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# Nanometer scale lithography at high scanning speeds with the atomic force microscope using spin on glass

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We have identified a resist material that is suitable for high-speed, nanometer-scale scanning probe lithography (SPL) using the atomic force microscope (AFM). The material is siloxene, commonly known as spin on glass (SOG). The SOG film is deposited on a silicon sample and exposed with a voltage applied between the AFM tip (negative) and the silicon substrate (positive). Voltages of 70 V and currents of 1 nA are typical. It is a positive resist where the etch selectivity between the exposed and unexposed areas is greater than 20. We have recorded line widths as narrow as 40 nm. The writing speed is greater than 1 mm/s, which we believe to be an important attribute in future systems for SPL. © 1995 American Institute of Physics.

The surge of activity over the past year in patterning with the scanning probes has centered on two different systems.<sup>1</sup> In the first system, electrons from the tip are used to expose electron resists<sup>2,3</sup> self-assembled monolayers (SAM)<sup>4</sup> and Langmuir–Blodgett (LB) films.<sup>5</sup> In the second system, the electric field at the tip of the scanning probe is used to selectively oxidize materials such as crystalline silicon,<sup>6–8</sup> amorphous silicon,<sup>9,10</sup> titanium,<sup>11</sup> tantalum,<sup>12</sup> and chromium.<sup>13</sup> In the latter system the region scanned by the tip is selectively oxidized when the water molecules in the atmosphere surrounding the tip are oxidized in the strong electric field. The etch rate of the oxidized area is less than that of the unoxidized material. This differential in etch rate means that the pattern can be easily transferred to the substrate with chemical etching. Enhanced oxidation has been used to pattern narrow metal lines and to fabricate working devices. Campbell and Snow<sup>14</sup> have fabricated a side-gate transistor in an epitaxial layer of silicon. Minne *et al.*<sup>15</sup> have fabricated a metal–oxide semiconductor field effect transistor (MOSFET) with reasonable values for the transconductance. Matsumoto *et al.*<sup>16</sup> have fabricated a single electron transistor (SET) in thin films of titanium with the staircase structure in the  $I$ – $V$  curve clearly visible at room temperature. These prototype devices with feature sizes in the range of 25–250 nm were patterned with tip scanning at speeds of 1–10  $\mu\text{m/s}$ .<sup>17</sup> A single tip moving at this speed cannot be used to pattern multiple devices on large area microchips. For this task, we must employ an array of tips for parallel processing. The number of elements in the array will decrease if we can increase the writing speed with a single tip.<sup>10</sup>

In this letter, we report on our results with patterning films of spin on glass (SOG) with the atomic force microscope (AFM) tip moving in excess of 1 mm/s. We have found that SOG is easily patterned with electrons emanating from the scanning tip. SOG is a material used for planarization in fabricating silicon integrated circuits.

The mechanism that underlies this experiment is illustrated in Fig. 1. The schematic of the molecular structure of siloxane SOG is shown in Fig. 1(a). The siloxane type of SOG consists of a methyl ( $\text{CH}_3$ ) group and silanol groups ( $\text{Si-OH}$ ) bonded to the Si atoms in the Si–O backbone. The organic content refers to the number of methyl groups, which is proportional to number of Si–C bonds. SOG films with high organic content have superior mechanical properties such as lower deposition stress, higher resistance to cracking, and lower film shrinkage. These all contribute to the planarizing properties. The silanol group ( $\text{Si-OH}$ ) with its high polarization ( $\text{Si-O-H}^+$ ) is a site for absorption of water. Thus SOG readily absorbs water molecules which must be removed during the processing.

In our opinion, the methyl group is decomposed, thereby decreasing the organic content, when SOG films are exposed with electrons from the tip. We believe that the generated dangling silicon bonds are filled with the silanol group. This is consistent with the results of T. Nakano *et al.*<sup>18</sup> shown in Fig. 1(b). This result shows increased formation of silanol as a function of decreasing organic content. Additionally, Fig. 1(c) shows that the wet etching rate of SOG film in buffered oxide etch (BOE) decreases exponentially with increasing organic content. Thus a large differential etch rate between exposed and unexposed regions makes SOG an effective resist material for scanning probe lithography (SPL).

In our experiments, we used methylsiloxane SOG (ACCUGLASS-111, Allied Signal, Inc.) with 11% organic content. Figure 2 shows the schematic diagram of the experimental system. The sample substrate is a 2.5  $\Omega$  cm,  $n$ -type Si (100) wafer. The SOG was spun onto the wafer at 5000 rpm for 20 s. The sample was ramped to 280  $^\circ\text{C}$  for 5 min. It was mounted in the atomic force microscope (AFM, Autoprobe CP, Park Scientific Instruments) operating in air with the contact mode. The sample was attached to the sample holder with conducting colloidal graphite and the sample holder was insulated from the piezoscanner. We used a silicon can-

