Evaluation of Molecular Interaction between Organic Molecules Physisorbed on Silicon Native Oxide Surface in Dry and Humid Atmosphere

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An experimental method for evaluating the intensity of molecular interactions between organic molecules and surfaces was studied. The organic compounds' physisorption and desorption rates to and from the silicon native oxide surface were in situ measured using a quartz crystal microbalance and analyzed following the multicomponent organic species adsorption-induced contamination model. Three organic compounds, isopropanol, octanol and diethylphthalate, in each single-component system and in a multicomponent system were used in dry and humid atmosphere. The physisorption and desorption rate constants of these compounds in the dry atmosphere were higher than those in the humid atmosphere. However, the molecular radii in both atmospheres were similar. Thus, the interaction between the organic molecules and the surface was influenced by the existence of a thin water layer, while the interaction between the organic molecules had less influence by the atmosphere.

In this study, the QCM and the MOSAIC model were applied to the in situ measurement of the airborne organic molecules physisorption and desorption in a dry atmosphere. The obtained rate parameters and the molecular radii were compared to those in the humid atmosphere which were previously obtained. Based on these results, the difference in the molecular interactions at the surface was evaluated in detail.

Experimental

Fig. 1 shows the QCM system used to measure the physisorption and desorption of organic compounds on a silicon surface in a dry ambient atmosphere and in a humid ambient atmosphere. The QCM sensor (Halloran Electronics Co., Ltd., Tokyo) consisted of a circular-shaped quartz plate, AT-cut, with an 8-mm diameter. Its front and back sides were circular silicon electrodes formed by sputtering. Because the silicon electrode surface was exposed to ambient air, it was covered with a silicon native oxide, the physisorption and desorption condition of which did not depend on the formation method, sputtering or crystal pulling. The difference between the silicon production methods was assumed to be ignorable. Additionally, because of the same reason, this measurement cannot be used for studying the crystal surface influences, such as crystal surface termination, crystalline defects, steps and kinks.

The amounts of the physisorbed and desorbed chemical compounds on the silicon surface were evaluated using the decrease and increase in the frequency of the QCM sensor, respectively. The frequency of the QCM sensor was adjusted to 25 MHz. A decrease in the frequency of 1 Hz showed an increase in the surface concentration of 0.5 ng cm$^{-2}$ for the QCM sensor used in this study.

The gas supply tube and the chamber were made of stainless steel. The gas mixture was introduced at 298 K and atmospheric pressure into the stainless steel chamber, which had a cylindrical geometry with a 200-mm length and a 30-mm inner diameter. The gas mixture flowed in a direction parallel to the surface of the QCM sensor in the chamber. The average gas velocity in the chamber was adjusted to 1,000 cm min$^{-1}$, which corresponded to the typical air velocity in a clean room. The geometry and the gas conditions were exactly the same as those used in our previous study of a humid atmosphere.

The liquid reservoirs were made of a fluorocarbon resin (polytetrafluoroethylene), which was found to cause a negligibly small amount of outgassing after a long purging with nitrogen gas based on our initial evaluation. The isopropanol (IPA), octanol (OCT) and diethylphthalate (DEP) were vaporized by nitrogen gas flowing through the liquid container. The vaporized amounts of these organic compounds in a multicomponent system were very often operated in a dry atmosphere. Additionally, the difference in the molecular interactions at the surface was evaluated in detail.

Preparing an appropriate material surface is a critical operation for producing specific functions at and on the surface. Actually, because various kinds of molecules, organic and inorganic, are adsorbed at the surface and remain there, often as surface contaminations, their behavior should be studied for removing them by any cleaning methods.

Generally, the surface cleaning process consisted of operations utilizing various molecular interactions including physisorption and desorption. Their elemental surface processes are the occupation of a molecule's own area with attraction and repulsion. Additionally, the critical surface contamination for manufacturing the advanced electronic devices is very often at a molecular level. Thus, these molecular interactions should be individually evaluated in order to choose and design the optimum surface preparation process. For this evaluation, a very sensitive and easy measurement method is expected.

