Supporting Information

Controlled electrochemical deposition of metal nanostructures on DNA origami templates

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Materials and Methods

Materials and instruments

M13mp18 DNA (7249 nucleotides) was purchased from BIORULER (Jiangsu, China). All short DNA strands were synthesized by Sangon (Shanghai, China) with ULTRAPAGE purified (sequences listed in Supplementary information). Reagents were purchased from Sinopharm (Shanghai, China) and Sigma-Aldrich (Shanghai, China) and used without further purification. All aqueous solutions were prepared with Milli-Q water (resistivity = 18.2 MΩ cm). Silicon wafer (single side polished with a thickness of 500 ± 10 μm, N-type, <111>) was purchased from Nanjing Jincaixin Technology Co., LTD (Nanjing, China). The samples were prepared by CHI660D electrochemical workstation (Shanghai, China). AFM imaging was performed using Bruker multimode VIII atomic force microscopy (Veeco Inc., USA). SEM measurements were obtained by HITACHI S-4800 scanning electron microscope (Tokyo, Japan).

Preparation of substrate

Firstly, the purchased silicon wafers were cut into 1 cm × 1 cm sizes. Next, the silicon wafers were soaked in ethanol and ultrasonically cleaned for 10 minutes, followed by soaking in ultrapure water for another 10 minutes of ultrasonic cleaning. After drying, the wafers were immersed in a piranha solution (concentrated H_2SO_4 and H_2O_2 volume ratio of 7:3) for 30 minutes. Subsequently, the piranha solution was washed off the surface of the wafers with ultrapure water, then immersed and ultrasonically cleaned with ethanol and ultrapure water for 10 minutes, respectively. Finally, the cleaned wafers were soaked in ultrapure water for later use.

Preparation of DNA origami template

The M13mp18 DNA scaffold, original staple strands, and extended staple strands were mixed at a molar ratio of 1:10:10 in 1× TAE-Mg^{2+} (40 mM Tris, 20 mM acetic acid, 2 mM EDTA, 12.5 mM magnesium acetate, pH 8.0) buffer. The mixture was annealed from 95 ℃ to 25 ℃ at a rate of 1 ℃/min. Subsequently, the assembled DNA origami was transferred to a 100 kDa (MWCO) centrifuge filters, and 1× TA-Mg^{2+} (40 mM Tris, 20 mM acetic acid, 12.5 mM magnesium acetate, pH 8.0) buffer was added. The sample was centrifuged three times for filtration to remove the extra staple strands. The concentration of the purified DNA origami was measured by UV-vis spectroscopy.

Electrochemical deposition process of metals

The DNA origami template and metal salts were mixed in 1× TA-Mg^{2+} solution with a final concentration of 3 nM for DNA origami and 0.5–8 mM for metal ions. The mixture was incubated for 3–4 h at room temperature to ensure sufficient interact between the metal ions and the extended DNA strands on the template.

Before performing electrochemical deposition of metals, the cleaned silicon wafers were ultrasonically cleaned again in ethanol and ultrapure water for 10 minutes each, followed by drying with nitrogen gas. Subsequently, the wafers were treated with a highly concentration Mg^{2+} solution (50 mM) for 10 minutes to enhance the adsorption between the DNA origami and the silicon wafer surface. After treatment, the wafers were dried again with nitrogen gas. Subsequently, 5 μL DNA origami template and metal salts incubated mixture was added onto the surface of the silicon wafer and adsorbed for 3–5 minutes.

The electrochemical deposition process was carried out using a three-electrode system, where the working electrode, reference electrode, and auxiliary electrode used were silicon wafer substrate, Ag/AgCl, and platinum wire, respectively. Then 50 μL of 1× TA-Mg^{2+} solution containing 0.5–8 mM metal ions were added to form a droplet on the silicon surface. The reference and counter electrodes were inserted into the droplet, and the system was connected to the electrochemical workstation for potentiostatic deposition. The determination of the metal deposition potential was based on the linear sweep voltammetry (LSV) curve of the metal salt solution. The appropriate potential for potentiostatic deposition was selected from the region where the current was negative and the slope increased rapidly.

The optimal deposition parameters (potential and time) were as follows: Ag (−0.8 V, 60 s), Cu (−1.5 V, 60 s), Au (0 V, 60 s), and Fe (−2.0 V, 60 s). For alloys, Ag-Cu was deposited at −2.0 V for 60 s, and Fe-Cu at −1.7 V for 60 s. For Ag/Cu composites, Ag was deposited at −0.8 V for 60 s, followed by Cu at −1.5 V for 40 s.

AFM characterization

The DNA origami adsorbed on the silicon wafer and the metal nanostructures fabricated via electrochemical deposition were imaged using the “PeakForce QNM in Fluid” mode with “SCANASYST-FLUID+” tips. The images were analyzed using NanoScope Analysis software.

SEM characterization

The metal samples deposited on the silicon wafer were gently rinsed with ultrapure water to remove the salt solution, followed by drying with nitrogen gas. Next, the wafer was secured to

the sample stage using conductive tape for SEM characterization, with a potential of 5 kV. The area containing the target structure was selected for elemental analysis.

Supplementary Figures

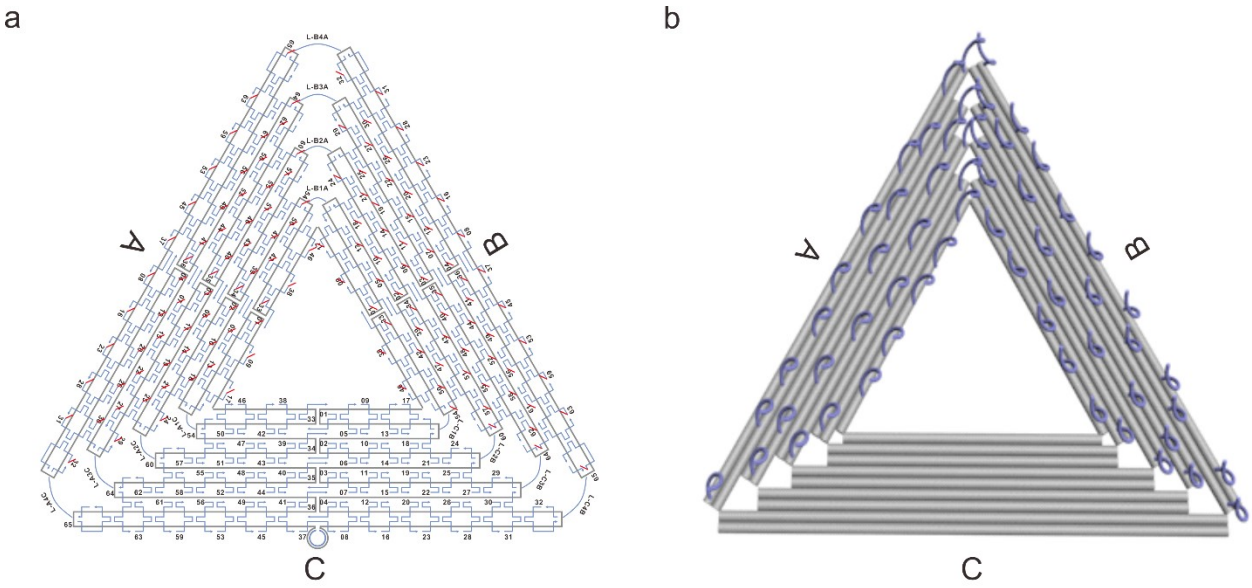

Figure S1. (a) Design of triangular DNA origami template with staples on both sides A and B extended by 15 bases. (b) Schematic diagram of triangular DNA origami template with a V-shaped pattern.

