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Nonuniform silicon oxidation and application for the fabrication of aperture for near-field scanning optical microscopy

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In this letter, a technological approach for the fabrication of a miniature aperture for near-field scanning optical microscopy using silicon micromachining technology is described. The aperture with diameter sizes from 10 to 500 nm at the apex of a SiO_2 tip on a Si cantilever is fabricated using a ''Low temperature Oxidation & Selective Etching'' technique. The SiO_2 tip is formed by nonuniform Si wet oxidation at 950 °C with a thickness of about 1 μm. The aperture is created by selective etching SiO_2 in a buffered-HF (50% HF:40% NH_4F, 9cc:100cc) solution at 36 °C using a thin chromium (Cr) layer deposited on the oxidized sample as a mask. Using the fabricated probe, atomic force microscopy and corresponding near-field scanning optical microscopy images of 300 nm diameter latex spheres on mica substrate are obtained. © *1999 American Institute of Physics.* [S0003-6951(99)05248-1]

Together with scanning tunneling microscopy (STM) and atomic force microscopy (AFM), which are capable of observation and modification of surfaces at atomic and nanometer scales, the near-field scanning optical microscopy (NSOM) has been extensively studied owing to the capabilities for optical imaging with a spatial resolution beyond the diffraction limit of the light,[1] for subwavelength photolithography,[2] and for next generation optical data storage[3] using the near-field light. The most crucial part of the NSOM probe is a subwavelength size aperture at the apex of the tip. Currently, tapered optical fiber probes are most widely used for the NSOM. Although improvements have been achieved in fabricating such optical fiber NSOM probes, problems remain. The fiber tip is very fragile. The shape of the tip and the size of the metallic aperture of the fiber tip are not reproducible. The opening angle of the fiber tip is small. Therefore, most of the light is absorbed at the metal wall, which leads to a low optical transmission efficiency. Moreover, it is very difficult to fabricate the fiber NSOM probe in a mass production.

Contrarily, Si based NSOM probe can be easily fabricated in a batch process. It is very easy to combine the microfabricated NSOM probe with a well-developed AFM as an AFM/NSOM probe.[4–8] In order to create the aperture at the apex of the tip, several technological approaches have been developed such as coating and selective etching metal at the apex of a Si_3N_4 tip,[5] metal molding with an aperture at the pyramidal etched pit on Si cantilever,[6] or creating the metallic aperture at the apex of a metal coated SiO_2 tip on Si cantilever by field evaporation,[7] etc. However, until recently a simple method to fabricate the aperture for the NSOM probe with a high reproducibility for mass production has not yet been achieved. In this letter, we present a simple method using a low temperature oxidation and selective etching (LOSE) technique to fabricate the aperture for the NSOM probe.

It is known that the thickness of oxide grown at a low temperature at convex and concave corners is thinner than that at a flat surface of Si due to the compressive stress at the corner structures (see Fig. 1).[9] The nonuniform Si oxidation effect at the convex corner has been used for fabrication of Si conical sharp tips for the AFM or field emitter.[10] The anomaly of the thermal oxidation at the concave corner has been used to produce a mold for fabrication of a Si_3N_4 pyramidal sharp tip for the AFM.[11]

On the other approach, we applied the nonuniform Si oxidation effect at the pyramidal etched pit to fabricate the aperture for the NSOM probe using the LOSE technique. The process flow of the LOSE technique is schematically shown in Fig. 2. First, a Si wafer (n-type, resistivity 0.01–0.1 Ω cm, 200 μm thick, (100) orientation) is thermally oxi-

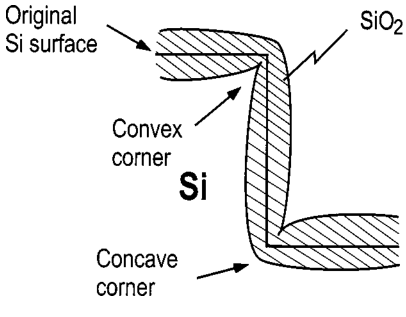

FIG. 1. Schematic diagram of Si thermal oxidation at convex and concave corners. The thickness of the oxide at convex and concave corners is thinner than that at the flat surface of Si due to the compressive stress (Ref. 9).