For this purpose, the quartz crystal microbalance (QCM) is expected to have a significant potential, because it has a very low detection limit in the ng/cm$^2$ range, which can detect the molecular layer formation, such as airborne molecular contamination. Additionally, by linking it with the rate theory, such as the multicomponent organic species adsorption-induced contamination (MOSAIC) model, the molecular radii of the organic molecules can be evaluated along with the rate parameters of physisorption and desorption. Additionally, this analysis could determine the molecular radii and their summation at the surface for showing the attraction and repulsion between the molecules in a multicomponent system.

In our previous study, the multicomponent organic compounds' physisorption and desorption were evaluated in detail in a humid atmosphere corresponding to that in a clean room for manufacturing advanced semiconductor materials and devices. For the three organic compound systems consisting of isopropanol, octanol and diethylphthalate, the interaction between these organic molecules were shown to be at an insignificant level. They are simply arranged on the thin water molecules layer surface and occupy their own molecular area the same as that in a single-component system. For further advancement to that in a humid atmosphere, these behaviors in a dry atmosphere should be evaluated, because the semiconductor and advanced materials surface are very often operated in a dry atmosphere. Additionally, the difference between the two atmospheres should be discussed. The organic compounds having the behaviors clarified in the previous study are convenient also for this study.

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Manuscript submitted December 8, 2014; revised manuscript received January 14, 2015. Published January 23, 2015.
compounds were obtained by measuring their weight decrease after
the measurement. The gas phase concentration was adjusted by
the nitrogen gas flow rate. The gas phase concentration of IPA was
1900, 3900, 7700 ng cm\(^{-3}\), respectively, in the single-component
system. For the measurement in the multicomponent system, the
gas phase concentration of IPA was 1900, 3900, 5800 and 7700 ng cm\(^{-3}\). That of OCT was 12, 25, 37 and 50 ng cm\(^{-3}\). Additionally, that of DEP was 2, 4, 6 and 8 ng cm\(^{-3}\).

For performing the measurement in a humid ambient atmosphere,
the water was vaporized by nitrogen gas flowing through the liquid
container, as shown in Fig. 1. The vaporized amount of water was
obtained by measuring their weight decrease after the measurement.
The humidity was adjusted by the nitrogen gas flow rate. The relative
humidity (RH) was obtained by dividing the gas phase concentration
of organic compound by the saturated vapor pressure of water. The
water film thickness under this condition is around 100 ng/cm\(^2\) as reported previously.\(^{16,19}\) Considering the water monolayer weight of about 30 ng/cm\(^2\), this suggests that
the water film is comprised of several molecular layers.

**Rate Theory**

**Rate parameters.**— The time-dependent physisorption and desorption behavior of airborne organic compounds on a silicon surface is described using the MOSAIC model\(^{11-13}\) as Eq. 1.

\[
\frac{dS_i}{dt} = (S_{\text{MAX}} - S_{\text{all}})k_{\text{ads},i}C_i - k_{\text{des},i}S_i, \tag{1}
\]

\[
S_{\text{all}} = \sum_j S_j, \tag{2}
\]

in a steady state,

\[
S_i = \frac{(S_{\text{MAX}} - S_{\text{all}})k_{\text{ads},i}C_i}{k_{\text{des},i}}, \tag{3}
\]

where \(i\) and \(j\) indicate the organic compounds, \(S_i\) is the surface concentration of organic compound \(i\) (ng cm\(^{-2}\)), \(t\) is the time (s), \(C_i\) is the concentration of organic compound \(i\) in the gas phase (ng cm\(^{-3}\)), \(k_{\text{ads},i}\) is the physisorption rate constant of organic compound \(i\) on a silicon surface (cm\(^3\) ng\(^{-1}\) s\(^{-1}\)), \(k_{\text{des},i}\) is the desorption rate constant of organic compound \(i\) from the silicon surface (s\(^{-1}\)), and \(S_{\text{MAX}}\) is the higher limit of the concentration of the total organic compounds physisorbed on a silicon surface (ng cm\(^{-2}\)). \(S_{\text{all}}\) is the summation of all organic compounds physisorbed on a silicon surface (ng cm\(^{-2}\)).

