

MATERIALS AND INTERFACES

Short Time Deposition Kinetics of Diethyl Phthalate and Dibutyl Phthalate on a Silicon Wafer Surface

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Short time adsorption and desorption behaviors of two alkyl phthalate esters, namely diethyl phthalate (DEP) and dibutyl phthalate (DBP), on silicon wafers exposed under various ambient concentrations were experimentally and theoretically investigated. The results showed that the surface density of DBP was significantly affected by both the length of exposure time and its ambient concentration, whereas that of DEP was only affected by its ambient concentration within the tested periods between 60 and 240 min. The determination of rate parameters for adsorption and desorption showed that the rate constants of DEP were always larger than those of DBP. Also, the sticking coefficient of DEP was larger during the initial adsorption stage due to its relatively lower molecular weight as compared to DBP. The value of the sticking coefficient for DEP, however, decreased much faster such that the value eventually became smaller than that for DBP. Therefore, for silicon wafers experiencing a short exposure time, organic compounds with lower molecular weights may be a more important source of airborne molecular contamination than those with higher molecular weights.

1. Introduction

Ever since fabrication technology advanced into a frontier in which the feature size of semiconductor devices is miniaturized below 100 nm, the airborne molecular contaminants (AMCs) in cleanroom environments have been recognized as a potential contamination source causing yield reduction and performance deterioration of semiconductor devices.^{1–3} Among all AMC classifications, organic species adsorbing on wafer surfaces have been probed to induce surface defects such as increased surface roughness, formation of haze, streaking, damage to epitaxial growth, and degradation of gate oxide integrity.^{4–7} Henceforth, the threshold limits of surface organic contamination that induce thin gate-oxide film deterioration have been reported in the literature.^{3,8–10} For instance, it was documented that device characteristics could be degraded if the organic molecular density on a bare silicon surface exceeds approximately 1×10^{13} C atoms cm^{-2} .⁹ The increasing concern over the problems associated with AMCs eventually led to a technical specification in the 2003 version of the International Technology Roadmap for Semiconductors (ITRS), which recommended that a maximum allowable carbon impurity of less than 1.5×10^{13} C atom cm^{-2} must be achieved for the 90-nm technology node.¹⁰

Trace condensable organic AMCs (oAMCs) are ubiquitously found in cleanroom ambience through outgassing from material surfaces and coatings. The organo-polymeric additives, most notably alkyl phthalate esters, outgassed from the polymeric materials generally exhibit the common properties of high

boiling points (bp's) as well as low vapor pressures, rendering these compounds highly susceptible to remaining on wafer surfaces once deposited.^{11,12} Consequently, although the ambient concentrations of the organic vapors with high bp's (e.g., phthalate esters) are much lower than those of the volatile organic compounds with low bp's (e.g. toluene) in a typical cleanroom, the condensable organic vapors with high bp's have generally been recognized to possess faster surface deposition rates than the volatile organic compounds.⁸

In a number of recent studies, the deposition kinetics of several phthalate compounds on silicon wafers have been reported for long exposure periods up to several days.^{13–16} However, in actual semiconductor fabrication processes, the wafer exposure time is most frequently limited to only a few hours. Nonetheless, it is possible that, even at a short exposure time, the wafer surface density of oAMCs with relatively lower molecular weight (M_w) could exceed the threshold contamination levels to result in device deterioration.^{17–19} Besides, the adsorption and desorption rate constants of oAMCs have usually been evaluated based on the actual data from cleanroom ambiances contaminated with several organic compounds.^{14–17} This approach may lead to biased results because the composition as well as the concentration of the oAMCs in cleanrooms may vary from plant to plant. In contrast, limited information on the deposition behavior of single phthalate compounds under a controlled environment is available in the literature. Furthermore, the influence of air velocity on the deposition of organic contaminants on silicon surfaces has been discussed in numerous studies,^{17,20,21} while the event of deposition under stagnant conditions (e.g., wafers in storage boxes or mini-environments) has not been adequately addressed.

