Adhesion of AFM Tip to Resist Surface due to Laplace Force

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1. Introduction

The resist pattern collapse has become serious problem in the formation of high-aspect ratio and fine-pitch resist patterns. In this regard, the present authors have already proposed the novel principle for analyzing resist pattern adhesion and cohesion, that is, DPAT (direct peeling with atomic force microscope (AFM) tip) method.[1] The collapsing load for resist pattern greater than 50 nm size can be determined as in the range from 20 to 100 nN.[2] However, in these days, high sensitive peeling test for resist pattern less than 50 nm size is required. The purpose of this paper is to discuss the possibility of the peeling test due to Laplace force, which is defined as the pressure difference between water and atmosphere[3]. We attempt to analyze interaction of tip-resist surface due to micro meniscus by using the AFM. In addition, by the nucelate simulation, the dependence of adhesion force on concavo-convex surface is discussed.

2. Experiment

2.1. Sample preparation

Chemically amplified positive type resist, consisting of hydroxystyrene as a base polymer, was used. The resist film of 550 nm in thickness was coated onto a Si(100) wafer by spinning method. Pre-baking was carried out at 100 °C for 90 s on a hot plate. The resist patterns were transferred to the resist film using the X-ray stepper in the SOR system with the irradiation energy of 227 mJ/cm². Post exposure baking was accomplished at 130 °C for 90 s.

2.2. Adhesion force measurement

A commercially available AFM, integrated with a micro tip, was used for the measurement of adhesion force. A Si₃N₄ conical tip mounted on the cantilever apex was used. The radius of curvature of tip apex and spring constant were approximately 8 nm and 0.35 N/m, respectively.

Figure 1 shows a schematic of the measurement of adhesion force between the AFM tip and the resist surface due to micro meniscus. The adhesion force depending on the relative humidity was measured in the ranging from 3 to 90 %RH. (i): The AFM tip was contacted to the top surface or side-wall of resist pattern. (ii): The cantilever was gradually apart from the resist surface. In this time, the AFM tip was adhering to the resist surface due to capillary-condensed water. (iii): When the AFM tip was jumped out from the resist surface, the adhesion force due to Laplace force was determined by multiplying the measured jump distance by the spring constant of the cantilever.

![Fig.1 Schematic diagram of the measurement of adhesion force with AFM tip due to micro meniscus.](image)

3. Results and Discussion

Figures 2 (a) and 2(b) show the AFM images of the top surface and side-wall of resist pattern, respectively. The concavity and convexity of the side-wall is larger than the top surface of resist pattern. Figures 3(a) and 3(b) show the dependence of the adhesion force of the AFM tip on relative humidity. The adhesion force increases with the relative humidity. Particularly, in Fig.3(a), the counterclockwise hysteresis curve was observed in the drying and humidifying stages. Therefore, these results clearly indicate the formation of meniscus between the tip and resist surface due to capillary condensation theory.[4] As comparing
Fig. 3(a) with 3(b), the humidity dependency is similar to each other, but the scatter of adhesion force on side-wall is fairly large.

![AFM image of resist surface.](image)

Fig. 2 AFM image of resist surface.

![Dependency of adhesion force on relative humidity.](image)

Fig. 3 Dependency of adhesion force on relative humidity.

We try to characterize this scatter by the nucleate simulation on concavo-convex surface as the following. Generally, condensation of water molecule to solid surface can be analyzed as the nucleate model of cluster. The curvature radius and the contact angle of the micro-cluster are $r$ and $\theta$, respectively. The total free energy $G$ of the cluster is defined as the following. [4]

$$G = 4\pi \left( \sigma_r r^2 + \frac{1}{3} \frac{8}{r} r^3 \right) \left( \frac{2 - 3\cos\theta + \cos^3\theta}{4} \right),$$

(1)

where $\sigma_r$ is liquid/vapor interface free energy, $g_r$ is free energy per unit volume of the cluster. Figure 4 shows the simulation results of nucleation on the flat, convex and concave surfaces. The size of critical nucleus $r_c$, which is defined as the radius at maximum of free energy, is strongly related to the concavo-convex surface. This result clearly indicates that the cluster growth and the meniscus shape depend on the concavo-convex surface. Therefore, we can safely state that the scatter of adhesion force depends on the concavo-convex surface, as shown in Fig. 3.

Moreover, it is found that the adhesion force of the AFM tip is ranging from 10 to 20 nN. By the DPAT method, the collapsing load for resist patterns of 60 ~ 100 nm size is approximately 20 ~ 50 nN.[2] It is considered that the peeling method by using the micro meniscus can be applied to collapse the pattern less than 50 nm, as shown in Fig. 5. Concretely, it is possible that the pattern collapse can be analyzed as the two peeling models, the pull-up and slide modes by the adhesion force due to micro meniscus.

![Peeling test of nano-pattern due to micro meniscus.](image)

Fig. 5 Peeling test of nano-pattern due to micro meniscus.

4. Conclusion

The condensation property of water vapor on the resist surface can be analyzed by measuring adhesion force between the tip and the resist surface. The possibility of adhesion analysis using micro meniscus is discussed.

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