

# Dual SAW Sensor Technique for Determining Mass and Modulus Changes

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**Abstract**—Surface acoustic wave (SAW) sensors, which are sensitive to a variety of surface changes, have been widely used for chemical and physical sensing. The ability to control or compensate for the many surface forces has been instrumental in collecting valid data. In cases in which it is not possible to neglect certain effects, such as frequency drift with temperature, methods such as the “dual sensor” technique have been utilized. This paper describes a novel use of a dual sensor technique, using two sensor materials (quartz and GaAs) to separate out the contributions of mass and modulus of the frequency change during gas adsorption experiments. The large modulus change in the film calculated using this technique and predicted by the Gassmann equation provide a greater understanding of the challenges of SAW sensing.

## I. INTRODUCTION

SAW DEVICES, because they are extremely sensitive to surface perturbations, have been utilized in a variety of sensor applications [1]. These include chemical sensing [2], vapor desorption and diffusivity [3], [4], conductivity changes [5], [6], pressure and temperature changes [7], and stress changes [8], [9]. SAW sensors have also been effectively used to determine the porous properties (surface area and pore size distribution) of some thin films [10], [11].

With advances in membrane sciences, the interest in microporous materials (pore sizes, < 2 nm), such as gas separation membranes, has increased. The use of SAW devices to characterize these microporous thin films has led to confusing results [12]. The attempt to understand these results has led to a novel use of the “dual sensor” technique. In a typical dual sensor experiment, one SAW device is kept in a controlled environment and used as a reference. This is usually an uncoated SAW device in the proximity of, but not actually exposed to, a chemical agent to measure the temperature drift of the sensor. The response of the reference device is subtracted from that of the sensor to determine the actual response caused by the chemical. In this experiment, the dual sensors were two different sensor materials. Both sensors were exposed to the same environment, and the different responses were due to the material properties of the sensors. Using this technique, the mass loading and modulus contributions to

the frequency response of the SAW sensors during the adsorption of methanol onto a microporous silicate thin film were separated [12].

## II. BACKGROUND

Porous properties of the materials are frequently derived from adsorption isotherms (the relationship, at constant temperature, between the amount of gas adsorbed and the corresponding pressure [13]), typically measured as mass changes. For thin films, for which the mass uptake is typically too low to measure with conventional means, SAW devices are used. The SAW devices, however, are also very sensitive to other surface changes. The response of SAW devices to common surface perturbations in gas adsorption experiments can be generalized as<sup>1</sup>

$$\frac{\Delta V_R}{V_{Ro}} = -\frac{\Delta \beta_R}{\beta_{Ro}} = \frac{\Delta f}{f_o} = -k_m \frac{\Delta m}{m_o} + k_s \frac{\Delta s}{s_o} + k_\sigma \frac{\Delta \sigma}{\sigma_o} + k_\gamma \frac{\Delta \gamma}{\gamma_o} - k_T \frac{\Delta T}{T_o} \quad (1)$$

where  $V_R$  = velocity of the Rayleigh mode acoustic wave,  $f$  = frequency of oscillation,  $\beta_R$  = the propagation factor equal to  $2\pi/\lambda_R$ ,  $m$  = mass density,  $s$  = the effective modulus (or stiffness),  $\sigma$  = stress in the film,  $\gamma$  = surface tension,  $T$  = temperature,  $k_m$  = mass sensitivity,  $k_\sigma$  = stress sensitivity,  $k_s$  = effective modulus sensitivity,  $k_\gamma$  = stress sensitivity, and  $k_T$  = temperature sensitivity.