This study intends to investigate the deposition rates of diethyl phthalate (DEP) and dibutyl phthalate (DBP), each representing a single-component contamination, on silicon wafers under

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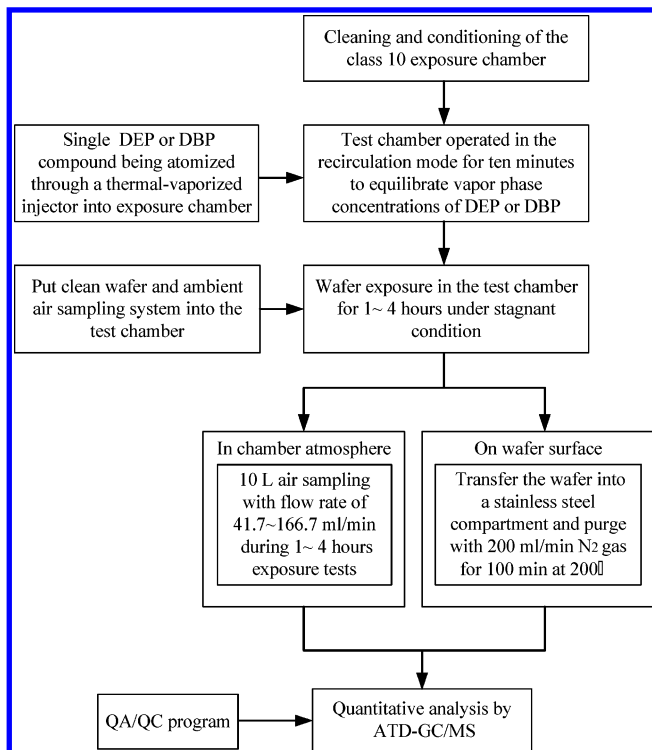


Figure 1. Experimental procedure of wafer exposure tests in the chamber.

various ambient concentrations and exposure times up to 4 h. The deposition rate parameters of DEP and DBP are determined by fitting the rate equation with the experimental data through an optimization algorithm. The differences in the deposition behavior between these two phthalate compounds under negligible flow conditions are discussed. In addition, a derived parameter, namely the sticking coefficient, is calculated based on the kinetic parameters.

2. Experimental Section

Standard 150-mm silicon wafers with crystal orientation in the (100) plane were used as the deposition “witness” wafers. The wafers were coated with an SiO₂ thin-film by a thermal oxidation furnace (980 °C, film thickness of 550 nm) to give a hydrophilic surface and were precleaned via the standard RCA method prior to the ensuing exposure experiments. Reagent-graded DEP (C₁₂H₁₄O₄, with a *M_w* of 222 g/mol and a bp of 299 °C) and DBP (C₁₆H₂₂O₄, 278 g/mol and 340 °C), Acros Organics, USA, both belonging to a group of phthalate compounds widely used as plasticizers in the production of polymeric materials such as poly(vinyl chloride) (PVC), were selected as the model contaminants representing the condensable oAMCs in this study. These two condensable compounds had also been demonstrated as the major components outgassed from wafer storage boxes.^{22,23}

The experimental procedure for the wafer exposure test in a test chamber was described in Figure 1. A clean gastight chamber (interior dimension: *L* × *W* × *H* = 120 cm × 60 cm × 180 cm) with the full function of air recirculation and filtration (equivalent to ISO class 4)²⁴ was employed in this study. The experimental setup allowed for precise temperature and humidity control at 22 ± 1 °C and 40 ± 3%, respectively, in the test chamber. To maximize the cleanliness prior to the wafer exposure experiments, the chamber was purged with a steady stream of purified air through activated carbon adsorption and HEPA filtration at flow rates ranging between 30 and 40 L

min⁻¹ for at least 24 h. In addition, the entire chamber system was located in an ISO class 6 cleanroom to minimize environmental contamination of organics and particles during wafer transfer in to and out of the chamber.

