Fabrication of high aspect ratio AFM probes with different materials inspired by TEM "lift-out" method

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The most commonly used materials in all commercially available high-aspect-ratio (HAR) nanowire's (NW) tips are made of silicon and carbon nanotube which limit their applications in other types of atomic force microscopy (AFM), such as conducting AFM and magnetic force microscope. Therefore, a simple process inspired by cross-sectional transmission electron microscopy sample preparation method was used to demonstrate the feasibility of fabricating HAR AFM probes, which can easily define the tilt angle of the NW tip with respect to the direction that is normal to the axis of the cantilever to which it is attached by simply tilting the sample stage where the cantilever is placed. This is very important as it enables precise control of the inclination angle of the NW tip and allows the tip to be made perpendicular to the probed surface for scanning with different AFM mounts. Two different tips were fabricated, one attached parallel and the other attached at an angle of 13° with respect to the normal of the cantilever axis. These tips were used to profile the topography of a silicon nanopillar array. Only the probe attached at an angle of 13° allowed mapping of the topography between nanopillars. This is the first successful demonstration of an HAR AFM tip being used to map the topography of a nanopillar array. In addition, the authors also demonstrated that this method can be extended to fabricate HAR AFM tips of different materials such as copper with a slightly modified approach.
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I. INTRODUCTION

The most common method for mapping topographies of three dimensional (3D) and one dimensional high aspect ratio (HAR) nanostructures, such as nanophotonic crystals and nanowires (NWs), is by cleaving samples and imaging cross-sections with a scanning electron microscope (SEM). This method is destructive to the sample. Atomic force microscopy (AFM) has also been widely used due to its nanoscale spatial resolution in mapping nanoscale surface topography.[1–3] However, conventional AFM cannot be used to map the topography of HAR structures with micrometer-scale walls/trenches and hundreds of nanometer or less widths/diameters/spacing. This is because conventional silicon AFM probes have tips of conical or pyramidal shape, which are too wide for the end of the tip to reach the bottom of narrow trenches. Therefore, AFM probes, with diameters smaller than the spacing of the topographic features and lengths longer than the height of the features, are required for such measurements.

The first HAR AFM probe was fabricated in the early 90s by using focused ion beam (FIB) milling[4] of a silicon tip on a silicon cantilever or focused electron beam induced deposition of an amorphous carbon tip on an AFM cantilever[5,6] for mapping of HAR features. These methods have been commercialized to produce HAR AFM tips. Several new methods, such as using a soft adhesive to attach a carbon nanotube (CNT) to the tip of a conventional AFM probe,[7] the use of a nanorobotic assembly to attach a CNT to the tip,[8] and the direct growth of a silicon NW or CNT tips on AFM cantilevers using a catalytic vapor-liquid-solid (VLS) growth or e-beam assisted plasma enhanced chemical vapor deposition (PECVD),[9] respectively, were developed over the past two decades. The e-beam assisted PECVD technique allows the growth of a wide variety of different NWs which have magnetic and piezoelectric properties[10–12] allowing for a wider range of applications such as magnetic force microscopy (MFM) and piezoresponse force microscopy (PFM). However, there are common limitations to these approaches, one of which is that it is difficult to define the angle of the NW probe on the cantilever. This is because the catalyst must be deposited on a flat surface to allow the growth of a NW, and to our knowledge, there is no way to control the direction of the NW growth at a precise angle to compensate for the tilt angle of the cantilever caused by the mount of the AFM head, which is typically 10°–15° (mostly commonly 13°)[13–15] with respect to the horizontal axis.

