DUV Hardened Layer of Resist Dot Pattern Detected by Tip Indentation Method

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1. Introduction
The importance of a scanning probe microscope (SPM) in material science has already been recognized.[1] Tomanek et al. analyzed slight deformation of a solid surface by indenting with an AFM tip theoretically.[2] Authors also analyzed surface indentation property of thin photore sist film experimentally.[3] Meanwhile, surface hardening, for example, deep ultra violet (DUV) light irradiation to polymer material has been adopted to enhance the thermal resistance and adhesion property. [4] In this paper, a DUV hardened layer of a resist pattern can be detected by indenting a micro tip into the side of a collapsed pattern.

2. Experiment
2.1 Pattern fabrication
The resist in this experiment was composed of m-cresol/p-cresol/xylenol/formaldehyde novolak resin and 2,3,4,4'-tetrahydroxybenzophenone 1,2-naphtoquinonediazide-5-sulfonate sensitizer.[5] The resist film of 1.03 μm in thickness was coated onto the Si(100) wafers by the spinning method. No silane-coupling treatment as an adhesion promoter was performed. The dot shaped patterns of 0.61 μm diameter were transferred to the resist film by using a stepper (Nikon, NSR1505-G6E, g-line, NA=0.54) at the exposing time of 300 msec. Then, they were developed by dipping into tetramethylammonium-hydroxide (TMAH) 2.38% aqueous solution for 1 min and rinsed in a pure water for 60 sec at 25°C. After the pattern development, in order to harden those surface layers, the resist patterns were exposed to DUV light (λ =250~360nm) for 1 min at 80 °C on a hot plate by using the DUV hardening system (Mark-Ⅱ, Tokyo Electron Ltd.). The resist pattern shape was observed by using a scanning electron microscope (SEM) JSM-35CF II made by JEOL LTD..

2.2 Tip indentation

3. Results and Discussion
3.1 Pattern collapse with AFM tip
Figure 2 shows the SEM photographs of resist patterns before and after collapse with the AFM tip. Most of patterns were collapsed without residue formation on the substrate. One collapsed pattern was selected and used for the indentation test. Figure 3 shows the AFM image of the collapsed pattern. The pattern image is not so clear but the pattern shape can be observed. The nine points in series on the pattern side were chosen as the indenting points.

The atomic force microscope (AFM) integrated with a gold-covered Si₃N₄ cantilever tips was used to the collapse and indentation investigations.[6] The radius of curvature of tip apex and its spring constant were 20 nm and 0.098 N/m, respectively. Figure 1 shows schematic diagram explaining this method. After the DUV irradiation, the pattern was imaged in so-called contact mode with applying load of 0.01nN which is much smaller than the load used for pattern collapse. Subsequently, the tip was contacted to the top corner of the pattern. By applying load up to 30 nN by using the cantilever tip, resist pattern can be collapsed as seen in Fig.1a. Subsequently, the micro tip was indented into the side of collapsed pattern at the some points in series as seen in Fig.1b.
3.2 Indentation investigation

Figure 4a shows the typical indenting curve of the tip. Zero value of displacement of piezo stage Z indicates an initial contact position of the tip with the resist pattern surface. In the repulsive region (the displacement Z of piezo stage less than zero), the force curve shows two kinds of curve slope, $S_1$ and $S_2$. Then, symbols P and D denote inflection point of the curve and its indenting depth, respectively. It is clear that the curve slope decreases as increasing the intending depth. In this regard, authors were already confirmed the correlation between indentation slope and polymer hardness.[3] Therefore, it seems reasonably to discuss that the regions $S_1$ and $S_2$ are correspond to the hardened surface layer by DUV irradiation and the inner region of resist pattern, respectively.

In this way, at the several indenting points of the resist pattern side, the meaningful values $S_1$, $S_2$, D can be obtained. Figure 4b shows the variation of values $S_1$, $S_2$, D at the indentation position of the pattern side defined in Fig.3. The depth D is relatively large at the pattern top but decreases rapidly approaching to the pattern bottom. The curve slope $S_1$ of hardened layer is about ten times as large as that of inner region $S_2$.

3.3 Cross sectional image of dot pattern

Figure 5 shows the schematic image of cross section of resist dot pattern obtained by the indentation investigation as shown in Fig.4b. The hardened layer of pattern surface can be imaged clearly. At the pattern top, the hardened layer is relatively thick, because the exposure dose of DUV light would be considerably large as compared with that at pattern bottom. Moreover, the image profile of surface hardened layer is similar to that shown by Hirose et al. [4] Therefore, it is fair certainly state that the surface hardened layer of micro pattern can be detected and imaged by means of the tip indentation.

4. Conclusion

By the tip indentation method, surface hardened layer formed on dot pattern can be clearly imaged. This technique can be applied to a chemically amplified resist pattern less than 100nm in size.

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References