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Temperature compensation of lamb wave sensor by combined antisymmetric mode and symmetric mode

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Both thermal sensitivity and mass sensitivity in liquid of the first antisymmetric (A_0) mode and the first symmetric (S_0) mode of Lamb wave biosensor were investigated. A_0 and S_0 modes are sensitive to the mass change on the surface of the sensor but A_0 mode is also sensitive to the liquid in the region of evanescent wave associated with Lamb wave. By combining A_0 mode and S_0 mode, the measurement error due to the environmental temperature drift decreased by a large factor, therefore, the environmental temperature was efficiently compensated without changing the structure of Lamb wave sensor. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2838313]

The fields of health care and environment control have an ever increasing demand for sensors which are able to detect low concentrations of particles such as molecular agents, warfare agents, or solid particles contained in gaseous or liquid samples.[1] The acoustic sensors, with rapid response, portability, ease of use, and small size are promising typically to be used in recognition of antibodies by specific antigens, absorption of proteins on chemical tailored surfaces, etc.[2,3] The detection principle of this sensor used the measurement of the variation of frequency of a resonant acoustic wave. Love wave and Lamb wave sensors are acoustic sensors which are more suitable to be used in liquid.[4] However, almost all the acoustic sensors are facing the difficulties for real use of online detection because of its undesired characters, such as their high temperature sensitivity. For Love wave sensor temperature, compensation can be done by choosing correct crystal cut direction or matching different materials,[5] but it is not suitable for the other sensors. Normally, dual devices configuration was used to compensate the phase or frequency drifts by temperature, but the thermal effect of both devices were not precisely matching. A crossed system using two A_0 modes of Lamb wave was reported in 2004 to compensate the drifts due to the temperature.[6] However, some technological problems did not allowed to get a temperature sensitivity difference large enough.

The variations of the frequency attributed to mass and temperature changes for two channels can be expressed as follows:

$$\frac{\Delta f_1}{f_1} = \alpha_1 \Delta m + \beta_1 \Delta T, \quad (1)$$

$$\frac{\Delta f_2}{f_2} = \alpha_2 \Delta m + \beta_2 \Delta T, \quad (2)$$

where Δf_1 and Δf_2 are frequency shifts for two channels, respectively, f_1 and f_2 are working frequencies for two channels, α_1 and α_2 are mass sensitivities expressed in $cm^2\ g^{-1}$, β_1 and β_2 are temperature sensitivities, Δm is mass changing due to the bioreactions and ΔT is temperature drifts. The temperature compensation depends on $(\alpha_1\beta_2 - \alpha_2\beta_1)$, the larger this difference is, and the better the correction will be. That was showed in previous studies for A_0 mode with a kind of cross system.[6]

The A_0 mode of Lamb wave is usually used because theoretically, its sensitivity could reach 200–1000 $g^{-1}\ cm^2$. Furthermore, for A_0 mode, an evanescent wave will be excited in liquid. Even the characters of the evanescent wave in the liquid layer could help to know the dynamic density changing in the liquid during bioreactions, the penetration depth and the density of the liquid will also be changed due to the temperature drifts which will affect the sensor output significantly. Instead using two A_0 Lamb wave devices, using the A_0 mode with the S_0 mode presents some interests. Because for a very thin membrane, the S_0 mode vibration is almost tangential to the sensor membrane, therefore, the wave almost does not penetrate in the liquid.

In this letter, we present the mass measurement and thermal characters of both A_0 and S_0 modes with experiments of mass sensitivity and temperature sensitivity both in air and liquid in order to understand its behaviors and to see the possibility to realize temperature compensation.

The Lamb wave sensors in the experiments were fabricated with clean room facilities. The thickness of the sensor membrane, where the Lamb wave propagates, was 16 μm and its dimension was $8000 \times 8000\ \mu m^2$; two pairs of interdigital transducers (IDTs) were built on 1 μm piezoelectric aluminum nitride layer and the period was 400 μm. One pair of IDT is connected with the output of gain-phase analyzer and excites Lamb wave on the membrane, the Lamb wave propagates on the membrane and transform into electrical signal on another IDT then the electrical signal is measured by the input of gain-phase analyzer. Since mass changes accompanying electrochemical reaction, this one can be accurately measured by frequency shifts of acoustic sensor.[7] In our case, we used copper electroplating on the Lamb wave membrane. The Lamb wave sensor was sputtered with gold electrode and then electroplated with copper in electrolyte solution. The electrolyte solution for copper electrodeposition was made of $10^{-2} M$ sulfuric acid and $10^{-2} M$ copper sulfate.[8] During the copper electrodeposition processes, the