Prior to the placement of a witness wafer into the test chamber, approximately 10–100 μg of liquid DEP or DBP was individually atomized through a thermal-vaporized injector (Perkin-Elmer, USA) into the test chamber. The test chamber was operated temporarily in a recirculation mode to help equilibrate the vapor-phase DEP or DBP concentration, which normally occurred within 10 minutes of mixing by air circulation in the chamber. The equilibrium vapor concentrations of DEP and DBP in the test chamber were in the range between 2 and 30 μg m⁻³.

The analytical procedures for the trace analyses of the phthalate esters in the chamber ambience as well as on the wafer surfaces were successfully developed in our previous study.²⁵ To briefly describe the procedure, 10 L of air sample from the test chamber was periodically collected by Tenax GR tubes (Supelco, USA) during the course of each wafer exposure experiment. The sampling flow rate was typically maintained at 41.7 mL min⁻¹, corresponding to a face velocity less than 0.02 m sec⁻¹ into the adsorption tube with a 1/4-in. opening. The sampling face velocity was considered sufficiently low such that perturbation by convective transport for deposition could be neglected at distances far away from the sampling location. The sampling tubes were then subjected to analysis by a gas chromatography/mass spectrometry (GC/MS) system (Perkin-Elmer, USA) equipped with an autothermal desorption (ATD) unit. For the surface analysis, a custom-designed heat-desorption compartment made of stainless steel and internally coated with Teflon was employed. The compartment containing the contaminated wafer was heated to 200 °C for 100 min in a stream of ultrahigh purity nitrogen purge gas at a flow rate of 200 mL min⁻¹, and the desorbed DEP or DBP molecules were then collected by Tenax GR tubes for the subsequent ATD-GC/MS analyses. This method is similar to the one specified in SEMI MF1982–1103.²⁶

3. Model

3.1. Deposition Kinetics of DEP and DBP on SiO₂ Wafers.

The Langmuir-type model has been demonstrated to adequately describe the time-dependent surface concentration for single-component oAMC contamination.^{14–17} This model consists of first-order rates of adsorption and desorption as expressed in the following equation:

$$\frac{dS}{dt} = (S_{\max} - S)k_{\text{ad}}C - k_{\text{de}}S \quad (1)$$

where *S* represents the surface density (ng cm⁻²), *C* is the ambient concentration (ng cm⁻³), *S_{max}* is the maximum surface adsorption capacity (ng cm⁻²), *t* is the exposure time (min), *k_{ad}* and *k_{de}* are the intrinsic rate constants for adsorption (cm³ ng⁻¹ min⁻¹), and desorption (min⁻¹), respectively. Integration of eq 1 leads to the following analytical solution:

$$S(t) = \left(\frac{S_{\max}k_{\text{ad}}C}{k_{\text{ad}}C + k_{\text{de}}} \right) [1 - e^{-(k_{\text{ad}}C + k_{\text{de}})t}] \quad (2)$$

In this study, the rate parameters (*k_{ad}* and *k_{de}*) are determined using a numerical algorithm based on the least-squares error method. By employing the measured value of *C* and a predetermined value of *S_{max}* (which will be described in the

following section), along with a set of trial values of k_{ad} and k_{de} , the value of S can be calculated from eq 2. The values of the rate parameters are then optimized based on the minimum mean-square error (E_{min}) between the measured ($S_{measured}(t)$) and the calculated ($S_{calculated}(t)$) values of S at different exposure times, as expressed in eq 3:

$$E_{min} = \sum_t [S_{measured}(t) - S_{calculated}(t)]^2 \quad (3)$$

3.2. Maximum Surface Adsorption Capacity. The values of the maximum surface adsorption capacity (S_{max}) have been estimated to be in the range from 2.5 to 54 ng cm⁻² in the literature.^{14–17} The broad range of the values of S_{max} reported in the literature could be attributed to a number of reasons, including the variation in the wafer surface properties as well as the coexistence of different compounds on a wafer surface. The results from the wafer exposure experiments in our previous study²⁷ revealed that the surface density of DEP was approximately 12 ng cm⁻² for ambient concentrations higher than 100 μg m⁻³, suggesting that S_{max} must be greater than 12 ng cm⁻². Therefore, the value of S_{max} in the range between 10 and 60 ng cm⁻² was employed in this study to evaluate the sensitivity of the rate parameters (k_{ad} and k_{de}) to the values of S_{max} .