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Currently, most commercial HAR AFM probes are made of either silicon[16–19] or CNT.[20–22] Commercial silicon HAR AFM tips are fabricated from conventional silicon tips and there are plenty of silicon materials that could be milled away during this sculptinglike process. Hence, the final shape and tilt angle of the tips could be easily defined during the milling process. On the other hand, CNT HAR AFM tips are fabricated by attaching them using a nanogripper or nanoassemly mentioned in the earlier paragraph.[8] The process of detaching the CNT with the nanogripper is very complicated and challenging. This is because the jaws of the nanogripper are big and it blocks the secondary electrons (SE) generated from the CNT to reach the SE-detector. This makes it very difficult to judge whether the CNT is in between the jaws of the nanogripper.[8]

As mentioned above, most commercial HAR AFM probes are made of silicon and CNT. Hence, if a conducting AFM tip is required, the silicon AFM tip needs to be coated with a metallic layer to make the tip conducting. One problem with this kind of metallic coated tip is that the metallic coating wears off easily after a few scans. Hence, it is advantageous for a tip to be made of entirely metallic materials for conducting AFM applications. In this article, a new and easy method for fabrication of high-aspect-ratio tips with precise control of the tilt angle of the tip with respect to the cantilever axis by using a FIB lift-out sample preparation technique, inspired by cross-section TEM sample preparation, is introduced. This technique allows the fabrication of perpendicular NW probes during scanning. Two silicon NW AFM tips with different inclination angles were fabricated. The scanning capabilities of these two different AFM tips on challenging closely spaced HAR NW arrays were demonstrated. In addition, we also demonstrated that this technique can be used to fabricate a HAR AFM tip that is made entirely of copper that can be used as a conducting AFM, which will prevent the wearing off effect of metal coating on existing conducting AFM tips.

II. EXPERIMENT

Figure 1(a) shows a large silicon NW array that was patterned using laser interference lithography followed by metal assisted chemical etching.[23] The details of the fabrication process can be found in the supplementary material.[24] A Stabila digital spirit level was used to ensure that the FIB stage was leveled to the horizon before the silicon substrate that contained the silicon NW array and a blunt AFM cantilever were placed on the stage. After the silicon NW array substrate and the AFM cantilever were placed on the FIB stage, the Stabila digital spirit level was employed to ensure both the substrate and the AFM cantilever were placed horizontally with inclination angles less than 0.1°. This is to ensure the silicon NWs were placed perpendicular to the stage and the AFM cantilever was leveled with the FIB stage that had been leveled to the horizon earlier on. An Omniprobe with x, y, and z micrometer controls inside an FEI Helios 600 dual-beam system was then lowered to touch the top of a single silicon NW [Fig. 1(a)], and the NW was then welded to the