Because the mass change is of primary interest in adsorption experiments, it is necessary to minimize, or account for, the numerous other surface perturbations that contribute to the frequency response. The temperature effects are eliminated by performing the gas adsorption experiments isothermally. The relatively thick (640  $\mu\text{m}$ ) substrate (compared with the 1000  $\text{\AA}$  film) used for the SAW sensors reduces the stress and surface tension contributions by minimizing the possible shape changes in the sensor during gas adsorption. For materials with pore sizes in the mesoporous regime (2 nm < pore diameter < 50 nm), the modulus term is minimized by the large differences in the gas size compared with the pore size. However, for microporous materials (pore diameter < 2 nm), the modulus term becomes significant. From calculation and experiment [12], the main contribution to the SAW response of

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<sup>1</sup>The subscript “o” refers to the initial conditions of the unloaded sensor; “ $\Delta$ ” indicates the difference between the current and initial conditions (e.g.,  $\Delta f = f - f_o$ ).

gas adsorption on the microporous film are the mass and modulus terms. Interestingly, these terms are coupled, primarily because of the small diameter of a micropore.

### III. THEORY

Using perturbation theory, Auld [14] derived the normalized phase delay change of a sensor loaded with a lossless isotropic thin film. This is, equivalently, the change in resonant frequency of a SAW sensor in a delay loop oscillator. For a Rayleigh surface wave, the normalized frequency shift is [14]

$$\frac{\Delta f}{f_o} = \kappa \Delta \left[ -\frac{V_R}{4} \left( \frac{|V_{Ry}|^2}{P_R} + \frac{|V_{Rz}|^2}{P_R} \right) h \rho' + \left( h \mu' \frac{\lambda' + \mu'}{\lambda' + 2\mu'} \right) \left( \frac{1}{V_R} \frac{|V_{Rz}|^2}{P_R} \right) \right] \quad (2)$$

where  $f$  is the frequency of oscillation,  $V_R$  is the velocity of the Rayleigh mode acoustic wave,  $h$  is the thickness of the thin film isotropic overlay,  $\rho'$  is the density of the thin film isotropic overlay,  $\mu'$  is the shear modulus of the thin film isotropic overlay,  $\lambda'$  is the bulk modulus of the thin film isotropic overlay,  $P_R$  is the acoustic power,  $V_{Ry,z}$  is the surface particle displacement velocity, and  $\kappa$  is the ratio of effective sensor coverage.

Note that the first term of (2) depends only on the mass density of the film ( $h \rho'$ ); whereas, the second depends on the film modulus. The film modulus changes have frequently been assumed to be negligible compared with the mass contribution for gas adsorption isotherm measurements of silicate films. This has proven to be a valid assumption, except for the case of microporous films, for which it is experimentally evident that modulus contributes a significant portion to the SAW sensor's response. This was initially noted by some quartz SAW samples exhibiting a positive frequency shift during increased mass loading. This experimental data necessitated the re-evaluation of (2) to determine methods to separate the mass and modulus contributions in the sensor's response.

Rearranging and redefining terms in (2) gives the following equation for a fully covered sensor:

$$\Delta f = -\frac{f_o V_R}{4} (R_y + R_z) \Delta m + \Delta s \frac{f_o}{V_R} R_z \quad (3)$$

where  $\Delta f$  is the frequency shift,  $f_o$  is the frequency of oscillation of an unloaded sensor,  $V_R$  is the velocity of the acoustic wave,  $R_{y,z} = |V_{Ry,z}|^2 / P_R$ ,  $\Delta m = \Delta(h \rho')$  is the surface mass density, and  $\Delta s = \Delta \left( h \mu' \frac{\lambda' + \mu'}{\lambda' + 2\mu'} \right)$  is the modulus term (sometimes referred to as the stiffness).

