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DNA-templated photo-induced silver nanowires: Fabrication and use in detection of relative humidity

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ABSTRACT

A very simple and novel approach of fabricating Ag–DNA network is herein reported. The Ag–DNA network can be formed from reduction of silver ion absorbed on DNA template by sunlight. Mesh size of the Ag–DNA network and the diameter of the Ag–DNA nanowire can be controlled by adjusting the concentration of DNA and irradiation time, respectively. Furthermore, the Ag–DNA network placed onto comb-like gold electrodes can be utilized as a sensor for humidity, which presents a good response to the detection of relative humidity (RH).

1. Introduction

As a natural template for nanofabrication [1], DNA ranging from nanometers to microns, can be realized with available technology in molecular biology such as DNA ligation, enzymatic digestion, and polymerase chain reaction. DNA-templated nanowires can be prepared with an almost unlimited range of aspect ratio. A DNA molecule has two binding sites, *i.e.*, negatively charged phosphate groups and aromatic bases. The polyanionic backbone of DNA, which is composed of alternating pentose and phosphate groups, binds to metal cation and nanoparticles by electrostatic interaction. Coordination between transition metal ions and nitrogen atoms of the DNA bases *via* d–p orbital bonding results in formation of metal–DNA complexes. DNA has been assembled into quite sophisticated and predesigned networks due to its excellent recognition ability. [2,3] Hence, a "smart glue" has also been implemented for guiding the self-assembly of nanostructures into functional circuits [4,5].

DNA has been exploited as a template for the fabrication of metal nanowires, *e.g.*, silver [6–11], palladium [12–14], platinum [15–18], gold [19–22], copper [23,24] and cobalt [25]. In general, the fabrication approach consists of two steps, that is, metallic clusters are first formed on the DNA, and then used as nucleation sites for selective metal deposition until a continuous wire is formed. The formation of metal nucleation centers relies on binding of metal ions or complexes to the DNA and their subsequent reduction to form metallic clusters, or binding of small metal particles to the DNA. Different metal wires can be deposited on DNA templates *via* the reduction of metal ions or metal complexes associated with DNA.

Current protocols for nonspecific silver deposition of DNA strands involving either photoreduction (254 nm) of Ag(I) ions complexed to DNA [26] or chemical reduction of Ag(I) ions by glutaraldehyde-modified DNA [6–11], should provide uniformly metallic DNA. Herein, a much simpler methodology, *i.e.*, taking advantage of DNA's great affinity for Ag(I) ions and performing *in situ* photoreduction of DNA-complexed silver ions to synthesize silver nanoparticles by sunlight, is being reported.

Humidity sensors are widely used in many measurements and control applications, including process control, meteorology, agriculture and medical equipment. At present, most humidity sensors, designed to detect humidity through changes of electrical properties, such as electrical resistance, use electrolytes [27], metal oxides, organic polymers [28] and porous semiconductors [29] as sensitive materials. Herein, we fabricate Ag nanowires with DNA template by the electroless plating method. Then, the Ag–DNA nanowires are deposited on the gold electrodes to measure the humidity.

There are several advantages in the present work, *i.e.*, firstly, method of synthesizing metallic DNA is remarkably simple and the Ag^+–DNA complexes are reduced only using sunlight-switchable reducing agents; Secondly, formation of Ag–DNA network is quite facile *via* a 3 μL drop of Ag–DNA solution placing onto the surface of comb-like gold electrodes; furthermore, two-dimensional Ag–DNA networks can be easily immobilized onto Au-electrode by self-gravity, avoiding that stretch and immobilization of DNA template molecule; finally, the sensor of Ag–DNA networks can work both in gas medium and solution. In addition, all of the experiments were replicated at least three times. We find that the reproducibility of the results is very well in all the study. Details of the experimental process will be demonstrated below.

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2. Experimental section

2.1. Reagents and apparatus

λ-DNA was purchased from Sangon Bioengineering Ltd. Company (Shanghai, China). $AgNO_3$ was A.R. grade and obtained from Shanghai reagent Co. without further purification. Millipore 18.2 MΩ/cm water was used for all solutions and obtained from a Millipore Simplicity 185 system.

