Engineering Silicon to Porous Silicon and Silicon Nanowires by Metal-Assisted Chemical Etching: Role of Ag Size and Electron-Scavenging Rate on Morphology Control and Mechanism

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Supporting Information

ABSTRACT: We demonstrate controlled fabrication of porous Si (PS) and vertically aligned silicon nanowires array starting from bulk silicon wafer by simple chemical etching method, and the underlying mechanism of nanostructure formation is presented. Silicon-oxidation rate and the electron-scavenging rate from metal catalysis play a vital role in determining the morphology of Si nanostructures. The size of Ag catalyst is found to influence the Si oxidation rate. Tunable morphologies from irregular porous to regular nanowire structure could be tailored by controlling the size of Ag nanoparticles and H2O2 concentration. Ag nanoparticles of size around 30 nm resulted in irregular porous structures, whereas discontinuous Ag film yielded nanowire structures. The depth of the porous Si structures and the aspect ratio of Si nanowires depend on H2O2 concentration. For a fixed etching time, the depth of the porous structures increases on increasing the H2O2 concentration. By varying the H2O2 concentration, the surface porosity and aspect ratio of the nanowires were controlled. Controlling the Ag catalyst size critically affects the morphology of the etched Si nanostructures. H2O2 concentration decides the degree of porosity of porous silicon, dimensions and surface porosity of silicon nanowires, and etch depth. The mechanisms of the size- and H2O2-concentration-dependent dissociation of Ag and the formation of porous silicon and silicon nanowire are described in detail.

INTRODUCTION

Silicon nanostructures have attracted a great deal of attention and interest because of their potential applications in various fields, such as nanoelectronics, optoelectronics, energy storage, and energy conversion, and in biological and chemical sensors. Nanostructures such as porous Si (PS), Si nanowires (SiNWs), nanogras, nanocrystals, and quantum dots are some of them. Out of these nanostructures, porous Si (PS) and Si nanowires (SiNWs) are widely studied and used in numerous applications. PS has been mostly exploited for its luminescent property and also as sensors. SiNWs have been mostly applied as chemical or biological sensors, solar cells, and anode for batteries. In addition, it has been demonstrated that SiNWs can serve as effective photocatalysts for the photocatalytic degradation of organic dyes and toxic pollutants. Controllable fabrication of Si nanostructures is therefore a prerequisite for various applications. Depending on the type of application, the use of nanostructures differs. For certain applications, highly porous Si is used and for certain other applications, nanowires are used. For example, porous Si is widely used in matrix-assisted laser desorption/ionization and optical sensing applications, whereas nanowire morphology is widely used in gas-sensing applications. So far, a number of methods have been developed to fabricate Si nanostructures using top-down or bottom-up approaches, such as vapor–liquid–solid (VLS), reactive-ion etching (RIE), electrochemical etching, and metal-assisted chemical etching (MACE), all of which aim to control various parameters of the Si structures. VLS and RIE methods need sophisticated instruments and toxic gases. Therefore, a simple, well-controlled, cost-effective method to fabricate Si nanostructures is necessary. Among these methods, metal-assisted chemical etching (MACE) has attracted increasing attention in recent years because of its simplicity and low cost for fabricating various Si nanostructures. The nanostructures fabricated by MACE have demonstrated their potential application in various fields, such as photovoltaics, energy storage, thermal power conversion, chemical and biological sensing, and biomimic superhydrophobicity.

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Fabrication of SiNWs and PS has been reported separately. However, deep understanding relating the combined effect of metal catalyst size and H$_2$O$_2$ concentration and its mechanism of formation is still lacking. This work gives a new understanding on the combined effect of size of metal catalyst and the H$_2$O$_2$ concentration to result in precise control over the etched nanostructures. This work brings into light one simple method for the controlled fabrication of porous Si and Si nanowires. Morphology was controlled by controlling the concentration of the AgNO$_3$, Ag deposition time, and the concentration of H$_2$O$_2$. Controlled fabrication thereby results in different morphology with one simple cost-effective technique that can be used for different applications. Evolution of nanostructures with respect to different experimental parameters and its possible mechanism are discussed in detail.