In a single-component system and in the steady state, the inverse values of \(S_i\) and \(C_i\) show a linear relationship as described by Eq. 4\(^{12-14}\)

\[
\frac{1}{S_i} = \frac{k_{\text{des},i}}{S_{\text{MAX}}k_{\text{ads},i}C_i} + \frac{1}{S_{\text{MAX}}}. \tag{4}
\]

The values of \(k_{\text{des},i}/k_{\text{ads},i}\) and \(S_{\text{MAX}}\) are obtained using the plot of \(1/S_i\) versus \(1/C_i\).

The occupied molecule area, \(A_i\) (cm\(^2\)), and the molecular radius of compound \(i\), \(r_i\) (cm), are calculated using the \(S_{\text{MAX}}\) value.

\[
A_i = r_i^2\pi = \frac{MW_i}{S_{\text{MAX}} \times N_A}. \tag{5}
\]

where \(MW_i\) is the molecular weight of organic compound \(i\) (g mol\(^{-1}\)). \(N_A\) is Avogadro’s number.

The desorption rate constant is obtained using Eq. 6 along with
the desorption process of an organic compound.\(^{9,14}\)

\[
\frac{dS_i}{dt} = -k_{\text{des},i}S_i. \tag{6}
\]

**S\(_{\text{MAX}}\) and molecular interaction radii in a multicomponent system.**— The \(S_{\text{MAX}}\) value is used for evaluating the molecular interaction radii of the organic compounds in a multicomponent system.\(^{13}\) When the unit area, 1 cm\(^2\), is fully occupied by an organic compound \(i\) (\(i = 1, 2, \ldots\)), its situation is assumed to be described using the maximum population of the organic compound \(i\), \(N_{\text{MAX},i}\) (molecules/cm\(^2\)) and \(r_i\). Thus, the summation of the occupation area by the organic molecules, \(A_{\text{all}}\) (cm\(^-2\)), is expressed as follows:

\[
A_{\text{all}} = 1 = \sum_i N_{\text{MAX},i} r_i^2 \pi. \tag{7}
\]

Here, \(N_{\text{MAX},i}\) is assumed to have a relationship with the \(S_{\text{MAX}}\) of the organic compound \(i\), \(S_{\text{MAX},i}\) (ng cm\(^{-2}\)).

\[
N_{\text{MAX},i} = \frac{S_{\text{MAX},i}}{MW_i \times 10^3} \times N_A, \tag{8}
\]

\[
S_{\text{MAX}} = \sum_i S_{\text{MAX},i}. \tag{9}
\]

10\(^3\) is for the unit conversion from g to ng. Taking into account Eq. 8, Eq. 7 becomes as follows:

\[
A_{\text{all}} = \frac{N_A}{10^3} \sum_i \left( \frac{S_{\text{MAX},i}}{MW_i} \times r_i^2 \pi \right). \tag{10}
\]

From Eq. 1 in the steady state, the ratio between the \(S_{\text{MAX},i}\) values is assumed to be the same as that of the \(S_i\) values and also as that of \(k_{\text{ads},i}/k_{\text{des},i}\) values\(^{13}\) as follows:

\[
S_1 : S_2 : \ldots : S_j : \ldots \equiv \frac{k_{\text{ads},1}C_1}{k_{\text{des},1}}, \frac{k_{\text{ads},2}C_2}{k_{\text{des},2}}, \ldots : \frac{k_{\text{ads},i}C_i}{k_{\text{des},i}}, \ldots \tag{11}
\]

Thus, Eq. 12 is obtained for evaluating the \(S_{\text{MAX},i}\) value.

\[
S_{\text{MAX},i} = S_{\text{MAX}} \left( \frac{k_{\text{ads},i}C_i}{k_{\text{des},i}} \right) \left( \frac{k_{\text{ads},1}C_1}{k_{\text{des},1}} \right)^{-1} \left( \frac{k_{\text{ads},2}C_2}{k_{\text{des},2}} \right)^{-1} \ldots \left( \frac{k_{\text{ads},i}C_i}{k_{\text{des},i}} \right)^{-1} \ldots \tag{12}
\]

When the molecular radii in the multicomponent system are the same as those in the single-component system, the right side of Eq. 10 can produce values near 1.0. However, because any significant attraction and repulsion between the molecules are expected to cause shrinking and expansion, respectively, of the molecular radii, Eq. 10 will not be equal to 1.0. Further details of the MOSAIC model were written in our previous report.\(^{15}\) The time dependent behavior of desorption and physisorption has already been verified by the MOSAIC model.\(^{9,10-15}\)