The modulus,  $\Delta s$ , can be readily related to Young's modulus. For an isotropic film,

$$\mu' = \frac{E}{2(1 + \nu)} \quad (4)$$

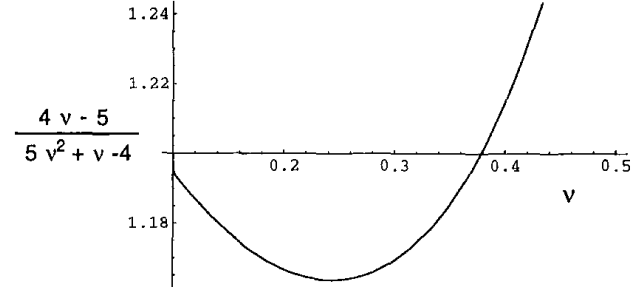


Fig. 1. Plot of prefactor from (7) vs Poisson's ratio,  $\nu$

and

$$\lambda' = \frac{E}{3(1 - 2\nu)} \quad (5)$$

where  $E$  is the Young's modulus and  $\nu$  is the Poisson's ratio. Substituting (4) and (5) into  $\Delta s$  gives

$$\Delta s = \Delta \left( h \mu' \frac{\lambda' + \mu'}{\lambda' + 2\mu'} \right) = \Delta \left( E \frac{h}{4} \frac{4\nu - 5}{(5\nu^2 + \nu - 4)} \right). \quad (6)$$

For typical Poisson's ratios of silicates (0.2 to 0.3) [15], a plot of the  $\nu$  terms in (6) shows the prefactor value to be approximately constant (Fig. 1) at a value of approximately 1.17. Therefore, in the regions of interest, the modulus is nearly proportional to Young's modulus.

The development of an approach to determine both the mass and modulus change contributions to total frequency change is desired. Referring back to (3), it is apparent that a given sensor's sensitivity to either surface change is dependent upon the substrate materials of the sensor. For identical films on sensors of different properties 1 and 2,  $\Delta m$  and  $\Delta s$  are identical, and (3) becomes

$$\Delta f_1 = -\frac{f_{o1} V_{R1}}{4} (R_{y1} + R_{z1}) \Delta m + \Delta s \frac{f_{o1}}{V_{R1}} R_{z1} \quad (7)$$

and

$$\Delta f_2 = -\frac{f_{o2} V_{R2}}{4} (R_{y2} + R_{z2}) \Delta m + \Delta s \frac{f_{o2}}{V_{R2}} R_{z2} \quad (8)$$

or

$$\begin{bmatrix} \Delta f_1 \\ \Delta f_2 \end{bmatrix} = \begin{bmatrix} -\frac{f_{o1}}{4} V_{R1} (R_{y1} + R_{z1}) & \frac{f_{o1}}{V_{R1}} R_{z1} \\ -\frac{f_{o2}}{4} V_{R2} (R_{y2} + R_{z2}) & \frac{f_{o2}}{V_{R2}} R_{z2} \end{bmatrix} \begin{bmatrix} \Delta m \\ \Delta s \end{bmatrix} = \mathbf{S} \begin{bmatrix} \Delta m \\ \Delta s \end{bmatrix}. \quad (9)$$

Eq. (9) represents two equations with two unknowns, which can be inverted to determine the mass and modulus changes:

$$\begin{bmatrix} \Delta m \\ \Delta s \end{bmatrix} = \mathbf{S}^{-1} \begin{bmatrix} \Delta f_1 \\ \Delta f_2 \end{bmatrix}. \quad (10)$$

The mass and modulus components may then be uniquely determined by substituting the known and measured values into (10).

#### IV. EXPERIMENTAL

To test this approach, ST-cut quartz and (001)-cut GaAs SAW sensors operating at 97 and 100 MHz, respectively, were coated with a microporous silicate thin film. The sensors were used as the feedback element of an oscillating circuit. The samples were inserted into a vacuum chamber at 300 K, and dry methanol was dosed into the chamber. The frequency changes for the sample under vacuum and at each relative pressure of methanol were recorded for each of the sensors.