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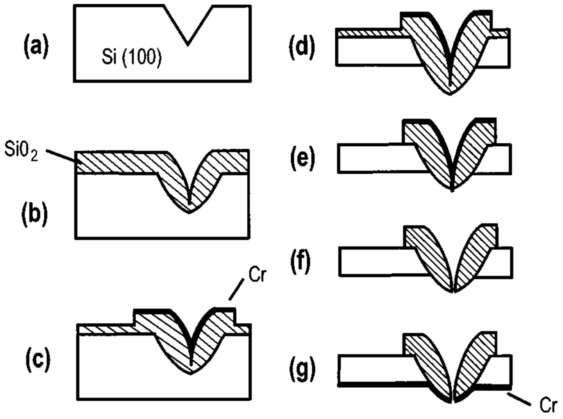

FIG. 2. Process flow for making aperture at the apex of the SiO$_2$ tip on Si cantilever for NSOM probe using the LOSE technique.

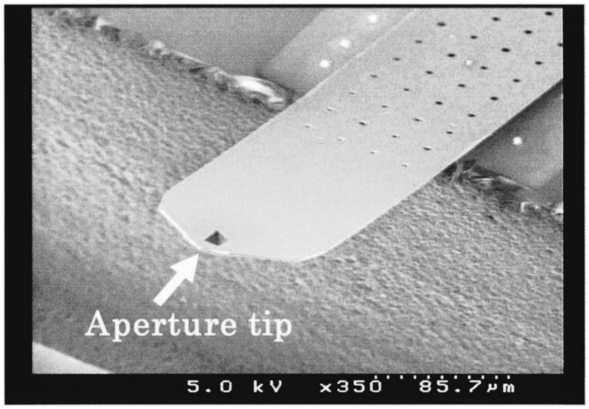

FIG. 4. SEM micrograph of a fabricated NSOM probe using the LOSE technique.

dized. Next, photolithography is done to define the mask patterns for etching SiO$_2$. In order to make a perfect pyramidal etched pit [i.e., four (111) surfaces should intersect at a point], electron beam lithography is used to define square patterns on a resist as a mask for SiO$_2$ opening. Consecutively, pyramidal etched pits and structure of cantilevers are formed by anisotropic etching of Si in a tetramethyl ammonium hydroxide (TMAH) (step a). Next, a SiO$_2$ film is thermally grown by wet oxidation at 950 °C with a thickness of about 1 μm (step b). Subsequently, a chromium (Cr) film of about 100 nm in thickness is sputtered and lifted-off to form a protective pattern on the upper side of the oxidized pit (step c). Using this Cr pattern as a mask, the SiO$_2$ of about 0.6 μm in thickness is partly etched as shown in step c. The remaining oxide of about 0.4 μm in thickness is needed for following steps. Next, the Si wafer is anisotropically etched from the back side in the TMAH solution until forming Si cantilever with the SiO$_2$ tip at the end of the cantilever (step d). The wafer is then dipped into the buffered-HF (50% HF:40% NH$_4$F, 9cc:100cc) (BHF) solution for selective etching SiO$_2$ until the Cr protrusion comes in sight (step e). At this step, only exterior wall of the SiO$_2$ tip is etched in the BHF while the interior wall is protected by the Cr pattern. It is found that the protective Cr film formed at step c was not etched or damaged during etching the sample either in the TMAH or in the BHF. Next, the protective Cr layer on the upper side is etched out in a conventional Cr etchant (step f). Finally, a Cr film with the thickness of about 40 nm is entirely deposited onto the back side of the cantilever to form an opaque layer (step g). It is found that the size of the aperture observed by scanning electron microscopy (SEM) at step f was almost identical to that of the aperture at step g. This means that the effect of filling the aperture by the Cr film deposited from back side at step g has not happened at least in the distinction of the SEM image.