### Results and Discussion

**Effect of AgNO$_3$ Concentration.** Field emission scanning electron microscopy (FESEM) images of Ag deposited over Si for 15 s, 30 s, 1 min, 2 min, and 5 min with 0.0017 M AgNO$_3$/4.8 M HF are shown in Figure 1a–e, respectively. FESEM images of Ag deposited over Si for 15 s, 30 s, 1 min, 2 min, and 5 min with 0.005 M AgNO$_3$/4.8 M HF are shown in Figure 1f–j, respectively. From Figure 1, it is evident that the Ag particle size grows from 30 to 200 nm as the deposition time increases for 0.0017 M AgNO$_3$ concentration. For 0.005 M AgNO$_3$ concentration, the Ag particle size grows from 50 nm until it percolates and forms discontinuous film/network structure for 5 min Ag deposition. From these observations, it can be concluded that for lower concentration of AgNO$_3$ (0.0017 M), the Ag particle starts growing slowly with initial small size. As the deposition time increases, there is increase in particle size from 30 to 200 nm. For higher AgNO$_3$ concentration (0.005 M), in case of 15 s Ag deposition, the particle size deposited is larger than that of the 15 s Ag deposition with 0.0017 M AgNO$_3$ concentration. Because the concentration of Ag ions is higher, the deposition rate is also higher. As the particle grows with time, the particles coalesce to percolate the whole substrate surface and form a discontinuous film, which can be seen in Figure 1j.

**Effect of Ag Particle Size on Si Etching.** FESEM images of etched samples of the Ag deposited over Si for 15 s, 30 s, 1 min, 2 min, and 5 min with 0.0017 M AgNO$_3$ concentration are shown in Figure S1 (see the Supporting Information). From the scanning electron microscopy images, we could notice that for 15 s Ag deposition time, the Ag particles deposited is ∼30 nm, and during etching in HF/H$_2$O$_2$ solution, these particles tend to etch the wafer in a random manner. Increasing the size of Ag particles reduces the randomness in the etching. This will be explained in detail in the following sections. FESEM images of etched samples of the Ag deposited over Si for 15 s, 30 s, 1 min, 2 min, and 5 min with 0.005 M AgNO$_3$ concentration are shown in Figure S2 (see the Supporting Information). In this case, random pores are observed for 15 and 30 s Ag depositions. These random pores get reduced and a definite vertical etching results for 2 and 5 min Ag depositions, thus
forming a nanowire-like morphology. This can be confirmed from the cross-section FESEM images given in the following sections.

**Effect of H$_2$O$_2$ Concentration.** Effect of H$_2$O$_2$ on the porosity, roughness, depth of etching, and SiNW formation is studied by varying the concentration of H$_2$O$_2$ from 0.05 to 0.3 M. Figure 2a–d shows the top-view FESEM images of the Si etched in 0.05 M H$_2$O$_2$/4.8 M HF solution of 30 s, 1 min, 2 min, and 5 min Ag-deposited Si, respectively. Figure 2e–h shows the corresponding cross-section FESEM images. Figure 2 reveals that 30 s Ag-deposited Si yields highly random porous structure after etching. The depth of the pores was found to be 4–5 μm. Because the Ag particles deposited on the Si are small and isolated, they form a highly random porous structure. As the Ag deposition time increases, the etching becomes directional.

For 1 min Ag-deposited Si, the etching is directional with distorted walls. Ag particles in 1 min Ag-deposited Si are larger than those in 30 s Ag-deposited Si, which could manage to form vertical downward etching to compete the etching in all other directions. However, the size of the Ag is still smaller and the Ag particles get separated from the mother Ag particle and participate in etching horizontally, which is the reason for the distortion in the wall of the vertical etched structures. In 2 and 5 min Ag-deposited samples, regular vertical etching was observed. The depth of the SiNWs was found to be 5–10 μm. For smaller Ag, the etch rate is low. From Figure 3, it is observed that for 30 s Ag-deposited Si, the etching is very porous as evident from the top-view FESEM images. The average depth of the pores was found to be 9 μm. As the concentration of H$_2$O$_2$ is increased, the porosity as well as the depth of the random pores increases. For 1 min Ag-deposited sample, the randomness of pores decreases compared to 30 s Ag-deposited Si. The 2 and 5 min Ag-deposited Si form a well-aligned vertical nonporous nanowire structure. Figure 4a–d shows the top-view FESEM images of the Si etched in 0.3 M H$_2$O$_2$/4.8 M HF solution of 30 s, 1 min, 2 min, and 5 min Ag-deposited Si. The 2 and 5 min Ag-deposited Si form a well-aligned vertical nonporous nanowire structure. Figure 4e–h shows the respective cross-section FESEM images.

