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Authors: Zhuoyao Li, Guilin Wang, Chengyu Zhang, Cong Wei, Xiang Wang, Yongqian Gao, Hai LI, Xiao Huang, Haifeng Yuan, and Gang Lu

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Silver nanowire-templated molecular nanopatterning and nanoparticle assembly for surface enhanced Raman scattering

Zhuoyao Li,^[a] Guilin Wang,^[a] Chengyu Zhang,^[a] Cong Wei,^[a] Xiang Wang,^[a] Yongqian Gao,^[a] Hai Li,^[a] Xiao Huang,^[a] Haifeng Yuan,^{*,[b]} and Gang Lu^{*,[a]}

Abstract: Developing simple and cost-efficient methods for fabricating molecular patterns is of great importance in the field of nanoscience and nanotechnology. Here, a simple and convenient method was developed for fabricating nanopatterns composed of positively charged silane molecules by using silver nanowires as templates. The as-obtained silane pattern copies the shape of the silver nanowires and is only 0.7 nm thick, which can later be used for templated assembling of small molecules and nanoparticles of opposite charges. As a proof of concept, the resulted assembly could be further used for surface-enhanced Raman scattering.

With the development of new technologies, the fields of nanoscience and nanotechnology experienced enormous progress in past decades, which boosts the developments in fields of electronics, optics and biophysics, etc.^[1-3] In these fields, it is of great importance to fabricate nanostructures, for both fundamental study and practical applications.^[4, 5] Nanolithography involves the precise fabrication and interconnection of structures on the nanometre scale,^[6] using techniques such as photolithography^[7, 8] and e-beam lithography^[9-11] (often combined with etching). These techniques are highly programmable and straightforward, and have been used to realize many complex structures. However, they are limited by the low yield and high cost, especially when used for fabricating structures with a precision of several or several tens of nanometres. In past years, ultrathin molecular nanopatterns have attracted much attention. Many techniques, such as micro-contact printing (μ CP) and dip-pen nanolithography (DPN), have been employed to fabricate various nanopatterns. Through conformal contact, μ CP replicates a pattern of self-assembled monolayers (SAMs) of molecules^[12] from the pattern on polydimethylsiloxane (PDMS) stamp. This method is simple, versatile and cost-efficient. However, it is still technically challenging to fabricate very small patterns, even though patterns of several tens of nanometres have been successfully fabricated using μ CP.^[13-15] On the other hand, DPN employs an ink-coated atomic force microscope (AFM) tip as a

pen to write molecular patterns directly. This method is highly flexible, and various patterns can be written directly.^[16-19] However, the throughput is usually low and the cost is high. Therefore, it is still necessary to develop simple, flexible and cost-efficient methods for molecular patterning.

One-dimensional nanostructure, such as nanowires, is an important member in the family of nanomaterials and has been widely used in research fields of nano-optics, molecular sensing, biology, etc.^[20-24] Silver nanowires (AgNWs) are one of the mostly studied nanowires due to its promising mechanical, optical and electronic properties. Until now, AgNWs have been used as plasmonic waveguides,^[25] surface-enhanced Raman scattering (SERS) platforms,^[4] single-cell endoscopy,^[26] tip-enhanced spectroscopy probe,^[27] etc. The polyol synthesized AgNWs possess the well-defined five-fold twin structure and pentagon-shaped cross section, and its physicochemical properties have been fully characterized by several research groups.^[28-30] Moreover, the length and diameter of the AgNWs can be easily tuned by varying the parameters during synthesis. Due to the easy preparation and convenient tuning of the AgNW morphology, it could also be used in many other applications.^[31, 32]

Herein, we used AgNWs as templates to prepare molecular patterns on solid substrate. The as-prepared pattern could be used as a platform for many other applications. We demonstrated its applications in fluorescence imaging and SERS sensing. This work provides a simple and convenient approach to prepare nanoscale molecular patterns for various applications.