The microporous thin film samples were prepared using a two-step acid-catalyzed (A2) silicate sol described by Brinker and Scherer [16]. The r ratio (water-to-silicon ratio) was 4, and the sol was aged 24 h before storage in the freezer. The A2 sol was allowed to reach room temperature before coating the samples. The SAW samples were cleaned using solvents (slightly heated acetone, methanol, and 1,1,1 trichloroethane) to remove organics. The humidity during coating was controlled using a dry box, and the samples were dip-coated at a rate of 8 in./min.

After coating, the samples were heated in a box furnace at a rate of 1 K/min to a temperature of 673 K and held for 3 h. Then, the samples were outgassed under vacuum for 12 h before analysis with "dry" gas.

#### V. RESULTS

Using the dual-sensor approach, two sensors (quartz and GaAs) were used to determine the mass and modulus contributions to the frequency response during gas adsorption. The velocity and displacement values were determined as accurately as possible from published data.

The surface wave velocity,  $V_R$ , and the normalized mechanical wave displacements,  $R$ , at the "free" electrical boundary condition are tabulated in Table I [14], [17], [18]<sup>2</sup>.

Substituting the values from Table I into (9),

$$\mathbf{S} = \begin{bmatrix} -\frac{f_{oQ}}{4} V_{RQ} (R_{yQ} + R_{zQ}) & \frac{f_{oQ}}{V_{RQ}} R_{zQ} \\ -\frac{f_{oGaAs}}{4} V_{R GaAs} (R_{y GaAs} + R_{z GaAs}) & \frac{f_{oGaAs}}{V_{R GaAs}} R_{z GaAs} \end{bmatrix} \quad (11)$$

$$= \begin{bmatrix} -1.19 \times 10^9 \text{ m}^2/\text{kg s} & 146.8 (N \cdot \text{s}/\text{m})^{-1} \\ -8.32 \times 10^8 \text{ m}^2/\text{kg s} & 390.62 (N \cdot \text{s}/\text{m})^{-1} \end{bmatrix}.$$

<sup>2</sup>Because of the difficulties in determining the "correct" values, GaAs values were determined experimentally, based on two assumptions: 1) the  $c_m$  of GaAs is 70% of that of quartz and 2) the mass uptake is based on measurements of A2 films with 20% porosity. In what was deemed the "most correct" published values for GaAs,  $V$  was 2763, the  $y$  term's coefficient was 3.9e-6, and the  $z$  was 2.9e-6.

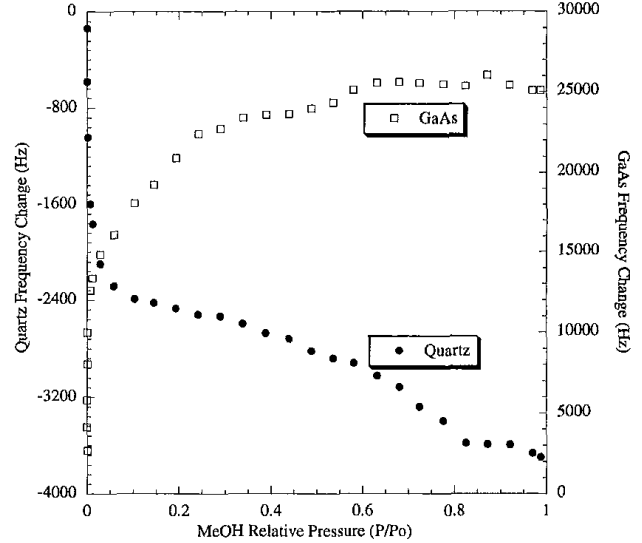


Fig. 2. Frequency response of A2-coated quartz and GaAs SAW sensors to adsorption of methanol.

Inverting (11) gives

$$\begin{bmatrix} \Delta m \\ \Delta s \end{bmatrix} = \begin{bmatrix} -1.141 \times 10^{-9} \text{ kg s}/\text{m}^2 & 4.288 \times 10^{-10} \text{ kg s}/\text{m}^2 \\ -0.00243 \text{ s}/\text{m} \cdot \text{Pa} & 0.00347 \text{ s}/\text{m} \cdot \text{Pa} \end{bmatrix} \begin{bmatrix} \Delta f_Q \\ \Delta f_{GaAs} \end{bmatrix}. \quad (12)$$

These equations are used to calculate the mass and modulus changes from the frequency change of the Quartz and GaAs sensors.

