Measurement of Lateral Removal Force for a Baked Polymer Particle on a Glass Plate

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It is important to evaluate the particle removal force in surface cleaning techniques using physical action. In this study, we measured a lateral removal force of highly adhered polymer particles on a glass plate. The polymer particles were baked on a glass plate at different temperatures to control adhesion. The removal force was measured with a self-sensitive cantilever in open-air and underwater conditions. As a result, the removal forces in both air and water were almost identical, of the order of $10^2 \mu N$. The adhesion forces of these particles were approximately $10^3$ times higher than particles sprinkled on the plate without baking. In addition, two types of force curves were obtained depending on the heating temperature and duration. At high and low heating temperatures, the force curve increased gradually, showing a clear maximum value and sharp decrease. The particles were removed when the force achieved its maximum. However, under the middle heating temperature and short duration, the force curve gradually increased and then gradually decreased. Several scanning times were needed to remove the particles. This result indicated that the particles gradually reduced their binding forces. It was found that duration is an important factor for particle removal, in addition to the force acting on the particles.

Keywords: Cleaning, Particle removal, Force measurement, Baking, Adhesion

1. Introduction

During the process of manufacturing semiconductor devices, particle contamination is among the critical problems; especially, high adhesion particles, which are often generated after dry etching or planarization processes are very difficult to remove [1]. In the evaluation of cleaning performance for such applications, especially using physical force cleaning techniques such as droplets impingements [2], ultrasonic cavitation [3], and brush scrubbing [4], standard contaminated samples are very important.

To evaluate the removal force for contaminating particles, many trials such as AFM measurements [5], wind tunnel tests [6], and so on, are reported. They measured the order of nN forces of particle adhesion. However, it is a difficult task to control the high adhesion force of particles and make such standard samples.

We proposed a contaminated sample with high adhesion force particles using resin particles [7]. In this method, particles were sprinkled on a glass plate and baked. Using a heating temperature that is higher than the heat resistance temperature, a high adhesion force was obtained. We demonstrated the potentiality of this sample through a comparison of the cleaning performance between PVA brush scrubbing and the droplet train [7].

In this study, we investigated these high adhesion force particles in more detail. We changed the heating temperature and measured its removal forces with a self-sensitive cantilever in
both open-air and underwater condition. We discuss the obtained force curves and different removal mechanisms depending on the heating temperature.

2. Experimental

2.1. Particle preparation

We produced high adhesion force samples with resin particles (cross-linked polyacrylic acid ester, AFX-30, Sekisui Plastics Co., Ltd.) on a glass plate (soda-lime silicate glass, S7224, MATSUNAMI). The average particle diameter was 30 μm. The schematic of the sample preparation is shown in Fig. 1(a). The resin particles were sprinkled using a sieve in a high moisture atmosphere. Afterwards, the samples were heated at $T_h = 100, 200, 290 \, ^\circ C$ for $t_h = 10$ or 60 min by changing the heating temperature and time. An example of temperature rise during heating is shown in Fig. 1 (b). The time to reach the target temperature was 15 min for 100 ºC, 30 min for 200 ºC, and 45 min for 290 ºC. The samples cooled naturally. Figure 1 (c) shows the case of temperature decreases at a room temperature of 18 ºC. The particle images after preparation are shown in Fig. 1 (c). When heated to 290 ºC, which is a temperature higher than the heat resistance temperature of the particles, the color of the particles turned brown. Note that the heat resistance temperature of this particles is around 240 ºC (230 ~ 250 ºC).

2.2. Removal force measurements

The adhesion force of the particles was measured using a self-sensitive cantilever (NPX1CTP004, Hitachi High-Tech Science). In this study, we selected the spring constant as 40 N/m. This measurement system was originally developed for the measurement of cell adhesion force [8]. The system was set on an inversed microscope (ECLIPSE Ti-U, Nikon) and the removal motion was observed from the bottom.

Figure 2 shows the overview of the experimental setup. The cantilever movements were controlled
by a pico-motor (8301NF, 8353, New Focus Technologies) and piezoelectric element (APA120S, CEDRAT Technologies) in coarse and micro motions, respectively. To control the piezoelectric element, a triangular waveform generated by a function generator was used. The traverse speeds of the cantilever were controlled by changing the signal frequency. In this study, we changed the traverse speed from 0.2 to 300 μm/s. In all experiments, the cantilever moved horizontally, and then returned to its original position. This method can be extended to measurements in liquids using platinum wire, as shown in Fig. 2 (b). The position of the contact between particles and cantilever is shown in Fig. 2 (c).

3. Results and discussion
3.1. Force curves
A typical example of the two kinds of force curves is shown in Fig. 3. The vertical axis denotes force $F$ obtained from the cantilever strain, and the horizontal axis is time $t$. As shown in Fig. 3 (a), after the contact of the cantilever and particle, the cantilever strain continues to increase, until it decreases immediately after the maximum force value $F_{\text{max}}$. However, in Fig. 3 (b), the force gradually rises, similar to the case represented in Fig. 3 (a); however, subsequently, the cantilever gradually returns to the receiving forces. Here, let us assume that the conditions representing the graphs in Figs. 3 (a) and (b) are Type 1 and Type 2, respectively. Type 1 particles could be removed with a single scan, but Type 2 particles returned to their original positions after scanning; multiple operations were needed to remove these particles. Figure 3 (c) shows the actual image observed from the bottom. As shown in the figure, the cantilever approaches the particle, and then the particle moves from its initial position. These images show that the particles were surely removed by the cantilever movement.