In addition to the knowledge of S_{max} value from the literature, its theoretical value can also be estimated by conceptualizing a full monolayer coverage (i.e., hexagonal close packed) with spherical DEP/DBP molecules on the wafer surface. Since the molar volume of the liquid is made up of the sum of the molecular volumes of N molecules, plus a negligible tiny volume between molecules,²⁸ the molecular volume occupied by each molecule can be obtained by dividing the liquid molar volume by Avogadro's number. Consequently, the molecular radii of DEP and DBP were computed to be 4.3 Å and 4.7 Å, corresponding to an S_{max} of 57.9 and 59.6 ng cm⁻², respectively, for DEP and DBP. These values, in turn, were regarded as the reference values for the determination of the rate parameters.

3.3. Sticking Coefficients. When a flux of molecules impinges onto a surface, only a fraction of the molecules will actually remain on the surface. This fraction is conventionally termed as the "sticking coefficient" to provide a measure of the molecular propensity to a surface. As proposed by Sematech International,⁶ the effective adsorption rate (R_{dep} , ng cm⁻² s⁻¹) of organic contaminants can be related to the sticking coefficient (E) by the following expression:

$$R_{dep} = E \frac{CV_T}{4} \quad (4)$$

In eq 4, V_T (cm s⁻¹) is the average thermal velocity of the approaching molecules given by

$$V_T = \sqrt{\frac{8k_B T}{\pi m}} \quad (5)$$

where k_B stands for the Boltzmann constant (1.38 × 10⁻¹⁶ dyne cm K⁻¹), T is the deposition temperature (K), and m is the molecular weight. Therefore, using eq 6, the time-average of the sticking coefficient, E_{ave} , for an organic compound can be calculated insofar as the change in the wafer surface density over a period of time ($\Delta S/\Delta t$) is experimentally determined.

$$E_{ave} = \frac{R_{dep}}{C \frac{V_T}{4}} = \frac{\Delta S/\Delta t}{J_{in}} \quad (6)$$

Realistically, the rate of molecular deposition (R_{dep}) is a dynamic event that decreases with increasing surface coverage and exposure time, and henceforth, the value of the sticking coefficient should also decrease as the exposure time is extended. The time-dependent function of the sticking coefficient, $E(t)$, under a stagnant condition, can thus be obtained by incorporating eq 1 or the differential form of eq 2 into eq 6:

$$E(t) = \frac{dS}{C \frac{V_T}{4}} = \frac{S_{max} k_{ad}}{V_T} e^{-(k_{ad}C + k_{de})t} \quad (7)$$

where the values of ($CV_T/4$) reflect the total mass impingement flux (J_{in}) of organic molecules onto the wafer surface under stagnant air conditions.