From Figure 4, it is observed that, for 30 s Ag-deposited Si, the etching is very porous, as evident from the top-view FESEM images. The depth of the pores was found to be 17 μm. As the concentration of H$_2$O$_2$ is increased, the porosity as well as the depth of the random pores increases as compared to 0.1 M H$_2$O$_2$. For 1 min Ag-deposited sample, the randomness of pores decreases compared to 30 s Ag-deposited Si. The 2 and 5 min Ag-deposited Si form a well-aligned vertical nonporous nanowire structure. Figure 4a–d shows the top-view FESEM images of the Si etched in 0.3 M H$_2$O$_2$/4.8 M HF solution of 30 s, 1 min, 2 min, and 5 min Ag-deposited Si. Figure 4e–h shows the respective cross-section FESEM images.
nanowire thickness is found to be low and the nanowires are found to be bundled at the top.

**Figure S3** (see the Supporting Information) shows the top view and 45° tilt view of a high-magnification FESEM image of Si etched in 0.6 M H₂O₂/4.8 M HF solution of 5 min Ag-deposited Si (with 0.005 M AgNO₃). Nanowires are bundled at the top. Also the thickness of the wires is low compared to that of the wires formed in 0.3 M H₂O₂/4.8 M HF solution (Figure 4d,h).

The thickness and porosity of the nanowire surface depend on the concentration of H₂O₂; for higher concentration, the surface is highly porous, and porosity decreases as the concentration is reduced. **Figure 5** shows the HRTEM images of the nanowires prepared at lower (0.05 M) and higher (0.6 M) H₂O₂ concentrations. The thickness of the porous layer surface increases as the H₂O₂ concentration is increased. When the H₂O₂ concentration is high, the conversion rate of Ag⁺ to Ag is high and hence it energetically favors more Ag nucleation on the walls, thereby making the surface more rough and porous.

The thickness of the wires decreases as the H₂O₂ concentration increases. The length of the nanowires increases from 7 to 20 μm for 5 min Ag film as the H₂O₂ concentration increased from 0.05 to 0.3 M. From HRTEM images, it is evident that the thickness decreases from 300 to 100 nm as the H₂O₂ concentration increases.

**Mechanism of Formation.** Step I. During Ag deposition, two simultaneous processes happen: deposition of Ag and etching of Si. Because the electrochemical potential of Ag²⁻/Ag is more positive than the Fermi energy of the Si substrate, holes are injected into the valence band of Si from Ag⁺ when Si substrate is immersed in HF/AgNO₃ solution. The Ag⁺ reduces to Ag, forming a nuclei in the local site. Simultaneously, the holes that are injected into the valence band of Si from the Ag nuclei locally oxidize Si, which gets dissolved by HF. Si atoms under the Ag nuclei get dissolved; as a result, the Ag particles sink into the Si substrate. This process is represented in eqs 1–3. The overall reaction during Ag deposition is given in eq 4. In this case, the etching of Si is meager.

At cathode

\[ \text{Ag}^+ + e^- = \text{Ag}, \quad E^o = 0.79 \text{ V} \]  

(1)

At anode

\[ \text{Si} + 2\text{H}_2\text{O} = \text{SiO}_2 + 4\text{H}^+ + 4e^-, \quad E^o = 0.91 \text{ V} \]  

(2)

\[ \text{SiO}_2 + 6\text{HF} = [\text{SiF}_6]^{2-} + 2\text{H}_2\text{O} + 2\text{H}^+ \]  

(3)

**Overall reaction**

\[ \text{Si} + 6\text{HF} + 4\text{Ag}^+ = 4\text{Ag} + [\text{SiF}_6]^{2-} + 6\text{H}^+ \]  

(4)

When the Si substrate is immersed in HF/AgNO₃ solution, Ag starts nucleating initially for very short deposition time (15 s) and small, well-separated nearly spherical-shaped nanoparticles are formed; as the deposition time is increased, the Ag nanoparticles continue to grow in size, increasing the surface coverage of the substrate. When the deposition time is increased further, the nucleated Ag particles coalesce with the neighboring particle. When the deposition time is further prolonged to 5 min, Ag particles grow and reach a percolation threshold, which forms a discontinuous thin film.