Next, we discuss the conditions of preparation of Type 1 and Type 2 samples. The relationship between the Type 1 emergence ratio and conditions is shown in Fig. 4. Here, the Type 1 emergence ratio $T_{1\text{ratio}}$ was defined as following.

$$T_{1\text{ratio}} = \frac{N_{\text{type1}}}{N_{\text{type1}} + N_{\text{type2}}}. \quad (1)$$

Here, $N_{\text{type1}}$ and $N_{\text{type2}}$ are the number of emergence of Type 1 and Type 2, respectively.

Fig. 3. Typically observed force curves and particle removal: (a) Type 1 ($T_h = 290 \, ^{\circ}\text{C}, t_h = 10 \, \text{min}, 8 \, \text{days elapsed since sample preparation}$), (b) Type 2 ($T_h = 290 \, ^{\circ}\text{C}, t_h = 10 \, \text{min}, \text{just after sample preparation}$), (c) Bottom view of Type 1 particle removal by cantilever movement (Scale bar, 30 μm).

It was found that the appearance of Type 1 particles varied with the number of days elapsed since sample preparation, heating temperature $T_h$, and duration of heating $t_h$. Particles baked with a relatively low $T_h$ of 100 °C were exclusively Type 1, regardless of the number of days elapsed. When $T_h$ was 290 °C, which is higher than the heat resistance temperature of the particles, the frequency of Type 2 particles was high, and the rate of Type 1 particles increased with an increasing number of elapsed days. It took almost one month for the emergence ratio $T_{1\text{ratio}}$ to become 1. When $T_h$ was 200 °C, only Type 2 particles were obtained. Therefore, the results of Type 1 at $T_h = 100 \, ^{\circ}\text{C}$ and 290 °C and the results of Type 2 at $T_h = 200 \, ^{\circ}\text{C}$, are separately discussed below.
3.2. Removal force for Type 1 particles

Figure 5 shows the relation between the maximum value of the force $F_{\text{max}}$ measured by the cantilever and the particle diameter $D_p$ for the case of Type 1 samples. Here, $F_{\text{max}}$ shows the largest force from several trials with a bin of $\Delta D_p = 5 \, \mu m$. Overall, $F_{\text{max}}$ was in the range of $10 - 100 \, \mu N$. This particle removal force was nearly $10^3$ times larger compared with the sprinkles particles without baking. This result is identical to the one from our previous study [7]. We believe that this is due to a chemical reaction occurring between the particles and the substrate during heating. Furthermore, the result at $T_h = 290 \, ^\circ C$ was approximately 10 times larger than at $T_h = 100 \, ^\circ C$. The condition of $T_h = 290 \, ^\circ C$ exceeded the heat resistance temperature so that the composition itself may have changed.

Next, we compare the results of the removal force in open-air and underwater conditions. As shown in Fig. 5, there was no significant difference. In general, the removal force measured in water was smaller compared with open-air conditions, because there was no influence of the capillary bridge. However, the capillary force is approximately several hundred nN, in general. Therefore, the removal force obtained in this experiment was not influenced by the capillary force.

3.3. Removal force for Type 2 particles

As shown in Fig. 3, Type 2 particles could not be removed by a single traversing. For such particles, we investigated the effect of the movement speed on the force curve. The typical force curves obtained for different traverse speeds are shown in Fig. 6. As shown in the graph, the shape of the curves is similar, but there are two different points of interest. First, the maximum force became almost two times the initial value when the scanning speed was higher. We consider this to be due to the impact force and viscoelasticity of the particles. In general, the dynamic load shows a higher value than the static load. Additionally, the particle should be deformed because of baking. Hence, we believe that part of the work done by cantilever movement was used for the deformation of viscoelastic polymer particles.
Second, the negative force for the case of high traverse speed was obtained when the cantilever returned. We consider this to be due to the adhesion of particles to the cantilever. These results were obtained in the case of speeds faster than 1 μm/s. This means that it takes several seconds to recover the particle shapes from the deformation by the cantilever; this is a significant time period.

Next, we repeatedly measured the force for an identical Type 2 particle. The result is shown in Fig. 7; the horizontal axis shows the displacement of the cantilever. As clearly shown in the figure, the triangular area, enclosed by the force curve and y-distance, reduced with the number of scans. This area corresponds to the work done on the particle by the cantilever. Thus, the continuous application of the force on the particles decreased the adhesion force, i.e., the work was used to decrease the adhesion energy between the particles and the surface. This result indicates that, not only the force acting on the particles, but also its duration is important for particle removal.

4. Conclusion

We experimentally evaluated the lateral removal force of a baked polymer particle on a glass plate. As a result, two types of force curves were obtained. In the first type, particles could be removed by a single scanning and the removal force was of the order of $10^2$ μN. In the second type, several scanning iterations were needed to remove the particles, and the particle adhesion force decreased with scanning repetition. This result indicates that duration is an important factor for particle removal, in addition to the force acting on the particles.

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