4. Results and Discussion

4.1. Determination of Kinetic Parameters of DEP and DBP. The value of the maximum surface adsorption capacity (S_{max}) has a significant impact on the values of k_{ad} and k_{de} . However, it was difficult to experimentally obtain the precise value of S_{max} due to a variety of uncertainties, as mentioned earlier in this article. Therefore, a sensitivity study on the values of S_{max} within the range reported in the literature was conducted to assess its effect on the values of the rate parameters (k_{de} and k_{ad}). Figure 2 shows the results of the sensitivity study for the values of S_{max} ranging from 10 to 60 ng cm⁻². It was observed that the value of k_{ad} was significantly affected by S_{max} , as manifested by the sharp decrease from 0.51 to 0.09 cm³ ng⁻¹ min⁻¹ for DEP and from 0.40 to 0.07 cm³ ng⁻¹ min⁻¹ for DBP when S_{max} was increased from 10 to 60 ng cm⁻². On the contrary, the value of k_{de} was less sensitive to S_{max} as it was only slightly increased from 0.022 to 0.030 min⁻¹ for DEP and from 0.008 to 0.014 min⁻¹ for DBP as S_{max} increased from 10 to 60 ng cm⁻². One can also note that the value of k_{de} approached an asymptotic value of around 0.03 and 0.014 min⁻¹ for DEP and DBP, respectively, at larger values of S_{max} . The independence of k_{de} to S_{max} at larger values of S_{max} could also be justified by eq 1, in which k_{de} was not directly linked to S_{max} as was k_{ad} . This result also validated the assertion that the reference values of $S_{max} = 57.9$ and 59.6 ng cm⁻² for DEP and DBP, respectively, were appropriate for the subsequent determination of the rate parameters. Furthermore, it was noted that the rate parameters of DEP were always greater than those of DBP regardless of the assumed values of S_{max} . In the present study, with the value of S_{max} at 57.9 ng cm⁻² for DEP and 59.6 ng cm⁻² for DBP, the resulting optimized values of k_{ad} were 0.091 and 0.072 cm³ ng⁻¹ min⁻¹ and those of k_{de} were 0.03 and 0.014 min⁻¹ for DEP and DBP, respectively.

4.2. Time Dependence of DEP and DBP Adsorption on a Wafer Surface. Figure 3 shows the simulated and measured surface concentrations of DEP and DBP on silicon wafers as functions of exposure time under different ambient concentrations. It was observed in Figure 3a that DEP reached a quasi-steady-state surface density in a rather short exposure time (within the initial 60 min). This result was further substantiated by the well-behaved linear correlation (linear-regression coefficient $R^2 = 0.82$ among 45 data points) between the surface

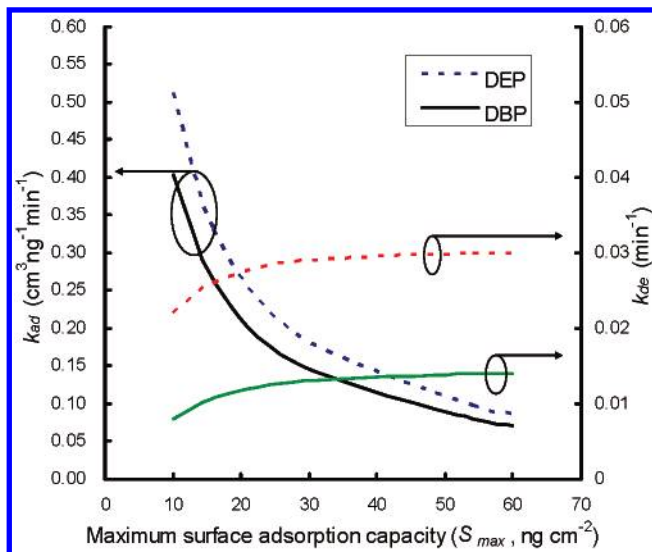


Figure 2. Optimum values of desorption rate constant (k_{de}) and adsorption rate constant (k_{ad}) computed based on the best-fit curves between the calculated and experimental profiles with different values of S_{max} .

density of DEP and its ambient concentration for the exposure durations of 60–240 min. Evidently, the surface density of DEP was predominantly dictated by its ambient concentration, whereas the exposure time (within the test periods of 60–240 min) had a minimal effect on the surface density of DEP.