**Step II.** In the second step, Ag-deposited Si substrate is immersed in HF/H₂O₂ solution and etching of Si at Ag-deposited sites takes place. The Ag nanoparticle possesses higher electronegativity than Si and therefore strongly attracts electron from Si. Si loses electron to the Ag nuclei and it gets oxidized, to form silicon dioxide underneath the Ag particles. The oxidized Si (SiO₂) is etched away by HF. The electrons are transferred to silver particles, which in turn reduces H₂O₂ on the surface of metal particle facing the electrolyte. Thus, the excess collected electrons are used to reduce H₂O₂. After reducing H₂O₂, the Ag particle collects electrons again and oxidizes Si. The collected electron in turn reduces H₂O₂. This process continues forming pits at the site where Ag was deposited. Here, H₂O₂ acts as a driving force in etching. It acts as scavenger of electrons from the Ag nanoparticle; in other words, H₂O₂ locally oxidizes Ag to Ag⁺. Ag⁺ ions formed tend to become Ag by quickly reacting with the Si atoms to get back as Ag. As H₂O₂ scavenges more electrons from Ag, more electrons from the Si is collected by the Ag and this process continues. This process is schematically shown in **Figure 6.** The possible chemical reactions during the etching process are given in eqs 5–8. Ag surface facing the electrolyte acts as cathode,

![Figure 6. Schematic to explain the etching of Si.](image)}
where H2O2 reduced to H2O, and is given in eq 5. At anode, Si is oxidized to SiO2 and the formed SiO2 simultaneously dissolves in HF solution; these reactions are given in eqs 6 and 7. The total reaction is given in eq 8.

At cathode (Ag surface facing electrolyte)

\[ \text{H}_2\text{O}_2 + 2e^- + 2\text{H}^+ = 2\text{H}_2\text{O}, \quad E^0 = 1.78 \text{ V} \]  
(5)

At anode (Ag at Ag/Si interface)

\[ \text{Si} + 2\text{H}_2\text{O} = \text{SiO}_2 + 4e^- + 4\text{H}^+, \quad E^0 = 0.91 \text{ V} \]  
(6)

\[ \text{SiO}_2 + 6\text{HF} = [\text{SiF}_6]^{2-} + 2\text{H}_2\text{O} + 2\text{H}^+ \]  
(7)

Total reaction

\[ \text{Si}^0 + 2\text{H}_2\text{O}_2 + 6\text{F}^- + 4\text{H}^+ = [\text{SiF}_6]^{2-} + 4\text{H}_2\text{O} \]  
(8)

The potential of the total reaction can be expressed as given below.

\[ \Delta E = \Delta E^0 - \frac{0.059}{4} \left[ \log \frac{[\text{SiF}_6^{2-}]}{[\text{H}_2\text{O}_2]^2} - \log[(\text{H}^+)^4][\text{F}^-]^6 \right] \]  
(9)

The etch rate of Si depends on scavenging rate of electrons by H2O2 from Ag; therefore, the concentration of H2O2 plays a key role in etching. At low H2O2 concentration, scavenging rate of electrons by H2O2 is low and therefore the etching rate is also low. At high H2O2 concentration, scavenging rate of electrons by H2O2 is high and therefore the etching rate is also high.

Depending on the size of the Ag and the H2O2 concentration, random-structured porous Si or Si nanowires are formed. Therefore, etch depth and the morphology are dependent on both the H2O2 concentration and the size of the deposited Ag. For isolated small Ag nanoparticles, the interaction between the Si and the Ag is not restricted by its surroundings. Once the Ag particles start etching, they interact with Si in all directions except the top.2 The electronegativity of the Ag particles tends to move the particles toward Si atoms. Thus, the Ag particle gets divided, which causes random etching and resulted in porous Si nanostructures. The randomness of the pores reduces as the Ag particle size increases. Unlike isolated particles, in discontinuous film, the clusters are bound to each other, and during etching, the movement of Ag clusters is restricted, resulting in collective sinking of the discontinuous Ag network vertically down. Very small Ag particles detach from the bigger clusters along their way vertical down, and this etches the side walls of the wires, causing porous structures to vertical Si nanowire structure, which is proved by elemental mapping. For this study, after etching in 0.3 M H2O2/4.8 M HF, the substrate was washed with deionized water and taken for elemental mapping study.

