz)</th>
<th>( f_n ) (MHz)</th>
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<tbody>
<tr>
<td>1st</td>
<td>131.752</td>
<td>131.332</td>
<td>132.071</td>
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<tr>
<td>2nd</td>
<td>262.221</td>
<td>260.999</td>
<td>262.353</td>
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### References