As shown in Fig. 2, the frequency response of the quartz SAW device for increasing relative pressure is a negative shift in frequency; whereas, the frequency response of GaAs SAW (in the same chamber, with the same film) for increasing relative pressure is in the positive direction. This result is less puzzling when two factors are taken into account. 1) Recall from (3) that the frequency responses for mass and modulus are in opposite directions. 2) The GaAs sensor has a greater sensitivity to modulus and is only 70% as sensitive to mass contributions as the quartz sensor [19].

An interesting aspect of this experiment is illustrated in Fig. 3 and Fig. 4, the mass and modulus change plotted as a function of relative pressure. The mass and modulus contributions to the frequency response track one another up until a relative pressure,  $P/P_o = 0.63$ . This is because at low relative pressures, the adsorbate molecule (methanol) adds in a linear way to the mass response,  $n\Delta m$ , as well as to the modulus response,  $n\Delta s$ , where  $n$  is a positive integer. If the adsorbate is of similar size to the pore (such as is the case here), the interaction is essentially adding spring constants to the system, all identical up to the point at which the pores are fully filled. When no more gas can condense in the pores, adsorption occurs on the surface, and there will no longer be additional contributions to the modulus. At that point, the modulus will reach a plateau,

TABLE I

SURFACE WAVE VELOCITIES, PARTICLE WAVE DISPLACEMENTS, AND OPERATING FREQUENCIES FOR QUARTZ AND GAAS SAW SENSORS. THE UNITS FOR  $V_R$  ARE M/S; UNITS FOR  $(R_x)^{1/2}$  ARE  $\frac{\text{M/S}}{(\text{W/M})^{1/2}}$ , AND UNITS FOR  $f_o$  ARE MHZ.

| Property      | ST quartz                         | GaAs                              |
|---------------|-----------------------------------|-----------------------------------|
| $V_R$         | 3158                              | 2800                              |
| $(R_y)^{1/2}$ | $4.2 \times 10^{-6} \omega^{1/2}$ | $1.2 \times 10^{-6} \omega^{1/2}$ |
| $(R_z)^{1/2}$ | $2.8 \times 10^{-6} \omega^{1/2}$ | $4.2 \times 10^{-6} \omega^{1/2}$ |
| $f_o$         | 97                                | 100                               |

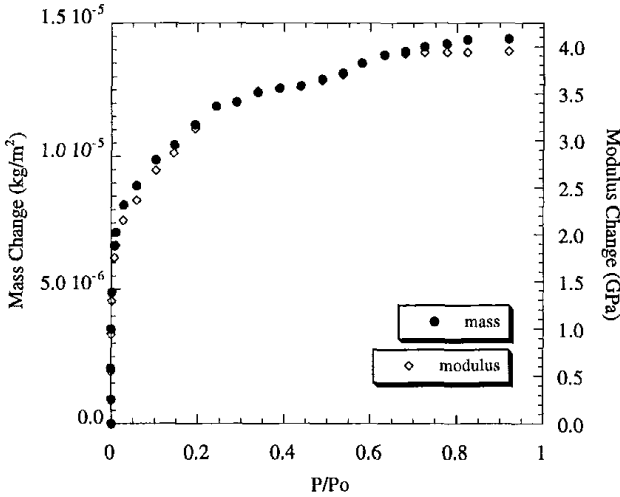


Fig. 3. Mass and modulus changes during adsorption of methanol on an A2 silicate film.