In contrast, the surface density of DBP progressively increased with the exposure time, as shown in Figure 3b, especially for the higher ambient concentration. Clearly, the surface density of DBP depended on both the exposure time and the ambient concentration. One can also note by comparing Figure 3a and b that, at the relatively longer exposure duration of 240 min, the surface density of DBP was larger than that of DEP under similar ambient concentrations. This observation can be interpreted by the greater surface adherence potential, i.e., the largest value of surface density obtained at equilibrium, for compounds with higher M_W or lower value of k_{de} (e.g., DBP) than compounds of low M_W (e.g., DEP).

As mentioned earlier, the value of k_{ad} for DEP was always larger than that for DBP, indicating that the impingement flux of DEP molecules on the wafer surface was larger than that of DBP molecules due to the smaller M_W of DEP. This result was consistent with the studies by Zhu,^{29,30} who reported that the adsorption rate constant was reversely proportional to the square root of the M_W of an organic compound. As a molecule of lower M_W approached a surface in stagnant air, it would carry a higher average thermal velocity (eq 5) that would lead to a faster arrival rate as it impinged onto the surface.

The DEP molecules also possess a larger value of k_{de} than the DBP molecules, reflecting that the surface density of DEP would reach a quasi-steady-state value faster than that of DBP. This could also be explained by the propensity of organic molecules having lower M_W (or higher saturation vapor pressure) to regain energy as they strike a wafer surface, and thus, they tend to desorb from the surface at much faster rates.

4.3. Determination of Sticking Coefficient. On the basis of eq 6, the time-average values of the sticking coefficient for DEP and DBP under stagnant air over 4 h of exposure were $(3.1 \pm 0.2) \times 10^{-6}$ and $(5.7 \pm 0.6) \times 10^{-6}$, respectively, while they were $(9.8 \pm 0.4) \times 10^{-6}$ and $(1.4 \pm 0.1) \times 10^{-5}$ over 1 h of exposure. As a comparison, Veillerot et al.²⁰ estimated the values of E_{ave} to be $(6.7 \pm 2.8) \times 10^{-5}$ and $(8.9 \pm 2.7) \times 10^{-5}$ for DEP and DBP, respectively, over 24 h of exposure with a