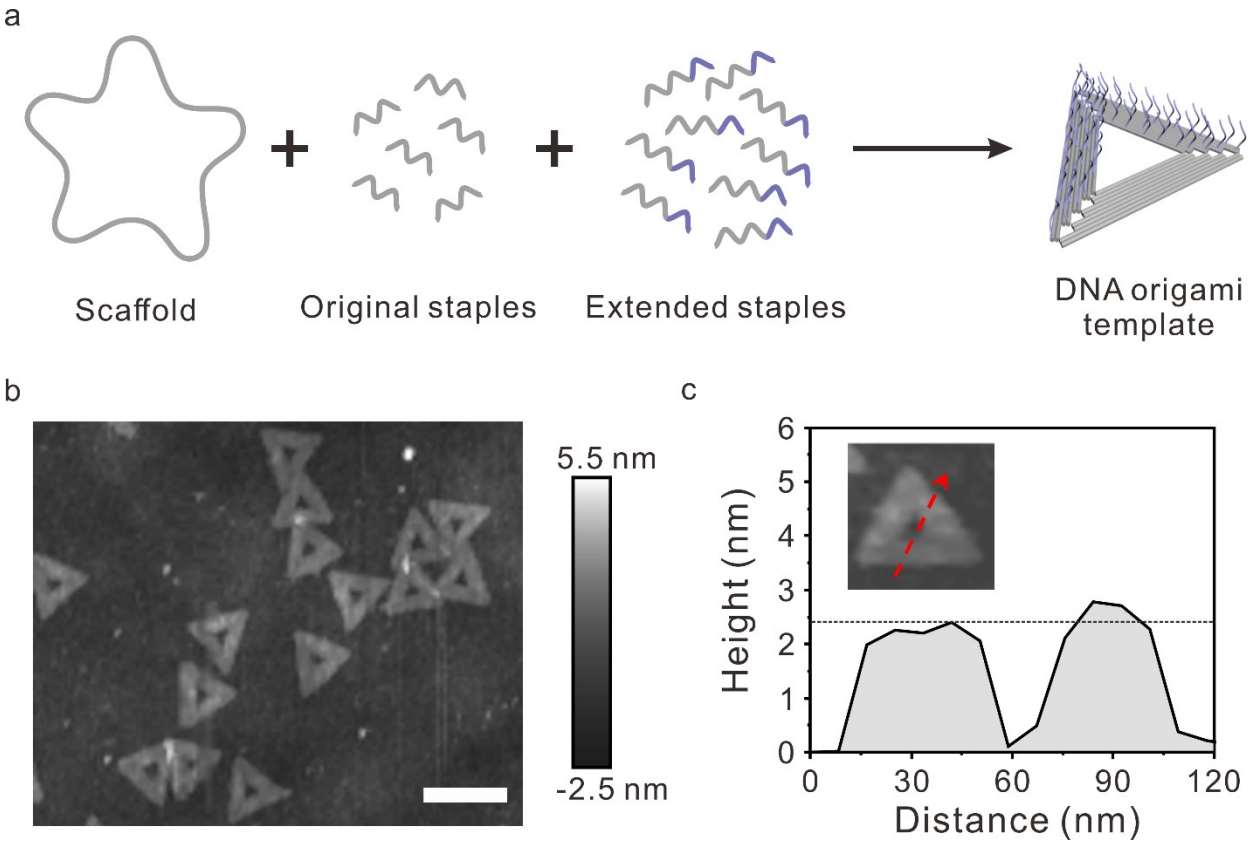

Figure S2. (a) Scheme depicting the assembly of triangular DNA origami template with a V-shaped pattern. (b) AFM image of triangular DNA origami template with a V-shaped pattern. Scale bars: 100 nm. (c) Height analysis.