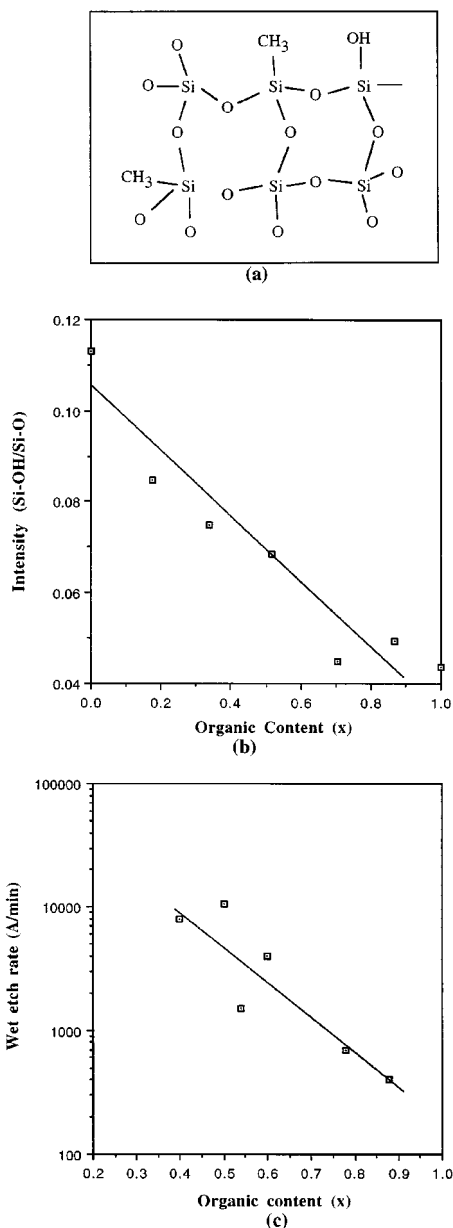


FIG. 1. (a) Schematic of the siloxane SOG structure. Si-CH<sub>3</sub> (siloxane) and Si-OH (silanol) are present. The highly polarized (Si-O-H<sup>+</sup>) bonds will absorb water molecules, which are not shown in the schematic. (b) Silanol peak intensity measured by Fourier-transform infrared spectroscopy as a function of organic content in SOG. Decreasing the organic content will favor (Si-OH) formation. (c) Wet etching rate as a function of organic content. As the organic content decreases, the wet etching rate increases exponentially.

tiler with a force constant of 2.8 N/m and with a nominal radius of 10 nm for the curvature of the tip. A positive voltage was applied to the sample to induce field emission from the tip. The current was measured with a current-voltage converting amplifier. A dc voltage of 70–100 V was applied to the substrate while monitoring the current. The measured current was in the range of 0.5–3.0 nA. One of the characteristics of field emission is Fowler–Nordheim (FN) behavior, in which the plot of  $\log I/V^2$  vs  $1/V$  is a straight line.<sup>19</sup> Our applied voltage and the measured current roughly followed the FN characteristic, as shown in Fig. 3. Higher cur-

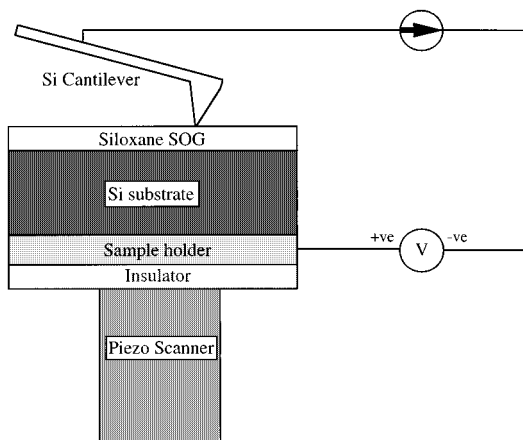


FIG. 2. Schematic diagram of the experimental setup for modification of siloxane SOG film. A voltage bias is applied across the 100 nm SOG film between the silicon tip and the underlying silicon substrate. The current is monitored with a current-voltage converting operational amplifier whose conversion ratio is set at  $10^8$  V/A.

rents were measured with silicon tips coated with 300 Å of gold compared to uncoated silicon tips with native oxide. Gold-coated tips, however, showed excessive wear; thus uncoated silicon tips were used exclusively for lithography.

After the current settled to the desired value, lines were scanned over the sample across a  $90 \times 90 \mu\text{m}$  area at a predetermined scan speed. The scan speeds were varied from 180–3024  $\mu\text{m/s}$ . Larger scan areas and higher scan speeds were limited by the response of the piezoscanner. After the exposure, the sample was etched in 100:1 BOE for 15 s, rinsed in deionized water for 2 min, blown dry with nitrogen, and transferred back to the AFM for measuring the topography. The latent image does not appear where the region is scanned with the AFM immediately after exposure and before development.