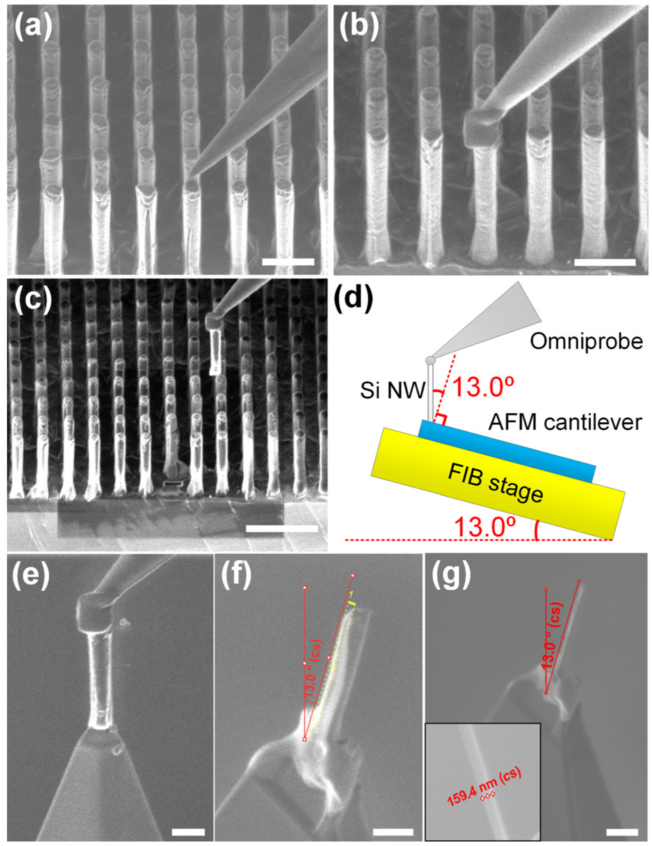

FIG. 1. (Color online) SEM images showing the fabrication process of HAR AFM probes with a precisely defined tilt angle. (a) A tungsten probe is approaching a single silicon NW in a large array. Scale bar: 2 μm. (b) The top of a silicon NW is welded to the tungsten probe. Scale bar: 2 μm. (c) The silicon NW is cut at the base by an ion beam and detached from the substrate. Scale bar: 5 μm. (d) The attachment of the silicon NW to the AFM cantilever by tilting the FIB stage at 13.0°. (e) The silicon NW is transferred to a blunt AFM probe. Scale bar: 1 μm. (f) The silicon NW is attached at an angle of 13.0° to the normal axis of the cantilever. Scale bar: 1 μm. (g) The final diameter was ion milled to around 159.4 nm as shown in the inset. Scale bar: 1 μm.

Omniprobe with a platinum source [Fig. 1(b)] and cut at the base using a Ga^{+} ion beam [Fig. 1(c)]. The silicon NW that was welded to the Omniprobe was then withdrawn vertically upward to allow the FIB stage to be tilted at 13.0° with an accuracy of 0.1°. The silicon NW was then lowered down to the blunt AFM probe [an illustration of this process is shown in Fig. 1(d)]. Figure 1(e) shows that the silicon NW was attached to the cantilever at an angle of 13.0° with respect to the normal axis of the cantilever. The silicon NW could also be attached at other angles with respect to the vertical axis of the cantilever by varying the tilting angles of the sample. Such attachment of silicon NW is similar to the method used in nanorobotic assembly[8] but is much easier and straightforward. A "fat" silicon NW that had a diameter of approximately 500 nm was attached to the blunt AFM tip. This is a contingency plan, in case the silicon NW is not lifted perpendicularly upright or the silicon NW array is placed at an inclination angle. The attached silicon NW has enough extra material that can then be milled to reduce its diameter using ion milling to produce exact orientation of the final tip [Fig. 1(e)]. The milling was done using the FEI Helios 600 dual beam. The initial milling was done at 30 kV and a beam current of 0.23 nA. Once the diameter has reached 250 nm, the voltage and beam current were dropped to 2 kV and 23 pA, respectively. The whole milling time takes less than 5 min. The final tilt angle of the tip with respect to the normal axis of the cantilever was 13.0° [Fig. 1(f)]. The width of the NW probe was around 160 nm [Fig. 1(f)]. The primary advantage of this method is that the tilt angle of the mounted NW tip can be controlled precisely with the last step ion-milling. A thinner NW say 250 nm could also be transferred directly (not shown in this work) so that less milling will be required.

It should be noted that this fabrication process is highly repeatable. Four probes have been fabricated and the final tip length is within $\pm 0.04\,\mu$m. In addition, this fabrication method can also be used on broken AFM tips, so that cantilevers can be recycled an unlimited number of times. This is not possible using electron beam induced deposition[5,6] or VLS NW growth, as it is not possible to control the deposition of carbon or wire growth at a specific angle. It is also not possible to regrow silicon NWs or CNTs at the same spot, as the surface would be rough and the position of the deposited catalyst material could not be positioned so as to control the orientation of the subsequent growth of the silicon NW or CNT.[9]

III. RESULTS AND DISCUSSION

A study of the effects of probe tilt angle on characterization of HAR structures was carried out. Two silicon HAR AFM probes with 0° and 13.0° tilt angles with respect to the vertical axis of the AFM cantilever were fabricated [Figs. 2(a) and 1(g), respectively]. These probes were used to scan an array of 2.22 μm-tall, 520 nm-diameter nanopillars that were spaced 690 nm apart [Fig. 2(b)]. The height of the nanopillars 2.22 μm was calculated based on the SEM software measurement and the inclination angle of the slope of the SEM sample mounting stub (see Fig. S1 in the supplementary material). The topographies obtained by the AFM