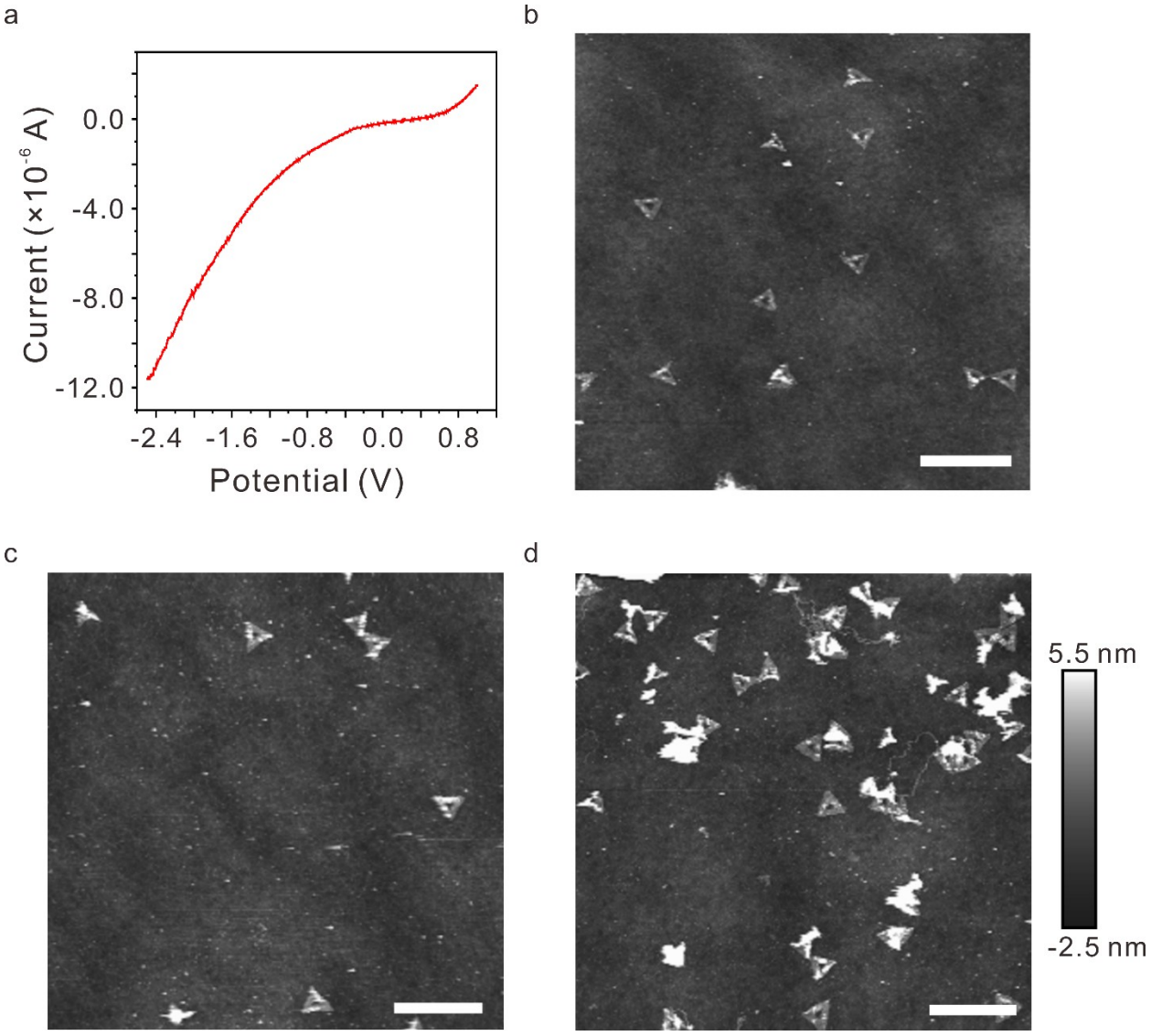

Figure S3. (a) LSV curves of the Cu^{2+}-containing system. (b–d) Electrodeposition of Cu at different potentials: (b) −1.3 V; (c) −1.5 V; (d) −1.7 V. Scale bar: 400 nm.

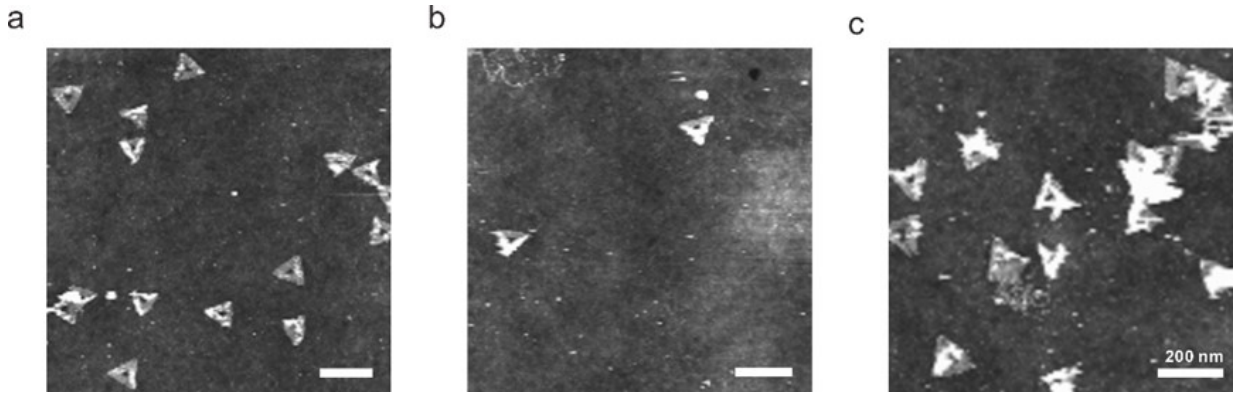

Figure S4. AFM images of Cu metal electrodeposition at different deposition times under a fixed potential of −1.5 V and a Cu^{2+} concentration of 4 mM: (a) 40 s, (b) 60 s, (c) 80 s. Scale bar: 200 nm.

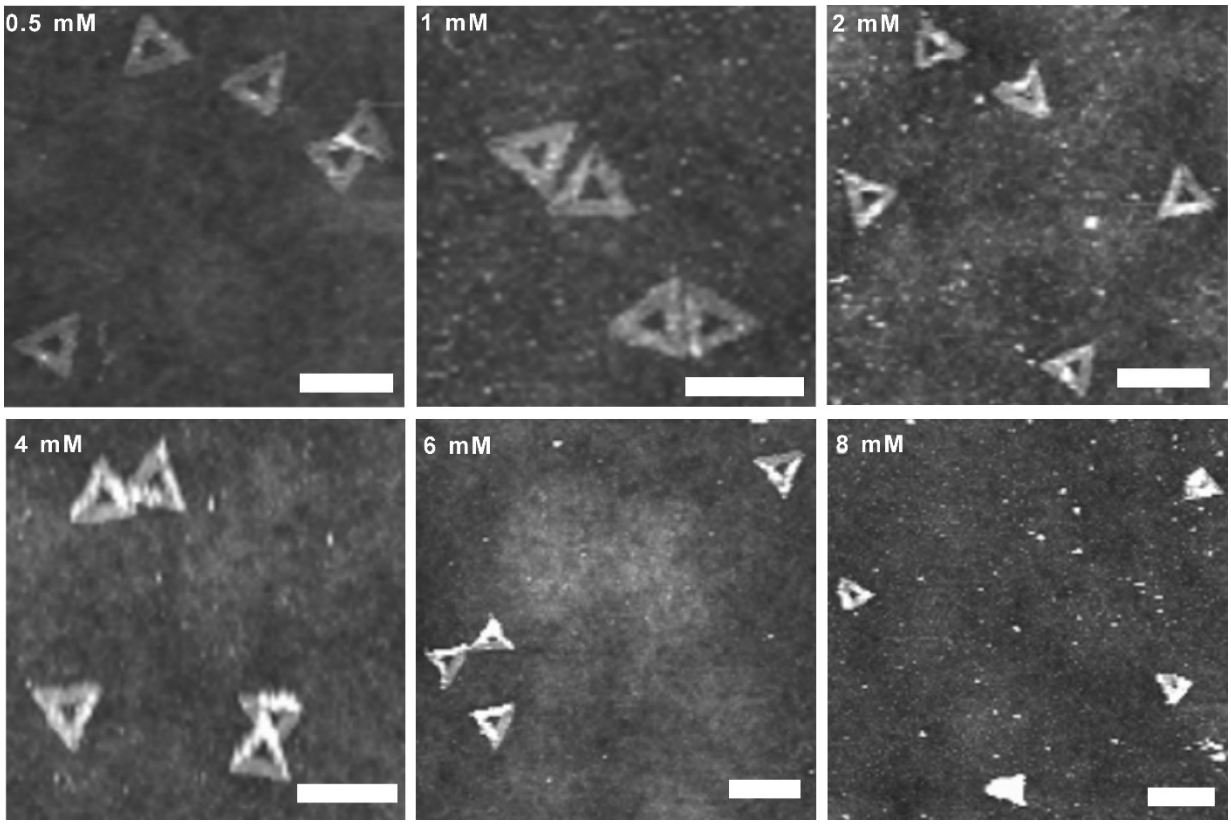

Figure S5. AFM images of Cu metal electrodeposition at different concentrations of Cu^{2+} (0.5 mM, 1 mM, 2 mM, 4 mM, 6 mM, 8 mM). Scale bar: 200 nm.