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**Figure 1.** Quartz crystal microbalance system used to measure the physisorption and desorption of molecules of isopropanol (IPA), octanol (OCT) and diethyphthalate (DEP) on a silicon surface in ambient nitrogen.
Interaction.— The interactions of molecules and surfaces in a multicomponent system are expressed in Fig. 2. Symbols I, D and O indicate the IPA, OCT and DEP, respectively. Fig. 2a is the surface condition in the humid atmosphere. The silicon surface has a native oxide film due to its exposure to the air. The water molecules are physisorbed on the native oxide surface and form a thin water layer. Finally, the organic molecules are physisorbed directly on the silicon native oxide surface. As shown in Figs. 2a and 2b, the organic molecules have an interaction with themselves, other organic compounds and the surface.

The interaction between three organic compound molecules, I_M,M, and that between the molecules and the surface, I_M,S, in the multicomponent system might be similar to or different from the single-component system. When there is a significant attraction and repulsion between the molecules, the \( r_i \) values are expected to decrease and increase, respectively. When the interaction between the molecules and the surface changes corresponding to the dry and humid atmosphere, the \( k_{ad,i} \) and \( k_{de,i} \) values are assumed to become different between the two conditions.

Thus, four possible combinations and behaviors are possible.

(i) \( k_{ad,i} \) and \( k_{de,i} \); same, and \( r_i \); same for the same I_M,S and I_M,M.
(ii) \( k_{ad,i} \) and \( k_{de,i} \); same, and \( r_i \); different for the same I_M,S and different I_M,M.
(iii) \( k_{ad,i} \) and \( k_{de,i} \); different, and \( r_i \); different for the different I_M,S and I_M,M.

Results and Discussion

Single-component system.— Fig. 3 shows the surface concentration of OCT in situ measured in a single component system, at the gas phase concentrations of 30, 60, 90 and 120 ng/cm\(^2\). At 0 s, the gas phase concentration of the OCT, \( C_{OCT} \), was increased from 0 to 30 ng cm\(^{-3}\). Additionally, the OCT value increased to 60, 90 and 120 ng cm\(^{-3}\). Corresponding to each \( C_{OCT} \) value, the surface concentration of OCT, \( S_{OCT} \), increased from about 13 to 23 ng cm\(^{-3}\), as shown in Fig. 3. The IPA and DEP showed a behavior similar to that of OCT.

Fig. 4 shows the desorption behaviors of IPA, OCT and DEP after 0 s, at which the organic compound vapor supply was terminated. The \( k_{de,i} \) was evaluated using these behaviors as list in Table II. The solid lines are the calculations using the obtained \( k_{de,i} \) values.

Figure 5 shows the relationship of \( 1/S \) and \( 1/C \). As shown in this figure, the relationship between these two parameters showed a linear relationship based on Eq. 4. From this figure, the \( k_{ad,i} \) and \( S_e,i \) values were obtained and listed in Table II.

Multicomponent system.— The surface concentration was measured at various combinations of IPA, OCT and DEP, as listed in Table I. Fig. 6 shows the measurement of three organic compounds in the multicomponent system. As shown in this figure, the surface concentration, \( S_{all} \), slightly increased with the increasing \( C_{OCT} \) and the decreasing \( C_{IPA} \) while fixing the \( C_{DEP} \). As listed in Table I, the

### Figures

- **Figure 2.** Interactions between molecules and silicon wafer surface, (a) in humid atmosphere and (b) in dry atmosphere.
- **Figure 3.** Surface concentration of octanol in a single component system at the gas phase concentration of 30, 60, 90 and 120 ng/cm\(^2\).
- **Figure 4.** Surface concentration of IPA, OCT and DEP octanol in a single component system during the desorption process.
- **Figure 5.** Relationship between surface concentration and gas phase concentration of isopropanol, octanol and diethylphthalate.
**Table I. Conditions for three component system.**