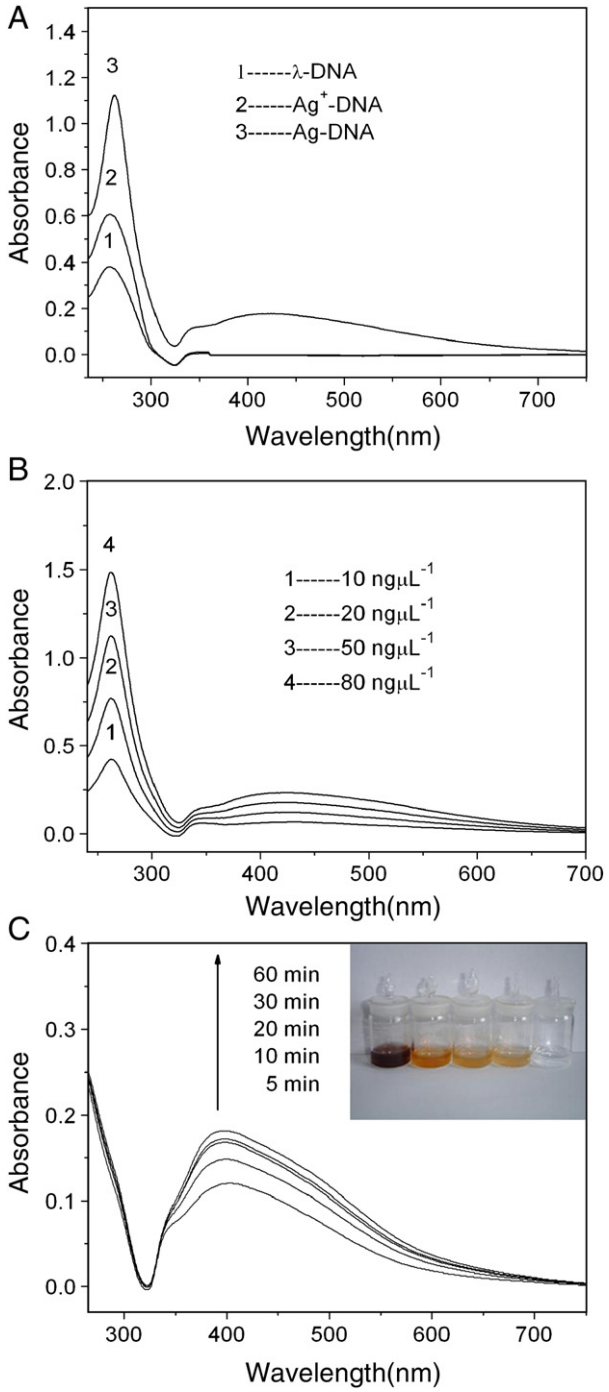

Fig. 1. (A) UV–vis spectra showing the photoreduction sequence for Ag^+–DNA complex. Pure λ-DNA (curve 1), Ag^+–DNA complex (curve 2), Ag–DNA synthesized by irradiation of Ag^+–DNA solution (curve 3). DNA: 50 ngμL^{-1}, $AgNO_3$: 1 mM. irradiation time: 30 min. (B) UV–vis spectra showing the effect of different concentration of DNA in silver complex. Curve 1–curve 4 correspond to 10 ngμL^{-1}, 20 ngμL^{-1}, 50 ngμL^{-1}, and 80 ngμL^{-1}, respectively ($AgNO_3$: 1 mM). (C) UV–vis spectra recorded for an aqueous mixture of Ag^+–DNA complex at different times of irradiation. Inset: the color of the solution of Ag–DNA at different time (Ag^+: 1 mM, DNA: 50 ngμL^{-1}) (from right to left, the color of reaction solution is deepened from colorless to dark at different stages of irradiation).