The procedure is schemed in Figure 1. Briefly, the polyol synthesized AgNWs are firstly deposited on a 3-aminopropyl triethoxysilane (APS) modified glass slide (Figure 1b). Afterwards, this AgNWs-coated substrate is immersed in a solution of sodium hydroxide (NaOH) to remove the APS molecules those are not masked by the AgNWs (Figure 1c). After etching away the AgNWs, we successfully obtain the patterns of APS molecules, which replicate the shape of the AgNWs (Figure 1d). Note that the APS patterns are prepared all in ambient conditions with wet-chemical processes, indicating that our method is convenient and cost efficient.

Figure 1. Schematic plots of the AgNWs-templated molecular patterning process. The glass substrate was first grafted with APS molecules (a), and then AgNWs, whose cross sections were pentagon shaped, were adsorbed on the APS modified surface (b). Afterwards, the AgNWs-coated glass substrate was

- [a] Zhuoyao Li, Guilin Wang, Cong Wei, Prof. Yongqian Gao, Prof. Hai Li, Prof. Xiao Huang, Prof. Gang Lu
Key Laboratory of Flexible Electronics (KLOFE) and Institute of Advanced Materials (IAM)
Jiangsu National Synergistic Innovation Center for Advanced Materials (SICAM)
Nanjing Tech University (NanjingTech)
30 South Puzhu Road, Nanjing 211816, China
E-mail: iamglv@njtech.edu.cn
- [b] Dr. Haifeng Yuan
Departement Chemie
KU Leuven
Celestijnenlaan 200F, 3001 Heverlee, Belgium
E-mail: haifeng.yuan@kuleuven.be

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immersed in NaOH solution to remove the APS molecules (c), and molecular pattern of APS molecules was formed after etching away the AgNWs (d). Note that the schemes were not drawn to scale.

The pattern fabrication process was discussed in detail in Figure 2. First, the polyol synthesized AgNWs with a diameter of 100 ± 20 nm were drop-casted or sprayed onto an APS-modified glass slide, which serve as templates in subsequent patterning process. As is shown in a scanning electron microscopy (SEM) image (Figure 2a), the deposited AgNWs were uniform in diameter and randomly distributed on the glass slide. The height of a typical AgNW was measured as 100 nm (Figure 2b) with AFM, revealing the diameter of the AgNW, which is consistent with the results from SEM measurements. It was found that the binding strength of the AgNWs on glass substrate had a crucial effect on the subsequent patterning process. If the binding was too weak, the AgNWs can be washed away in the subsequent steps, or cannot work as efficient masks during the removal of the APS molecules (Figure 1c). Therefore, the AgNWs coated glass substrates were usually annealed on a hot plate at 60 – 100 °C for more than 15 minutes to increase the binding force between the AgNWs and glass substrate, which is possibly due to the capillary force during the evaporation of solvent that residences in the AgNW-glass gap. Without annealing, the molecular pattern of APS can hardly be prepared. Thereafter, in aqueous solution of NaOH, the APS molecules were removed and hydroxyl groups were grafted on the glass surface instead, leaving only the parts underneath the AgNWs unchanged. Etching away the AgNWs in an aqua regia solution, line patterns of APS molecules were exposed (Figure 2c), resembling the shape of the deposited AgNWs. The quality of the as-obtained APS pattern is highly related to the immersion time in NaOH solution. The APS molecules cannot be effectively removed within half an hour. Increasing the immersion time to one hour, the APS molecules were effectively removed except for the parts masked by the AgNWs, generating the APS line patterns. However, the boundaries of the line patterns appeared very rough (Figure S1 in ESI). This is probably due to the less hydrophilicity of the APS-coated region, where air bubbles may exist at the AgNW-glass boundary at the beginning of APS removal, hindering the APS removal process. Extending the etching time to two hours, the edges of the line patterns became sharp and smooth (Figure 2c&e), perfectly resembling the shape of the AgNWs. The APS patterns showed negligible change when the immersion time was further extended, indicating that the AgNWs could provide perfect protection for the underneath APS molecules. Note that the surface without AgNWs templates is very smooth (RMS = 0.38 nm) after immersion (Figure 2d), indicating the smooth feature of the glass surface, even after removal of the APS molecules on surface. From the AFM image of a typical APS line pattern (Figure 2e), we estimate that the molecular pattern has a height of ~ 0.7 nm and a width of 86 ± 5 nm. As is known, the grafted APS molecules are silanes terminated with 3-aminopropyl groups, which are positively charged and have a length of roughly 0.8 nm. Therefore, the 0.7 nm height indicates that the APS pattern should be composed of monolayer or sub-monolayer APS molecules, which double confirms the perfect masking of the AgNWs during the APS removal. The width of the APS pattern is slightly smaller