| Condition No. | IPA   | DEP   | OCT   | \( S_{\text{eff}} \) (ng cm\(^{-2}\)) | \( A_{\text{eff}}/A_{\text{Si}} \) |
|---------------|-------|-------|-------|-------------------------------------|-------------------------------|
| 1             | A 7703 | 9.6   | 12    | 17                                  | 1.1                           |
|               | B 5777 | 9.6   | 25    | 19                                  | 1.1                           |
|               | C 3852 | 9.6   | 37    | 20                                  | 1.1                           |
|               | D 1926 | 9.6   | 50    | 22                                  | 1.0                           |
| 2             | A 7703 | 1.9   | 62    | 18                                  | 1.1                           |
|               | B 5777 | 3.8   | 62    | 19                                  | 1.0                           |
|               | C 3852 | 5.8   | 62    | 21                                  | 1.1                           |
|               | D 1926 | 7.7   | 62    | 22                                  | 1.1                           |
| 3             | A 1926 | 7.7   | 62    | 22                                  | 1.1                           |
|               | B 3852 | 5.8   | 62    | 20                                  | 1.1                           |
|               | C 5777 | 3.8   | 62    | 18                                  | 1.1                           |
|               | D 7703 | 1.9   | 62    | 17                                  | 1.0                           |

**Table II. Rate parameters and molecular radii, \( R \).**

|          | IPA       | OCT       | DEP       |
|----------|-----------|-----------|-----------|
|          | Dry       | Humid     | Dry       | Humid     | Dry       | Humid     |
| \( k_{\text{ad}} \) (cm\(^3\) ng\(^{-1}\) s\(^{-1}\)) | 4.4 \times 10\(^{-4}\) | 3.4 \times 10\(^{-5}\) | 5.5 \times 10\(^{-3}\) | 1.8 \times 10\(^{-3}\) | 6.5 \times 10\(^{-3}\) | 3.4 \times 10\(^{-3}\) |
| \( k_{\text{de}} \) (s\(^{-1}\)) | 1.33      | 0.18      | 0.293     | 0.109     | 0.046     | 0.014     |
| \( S_{\text{eff}} \) (ng cm\(^{-2}\)) | 18        | 23        | 38        | 50        | 27        | 23        |
| \( A \) (cm\(^2\)) | 5.5 \times 10\(^{-15}\) | 4.3 \times 10\(^{-15}\) | 5.7 \times 10\(^{-15}\) | 4.3 \times 10\(^{-15}\) | 1.4 \times 10\(^{-14}\) | 2.3 \times 10\(^{-14}\) |
| \( R \) (cm) | 4.2 \times 10\(^{-8}\) | 3.7 \times 10\(^{-8}\) | 4.3 \times 10\(^{-8}\) | 3.7 \times 10\(^{-8}\) | 6.7 \times 10\(^{-8}\) | 8.6 \times 10\(^{-8}\) |
| \( R/R_{\text{IPA}} \), Humid (+) | 1.1       | 1.0       | 1.2       | 1.0       | 1.8       | 2.3       |
not change. Based on the information obtained in this study, the dry and humid atmospheres only change the surface condition, while they do not change the interactions between the organic compounds. This information helps in designing the surface preparation process.

This study was concentrated to the significantly low level surface contamination, such as the monolayer physisorption occurring in a clean room at room temperature. Generally, there are more complicated surface contamination, such as the quick and multilayer physisorption at higher concentrations, pressures and temperatures. The information obtained from the complicated conditions will show the details of hydrogen bonding and the temperature low enough not to cause the molecular physisorption.

Conclusions

The interactions among various organic molecules and surfaces were experimentally evaluated using a quartz crystal microbalance, the information of which was analyzed based on the rate theory. This study used three organic compounds, i.e., isopropanol, octanol and diethylphthalate, each in a single-component system and in a multi-component system. The physisorption and desorption rate constants of these compounds in a dry atmosphere were different from those in the humid one. However, the molecular radii in both atmospheres were similar. Thus, the interaction between the organic molecules and the surface was considered to be influenced by the existence of a thin water layer, while the molecular interaction between the organic molecules had negligible influence by the atmosphere.

Acknowledgments

The authors thank Nobuyoshi Enomoto of Halloran Electronics Co., Ltd., for his helpful suggestions and support.

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