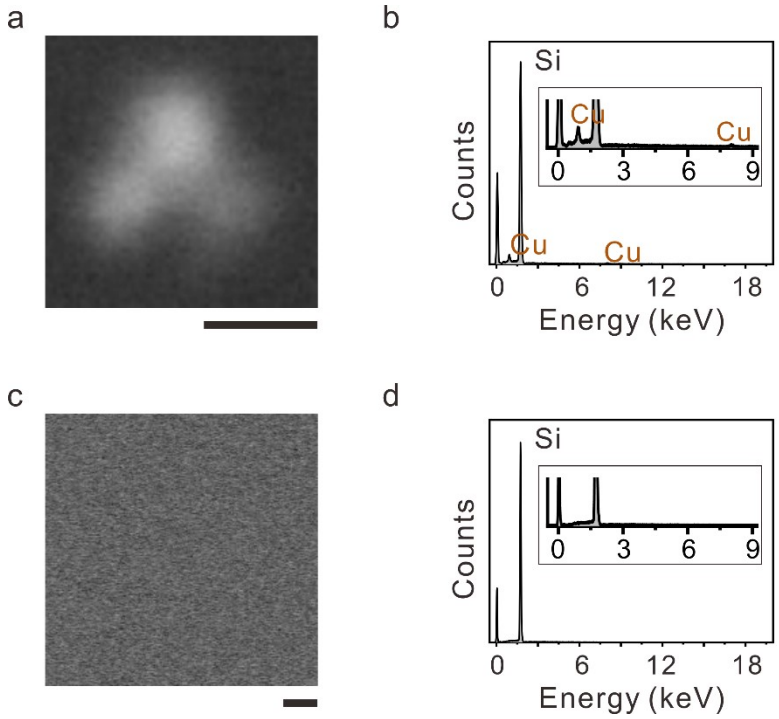

Figure S6. (a) SEM image of a Cu nanostructure formed on DNA origami template. Scale bar: 50 nm. (b) Element component analysis of the Cu nanostructure by EDX. (c) SEM image of a bare Si region without the nanostructures. Scale bar: 50 nm. (d) EDX spectrum collected from the bare Si region, showing no characteristic Cu peaks.

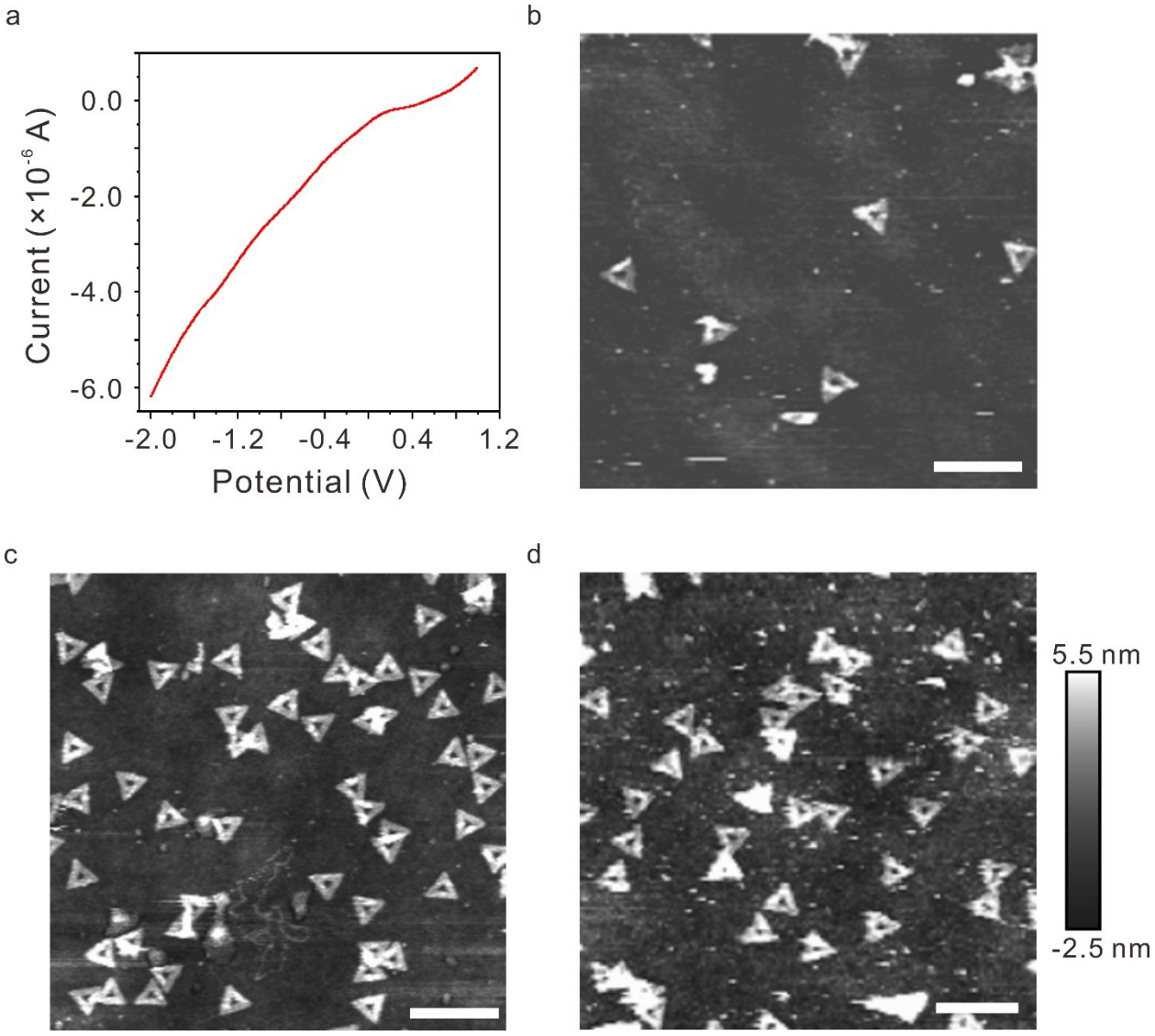

Figure S7. (a) LSV curves of the Ag^+-containing system. (b–d) Electrodeposition of Ag at different potentials: (b) −0.6 V; (c) −0.8 V; (d) −1.0 V. Scale bar: 400 nm.