2.2. Synthesis of silver nanowires in DNA

An aqueous solution of $AgNO_3$ (100 μL, 1 mM) was added to a solution of λ-DNA (20 μL, 300 ngμL^{-1}). The diluted DNA solution is 50 ngμL^{-1}, and the solution was mixed thoroughly and kept for 3 h at room temperature. Then photoreduction performed directly by exposure to the sunlight. The color of the solution changed from colorless to yellow, and to gray gradually, indicating the formation of silver nanoparticles. The exposing time was controlled from several minutes to several hours. The samples for electrical measurement were prepared by depositing a drop (3 μL) of the Ag–DNA solution onto the surface of comb-like gold electrodes.

The processes for the corresponding controllable experiments under different concentration of DNA, were similar to the experiments described above.

2.3. UV–vis spectroscopy studies

UV–vis spectroscopy measurements of the samples were carried out on a TU-1901 model UV–vis double beam spectrophotometer (Beijing Purkinje General Instrument Co., Ltd, China) operated at a resolution of 2 nm.

2.4. X-ray photoemission spectroscopy (XPS) measurements

XPS measurements of a film of silver–DNA complexes were carried out on a VG ESCALAB MKII instrument at a pressure lower than 10^{-6} Pa. The general scan and C 1s, Ag 3d, P 2p, and N 1s core-level spectra were recorded with un-monochromatized Mg K_α radiation (photon energy = 1253.6 eV). The core level binding energies (BEs) were assigned with respect to the C 1s binding energy (BE) of 285 eV.

2.5. Atomic force microscopy (AFM) measurements

AFM measurements were performed with a digital Nanoscope IIIa multimode system (DI, Santa Barbara, CA). The image was acquired in the tapping mode. The AFM measurements were made in air at room temperature with the Si cantilever. The force constant of the cantilever was 0.1–0.6 N/m with the scan rate of 1–2 Hz.

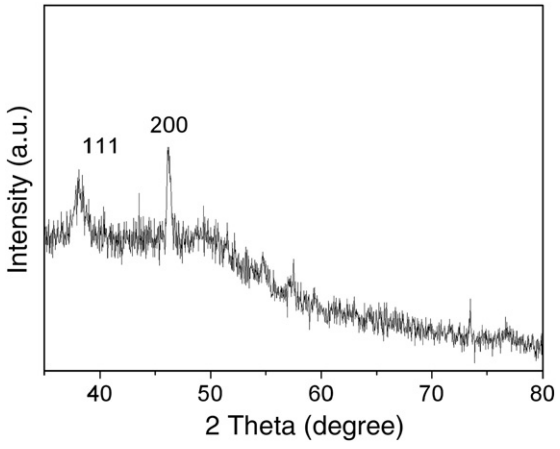

Fig. 2. XRD pattern recorded from drop-coated films on a silica substrate of the Ag–DNA synthesized by irradiation of the Ag^+–DNA solution.