Figure 7 shows the elemental analysis of Si nanowires without removing Ag to show the disintegration of Ag and the presence of Ag in the nanowire walls. Figure 7a shows the cross section of the nanowire etched in 0.3 M H2O2/4.8 M HF solution with 5 min Ag-deposited Si with 0.005 M AgNO3. In the figure, the Ag particles present at the root of the nanowires can be seen. Figure 7b shows the corresponding combined (Si and Ag) elemental map, where pink represents Si and yellow represents Ag. Figure 7bc shows the corresponding individual elemental maps of Si and Ag, from which it could be seen that, at the root of the nanowires, the presence of Ag is large, which represents the bigger particles. The presence of lower amount of Ag in the nanowire walls is also seen. Elemental mapping analysis thus confirms the dissociation of Ag and deposition of Ag in the nanowire walls.
For medium-sized Ag particles, the etching in lower \( \text{H}_2\text{O}_2 \) concentration is directional. At high \( \text{H}_2\text{O}_2 \) concentration, the etching is directional, but there is disintegration in the wall structure. Because the electron-scavenging rate (from Ag to \( \text{H}_2\text{O}_2 \)) and the electron collecting rate (from Si to Ag) are not at equilibrium, the disassociation of Ag takes place from the mother nuclei. The disassociated Ag etches the side walls therefore resulting in distorted wall structure. When we increase the \( \text{H}_2\text{O}_2 \) concentration, the potential for the etching process (hole supply) also increases, which can be known from eq 9. The driving force for the etching process becomes larger and thus increases the etching rate.

When the \( \text{H}_2\text{O}_2 \) concentration is high, the concentration of Ag\(^+\) is also high. The system tries to equilibrate by collecting the electrons through Si/Ag interface. If the equilibrium is not attained by converting Ag\(^+\) to Ag, then excess Ag\(^+\) ions remains. These being highly electronegative will seek and diffuse into the side wall of Si, where it collects electrons from the side wall of Si and gets detached from the mother Ag cluster and renucleates, forming a new silver nanoparticle and a new etch path. Thus, the increase in \( \text{H}_2\text{O}_2 \) concentration increases the amount of free Ag\(^+\), which tends to increase the porosity as well as the etching rate.\(^{35,44}\) For discontinuous Ag film, that is, for 5 min Ag deposition and lower \( \text{H}_2\text{O}_2 \) concentration (0.05 and 0.1 M), formation of wall-like structure results, as seen in the FESEM images of Figures 2d,h and 3d,h. The overall thickness of the wall structure is around a micron. For higher \( \text{H}_2\text{O}_2 \) concentration (0.3 M), wire morphology is obtained with lower thickness. At higher \( \text{H}_2\text{O}_2 \) concentration, the Ag to Ag\(^+\) conversion rate is high and as a result Ag particle film tends to equilibrate the reaction by disintegrating themselves and finding new etch paths. Thus, small Ag particles tend to disintegrate into even much smaller fragments and exhibit random etching, forming porous silicon, whereas larger Ag particles lead to vertical etching and form nanowire structure.

Figure 8A1 shows the schematic of the etching process of small Ag particles with low \( \text{H}_2\text{O}_2 \) concentration. Small Ag particles travel in all directions and thus finally create a random porous structure. The Ag particles get separated and renucleate to form newer nuclei for etching, which further get divided and the process continues. Figure 8A2 shows the schematic of the etching process of small Ag particles with high \( \text{H}_2\text{O}_2 \) concentration. Here, the high \( \text{H}_2\text{O}_2 \) concentration increases the etching rate and results in more randomness in the etching direction; therefore, the pores reach higher depth than those in the low \( \text{H}_2\text{O}_2 \) concentration. When the Ag particle size increases over 100 nm, the etching becomes directional, that is, vertically down; here, the diameter of the etched nanowires is high and the surface porosity will be very low.