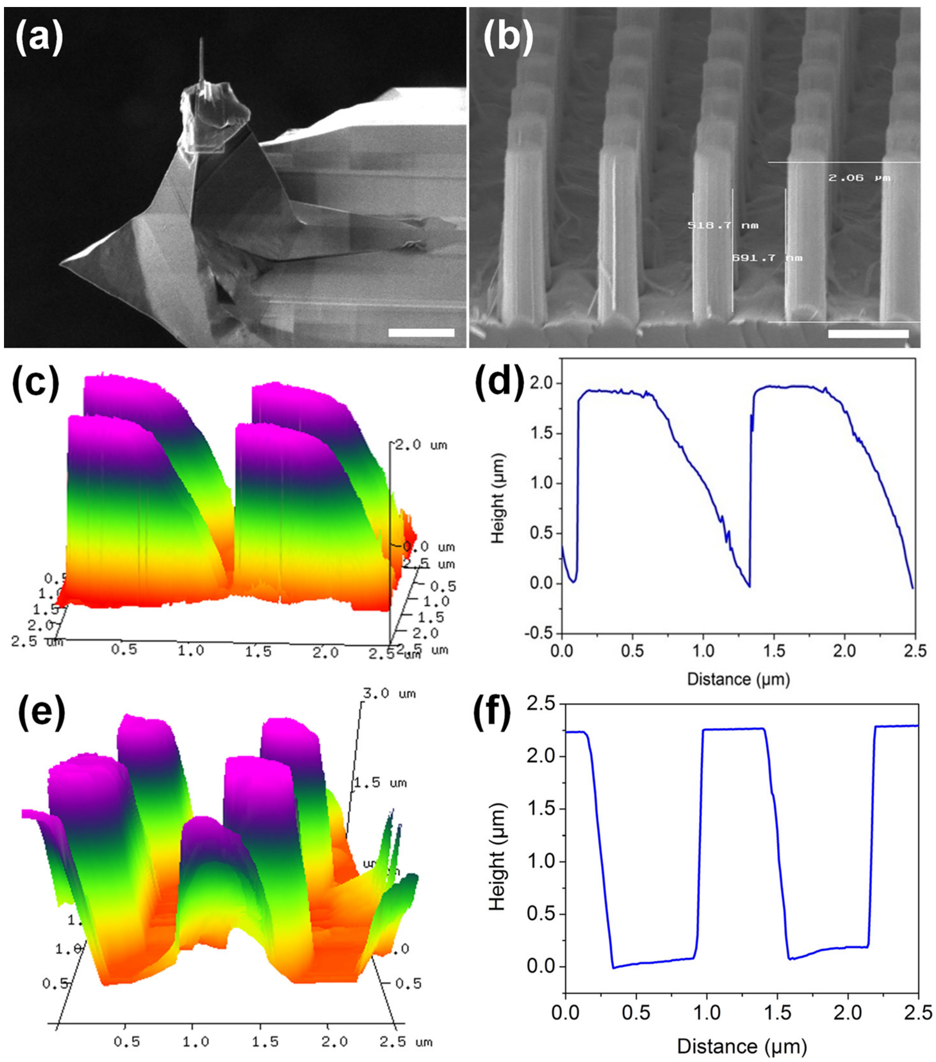

FIG. 2. (Color online) AFM probe tilt angles with respect to the cantilevers and mapping of HAR pillar arrays. (a) A silicon NW probe is mounted normal to a horizontal AFM cantilever. Scale bar: 5 μm. (b) SEM image of an array of silicon NWs to be mapped by the as-fabricated AFM probes. Scale bar: 1 μm. (c) The 3D mapping of the silicon nanopillar array's topography by the silicon NW AFM probe shown in (a). (d) The scanning profile of the nanopillar array extracted from (c). (e) The 3D mapping of the silicon nanopillar array's topography by the 13.0° tilt compensated silicon NW AFM probe. (f) The scanning profile of the nanopillar array extracted from (e).

probes are presented in Figs. 2(c)–2(f). The silicon HAR AFM probe that was vertically aligned with respect to the longitudinal axis of the AFM did not give an accurate profile of the nanopillars [Fig. 2(c)]. Figure 2(d) shows the cross-section profile of the scan and the height measured from the AFM is 0.3 μm shorter than the height obtained from the SEM.