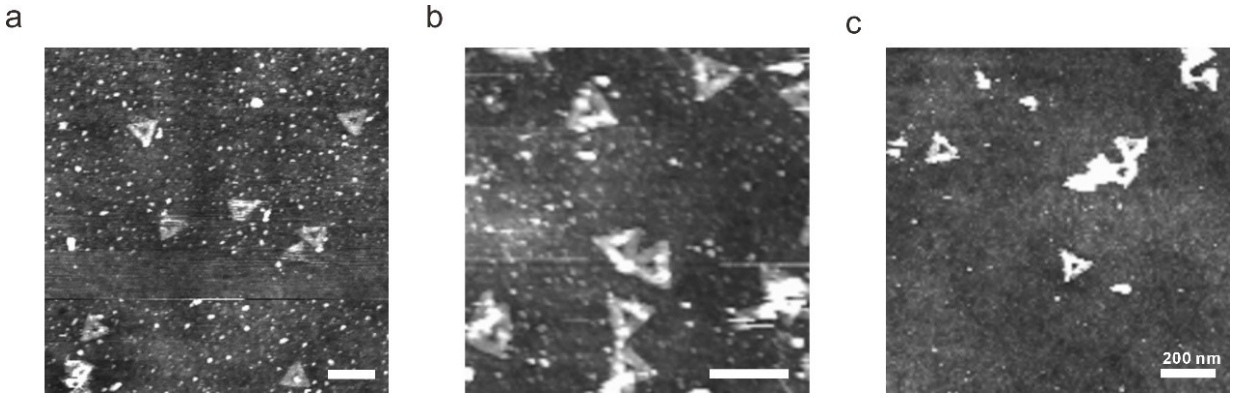

Figure S8. AFM images of Ag metal electrodeposition at different deposition times under a fixed potential of −0.8 V and an Ag^+ concentration of 4 mM: (a) 40 s, (b) 60 s, (c) 80 s. Scale bar: 200 nm.

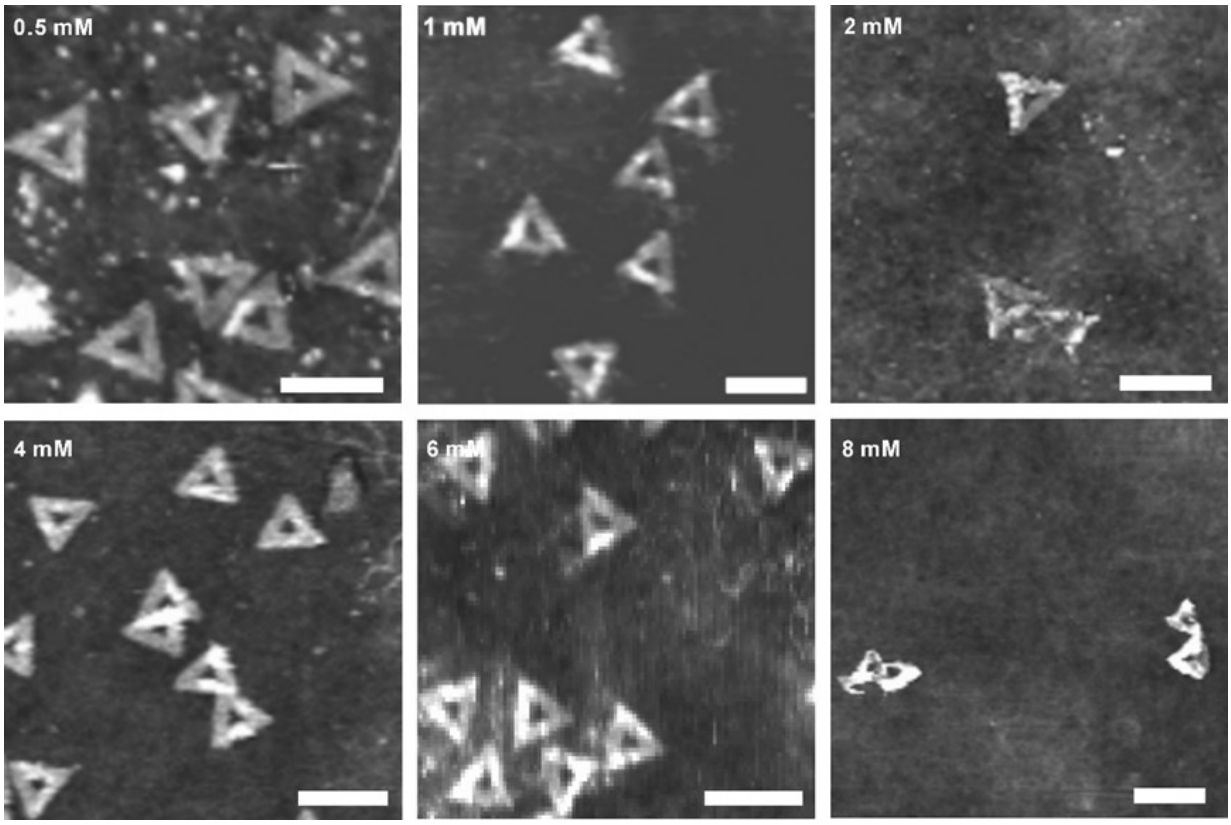

Figure S9. AFM images of Ag metal electrodeposition at different concentrations of Ag^+ (0.5 mM, 1 mM, 2 mM, 4 mM, 6 mM, 8 mM). Scale bar: 200 nm.

