Batch fabrication of AFM probes with direct positioning capability

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One major problem for most commercial atomic force microscope (AFM) probes is the uncertainty of the tip location relative to its cantilever. In most scenarios, AFM probes have tips 5–25 μm away from the very end of the cantilever, and it is thus impossible to know where exactly the tip is because the camera in an AFM system shows only the backside of the AFM cantilever. This uncertainty of the tip location has raised some major problems, e.g., the initial scanning area must be set very large to ensure that the area of interest is within the scanning field. Here, the authors will show a straightforward fabrication method that can convert a wafer of regular pyramidal-shaped probes into direct positioning probes, for which the tip is located either at the very end of its cantilever or next to a through-cantilever hole that is visible when viewed from the backside of the cantilever. Our method involves angle evaporation of a hard mask layer onto the AFM probe, followed by dry etching of silicon that etches the area not covered by the metal layer, i.e., the shadow area of the pyramid-shaped tip. As an additional benefit, because our process etched away half of the tip pyramid, the resulting tip is sharper with a smaller half cone angle than the original one, leading to higher resolution imaging. Published by the AVS. https://doi.org/10.1116/1.5010814

I. INTRODUCTION

After Binnig and his colleagues first proposed the atomic force microscope (AFM) in 1986,1 it shortly became a versatile and popular nanocaracterization tool. Commercial products were first available in 1988, and the gold/diamond combined lever used by Binnig and his colleagues was replaced by much more reproducible cantilevers manufactured by silicon.2 Many developments were achieved, and new modes of operation have been invented since then.3,4 In an AFM, the entire probe includes a probing tip, a spring-like cantilever, and a millimeter-scale handle. There are two conventional methods of fabricating AFM probes. One method first defines a silicon mold with tip-shaped holes throughout the surface and then fills them with the desirable tip and cantilever materials. The probes are obtained after attaching handles and removing the silicon mold by a wet silicon etchant. The other method micro-machines a silicon wafer by photolithography and anisotropic wet etching steps to obtain the structures directly. In both methods, the tip shape is almost exclusively defined by potassium hydroxide wet etching of crystalline silicon, which results in a pyramidal shape.5 At present, there are more options for the shape of the tip depending on specific applications;6 yet, the pyramidal shape is still the most common one for commercial AFM tips.

One common issue for almost all commercial AFM probes is that the location of the tip relative to the cantilever is uncertain. Due to issues such as alignment accuracy in photolithography,7 most commercial AFM probes have tips 5–25 μm away from the very end of the cantilever, and it is thus impossible to know where exactly the tip is during scanning because the optical microscope in an AFM system shows only the backside of the AFM cantilever. This uncertainty of the tip location has raised some concerns because one has to set the initial scanning area large in order to ensure that the area of interest is within the scanning area, and/or one has to make multiple attempts. However, in many biological applications, it is often desired to scan the specimen with least attempts and the small scanning field to avoid damaging the soft delicate samples like cells or membranes. In addition, for applications like tip-enhanced Raman spectroscopy (TERS), it is important to know the tip apex location to shine the excitation laser beam onto it; the gold coated on the TERS tip is unstable and may be damaged by multiple trial scans if using regular tips. Hence the direct view of the tip location over the sample is indispensable for such applications.

In this study, we processed the commercial AFM probes such that the resulting tip is located either at the very end of the cantilever or right in front of a through-cantilever hole, and thus, the tip location can be precisely determined in the view of the integrated optical microscope in an AFM system. In addition, the resulting tip offers higher resolution imaging when scanning high aspect ratio (HAR) structures because half of the tip pyramid is etched away, which leads to a much smaller half-cone angle along the cantilever direction. Hence, this feature is particularly important because for a regular AFM tip, it is more challenging to reach the HAR structure bottom along the cantilever direction as the tip is tilted by 10°–13° (rather than being perpendicular to the sample surface) during the scanning, whereas our direct positioning tip (with half pyramid etched away) is inherently tilt-compensated.

II. EXPERIMENT

We started with regular commercial AFM probes having pyramidal-shaped tips. They were first attached to a carrier wafer for easier handling. Figure 1 shows the fabrication process steps that convert normal AFM probes into direct-positioning ones. First, we carried out angle-evaporation to
deposit a metal hard mask layer that covers only one or two sides (depending on whether the starting tip is a three-sided or four-sided pyramid) and leaves the other side or sides uncovered. Although Cr is arguably the most popular hard mask material for dry etching of silicon, the evaporated Cr film has high stress that can result in film detachment, and thus, we chose Al as the hard mask material. The vapor incident angle must be large enough to avoid coating all the sides, and we have chosen 60° (with 0° being perpendicular to the cantilever surface) for angle evaporation.

After angle evaporation, the probes were etched by inductively coupled plasma reactive-ion etching (ICP-RIE) (Oxford Instruments ICP380 system) using a nonswitching process with SF₆ and C₄F₈ gas. Only the shadow area not covered by the metal mask was etched away within the 20 min etching. The conventional Bosch deep silicon RIE process is not suitable for our purpose because of its inherent sidewall scallop (a rough wavy sidewall formed as a result of the switching between etching and passivation steps). Therefore, here, we employed a nonswitching pseudo-Bosch process that can give a smooth sidewall with a tunable sidewall taper angle by adjusting the ratio of SF₆ and C₄F₈ gas. With the optimal ratio of SF₆/C₄F₈ = 22:38, a smooth vertical etching profile can be obtained, with a silicon etching rate of 390 nm/min and a high etching selectivity between Si and Al of 100:1.

