Wetting Analysis of Hydrophobic Substrate treated by HMDS Primer

Akira Kawai* and Junko Kawakami

Department of Electrical Engineering, Nagaoka University of Technology
1603-1 Kamitomioka, Nagaoka, Niigata 940-2188 Japan
*Corresponding author, kawai@nagaokaut.ac.jp

Keywords: HMDS, primer, contact angle, hydrophobic, wetting, adhesion

1. Introduction
The hydrophobic primer treatment is a well-known process in order to improve a micro pattern adhesion in a liquid environment. So far, in micro electronic industry, HMDS (hexamethyl-disilazane) has been employed as a typical primer source. There are various reports for the micro pattern adhesion by the primer treatment.[1,2] In this paper, we focus on an ideal condition of primer treatment with HMDS. Particularly, as optimum parameters, dependency on primer time and heating temperature after primer are investigated.

2. Experiment
A Si(100) wafer was used as a substrate for the primer treatment. The Si substrate was cleaned by dipping into acetone with ultrasonic vibration for 5min. The thickness of native oxide layer was approximately 2nm. As a primer chamber, a sealed glass chamber was used. In the glass chamber, the concentration of HMDS vapor was saturated. The primer treatment was conducted at 29°C by setting the samples in the glass chamber. Following two kinds of primer conditions were employed in order to optimize the hydrophobic condition.

2.1 Heating temperature after primer treatment
The Si(100) substrates were stored in the primer chamber for 5h. After exposing to air, the substrates were heated on a hot plate. The heating temperature was changed from 50 to 300°C. The heating was conducted for 1 and 5min at each heating temperature.

2.2 Exposure time in HMDS vapor
In the primer chamber, the storage time of the substrates was changed up to 6h. After exposure to air, the samples were heated at 150°C for 1min.
A contact angle of DI-water was employed to determine the degree of hydrophobic of the sample surfaces. The contact angles were measured by using a goniometer at 23°C and 55%RH after 1min of sessile drop.

3. Results and Discussion
Figure 1 shows a contact angle change depending on the heating temperature. The contact angle on the Si substrate after the organic cleaning treatment is 31 degree. By heating the sample, the contact angle decreases slightly which means slight growth of native oxide layer on the sample surface. No difference property for the heating time can be observed.

By exposing to the HMDS vapor, the contact angle of the sample increases up to 83 degree without heat treatment. For the 1min heating time, the contact angle increases with the heating temperature and reaches a maximum of 99 degree at 150°C. By heating over 150°C, the contact angle
decreases and becomes 65 degree at 300°C heating temperature. It can be explained that the excess HMDS molecular adsorbed on the substrate would evaporate around the boiling temperature 156°C of HMDS. The ideal monolayer of hydrophobic coupling layer would be formed. At higher temperature around 300°C, decomposition of primed layer would occur.

For the 5min heating time, the contact angle decreases gradually as increasing the heating temperature, and the maximum of contact angle cannot be observed. It can be explained that the surface concentration of hydrophobic group decreases as similar to the 1min heating. At 300°C heating, the contact angle reaches to 25 degree, which is mostly same as that for the cleaned Si surface. Manaka et al. reported the pattern adhesion properties depending on the heating temperature after HMDS primer treatment [2]. They obtained the heating temperature at 275°C as the better adhesion condition. In this case, the HMDS treatment was conducted by the spin coating, therefore the considerable excess HMDS molecule should remain on the substrate. Consequently, relative higher heating temperature was required.

Figure 2 shows the contact angle change depending on the exposure time to the HMDS vapor. The heating treatment is carried out at 150°C which corresponds to the maximum contact angle condition as shown in Fig.1.

Fig. 2 Exposure time dependence to HMDS vapor.

the contact angle rapidly increases to 70 degree. Then, the contact angle continues to increase. After 60min exposure, the contact angle reaches to 80 degree and mostly saturates. After 6h exposure, the contact angle becomes 86 degree. It can be considered that the relative short primer time is required in order to obtain enough hydrophobic surfaces.

4. Conclusion

The contact angle change by the HMDS primer treatment is discussed in order to obtain the ideal hydrophobic condition. The wetting behavior is considerably sensitive to the heat treatment and the primer time. The optimum condition of the HMDS primer is determined as the heating condition (150°C for 1min) and primer time more than 5min in order to obtain a hydrophobic surface.

Acknowledgements

This present work was partially supported by Grant-in-Aid for Scientific Research from Japan Society for the Promotion of Science (Scientific Research (B) 19350157) and A Research for Promoting Technological Seeds 2007 (Japan Science and Technology Agency).

References