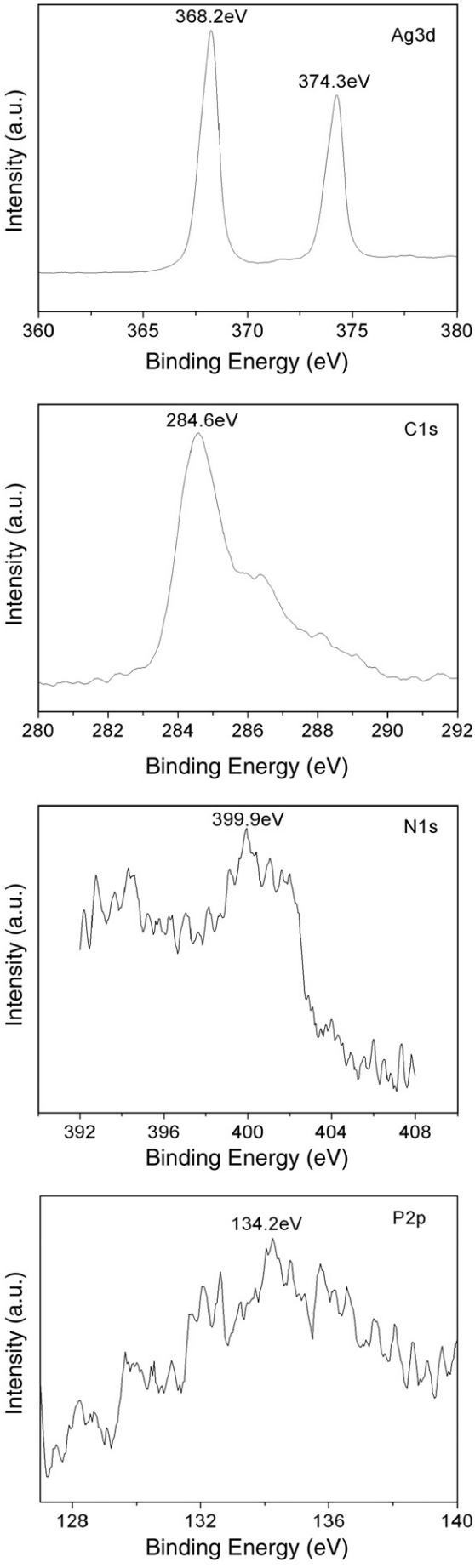

Fig. 3. XPS spectra of Ag 3d, C 1s, N 1s and P 2p core-level recorded from Ag–DNA sample.

2.6. Scan electron microscopy (SEM) measurements

SEM measurements were carried out with a Leica Stereoscan-440 instrument equipped with a Phoenix energy dispersive analysis of X-rays (EDAX) attachment.

All of the measurements (AFM/SEM) were replicated at least three times.

2.7. X-ray diffraction (XRD) measurements

XRD analysis of drop-coated films of the Ag–DNA solution on silicon substrates was carried out on a MAP18AHF instrument (Japan MAC Science Co.).

2.8. Electrical measurements

Two-terminal two-probe current measurements were taken using a picoammeter/voltage source (Keithley 6487) in the range 0–500 V, the dc current sensitivity of the system is in the range 10^{-14} A. Timed data acquisition was performed using a LabView® program and the current sampling frequency was set at two samples per second. Bias voltage was set at $V = 0.1$ V in all of the experiments. The electrical measurements were repeated many times until the signal is stable.

3. Results and discussion

Referring that DNA can form stable complexes with Ag(I) by embedding the ions inside the double helix [30], Lorenzo et al. [26] reasoned that it would be possible to grow chains of silver nanoparticles through photoreduction of silver-laden DNA. Also, they believed that DNA as such would act as photosensitizer due to short-wavelength UV irradiation causing photooxidation of DNA bases [31] which is similar to the present case, *i.e.*, ultraviolet radiation in the sunlight. To test our hypothesis, we formed Ag^+–DNA complex and attempted silver reduction by irradiating at different wavelength radiations such as, 366 nm, visible light and infrared ray, respectively. However, the formation of silver nanoparticles could not be detected under these conditions because of the absence of the characteristic plasmon resonance peak. Only exposing the Ag^+–DNA complex to 254 nm UV light resulted in the appearance of a peak centered at *ca.* 410 nm, which can be attributed to the surface plasmon resonance of small silver nanoparticles [32] As shown in Fig. 1A, the DNA absorption peak appeared at 260 nm concomitantly. Compared to curve 2 in Fig. 1A, the intensity of the DNA absorption peak in curve 3 is increased. Because of the intimate nature of the Ag^+–DNA complex, this effect could be attributable to decomplexation of the bases and to changes in the duplex structure during the irradiation process. It should be noted that the absorption band in Fig. 1B is changed sharply and the resonance intensity increased. All of these observations might result from the increasing concentration of DNA. In addition, UV–vis spectra of the Ag^+–DNA complex at different stages of irradiation are obtained, suggesting that the absorption peaks of the silver nanoparticles increases after silver photoreduction occurs and the color of the reaction solution is gradually deepened from colorless to dark (see the inset in Fig. 1C) [33]. The photo-reduction process is considered complete when the extension of the irradiation time does not cause any changes in the Plasmon resonance peak intensity or shape [26].