The other AFM NW tip which was placed and ion-milled to 13.0° with respect to the normal axis of the cantilever [Fig. 2(e)] was used to map the topography of the same nanopillar array that is shown in Fig. 2(b). An accurate representation of the silicon nanopillar array's 3D topography was mapped out as shown in Fig. 2(e). In addition, a cross section profile along the arrow indicated in Fig. 2(f) shows that the height obtained from the AFM scan (2.25 μm) matched the height measured from the SEM image with only an error of 1.4%. This probe gave successful mapping of the topography of the silicon NW array in all directions. This is the first successful demonstration of an HAR AFM tip being used to map the topography of a nanopillar array.

In addition to silicon and CNT, other materials that have desirable electrical and magnetic properties could be mounted to the tip of the AFM probe for specific applications such as MFM and PFM. Current conducting and magnetic AFM tips are mostly coated with a platinum film and a magnetic film, respectively. These films tend to wear off easily. Although there are harder, more wear resistant coatings such as doped diamond coatings, they usually suffer from large radius or low conductivity.[25] Hence, it would be more practical to have probes that are made entirely from electrically conducting materials or magnetic materials. The method described here could also be used to fabricate HAR tips made of various materials. Hence, a copper NW AFM tip is fabricated using the same lift-out technique. Instead of using a NW array, a copper substrate was used as the starting material. The first step is to place a copper substrate and an AFM cantilever on the FIB stage. Opposing trenches are then milled out with the Ga^+ ion source and approximately 1.0 μm thick lamella is left free standing in the wafer [Fig. 3(a)].