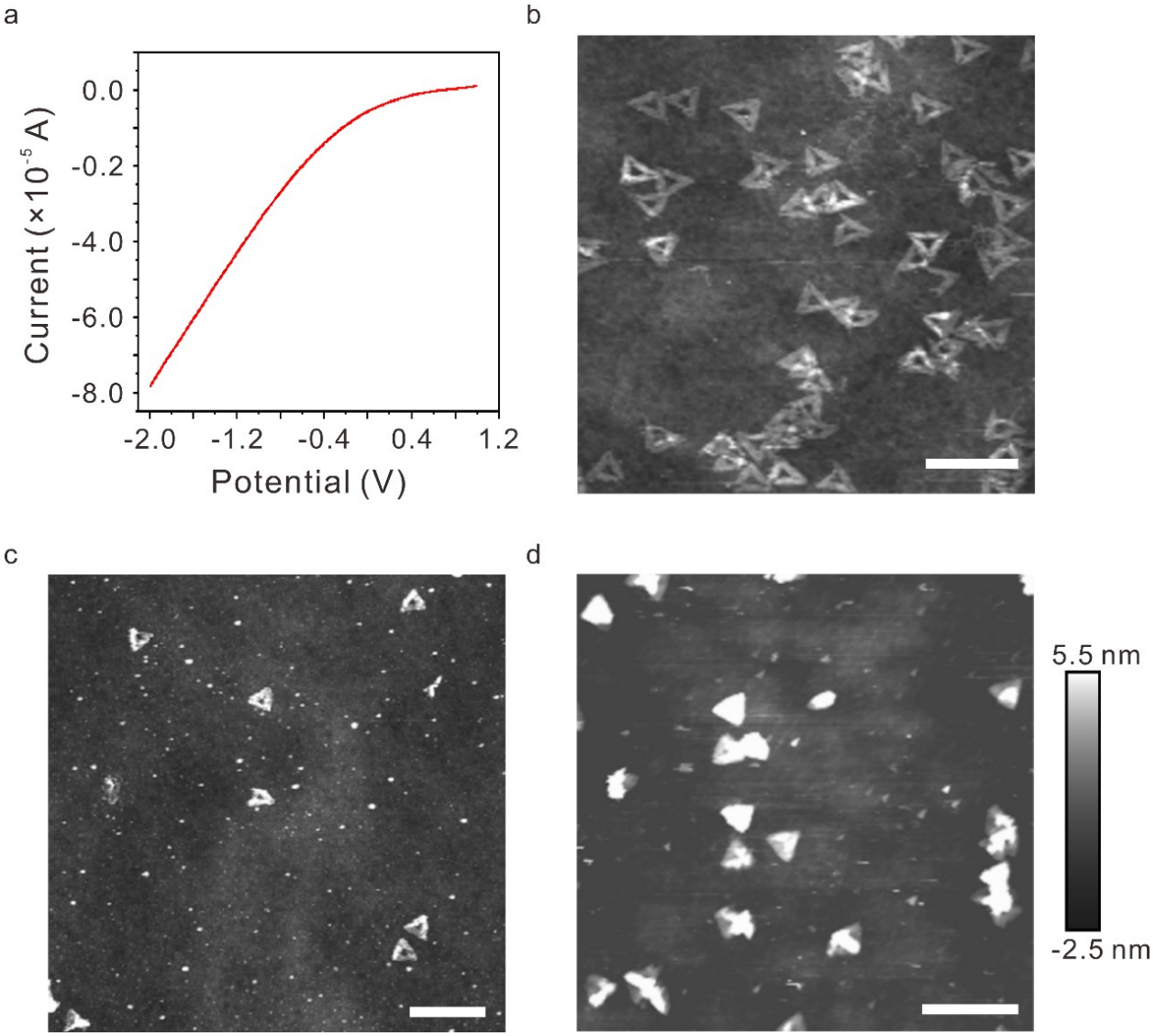

Figure S10. (a) LSV curves of the $AuCl_4^-$-containing system. (b–d) Electrodeposition of Au at different potentials: (b) +0.2 V; (c) 0 V; (d) −0.5 V. Scale bar: 400 nm.

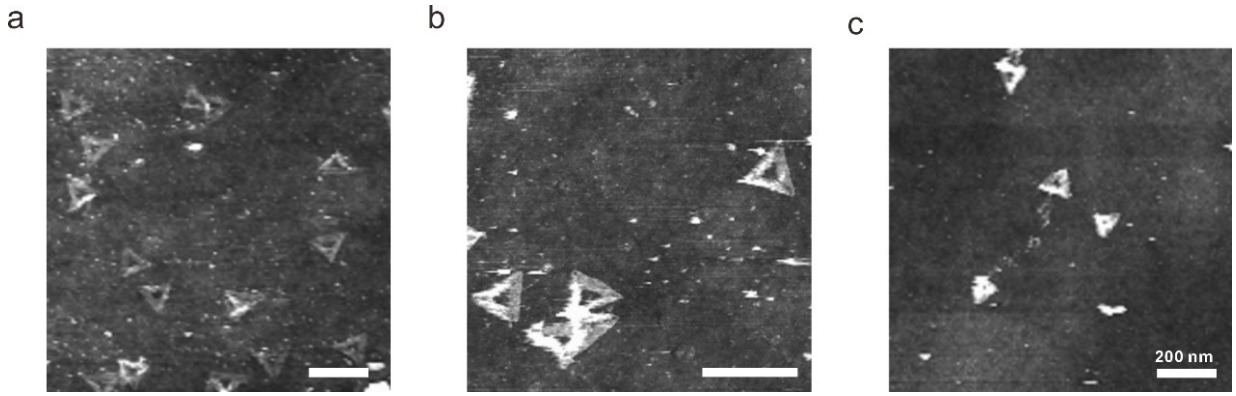

Figure S11. AFM images of Au metal electrodeposition at different deposition times under a fixed potential of 0 V and an $AuCl_4^-$ concentration of 4 mM: (a) 40 s, (b) 60 s, (c) 80 s. Scale bar: 200 nm.

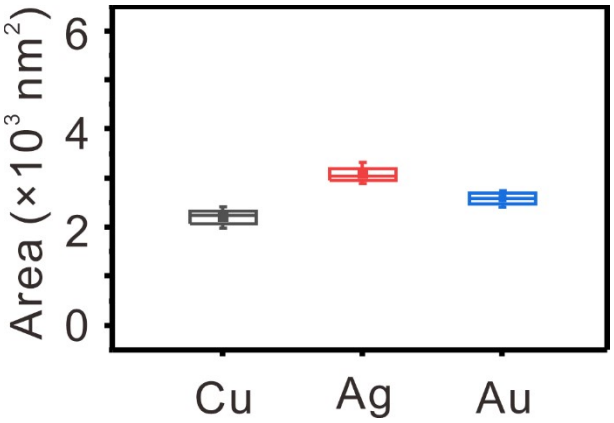

Figure S12. Area analysis and comparison of Cu, Ag, and Au structures prepared with 4 mM concentration precursor conditions.

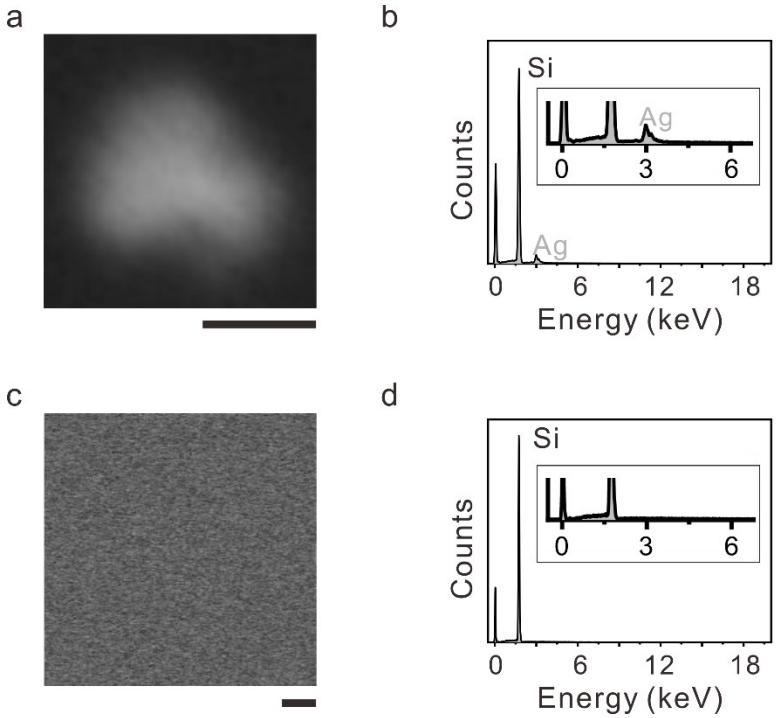

Figure S13. (a) SEM image of an Ag nanostructure formed on DNA origami template. Scale bar: 50 nm. (b) Element component analysis of the Ag nanostructure by EDX. (c) SEM image of a bare Si region without the nanostructures. Scale bar: 50 nm. (d) EDX spectrum collected from the bare Si region, showing no characteristic Ag peaks.