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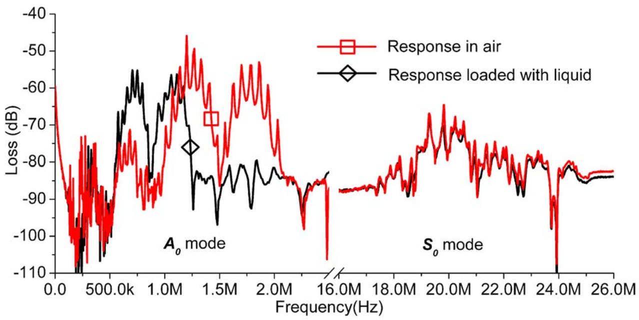

FIG. 1. (Color online) The comparison of the Lamb wave sensor's responses in air and liquid.

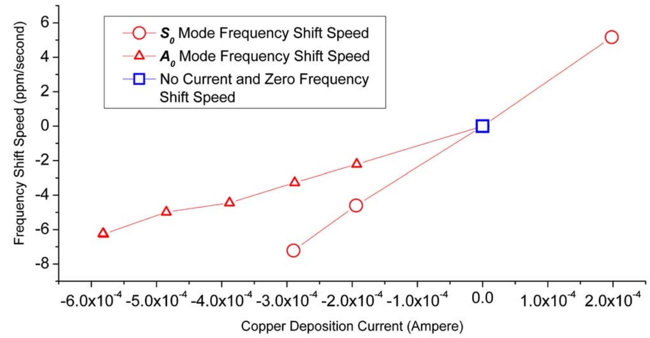

FIG. 2. (Color online) The frequency shift speeds as a function of current used for metal deposition.

frequency responses of both A_0 and S_0 modes were alternatively tested using a HP4194 gain-phase analyzer. The deposition current was square wave with a period of 200 s, the duty cycle was 30% and the maximum current was 5.81 $\times 10^{-4}$ A.

The temperature sensitivity of sensor was tested both in air and liquid. The acoustic sensor was placed in a computer controlled oven and the temperature varied between 20 and 39 °C back and forth. The response time of Lamb wave sensor was about 2 s, which was measured by Shibaura PSB-S7 high speed temperature sensor. The temperature in the oven chamber changed at the speed of 1 °C per 10 min, which was slow enough for temperature sensor and the Lamb wave sensor to respond; both A_0 and S_0 modes were tested alternatively. During the temperature sensitivity measurement in liquid, the sensor was loaded with DI water downside and the temperature in the oven also varied between 20 and 39 °C, the liquid cavity was opened to avoid the pressure variation and the Lamb wave sensor was suspended in the oven chamber to avoid vibration.

The calculated Lamb wave phase velocity of A_0 mode in air was 741 m/s at a frequency of 1.8525 MHz. Theoretically, when the sensor is loaded with liquid on a single side, an evanescent wave appears and the length decay is about 67 μm then the velocity decreases to 489 m/s at a frequency of 1.2225 MHz. As the particle motion of S_0 mode is predominantly tangential to the sensor membrane and the length decay of the evanescent wave due to the viscosity is only 0.13 μm which can be neglected, therefore, this mode is insensitive to the liquid loading. The calculated velocity of S_0 mode in air is 8635 m/s. The comparison between S_0 mode and A_0 mode is shown in Fig. 1: the A_0 mode shifted to lower frequency when the sensor was loaded with liquid (de-ionized water), whereas S_0 mode frequency stills the same. By taking frequency shifts of A_0 mode and using Eq. (3),

$$\frac{\rho_{\mathrm{Si}} d}{\rho_{\mathrm{Si}} d + \rho_l \delta_e} = \left(\frac{f}{f_0}\right)^2, \tag{3}$$

where ρ_{Si} and ρ_{l} are the densities of silicon and liquid, d is the membrane thickness, δ_e is the evanescent wave penetration thickness, and f and f_0 are the working frequencies of the sensor loaded with water and without water, the actual evanescent wave penetration depth value (71 μm) could be obtained.

