

# 100 nm period silicon antireflection structures fabricated using a porous alumina membrane mask

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An ordered anodic porous alumina membrane has been used as a lithographic mask of SF<sub>6</sub> fast atom beam etching to generate a 100 nm period antireflection structure on a silicon substrate. The antireflection structure consists of a deep hexagonal grating with 100 nm period and aspect ratio of 12, which is a fine two-dimensional antireflection structure. In the wavelength region from 400 to 800 nm, the reflectivity of the silicon surface decreases from around 40% to less than 1.6%. The measured results are explained well with the theoretical results calculated on the basis of rigorous coupled-wave analysis. © 2001 American Institute of Physics. [DOI: 10.1063/1.1339845]

A subwavelength structured (SWS) surface, which is the surface-relief grating with the period smaller than the light wavelength, behaves as an antireflection surface.<sup>1-4</sup> The SWS surface with a deep tapered shape grating especially suppresses the reflection over a wide spectral bandwidth and a large field of view. According to the analysis, antireflection properties are improved by decreasing the grating period and increasing the grating depth.<sup>4</sup> Lately, silicon SWS surfaces consisting of very fine gratings have been fabricated using an electron beam or laser interference lithographies.<sup>1-3</sup> In the technical aspects, the electron beam writing is a time consuming method and the periodicity of the laser interference is limited by the wavelength. The smallest period and the highest aspect ratio of the two-dimensional antireflection surfaces reported so far are 150 nm and 2.3, respectively.<sup>1</sup>

In this letter, SWS surfaces consisting of hexagonal gratings have been fabricated on crystal silicon substrates. In the fabrication, an ordered anodic porous alumina (OAPA) membrane has been used as an etching mask<sup>5</sup> and the surface has been etched by an SF<sub>6</sub> fast atom beam (FAB). The period of our fabricated grating has been 100 nm and the aspect ratio has been higher than 6.

The process steps used in the fabrication of the SWS surfaces are shown in Fig. 1. For the preparation of the OAPA membrane as the etching mask, we use a two-step anodizing process reported by Masuda and Satoh.<sup>6</sup> The two-step anodizing process improves the periodicity of the hole because the periodic seeds are generated by the first anodization. An Al sheet (99.999%) polished electrochemically is first anodized at the voltage of 40 V in 0.3 M oxalic acid solution at 20 °C for 5 h. The OAPA layer is removed in a mixture of phosphoric acid (6 wt %) and chromic acid (1.8 wt %) at 60 °C for 10 h. The Al sheet is anodized again for 5 min under the same condition as used in the first anodization. Next, the OAPA layer is removed by immersing it in a saturated HgCl<sub>2</sub> solution for 50 min. The OAPA membrane is

placed on a 200 μm thick polished Si substrate [*n* type, 5–8 Ω cm, (100)] directly after rinsing in acetone, and it adheres to a Si substrate after drying. The upper part of the OAPA membrane is etched in 5 wt % phosphoric acid at 30 °C for 50 min to make the hole array and to adjust the hole diameter. The fabricated OAPA membrane is used as a mask for etching Si substrate with the FAB (EBARA Co., Ltd.; FAB-60 ML). Since the FAB has good directionality and deposits no charge on the surface, the surface is etched anisotropically and deeply.<sup>7</sup> The Si substrate is etched by the FAB at the SF<sub>6</sub> flow rate of 6.7 sccm, the discharge voltage of 2 kV and the discharge current of 35 mA. After etching, the OAPA membrane is removed with H<sub>2</sub>SO<sub>4</sub> and H<sub>2</sub>O<sub>2</sub>(1:1) solution.

Figures 2 and 3 show the fabricated SWS surfaces. The etching times of FAB are 30 and 50 min for the results shown in Figs. 2 and 3, respectively. The SWS surface in Fig. 2 have holes in a hexagonal arrangement, the period of the holes is 100 nm and they are about 700 nm deep. The

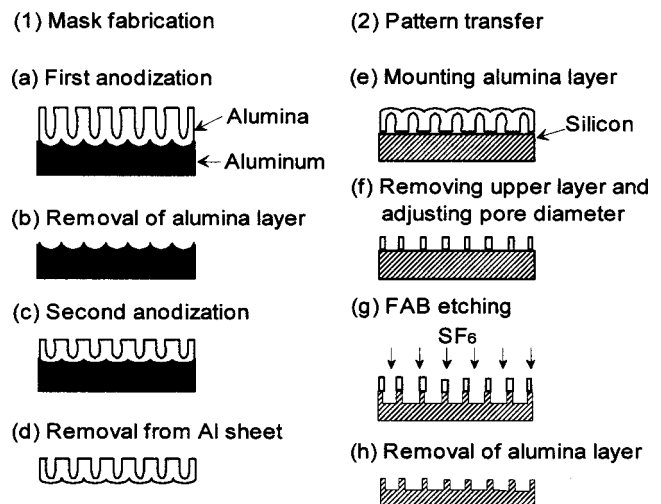


FIG. 1. Lithographic steps for the fabrication of SWS surface.