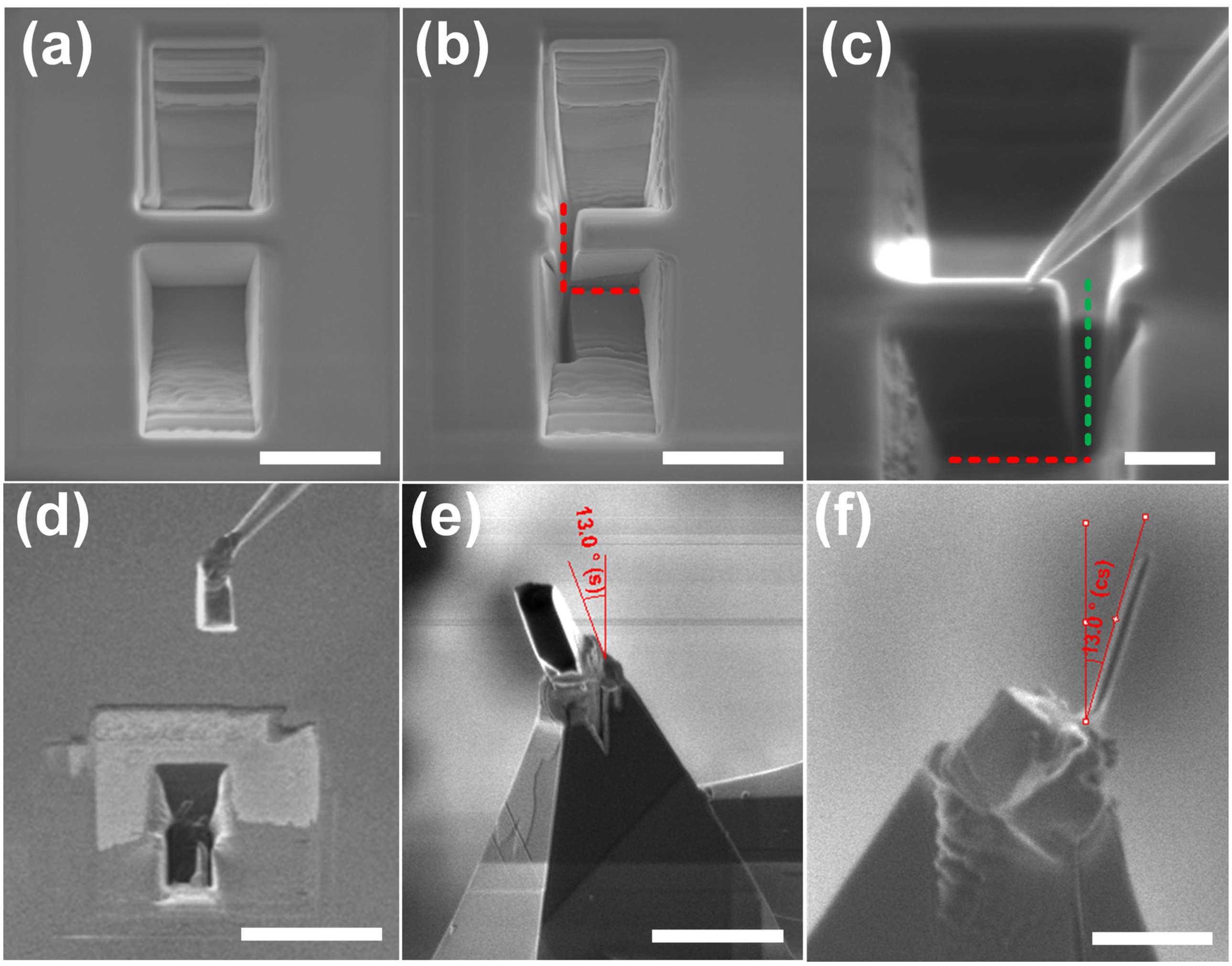

Fig. 3. (Color online) Fabrication of a HAR tip that is made entirely of copper. (a) Opposing trenches were milled out with the Ga^+ ion source and a 1.0 μm thick lamella is left free standing in the wafer. Scale bar: 5 μm. (b) An L shaped cut is then made on the lamella by Ga^+ ion source leaving it to be hung on the right hand side of the trench. Scale bar: 5 μm. (c) A micromanipulator was then lowered down to touch the top of the lamella and the both objects were welded with platinum (not shown in this figure). Scale bar: 2 μm. (d) The lamella was then cut free, extracted from the wafer. Scale bar: 10 μm. (e) The stage was tilted at 13.0° when the copper lamella was attached to the AFM cantilever. Scale bar: 5 μm. (f) The lamella was then milled down to a NW with an inclination of 13.0° with respect to the normal axis. Scale bar: 2 μm.

An "L" shaped cut is then made on the lamella by Ga^{+} ion source leaving it to be hung on the right hand side of the trench [Fig. 3(b)]. This lamella is then welded to the micromanipulator [Fig. 3(c)], "cut" free, extracted vertically from the substrate, the FIB stage was then tilted 13.0° as shown in Fig. 2(d). The copper lamella that was welded to the micromanipulator was then brought down to touch the AFM probe. The micromanipulator was kept at this position while a Gas Injection System was used to deposit platinum to weld the Omniprobe and the copper lamella together. The left hand side of the lamella [Fig. 3(c)] was cut using the Ga^{+} ions. The copper lamella was then raised vertically upward [Fig. 3(d)]. The FIB stage was tilted 13.0° as described previously. The copper lamella was brought downwards to contact the AFM probe. The Omniprobe was then cut free. The FIB stage was tilted back to the horizon level, and it shows that the copper lamella was standing at 13.0° with respect to its normal axis [Fig. 3(e)]. The lamella was then shaped to 160 nm in diameter by using the Ga^{+} ion beam [Fig. 3(g)].

The fact that the measured height of the nanopillars is 0.3 μm shorter than the height obtained from the SEM image indicated that the silicon NW probe mounted perpendicular to the cantilever [i.e., 0° with respect to the normal axis-Fig. 4(a)] cannot reach the bottom of the pillars due to the mount of the probe on the AFM head. Hence, when the tip was scanned along the direction indicated by the red arrow, it was blocked by adjacent nanopillars as shown in the schematic in Fig. 4(b). This is because if the NW tip is placed perpendicular to the cantilever, it will be inclined at an angle of 77.0° with respect to the substrate. This may not be a critical issue in mapping the topography of HAR trenches or walls because the NW tip can still scan along the wall or trenches as illustrated in Fig. 4(c). However, when this perpendicular aligned AFM tip is scanned across the structure, the close proximity of the tall walls prevents the NW tip from touching the bottom of the trenches [Fig. 4(d)]. Hence, this becomes a critical issue when mapping NWs or nanopillars. Therefore, it is impossible to obtain a 3D mapping of NWs or nanopillar array topography if the NW tip angle is not compensated for the tilt of the cantilever caused by the mount of the AFM head. To solve this problem, the NW tip must be attached to the cantilever in such a way that the NW probe remains vertical with respect to the substrate during AFM scanning. To achieve this, the NW probe must attach to the AFM cantilever at 13.0° [Fig. 4(e)]. Hence, when the silicon NW AFM tip as shown in Fig. 4(e) was used to scan the nanopillar topography, it was able to map out an accurate 3D topography of the NW array as shown in Fig. 4(f). Proper choice and control of the tilt angle of the NW probe on the cantilever mount is extremely important in defining the trajectory of the silicon NW tip for scanning of the complete topography of any HAR structure.