In the case of higher \( \text{H}_2\text{O}_2 \) concentration, the Ag particles will diffuse the side walls and renucleation takes place around the mother Ag particle, which will further undergo renucleation and etching. For discontinuous Ag film, when the \( \text{H}_2\text{O}_2 \) concentration is low, the etching is directional, with etching shape dependent on the deposited Ag film. For medium \( \text{H}_2\text{O}_2 \) concentration, the discontinuous Ag film subdivides and etches vertically downward. When the \( \text{H}_2\text{O}_2 \) concentration is very high, that is, above particular limit, the discontinuous Ag film subdivides and etches vertically downward, with thickness of the wires lower compared to that at lower \( \text{H}_2\text{O}_2 \) concentration. These etching pathways are depicted in Figure 8B1,B2, respectively.

**CONCLUSIONS**

Strategy for fabrication of the porous Si and Si nanowires has been developed by controlling various parameters like AgNO\(_3\) concentration, Ag size, and \( \text{H}_2\text{O}_2 \) concentration. The size of the Ag particles and \( \text{H}_2\text{O}_2 \) concentration determine the Si etching morphology. For isolated small Ag nanoparticles, the etching is random irrespective of the concentration of \( \text{H}_2\text{O}_2 \). For lower \( \text{H}_2\text{O}_2 \) concentration, the etch depth and randomness are less, whereas for higher \( \text{H}_2\text{O}_2 \) concentration, these are high. Equilibrium electron supply from Si and equilibrium scavenging rate by \( \text{H}_2\text{O}_2 \) (lower concentration) lead to directional etching, forming wall structure. Higher scavenging rate causes higher conversion rate of Ag to Ag\(^+\), where the system tries to equilibrate the reaction by collecting more electrons from Si. Under this condition, Ag becomes unstable and gets disassociated and forms new etch paths so that more electrons can be collected from the Si. The thickness of the nanowires can be controlled by controlling the \( \text{H}_2\text{O}_2 \) concentration. With discontinuous Ag film structure, increasing the \( \text{H}_2\text{O}_2 \) concentration, the thickness decreases and the length of the nanowire increases. Thus, in this work, we demonstrate by optimizing Ag size and \( \text{H}_2\text{O}_2 \) concentration that we can control the Si etching from highly porous random structure to vertically aligned Si nanowires for various potential applications. We anticipate that this work might be useful for the researchers to fabricate desirable nanostructures for their potential applications.

**EXPERIMENTAL SECTION**

Si etching was done in two steps: Ag deposition and etching of Si in HF/\( \text{H}_2\text{O}_2 \).

**Ag Deposition.** Ag deposition was done in two different AgNO\(_3\) concentrations (0.0017 and 0.005 M). The N-type Si wafer of \( \langle 100 \rangle \) orientation with 1–10 \( \Omega \) cm was sequentially cleaned in acetone, 2-propanol, and deionized water (three times) and in boiling piranha solution (\( \text{H}_2\text{SO}_4/\text{H}_2\text{O}_2 \) in the volume ratio of 3:1) for 1 h (caution: extremely corrosive and highly exothermic reaction) and then rinsed thoroughly with deionized water and dipped into a 10% HF solution for 1 min. A cleaned Si wafer was immersed in the mixture of 4.8 M HF and 0.005 M AgNO\(_3\) solution for different time periods (30 s, 1 min) for Ag deposition.
min, 2 min, and 5 min) at room temperature. Ag nanoparticles were deposited on Si wafer surfaces. Then, the Si wafer was taken out of the solution rapidly and rinsed with deionized water.

**Etching in HF/H₂O₂.** Si substrate covered with Ag nanoparticles was immersed in the etching solution containing 5 M HF (0.05, 0.1, 0.3, and 0.6 M), H₂O₂, and H₂O in the volume ratio of 1:5:10 for 1 h at room temperature. Finally, the Si wafer was immersed in concentrated nitric acid for 1 h at room temperature to remove residual Ag nanoparticles from the surfaces.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.7b00584.

FESEM images of Ag deposited with 0.0017 M AgNO₃ and the respective etched samples in 4.8 M HF/0.3 M H₂O₂ solution; FESEM images of Ag deposited with 0.005 M AgNO₃ and the respective etched samples in 4.8 M HF/0.3 M H₂O₂ solution; top-view and 45° tilt-view FESEM images of the Si etched in 0.6 M H₂O₂/4.8 M HF solution of 5 min Ag-deposited Si (with 0.005 M AgNO₃) (PDF)

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**Notes**

The authors declare no competing financial interest.

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