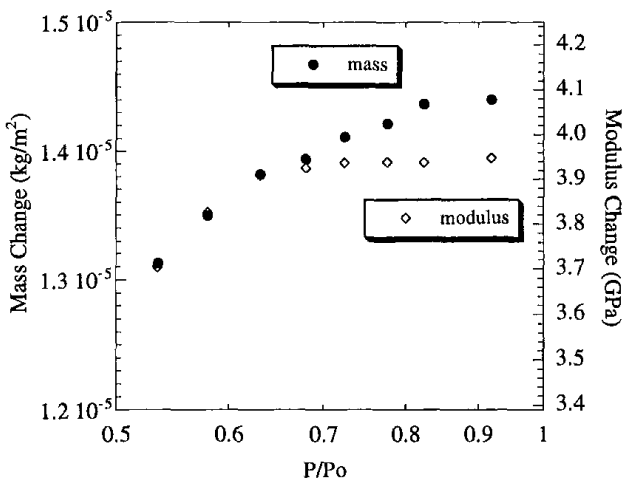


Fig. 4. Detail of the diverging modulus change compared with the mass change (at  $P/P_o > 0.63$ ) during adsorption of methanol on an A2 silicate film.

and the mass will continue to increase linearly with each molecule. Thus, there will be a divergence, as shown in detail in Fig. 4.

The mass and modulus are directly proportional to one another, at low relative pressures, as a result of microporosity. Modulus changes are rarely considered in the studies of mesoporous materials because the pore size is so much greater than the adsorbate molecule. The compressibility of the gas molecule does not enter into the results because the gas molecule is not bounded by both sides to a pore wall. Thus, the modulus effect can be ignored, as is the case for microporous materials after the pores have filled.

Another interesting aspect of this experiment was the calculated modulus change. The modulus changes 34% based on an initial Young's modulus of 9.95 GPa [15] for a silicate film. (The modulus can be calculated by factoring in the prefactor term of 1.17 for a Poisson's ratio of 0.245 [15].) This appears to be a tremendous change based on a porosity of 20% because the bulk modulus of methanol is only 0.773 GPa [20]. However, Gassman studied compressibilities of composite materials and derived an equation to calculate the expected effect. Solving Gassmann's equation for  $\kappa^*$ , the compressibility of the closed container, a 37% change, is calculated, where [21]

$$(\kappa^* - \kappa_M)^{-1} = (\kappa_A - \kappa_M)^{-1} + [(\kappa_F - \kappa_M) \phi_o]^{-1} \quad (13)$$

and  $\kappa_M$  is the material compressibility exclusive of pores (37 GPa for a dense silica) [22],  $\kappa_A$  is the compressibility of the container with the fluid pressure held constant in the interconnected pore system (6.53 GPa for the silica film) [15],  $\kappa_F$  is the fluid compressibility (0.77 GPa for methanol) [20], and  $\phi_o$  is the porosity (20% in this case).

The value calculated from Gassmann's equation is consistent with the value calculated from the SAW data. One criterion of the Gassmann equation is that the system be closed, i.e., that no fluid escapes during the compression of the "container." A quick calculation of the inertial energy term shows that there would have to be 100 times more energy in a SAW wave to liberate the methanol [12].

## VI. CONCLUSIONS

The dual sensor technique, using two different sensor materials, allows for the separation of convoluted contributions to the frequency response. In this example, the mass and modulus contributions were separated out of the frequency response of A2 silicate-coated quartz and GaAs sensors exposed to methanol. With increasing methanol concentration (mass loading), the quartz exhibited a negative frequency shift; whereas, the GaAs sensor exhibited a positive frequency shift.

This was not surprising because Auld's perturbation equations predict a negative contribution to the frequency response for a mass change and a positive contribution to the frequency response for a modulus change. The surprising result from this work was the large magnitude of the calculated modulus change induced in the film, which was caused by the similarity in size of the pores and the adsorbate. The large modulus change was also predicted by the Gassmann equation, which describes the relationship between the elastic properties of a material and the compressibility of a pore fluid.

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