The unexposed region has an etch rate of 3.6 Å/s, and the etch selectivity was 20:1 between the exposed and unex-

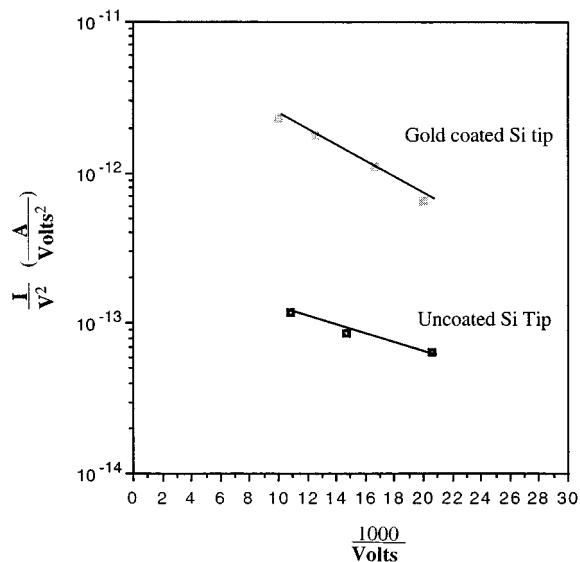


FIG. 3. Measured emission current vs applied voltage. The emission characteristics show Fowler–Nordheim behavior.

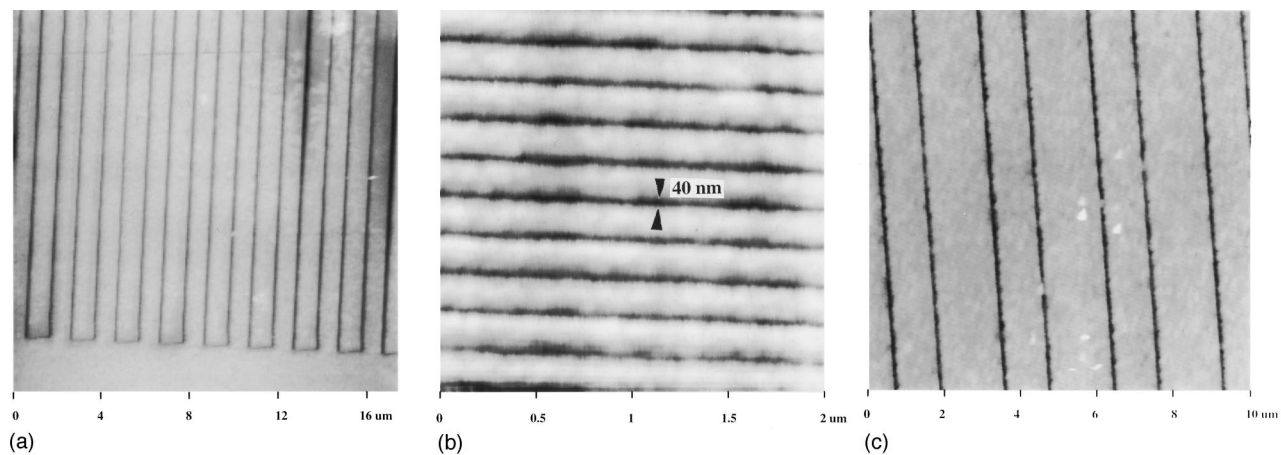


FIG. 4. (a) SOG patterned at 1.0 nA at 180  $\mu\text{m/s}$ . The thickness of the film is 1000  $\text{\AA}$ . The line spacing is 1  $\mu\text{m}$  and the linewidth is 100 nm. The proximity effect is not observed at the corners. (b) SOG pattern of a fine line at 0.8 nA and 180  $\mu\text{m/s}$ . The linewidth is 40 nm and the spacing between the lines is set to 200 nm. (c) SOG patterned at 2.0 nA and at 1 mm/s. The linewidth is 100 nm and the spacing is approximately 1.4  $\mu\text{m}$ .

posed regions. Figure 4(a) shows an AFM micrograph of SOG structure after it is developed with BOE. The pattern was written with a 74 V bias and 1.0 nA, with a writing speed of 180  $\mu\text{m/s}$ . The software for controlling the tip scanning motion was modified for lithography to accommodate the patterns shown in Fig. 4. The depression (darker contrast) corresponds to the regions of the SOG scanned by the tip. The linewidth obtained from these AFM images was near 100 nm. The corners of the lines do not exhibit any proximity effect, which is a problem with direct writing lithography using the scanning electron microscope. In Fig. 4(b) we show an AFM micrograph of a fine pattern with 200 nm spacing patterned with a current of 0.8 nA and a scan speed of 180  $\mu\text{m/s}$ . The linewidth is near 40 nm. In Fig. 4(c) we show the structure written with a scan speed of 1 mm/s and a current of 2.0 nA at 78 V. The linewidth in the image is near 100 nm. We have written patterns with linewidths of 100 nm in SOG with scan speeds as high as 3 mm/s.

In summary, we have demonstrated nanometer-scale AFM lithography of siloxane SOG films with writing speeds in excess of 1 mm/s. In our case, the speed of lithography was limited by the response of the scanner. A minimum resolution of 40 nm has been demonstrated. The patterns show no proximity effects.

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