In addition, irradiating an aqueous silver nitrate solution without DNA under the same conditions does not result in formation of silver nanoparticles (data not shown), indicating that DNA plays an active role in the reduction process. DNA bases, as photosensitizers in UV

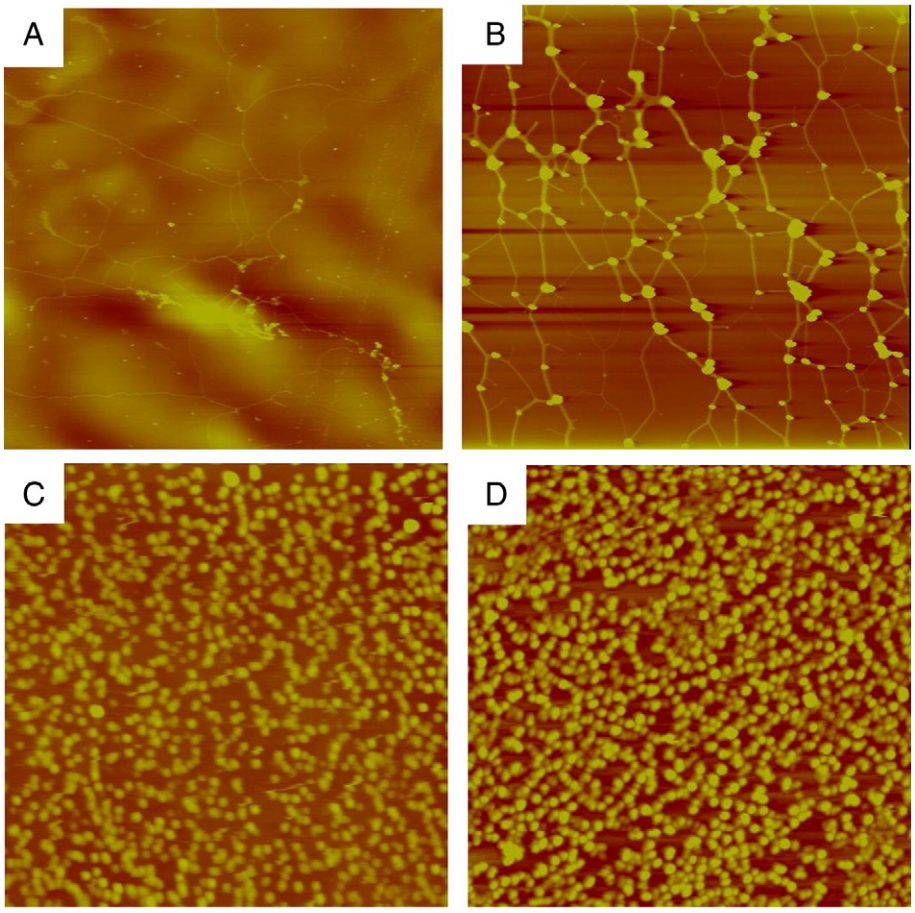

Fig. 4. AFM images of DNA network (50 ng μL^{-1}) (A) and synthesized nanoparticles on DNA network (50 ng μL^{-1}) for different irradiation times: 10 min (B), 30 min(C), and 60 min (D). The *z*-scale is 20 nm for all images. Scan size for all images: 5 μm × 5 μm.

radiation, undertakes photooxidation to form very reactive radical intermediates or generate singlet oxygen contributing to possible reduction of the complexed silver ions [26].

As shown in Fig. 2, the (111) and (200) Bragg reflections of face-centered cubic (fcc) silver are clearly observed, which are good agreement with previously reported data [34]. In contrast to metallization of DNA from cluster deposition in the previous work [6–11], metallic DNA could be easily fabricated *via* activating the DNA with silver ions in the present experiment. The positively charged silver ions can be associated with the negatively charged DNA phosphate groups during the activation. After the activation time (3 h) elapsed, the sunlight is used to reduce the silver ions binding to the DNA double strands. The background of XRD pattern is most likely caused by Brownian motion of the particles during the scan, solvent scattering, or the short-range order of the solvation cage around the particles.