SEM image of a typical Cr coated SiO$_2$ tip with an aperture of about 500 nm diameter fabricated using the LOSE technique is shown in Fig. 3. It can be seen that the aperture is exactly situated at the apex of the tip. Pinholes were not found on the side wall of the tip. The aperture sizes from 10 to 500 nm is experimentally achieved corresponding to etching time ranging from 3 to 6 min in BHF at 36 °C. SEM images of the fabricated probe and a close up view of the aperture with about 25 nm diameter are shown in Figs. 4 and 5, respectively. Using the fabricated NSOM probe with an aperture of about 130 nm diameter, the contact mode AFM and the corresponding NSOM images of latex spheres with 300 nm diameter on mica substrate are simultaneously obtained as shown in Figs. 6(a) and 6(b), respectively. The experimental setup for the AFM/NSOM system was presented in Ref. 8. The NSOM image is obtained in illumination mode using a laser diode with 780 nm wavelength. From

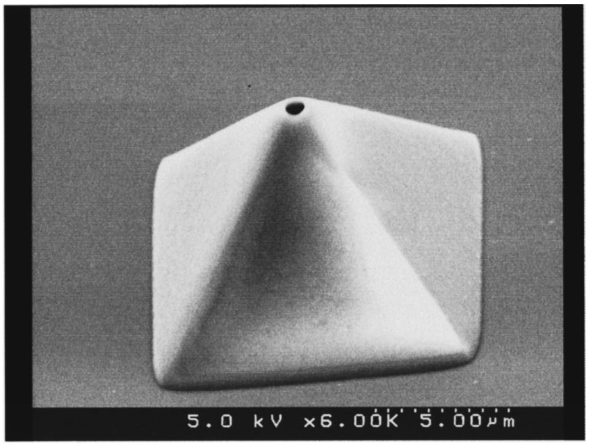

FIG. 3. SEM micrograph of a typical NSOM tip with an aperture of about 500 nm diameter.

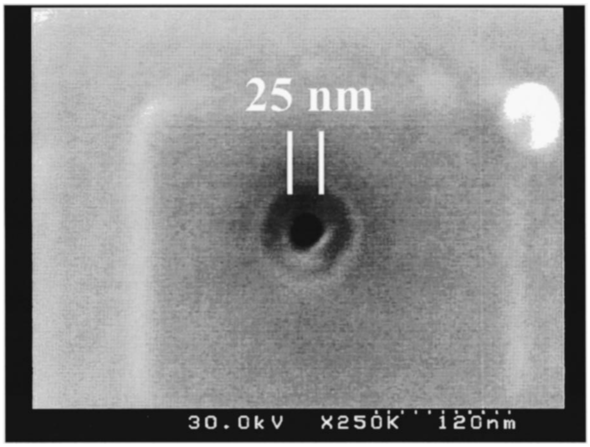

FIG. 5. SEM micrograph of an approximately 25 nm diameter aperture at the apex of the tip on the fabricated NSOM probe as shown in Fig. 4.

Fig. 6(b), a resolution of much smaller than the diameter of the aperture can be achieved. The probe was scanned many times on the surface of the sample without damaging either the sample or the tip.

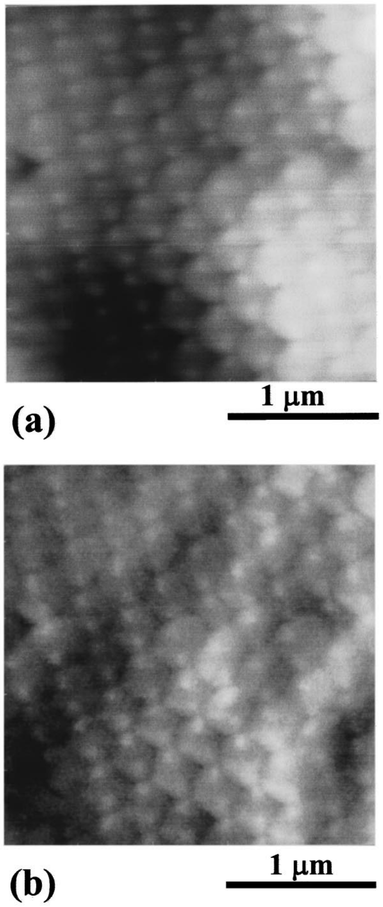

FIG. 6. The AFM (a) and corresponding illumination mode NSOM (b) images of 300 nm diameter latex spheres on mica substrate using the fabricated NSOM probe with an aperture of about 130 nm diameter.

In summary, the nonuniform Si oxidation at low temperature is a very interesting effect. The effect has been effectively used for the fabrication of the Si or Si_3N_4 cantilever having a sharp tip for AFM probe. The effect is also very useful for making a small aperture for NSOM probe as presented in this letter. The process is quite simple, low cost, mass productive, and highly reproducible. The fabricated probe is actually operated as an AFM/NSOM probe.

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