When the target molecules are being selectively adsorbed on the Lamb wave membrane, the frequencies of both modes will decrease because of molecules mass loading. The frequency shifts are different because of the existence of liquid evanescent layer. The mass sensitivity of A_0 mode α_{A0} is[9]

$$\alpha_{A_0} = \frac{1}{2}\frac{1}{\rho_{\mathrm{Si}} d + \rho_l \delta_e}. \tag{4}$$

Using the same theory, the mass sensitivity of S_0 mode α_{S0} can be got,

$$\alpha_{S_0} = \frac{1}{2}\frac{1}{\rho_{\mathrm{Si}} d}. \tag{5}$$

In theory, the sensitivity of S_0 mode and A_0 mode were 110.7 and 39.4 $\mathrm{g}^{-1}\,\mathrm{cm}^2$ respectively; the sensitivity ratio was 2.8, which was much larger than the ratio of cross-system mass sensitivity. The mass sensitivity was measured using electrochemical deposition of copper.

Different deposition currents were taken and the frequency shift speeds are shown in Fig. 2. The frequency shift speed shows the good linearity of the sensor's response and both curves cross the origin point. In Fig. 2 the point (2.0 $\times 10^{-4}$ A, 5.49 ppm/s) meant that the copper was being removed from the Lamb wave sensor and the center frequency was increase. While the copper was being removed from the membrane small quantity of hydrogen may be produced near the gold surface. It was found that even tiny hydrogen gas bubbles could distort A_0 mode measurement results whereas S_0 was immune to the bubbles. Therefore, the distorted response of A_0 with 2.0×10^{-4} A was not shown in Fig. 2.

The measured sensitivities of S_0 mode and A_0 mode were 87.6 and 36.4 $\mathrm{g}^{-1}\,\mathrm{cm}^2$, respectively. The sensitivity of S_0 mode was 2.40 times that of A_0 mode, which agreed with calculation.

In order to compare the temperature sensitivity, the temperature sensitivity was measured in air as shown in Fig. 3.

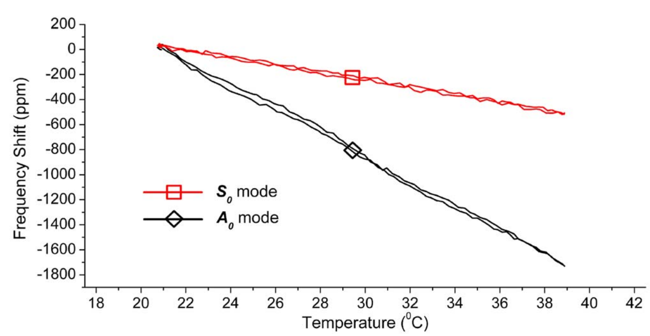

FIG. 3. (Color online) The thermal responses of A_0 and S_0 Lamb wave modes in air.