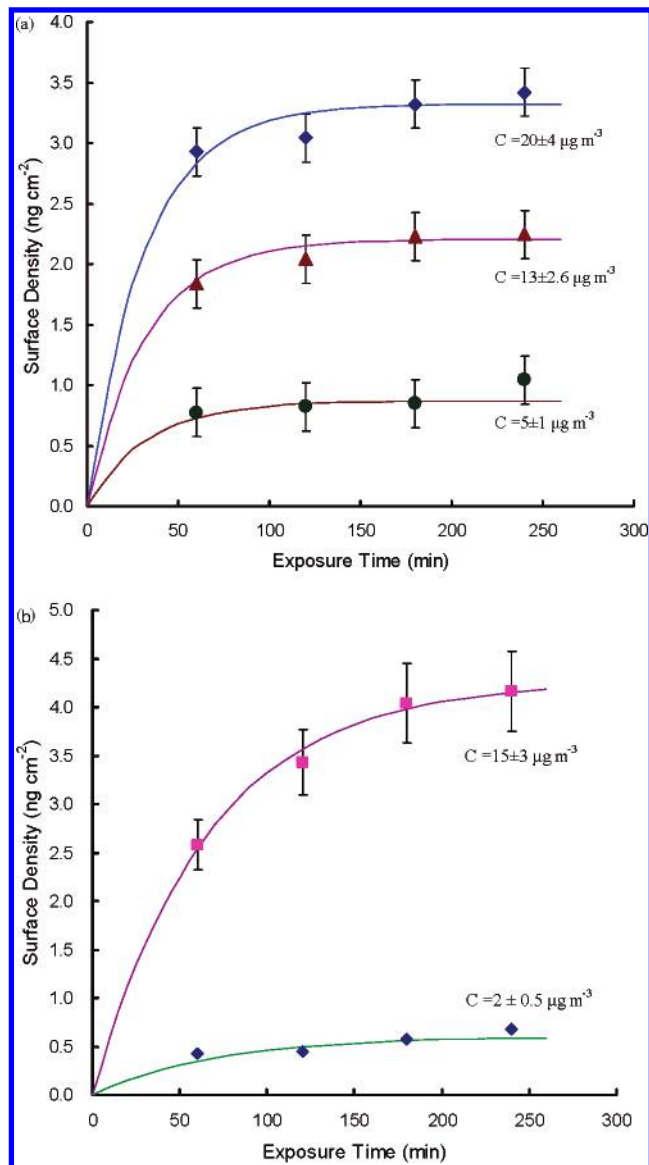


Figure 3. (a) Simulated and measured surface DEP concentration as a function of exposure duration. The estimated value of k_{ad} was $0.091 \text{ cm}^3 \text{ min}^{-1} \text{ ng}^{-1}$, k_{de} was 0.03 min^{-1} , and S_{max} was 57.9 ng cm^{-2} . (b) Simulated and measured surface DBP concentration as a function of exposure duration. The estimated value of k_{ad} was $0.072 \text{ cm}^3 \text{ min}^{-1} \text{ ng}^{-1}$, k_{de} was 0.014 min^{-1} , and S_{max} was 59.6 ng cm^{-2} .

vertical air flow velocity of 0.44 m s^{-1} , as referred to in Table 1. These values were greater than those obtained in this study by an order of magnitude. One of the major causes of the discrepancies was the difference in the flow condition (i.e., stagnant air vs laminar flow). In their study, the molecular movement was dominated by the external laminar flow field, and thus, the thermal velocity ($V_T/4$) in eq 6 should be replaced by the vertical laminar flow velocity, u . Therefore, the expression for the averaged sticking coefficient (E_{ave}) could be modified into

$$E_{ave} = \frac{\Delta S / \Delta t}{Cu} \quad (8)$$

As a consequence, the average thermal velocities of DEP and DBP used in the present study were both nearly 2 orders of magnitude greater than the laminar velocity used in Veillerot's study, thereby yielding much smaller values of E_{ave} in this study.

Another important factor for determining E_{ave} was the length of the exposure time (≤ 4 vs 24 h). As an illustration, the time-

Table 1. Sticking Coefficients of DEP and DBP on the Silicon Wafer Surface

authors	compounds	sticking coefficient	conditions
Veillerot ²⁰ (2003)	DEP	$(6.7 \pm 2.8) \times 10^{-5}$	1. under laminar air flow velocity of 0.44 m s^{-1}
	DBP	$(8.9 \pm 2.7) \times 10^{-5}$	2. determined for first 24 h of exposure.
this study	DEP (0–1 h)	$(9.8 \pm 0.4) \times 10^{-6}$	1. $V_T/4 = 41.94$ and 37.48 m s^{-1} for DEP and DBP, respectively, under stagnant air
	DEP (0–4 h)	$(3.1 \pm 0.2) \times 10^{-6}$	2. determined over 1 h and 4 h of exposure
	DBP (0–1 h)	$(1.4 \pm 0.1) \times 10^{-5}$	
	DBP (0–4 h)	$(5.7 \pm 0.6) \times 10^{-6}$	

dependent sticking coefficients of DEP and DBP were calculated according to eq 7 for ambient concentrations of 2 and $20 \mu\text{g m}^{-3}$, as shown in Figure 4. It was observed that the values of the sticking coefficient for both DEP and DBP declined with the exposure time and asymptotically approached zero. In addition, the profile of E at the higher ambient concentration ($20 \mu\text{g m}^{-3}$) declined faster than that at the lower ambient concentration ($2 \mu\text{g m}^{-3}$), a result that could be attributed to a faster monolayer surface coverage rate at a higher ambient concentration. This tendency was particularly revealing for DBP because the value of k_{de} was significantly smaller than that of DEP, and thus, the $k_{\text{ad}}C$ term in eq 7 became increasingly predominant in determining the rate of exponential decay of $E(t)$. Indeed, if one were to characterize the sticking coefficient of DOP, which essentially does not desorb (i.e., $k_{\text{de}} \approx 0$) from wafer surfaces,^{14,15} the rate of exponential decay between high and low ambient concentrations would have resulted in an even greater difference.