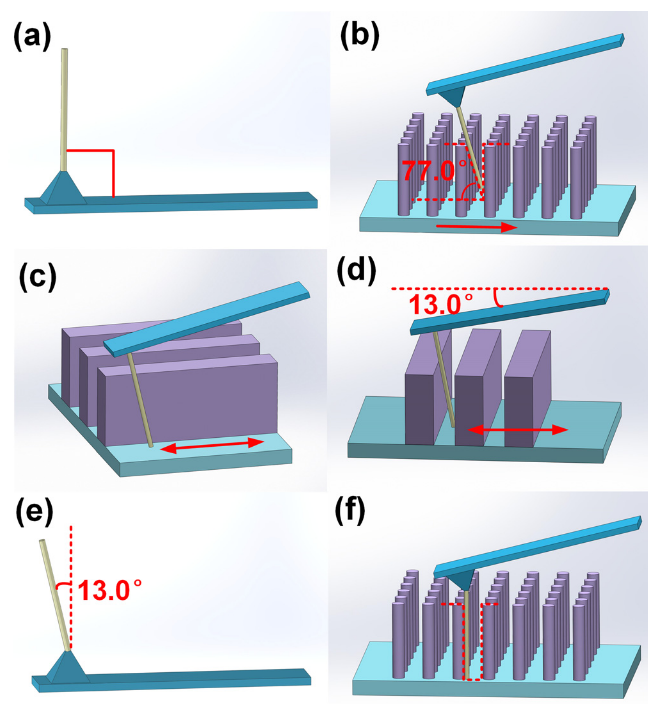

Fig. 4. (Color online) Effects of silicon HAR AFM probe tilt angle on mapping of HAR dense silicon nanopillar arrays. (a) A schematic of a silicon NW probe that is attached perpendicular to the AFM cantilever. (b) An illustration showing the silicon NW probe scanning an array of silicon nanopillar. (c) An illustration showing how a perpendicularly aligned silicon NW probe could be used to scan a set of HAR walls without any issue. (d) When the HAR walls sample is rotated 90°, the silicon NW cannot reach the bottom of the HAR walls. (e) A cartoon showing how the tilt compensation silicon NW probe looks like. (f) The probe in (e) does not have any problem in mapping the HAR nanopillars regardless of the sample orientation with respect to the probe.

IV. SUMMARY AND CONCLUSIONS

In summary, we have demonstrated a simple lift-out and ion milling process for fabrication of high-aspect-ratio NW probes by attaching a NW to AFM tips with precise control of the angle of the NW with respect to the AFM cantilever axis. We demonstrated the strong effect the tilt angle of the NW tip has on the mapping of topography of closely spaced HAR structure arrays. We also demonstrate that this method can be extended to fabricate a wide range of NW probes from a wide range of materials chosen for different applications, and can be mounted on blunted AFM tips. The probes themselves can be reshaped after extended use through the use of focused ion milling. These techniques provide a powerful and versatile approach in the production of low-cost HAR AFM tips for characterization of closely spaced high-aspect-ratio surface topography.