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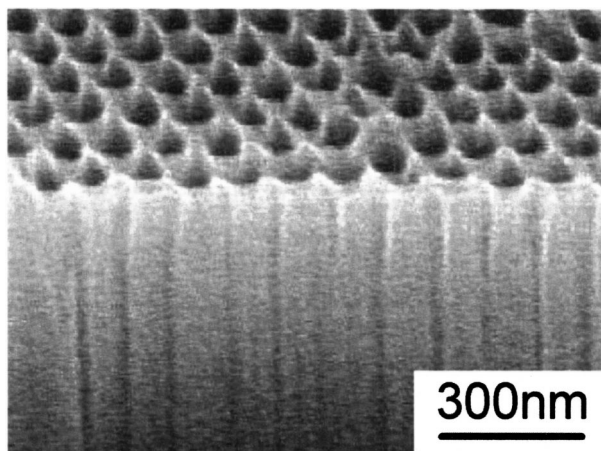


FIG. 2. Scanning-electron micrograph of the SWS surface.

etching rates of silicon and the OAPA membrane are 23.3 nm/min and 9.35 nm/min, respectively. Since the hole diameter is 60 nm, the aspect ratio of the etched hole is 12. The 60 nm diameter alumina holes of the mask have been well transferred to the Si substrate. The SWS surface shown in Fig. 3 is obtained by overetching of that shown in Fig. 2. In the bottom layer, the etched substrate also consists the circular holes in the hexagonal arrangement. In the upper layer, however, the Si surface is tapered periodically at six-fold rotation symmetry since the thin walls of the hexagonal structure are partially removed by the overetching. There-

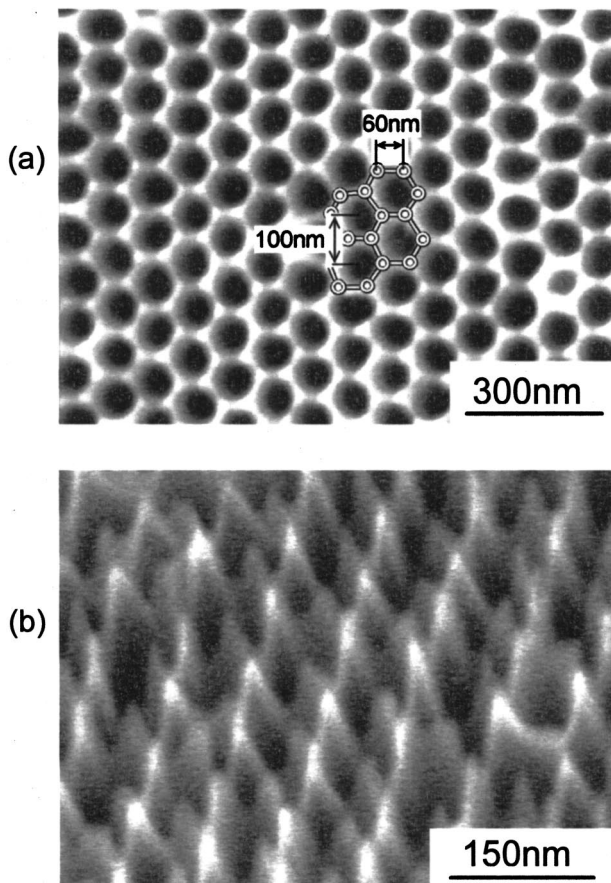


FIG. 3. Scanning-electron micrograph of overetched SWS surface. (a) top view. (b) oblique view.

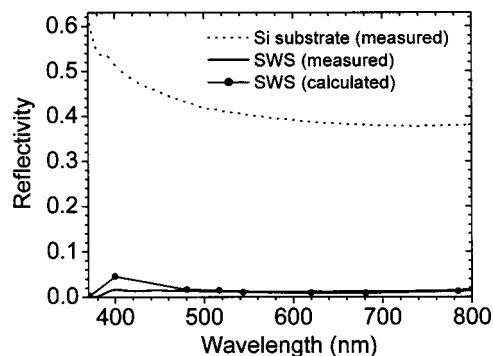


FIG. 4. Reflectivities as a function of wavelength.

fore, the smallest distance between the elongated structures in the upper layer is 60 nm.

Figure 4 shows reflectivities measured as a function of the wavelength. The incident light is randomly polarized. The dashed and solid curves show the reflectivities of the polished Si substrate and the SWS surface shown in Fig. 3, respectively. It is shown that the SWS surface decreases the reflection drastically compared with that of the polished Si surface. The reflectivity of the SWS surface is lower than 1.6% at the wavelengths between 370 and 800 nm. The theoretical calculations were carried out by using the rigorous coupled-wave analysis (RCWA) proposed by Moharam<sup>8,9</sup> and others.<sup>10</sup> The calculation model was constructed by the observation of the fabricated SWS surface with scanning electron microscope. The model consisted of 12 layers by slicing the grating of the SWS surface. The calculated reflectivity is also shown in Fig. 4. The calculated reflectivity agrees well with the measured results.

In conclusion, we have shown that the porous alumina membrane is useful as a mask for the fabrication of deep SWS surface with the FAB etching. 100 nm period gratings with aspect ratio higher than six were generated on a silicon substrate. At the wavelength from 370 to 800 nm, the reflectivity of the SWS surface decreased to be lower than 1.6% from around 40% of the polished silicon surface. The theoretical calculation based on RCWA explained the measured reflectivities well. Since the porous alumina membrane is easily widened, the proposed process will be useful for the fabrication of the antireflection surface in a large area on a silicon substrate.

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