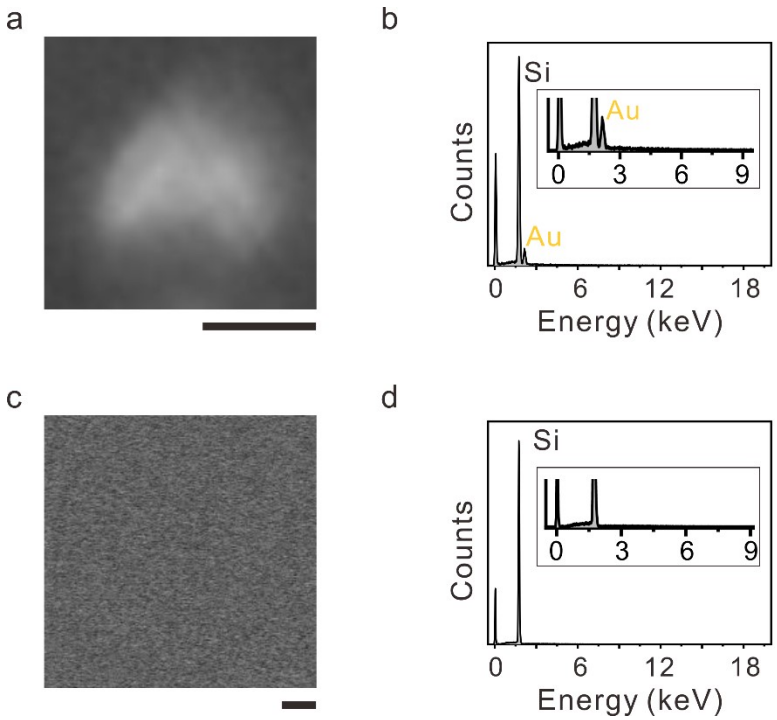

Figure S14. (a) SEM image of an Au nanostructure formed on DNA origami template. Scale bar: 50 nm. (b) Element component analysis of the Au nanostructure by EDX. (c) SEM image of a bare Si region without the nanostructures. Scale bar: 50 nm. (d) EDX spectrum collected from the bare Si region, showing no characteristic Ag peaks.

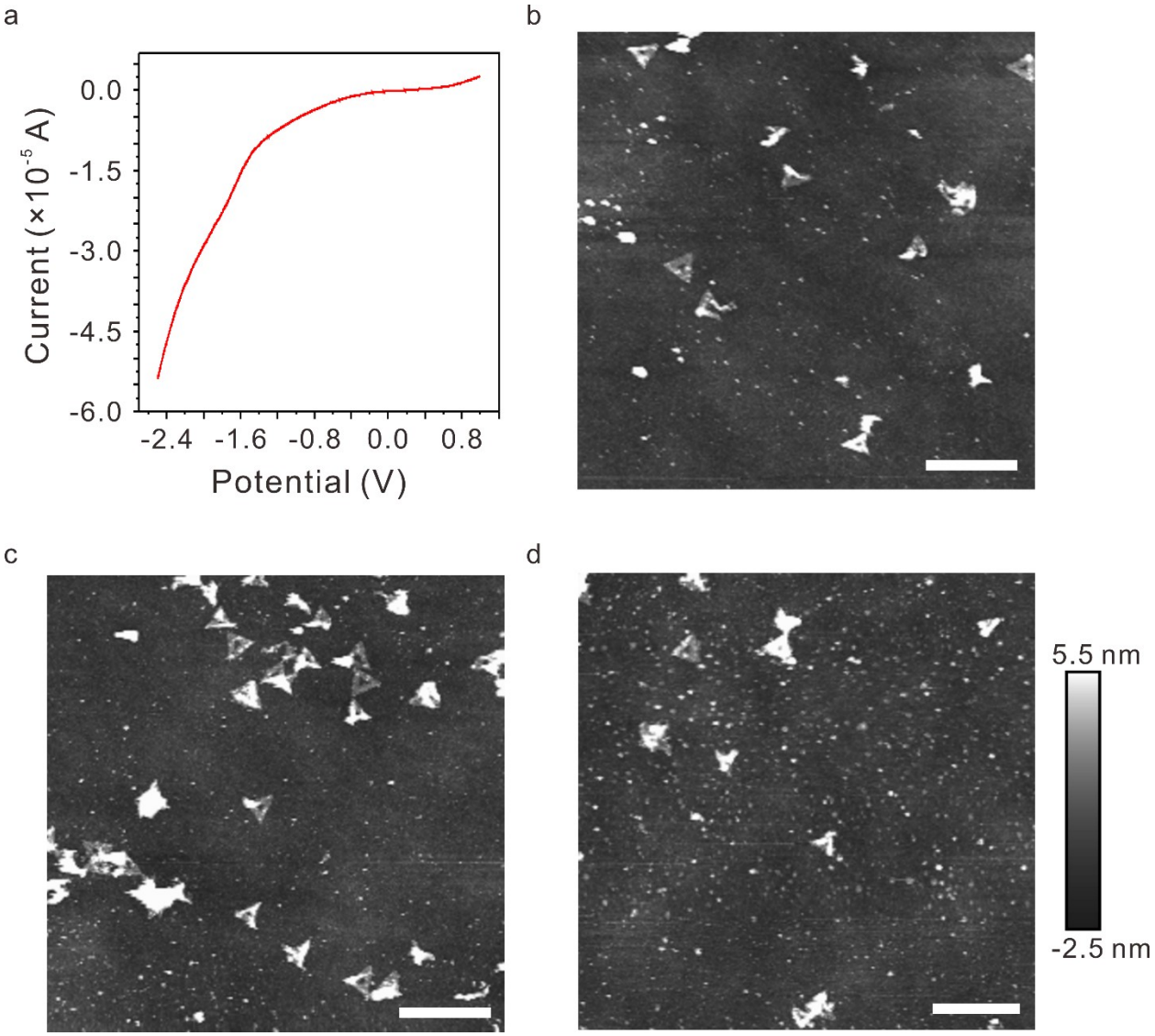

Figure S15. (a) LSV curves of the Fe^{2+}-containing system. (b–d) Electrodeposition of Fe at different potentials: (b) −1.6 V; (c) −1.8 V; (d) −2.0 V. Scale bar: 400 nm.

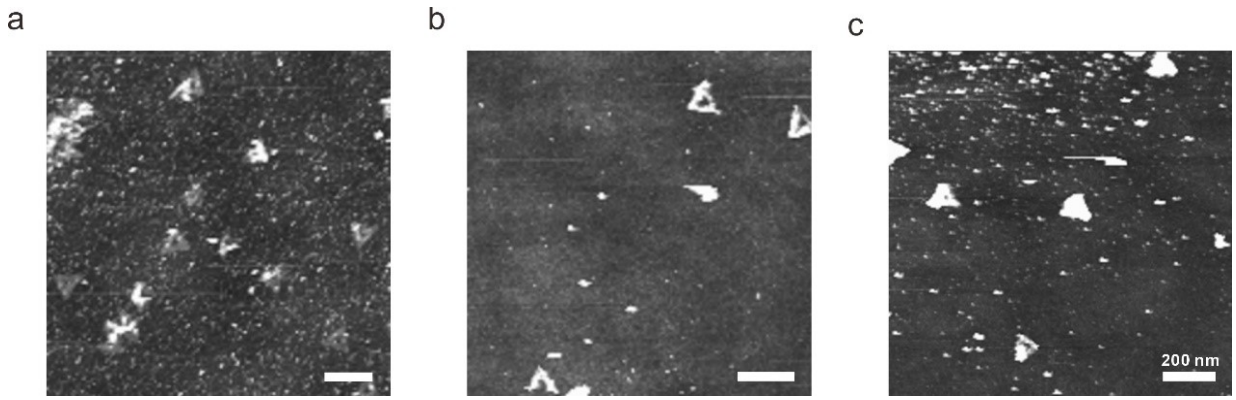

Figure S16. AFM images of Fe metal electrodeposition at different deposition times under a fixed potential of −1.8 V and a Fe^{2+} concentration of 4 mM: (a) 40 s, (b) 60 s, (c) 80 s. Scale bar: 200 nm.