than the diameter of the single AgNW. It has been reported that the polyol synthesized AgNWs possess five-fold twin structures and their cross sections are pentagon shaped.^[28] When the AgNWs were positioned on a flat surface, only one side of the pentagon contacted with the surface (Figure 1b), which serves as a mask in the APS removal step. Note that the width of the contact region is equal to the edge length of the pentagon, which is slightly smaller than the diameter of the AgNW, explaining the observed smaller width of the APS line pattern. It needs to be pointed out that the diameter of the AgNWs can be easily tuned in a range of several tens to hundreds of nanometres and the AgNWs can assemble to form various complex patterns.^[33–35] This makes it possible to prepare variously sized (from several tens to hundreds nanometres) and shaped APS patterns by using this simple method.

Figure 2. Evolution of the patterns on a glass slide. (a) SEM image showing that AgNWs were adsorbed on glass slide surface. (b) AFM image showing the morphology of the deposited AgNWs. (c) AFM image of the APS patterns that were copied from the AgNWs. (d) AFM image of the region without AgNWs template. (e) Zoomed in AFM image of a line pattern of APS molecules with a width of 86 ± 5 nm and a height of 0.7 nm that was copied from single AgNW.

The APS patterns are composed of monolayer or sub-monolayer silane molecules terminated with the positively charged amino groups. Thus, negatively charged molecules and nanoparticles can be adsorbed onto it via electrostatic interaction. Figure 3a shows that the negatively charged fluorescein molecules were successfully adsorbed on the positively charged APS patterns, generating fluorescent nanowires on the glass substrate, which emit yellow-green light (Figure 3a&b). The shape of the fluorescent patterns is determined by the APS pattern, so that the width of the fluorescent pattern can also be controlled by tuning the diameter of the AgNWs templates, although the fluorescent lines look wider due to the limited resolution of fluorescence microscope. Note that the fluorescence along single nanowire is uniform in strength (Figure 3a), indicating a uniform adsorption of the fluorescein molecules, probably monolayer or sub-monolayer, on the APS pattern. Besides physical adsorption, the APS molecules may also chemically react with other

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functional molecules (such as glutaraldehyde),^[36] which could be used for chemical and bio-sensing.

Besides small molecules, nanoparticles can also be adsorbed on the as-prepared APS patterns. Due to the citrate ions bounded on surface, the citrate synthesized gold or silver nanoparticles are negatively charged thus can also be selectively adsorbed on the as-prepared APS patterns.^[37] As is shown in Figure 3c-d, gold nanoparticles (AuNPs, 13±2 nm in diameter) were precisely adsorbed on the as-prepared APS patterns, leaving the APS uncoated region blank. We found that the ion strength in the AuNPs colloid was crucial for the successful AuNPs adsorption, which is consistent with the reported work.^[38] When the as-synthesized AuNPs colloid was used directly (No salt was added), only low-density AuNPs were adsorbed on the APS patterns. If sodium chloride (NaCl) was added to AuNPs colloid to get a final concentration of 10 mM, high-density AuNPs were assembled on the APS patterns, and the APS uncoated region was free of AuNPs (Figure 3c-d). Further increasing the NaCl concentration to 20 mM, higher density of AuNPs were adsorbed on the APS pattern, however, the APS uncoated region was also covered with non-specifically adsorbed AuNPs (Figure S2 in ESI). Except for the 13 nm ones, larger AuNPs could also be adsorbed on the APS line pattern, however with a poor quality, which is consistent with a reported work.^[37] These AuNPs patterns provide a tunable plasmonic structure, which has potential applications in the fields of SERS sensing and nanophotonics.