As shown in Figure 4, the sticking coefficient of DEP appeared to be larger than that of DBP for the initial 10 min of exposure time, followed by a rapid decline, and eventually became smaller than that of DBP. This result revealed that the phthalate compounds with lower M_{W} had relatively larger values of sticking coefficient and surface density during the initial adsorption stage. These observations were consistent with the experimental results shown in Figure 3a and b. Furthermore, for a short exposure time, the condensable oAMCs with lower M_{W} might be a more important source of contamination than the ones with higher M_{W} .

It was also observed from Figure 4 that only slight differences exist between the sticking coefficient profiles corresponding to high ($20 \mu\text{g m}^{-3}$) and low ($2 \mu\text{g m}^{-3}$) ambient concentrations for both DEP and DBP. This result was due to the large values of S_{max} (corresponding to larger k_{ad}) employed in this study. For smaller values of S_{max} , the differences would be more

significant. In addition, due to the smaller k_{de} for an organic compound with relatively higher M_{W} , one can expect that the values of E at a higher ambient concentration would decline at a much faster rate than those at a lower ambient concentration.

5. Conclusions

This study investigated the surface deposition behavior of DEP and DBP at short exposure times (60–240 min) in a well-controlled test chamber. The results showed that the surface density of DBP was strongly affected by both exposure time and ambient concentration, whereas the surface density of DEP was only influenced by the ambient concentration within the tested periods of 60–240 min. The surface adherence potential of DBP on a wafer surface was generally greater than that of DEP. However, this may not be true for short exposure times within 60 min and/or high ambient concentrations because of the relatively slower rate of DBP reaching an equilibrium surface concentration.

S_{max} was a critical parameter to the deposition kinetics, as evidenced by its profound influence on the values of k_{ad} for both DEP and DBP. In comparison, the values of k_{de} were less sensitive to S_{max} . Furthermore, the rate parameters (k_{ad} and k_{de}) of DEP were always larger than those of DBP regardless of the values of S_{max} . Generally speaking, phthalate compounds with lower M_{W} exhibit a relatively higher time-dependent sticking coefficient during the initial adsorption stage, but it also decreases with time faster than the phthalate compounds with high M_{W} . Therefore, for longer exposure time, the sticking coefficient of high M_{W} phthalate compounds became larger.

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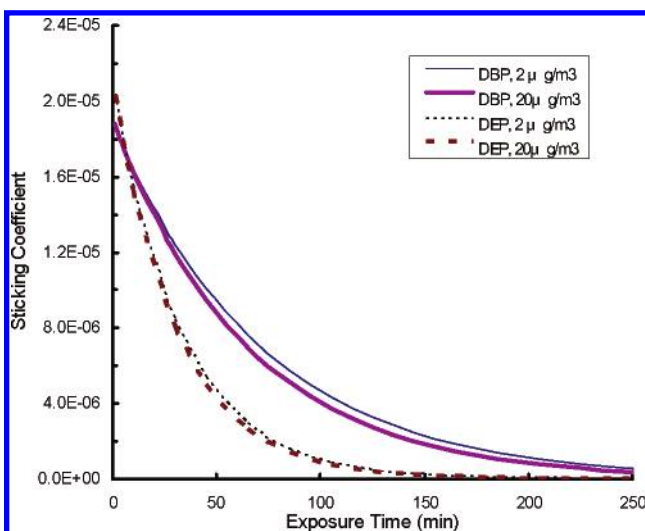


Figure 4. Time-dependent function of the sticking coefficient for single DEP and DBP compounds at ambient concentrations of 2 and $20 \mu\text{g m}^{-3}$.

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