As shown in Fig. 3, the Ag 3d spectrum from the XPS experiment of metallic DNA networks consists of two components corresponding to 368.2 and 374.3 eV, which are consistent with the binding energy of Ag 3d5/2 core level [35]. A single component at 284.6 eV can be assigned to C 1s spectrum of the carbons in the DNA sugars and bases, The signals at 399.9 eV and 134.2 eV can be assigned as the N 1s and P 2p spectrum, respectively, due to DNA containing nitrogen atoms and phosphate backbone [19]. Moreover, the signals of the P 2p and N 1s are not very strong, indicating that the backbone of template DNA molecule is mainly coated by silver nanoparticles grown along the DNA networks of the DNA template.

Direct evidence for the formation of network assemblies of silver nanoparticles driven by the DNA template is acquired from the AFM measurements. As shown in Fig. 4A, the as-prepared DNA network has been formed in an ordered manner and the apparent height of λ-DNA is 0.7–1.3 nm. The immobilization of the DNA strands on the substrate is realized by the extremely stable DNA networks, even if they were repeatedly scanned many times. The AFM images of silver nanoparticles on DNA network at the different irradiation time have been obtained in order to examine the effect of reduction time on the growth of silver nanoparticles. As observed from Fig. 4B, uniform and monodisperse silver nanoparticles (6.6–8.6 nm) can grow along the DNA strands at the irradiation time of 10 min. Subsequently, when the

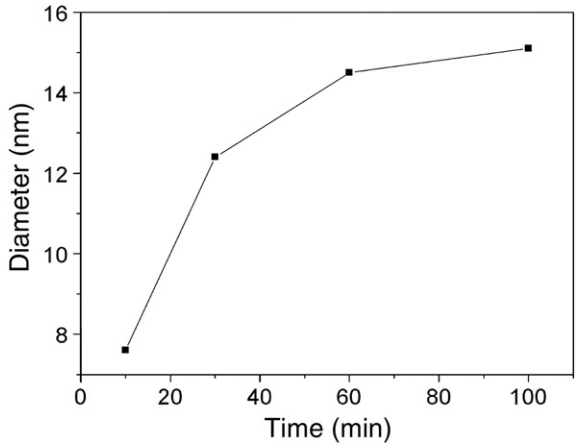

Fig. 5. Relative plot of the diameter of synthesized nanoparticles versus different irradiation time.