Figure 3. (a) Fluorescent image of the patterned fluorescein molecules. (b) Typical emission spectrum of the fluorescein molecules. (c) SEM image of the AuNPs assembled on the as-prepared APS pattern. (d) Zoom in image of the rectangular area shown in (c).

It has been reported that AgNWs can be orderly deposited on solid substrate by using the grazing incidence spraying method (Scheme shown as Figure S3 in ESI).^[39-41] Using these ordered AgNWs (Figure S4a in ESI) as templates, it is possible to prepare aligned APS line patterns, which could also be used for assembly of small molecules and nanoparticles, generating corresponding aligned line patterns (Optical image of the assembled AuNPs was shown as Figure S4b in ESI). In addition, more complex molecular

patterns could also be prepared by controlling the assembly of AgNWs.

The assembled nanoparticle patterns could be used for SERS detection. As known, SERS has been widely used in fields of chemistry, physics, biomedicine and biology,^[42, 43] thanks to the huge Raman enhancement. Various SERS substrates have been prepared for detection of molecules,^[44] however, it is still necessary to fabricate patterned SERS substrates, especially nanopatterns, for multi-functional detections.^[45-47] As discussed, the aligned APS line patterns can be used as templates for assembly of nanoparticles. As a proof of concept, 13 nm AuNPs were selectively adsorbed on the ordered APS pattern, forming aligned line patterns of AuNPs (Figure 4a). It can be observed that all the AuNPs lines oriented to a given direction, although with a certain degree of deviation, which originates from the AgNWs templates (Figure S4a in ESI). We found that the as-obtained AuNPs pattern could serve as an efficient SERS platform. After being functionalized with Raman probe molecules of *p*-aminothiophenol (PATP) or methylene blue (MB), SERS property of the substrates were investigated. We observed SERS-active line patterns (Figure 4b) with identical SERS spectra across the line patterns. These uniform SERS intensities across the line pattern indicated uniform adsorptions of the AuNPs and uniform Raman enhancements. The Raman enhancement could be further increased by extending the immersion period in AuNPs colloid or re-adsorbing another layer of nanoparticles. The average enhancement factor (EF) was calculated (The details of the EF calculation was already described in our previous report^[48]) as 2.2×10^5 for the Raman band of PATP (typical spectrum shown in Figure 4c) at 1076 cm^{-1} , by using this equation $EF = (I_{\text{SERS}}/I_{\text{bulk}}) \cdot (N_{\text{bulk}}/N_{\text{SERS}})$.^[31] This EF is slightly smaller than the reported one, which is probably due to the limited width of the line pattern. Moreover, our SERS substrate was stable after being immersed in aqueous solutions at various pH value (Figure S6a). Furthermore, the thermal stability of the substrate was quite high, and no SERS intensity drop could be observed after being heated at 160°C for one hour (Figure S6b).

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Figure 4. Typical optical image of the APS line patterns adsorbed with AuNPs (a) and SERS mapping of the pattern (b) after functionalized with Raman probe molecules (band intensity in 1500–1650 cm^{-1} was used for Raman mapping). (c) A typical SERS spectrum of the PATP molecules on the assembled AuNP pattern.

In conclusion, a simple and convenient method was developed for nano-patterning of silane molecules on glass substrate by using AgNWs as templates. The APS patterns closely resemble the shape of the AgNW templates. Moreover, as a proof of concept, the as-prepared APS patterns can be used to guide the assembly of small molecules and nanoparticles for various applications, including SERS. We believe that more complex molecular patterns can also be fabricated by preparing complex AgNWs templates, which will extend our method practically useful in many fields.

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Entry for the Table of Contents (Please choose one layout)

Layout 1:

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Nanopatterns of silane molecules were fabricated by using silver nanowires as templates, and the obtained molecular patterns could be used for assembly of small molecules and nanoparticles, which could be used in many applications, including surface-enhanced Raman scattering.

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Yongqian Gao, Hai Li, Xiao Huang,
Haifeng Yuan,* and Gang Lu*

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