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[1]G. Binnig and C. F. Quate, Phys. Rev. Lett. **56**, 930 (1986).
[2]B. Cappella and G. Dietler, Surf. Sci. Rep. **34**, 1 (1999).
[3]F. J. Giessibl, Rev. Mod. Phys. **75**, 949 (2003).
[4]M. J. Vasile, D. Grigg, J. E. Griffith, E. Fitzgerald, and P. E. Russell, J. Vac. Sci. Technol., B **9**, 3569 (1991).
[5]Y. Akama, E. Nishimura, A. Sakai, and H. Murakami, J. Vac. Sci. Technol., B **8**, 429 (1990).
[6]I.-C. Chen, L.-H. Chen, C. Orme, A. Quist, R. Lal, and S. Jin, Nanotechnology **17**, 4322 (2006).

[7]H. Dai, J. H. Hafner, A. G. Rinzler, D. T. Colbert, and R. E. Smalley, Nature **384**, 147 (1996).
[8]V. Eichhorn, M. Bartenwerfer, and S. Fatikow, IEEE Trans. Autom. Sci. Eng. **9**, 679 (2012).
[9]D. S. Engstrom, V. Savu, X. Zhu, I. Y. Y. Bu, W. I. Milne, J. Brugger, and P. Boggild, Nano Lett. **11**, 1568 (2011).
[10]N. Silvis-Cividjian, C. W. Hagen, P. Kruit, M. A. J. vd. Stam, and H. B. Groen, Appl. Phys. Lett. **82**, 3514 (2003).
[11]K. Teker, Sens. Actuator, A **216**, 142 (2014).
[12]Q. B. Zhang, H. X. Chen, J. X. Wang, D. G. Xu, X. H. Li, Y. Yang, and K. L. Zhang, ChemSusChem **7**, 2325 (2014).
[13]M. J. D'Amato, M. S. Marcus, M. A. Eriksson, and R. W. Carpick, Appl. Phys. Lett. **85**, 4738 (2004).
[14]M. S. Marcus, R. W. Carpick, D. Y. Sasaki, and M. A. Eriksson, Phys. Rev. Lett. **88**, 226103 (2002).
[15]A. Savenko, I. Yildiz, D. H. Peterson, M. Bartenwerfer, F. Krohs, M. Oliva, and T. Harzendorf, Nanotechnology **24**, 465701 (2013).
[16]NanoAndMore GmbH, "High aspect ratio (HAR) AFM probes," 2016, http://www.nanoandmore.com/High-Aspect-Ratio-AFM-Probes.html.
[17]Windsor Scientific Ltd. "AFM probe AR10T-NCHR," 2016, http://afm-probes.windsorscientific.co.uk/AFM-Probe-AR10T-NCHR.html.
[18]NanoWorld AG. Pointprobe®, "High aspect ratio AFM probes AR5T-NCHR," 2016, http://www.nanoworld.com/pointprobe-high-aspect-ratio-afm-tip-ar5t-nchr.
[19]Nanosensors™, "High aspect ratio tilted non-contact tapping mode high resonance frequency reflex coating AFM tip AR10T-NCHR," 2016, http://www.nanosensors.com/High-Aspect-Ratio-Tilted-Non-Contact-Tapping-Mode-High-Resonance-Frequency-Reflex-Coating-afm-tip-AR10T-NCHR.
[20]Nanotools GmbH, "CNT AFM tips," 2016, http://www.nanotools.com/products/blue-line/m-cnt/.
[21]Bruker AFM probes, "CNT probes," 2015, http://www.brukerafmprobes.com/Product.aspx?ProductID=3245.
[22]NanoAndMore GmbH, "AFM probe M-CNT-100," 2016, http://www.nanoandmore.com/USA/AFM-Probe-M-CNT-100.htm.
[23]W. Choi, T. H. Liew, M. K. Dawood, H. I. Smith, C. V. Thompson, and M. H. Hong, Nano Lett. **8**, 3799 (2008).
[24]See supplementary material at http://dx.doi.org/10.1116/1.4961595 for details of the Si NW fabrication and the measurement of the nanopillar height.
[25]N. S. T. I. Team, N. S. T. I. Fellows, and D. E. Summit, *Nanotechnology 2010: Advanced Materials, CNTs, Particles, Films and Composites* (NSTI-Nanotechnology, 2010), Vol. 1, www.nsti.org.