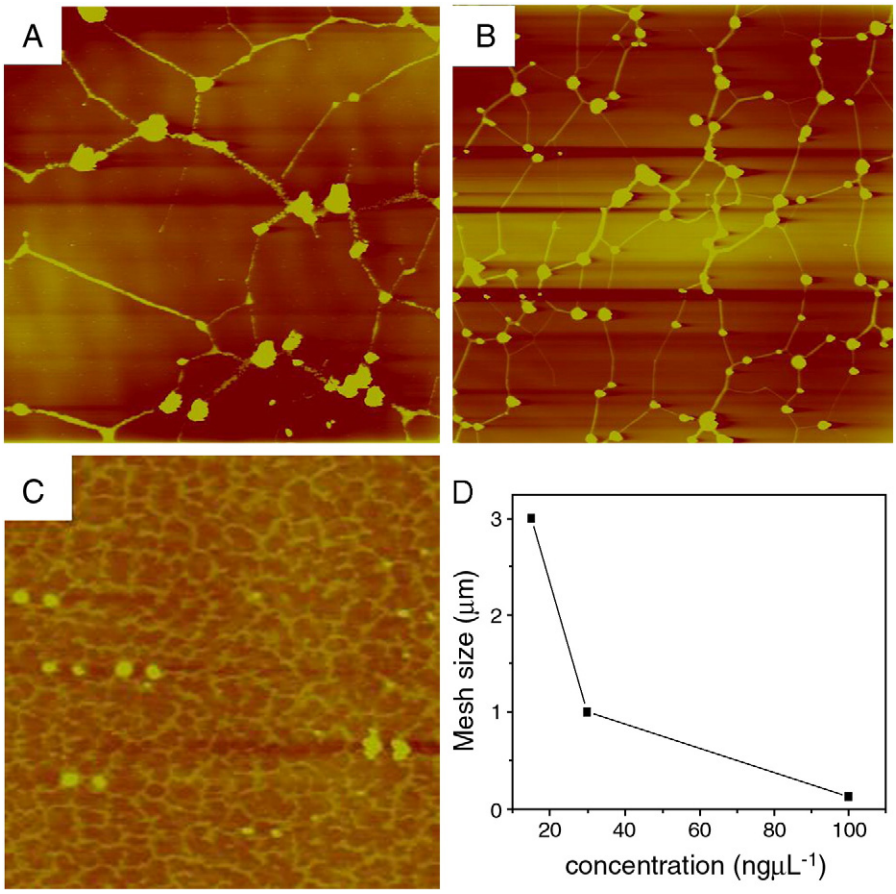

Fig. 6. AFM images of the silver nanoparticles on DNA networks with different concentration (A) 30 ngμL^{-1}, 10 min, (B) 50 ngμL^{-1}, 10 min, (C) 100 ngμL^{-1}, 10 min, and (D) relative plot of the mesh of networks versus different concentration of DNA. The *z*-scale is 20 nm for images of A, B, C. Scan size for images of A, B: 8 μm × 8 μm and C: 4 μm × 4 μm.

irradiation time is increased to 30 min, the silver nanoparticles with the mean diameters at *ca.* 11.4–13.4 nm are totally formed on the DNA strands instead of forming in the DNA mesh, suggesting that the strong electrostatic interaction between silver ions and phosphate backbone of DNA strands can decrease the mobility of silver ions and accelerate the nucleation of silver nanoparticles on DNA strands. Furthermore, when the treatment time is beyond 60 min (see Fig. 4D), larger nanoparticles are shown on the AFM image and their mean diameter of particles are estimated to be 13.3–15.7 nm according to statistical analysis, indicating that the unstable silver nanoparticles can move along the DNA strand.

Growth mechanism of the silver nanoparticles may be given on the basis of the present results. Initially, silver ions are equably absorbed onto DNA strands accompanying with immersion of $AgNO_3$ solution into the porous DNA nano-network. Furthermore, when silver ions in the DNA network are reduced by the sunlight, the newly formed silver particles could move along the DNA strands to the nearest spot of nucleation so as to increase silver nanoparticle core within short time, which was seen as the growth step. As reported by Li et al. [36], the formation of uniform crystalline particles arose from a template-directed aggregation of small particles and subsequent recrystallization rather than simple aggregation of small particles. This main principle is similar to the explanation for the preparation of noble metal nanoparticles on nanoporous TiO_2 films. This explanation considered that the size of nanoparticles could increase with the reduction time [37]. That is to say, with prolonging irradiation time, some silver monomers can easily move along the DNA strands and be adsorbed onto the deposited seeds, and these aggregated seeds can form nanowire (see Fig. 4C, D) by subsequent recrystallization. We think the growth mode in our work is the same as the seed-mediated growth mechanism [36], furthermore, it should also be adsorption and diffusion-controlled, as reported by Hu et al. [38].

As shown in Fig. 5, the diameter of silver nanoparticles increased continuously with the prolongation of reduction time; however, the growth rate decreases gradually until 60 min. Furthermore, the diameter of silver nanoparticles increased slightly after 60 min.

As seen from Fig. 6A–C, silver nanoparticles are highly orientedly assembled by DNA network temples and appear much dense along the fringe of DNA network on mica substrates. Additionally, one notes

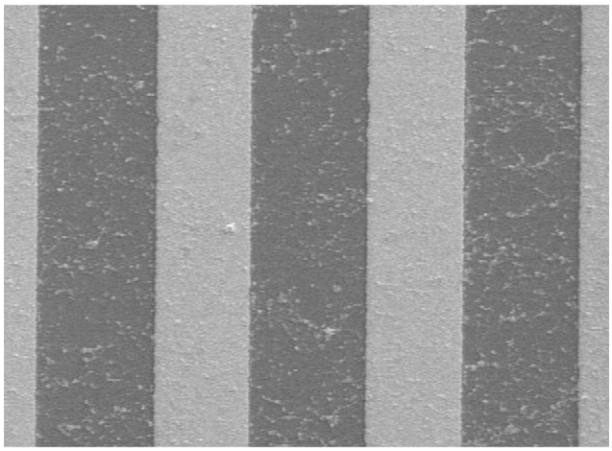

Fig. 7. SEM image of Ag–DNA network placed onto comb-like gold electrodes on a silicon (400 nm oxide) substrate. The bar scale is 8 μm.