Next, the probes were dipped into 1:20 diluted hydrofluoric (HF) acid for 2 min to remove the remaining Al layer. Finally, to further sharpen the tip apex, we performed thermal oxidation sharpening that was first reported by Marcus and his colleagues. To do so, we first conducted thermal oxidation at 950°C for 30 min and then removed the oxide by HF etching. The resulting tip is sharper than the original one because for oxidation of less than roughly 100 nm when the oxide growth rate is limited by chemical reaction (rather than by the diffusion of oxygen or H₂O through the already grown oxide film), the oxidation rate is lower for the curved surface. Hence, much less silicon is oxidized near the tip apex where the curvature of the radius is smaller than the area farther away from the tip apex.

III. RESULTS AND DISCUSSION

Figure 2 shows angle evaporation of 200 nm Al at a deposition rate of 2 A/s onto AFM probes from two opposite directions. As expected, when deposited along the cantilever direction, only the cantilever surface and two sides of the four-side tip pyramid were coated with Al [Fig. 2(a)]; when coated along the opposite direction onto a three-side tip pyramid, a clear triangular shape shadow area was formed in front of the pyramid. A similar method using the shadow of an array of pyramid structures has been utilized to fabricate nanoprisms structures for plasmonic applications.

After etching away the silicon in the area not protected by Al mask using the pseudo-Bosch process for 20 min, followed by the removal of the remaining Al mask layer by diluted HF, the completed AFM probes with direct positioning capability are shown in Fig. 3. As seen, when the Al mask was evaporated along the cantilever direction [Fig. 3(a)], half of the pyramid not covered by the mask was etched away, resulting in the tip located at the very end of the cantilever. With a typical tip height of 10 μm and an evaporation angle of 60°, the shadow area has a length of 20 μm that is larger than the typical distance between the end of the cantilever and the tip for the unprocessed original probe, which ensures that these areas will be exposed during the RIE. When the mask was evaporated along the opposite direction, a through-cantilever hole with a triangular shape is obtained, with a size of several micrometers to make it visible under the optical microscope integrated in an AFM system. To further sharpen the tip, we oxidized the tip and then removed the oxide by HF. Figure 4 shows an AFM probe with a radius of curvature at a tip apex of 15 nm.

Figure 5 shows the optical image of the AFM cantilever viewed from backside by the optical microscope integrated in a Veeco Dimension 3100 AFM, and the arrow indicates the location of the AFM tip apex that is either at the end of a cantilever [Fig. 5(a)] or in front of an etched through hole [Fig. 5(b)]. Since half of the pyramid is etched away, the cantilever resonant frequency (f ∝ L⁻², where L is the cantilever length) should be increased. We measured f for five tips having a cantilever length of approximately 100 μm and resonant frequency ranging from 343 to 353 kHz. After our process, the frequency was measured again, with a range of 386 to 401 kHz or an approximately 13% increase from the original unprocessed tips. This slight change would not cause any serious concern because the acceptable range for resonant
FIG. 2. SEM images of two different AFM probes coated with 200 nm Al by 60° angle evaporation along two opposite directions.

FIG. 3. SEM images of the completed AFM probes with direct positioning capability, with the tip located at the very end of the cantilever (a) or right in front of the through-cantilever hole (b).

FIG. 4. SEM images of a fabricated AFM probe after oxidation sharpening, captured at low (a) and high (b) magnification, showing a tip apex radius of 15 nm.

FIG. 5. (Color online) Optical images showing the backside of two processed tips captured by the built-in optical microscope. The tip is right next to the edge of the cantilever in (a), and the through-cantilever hole is very clear to be seen in (b). The location of the tip can be speculated simply and is indicated by the red arrow.
frequency, as specified by AFM tip manufacturers, is usually very broad (e.g., from 200 to 500 kHz for the tips we studied here). As for the force constant, there should be no change because the distance from the cantilever fixed end to the tip apex (where force is exerted onto the specimen) is not altered by our process.

An additional advantage of our AFM probe is the reduced half cone angle along the cantilever direction since our process etched away half of the tip pyramid. As is known, when the tip is landed onto the sample surface, the tip axis is not perpendicular to the sample surface; instead, it is typically tilted by $10^\circ$–$13^\circ$, making it more difficult for the tip to reach the bottom of the high/deep structures. AFM probes with tilt compensation, for which the tip axis near the apex is $10^\circ$–$13^\circ$ tilted relative to the cantilever normal direction, are commercially available, yet at roughly twice the price of the same tip without tilt compensation. Our tip is inherently tilt-compensated and thus can give higher image quality particularly when scanning tall or deep and narrow structures, as demonstrated in Fig. 6.

IV. CONCLUSIONS

We presented a simple and low-cost batch fabrication process to convert commercial AFM probes into probes whose tip apex position can be determined precisely by the optical microscope integrated with an AFM instrument, thus facilitating fast locating the area of interest on the sample. The process consists of three major steps: angle evaporation of a hard mask layer, etching of the areas not covered by metal due to the shadowing effect during angle evaporation, and the removal of the remaining mask layer by wet etching. Tip sharpening by thermal oxidation can also be applied to further sharpen the tip apex. The fabricated probe has the pyramid tip located either at the very end of the cantilever or right in front of a hole etched through the cantilever, which is visible in the optical microscope when viewed from the backside of the cantilever. As a side benefit, the fabricated tip has a much smaller half-cone angle and is tilt-compensated, which offers better imaging of high aspect ratio structures.