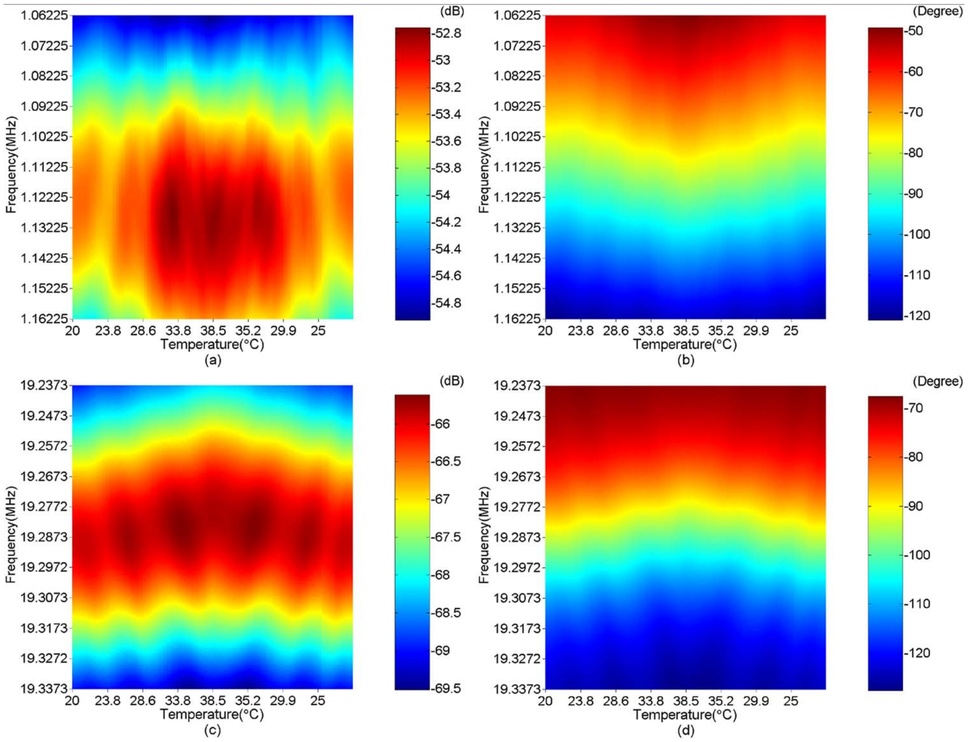

FIG. 4. (Color online) The thermal responses of A_0 and S_0 modes of Lamb wave sensor loaded with liquid (a is amplitude response of A_0 mode, b is phase response of A_0 mode, c is amplitude response of S_0 mode, and d is phase response of S_0 mode).

For both modes, the frequencies decrease with temperature; A_0 mode temperature sensitivity was −96.2 ppm/°C and S_0 mode was −29.6 ppm/°C.

While the sensor was loaded with liquid the gain-phase responses changed: the gain-phase two-dimensional (2D) plots are shown in Fig. 4: the frequency of S_0 mode loaded with liquid decreases with temperature which agreed with its response in air (Fig. 3), the frequency of A_0 mode increases with temperature which was reverse to its response in air. It is valuable for temperature compensation in liquid that the sign of temperature sensitivity for these two modes are opposite.

However, "periodic" variation obviously appears including a frequency decreasing or increasing. By comparing 2D plots, nonlinearity was clearly seen and the gain and phase response of each mode no longer vary synchronously as in air. Even with this nonlinearity phenomenon, the repeatability has been proved in Fig. 4.

Using this last measurement, the precision on mass and temperature can be evaluated. In order to avoid the possible nonlinearity phenomena, the precision was calculated with linear approximation around one given temperature. From Eq. (1) and (2), the error on the mass measurement can be evaluated as

$$\delta_{\Delta M}^2 = \left(\frac{\partial \Delta M}{\partial \alpha_1}\right)^2 \delta\alpha_1^2 + \left(\frac{\partial \Delta M}{\partial \alpha_2}\right)^2 \delta\alpha_2^2 + \left(\frac{\partial \Delta M}{\partial \beta_1}\right)^2 \delta\beta_1^2 + \left(\frac{\partial \Delta M}{\partial \beta_2}\right)^2 \delta\beta_2^2, \tag{6}$$

where $\delta_{\Delta M}$ is the error on mass measurement, $\delta\alpha$ and $\delta\beta$ are the errors on coefficients α and β, subscripts 1 and 2 are for A_0 and S_0 modes. At 23.0 °C, β_1 and β_2 are 105.4 and −51 ppm/°C, and $\alpha_1 = 36.4$ and $\alpha_2 = 87.6$ g^{-1} cm^2. Furthermore, $\delta\alpha_1$ and $\delta\alpha_2$ are 0.3 and 0.8 g^{-1} cm^2, and $\delta\beta_1$ and $\delta\beta_2$ are 3 and 3 ppm/°C. The relative error can be expressed as a function of ΔT and Δm. For example, with $\Delta T = 0.1$ °C and $\Delta m = 10^{-7}$ g cm^{-2}, the relative error on the mass measurement is about 290% when using A_0 mode, 59% with S_0 mode and only 3.2% when combined. It has to be noted that the advantage of the combined measurement is better as the ratio $\Delta T/\Delta m$ increases.

In summary, we investigated the mass and thermal characters of Lamb wave sensor in liquid, especially the characters of S_0 mode. The A_0 mode is sensitive not only on the surface of the sensor but also in the liquid layer itself where the evanescent wave is present, whereas S_0 mode is sensitive only to the mass changes on the surface; the S_0 mode is even more sensitive than A_0 mode of Lamb wave sensor, which is promising in biochemical fields. Their responses to temperature variation are fundamentally different due to the existence of evanescent wave. By combined those two modes it is possible to measure both mass and temperature variations with the same device without changing the structure of Lamb wave sensor therefore the temperature drift is efficiently compensated.

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