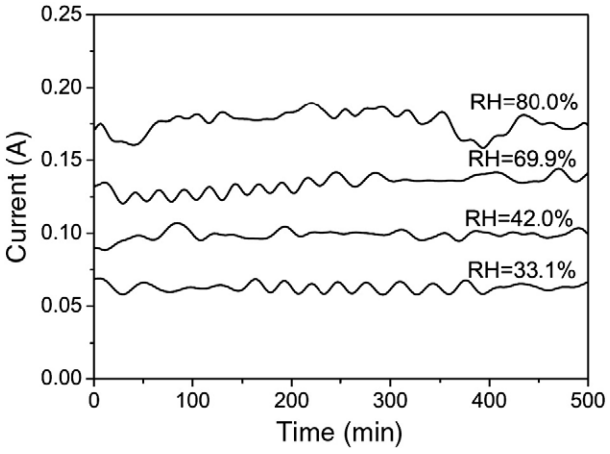

Fig. 8. Characteristics of the humidity sensor at various RH at $V = 0.1$ V.

that the mesh becomes smaller (shown in Fig. 6A–C) and the pore on the Ag–DNA shrank from approximately 3.1 μm, 1.1 μm to 130 nm (Fig. 4D) according to statistical analysis, *i.e.*, the dense of the Ag–DNA networks can be controlled by changing the concentration of the DNA.

To test the electrical transport capabilities of the Ag–DNA networks, electrically contacted networks of wires were fabricated by dropping Ag–DNA solutions onto 15 nm thick comb-like gold electrodes which were setup on silicon (400 nm oxide) substrate. Fig. 7 shows that the Ag–DNA networks are homogeneously covered on electrodes. From the SEM image it is showed that the DNA nanowires are not continuous, which is because of poor conductivity of DNA and some parts of the DNA strands are not metalized. It is also clear from this image that the metallization was restricted almost entirely to the DNA [6]. Also, it should be pointed out that the SEM image is compatible with the images of AFM.

Fig. 8 shows the characteristics of the humidity sensor at various RH. The humidity sensing is based on a water-adsorpting process. At first, water vapour is chemisorbed on the surface of the Ag–DNA nanowires, and then hydroxyl can be formed at the surface [39–42]. In this situation, proton transfer occurs among the hydronium (H_3O^+). The electrical response depends on the number of water molecules adsorbed on the surface. As we know, the more the water vapour adsorbed is, the better the electrical response is. Usually, there is one monolayer of water of covering at around 20% relative humidity, and more layers at even higher humidity [43]. From the Fig. 8, it can be seen that the current of the sensor increases with increasing relative humidity from 33.1% to 80.0%. The current variations with time for the sample are very little. The measurements are repeated at 25 °C for 500 min. Slight variation in current is observed over time. The current variations have a maximum of 3.1% at different RH. All of these demonstrate that the Ag–DNA nanowires are relatively stable to the exposure to water in air. For the detailed parameters and deep understanding of the mechanism, further investigation is ongoing.

4. Conclusions

We have presented a very simple method for fabrication of metallic DNA network, which is a controllable one-step, photo-induced synthesis of silver nanoparticles on the DNA network. In the synthesis process, the mesh size of Ag–DNA network and the diameter of the Ag–DNA nanowires are mainly determined by the concentration of the DNA and reduction time of silver cations, respectively. The mesh size of the Ag–DNA network shrinks with the increase of the concentration of DNA and the diameter of the Ag–DNA nanowires is enlarged with the prolongation of reduction time. Furthermore, the Ag–DNA network exhibits an excellent current response for the detection of relative humidity.

Acknowledgement

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