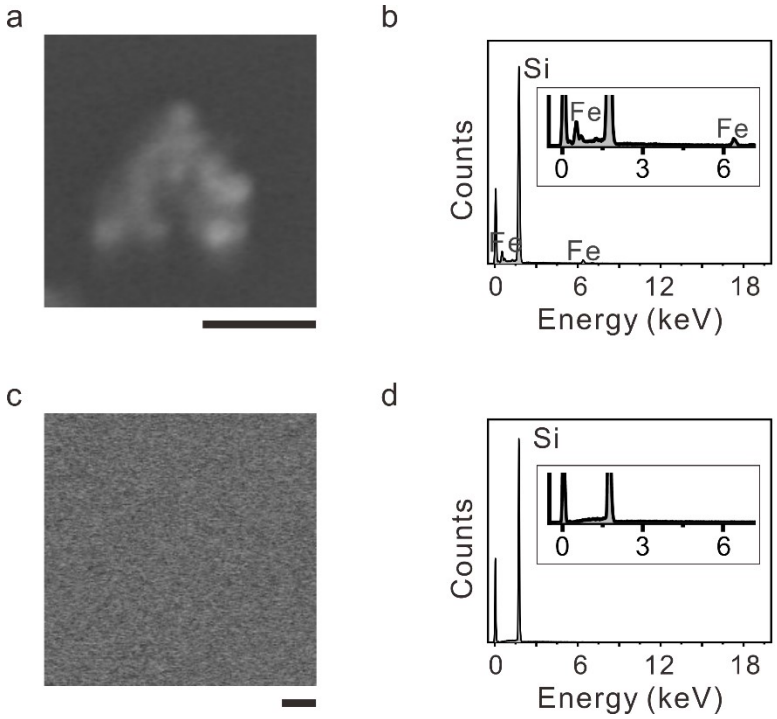

Figure S17. (a) SEM image of a Fe nanostructure formed on DNA origami template. Scale bar: 50 nm. (b) Element component analysis of the Fe nanostructure by EDX. (c) SEM image of a bare Si region without the nanostructures. Scale bar: 50 nm. (d) EDX spectrum collected from the bare Si region, showing no characteristic Ag peaks.

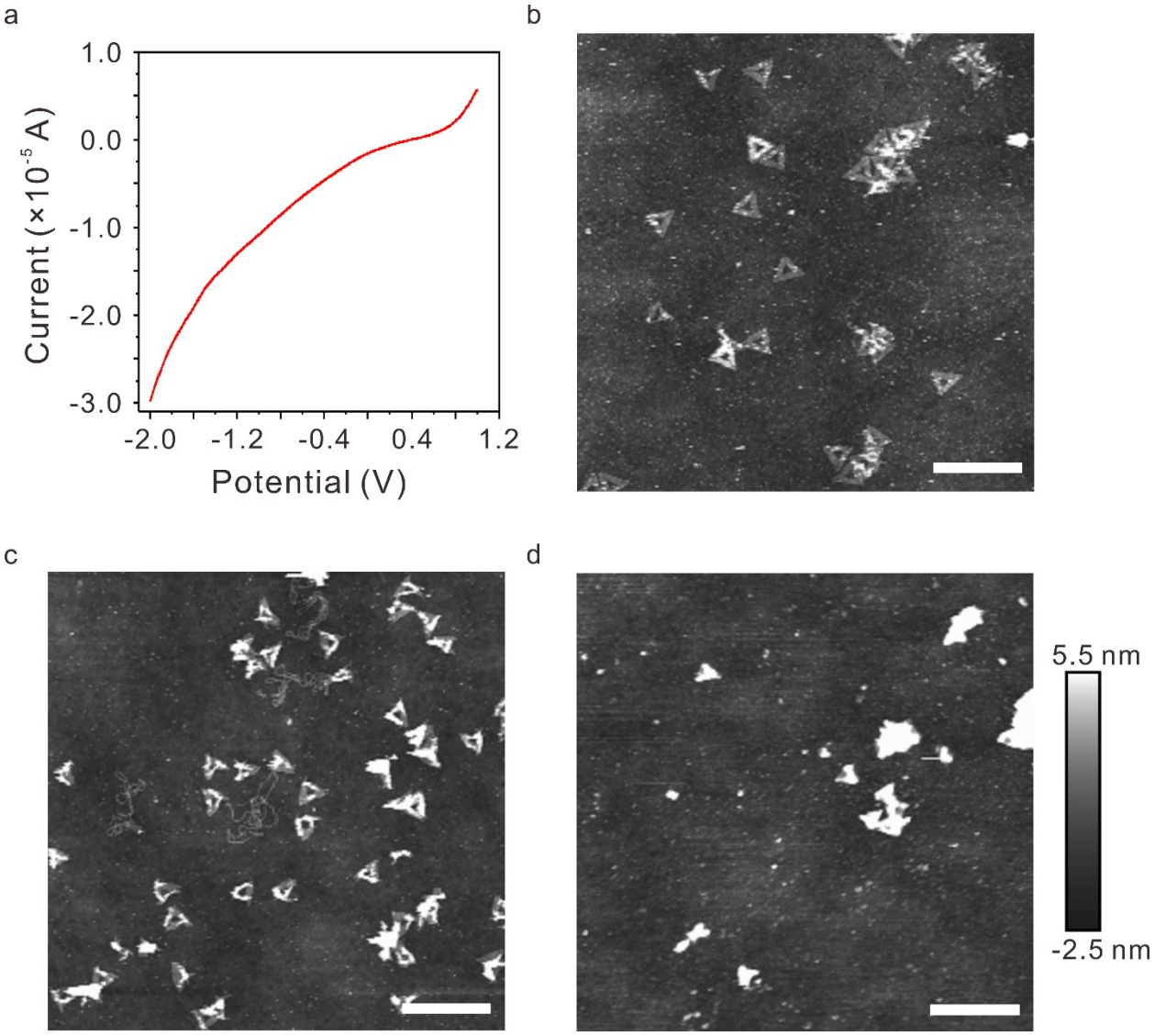

Figure S18. (a) LSV curves of the Cu^{2+} and Ag^{+}-containing system. (b–d) Electrodeposition of Cu-Ag alloy at different potentials: (b) −1.5 V; (c) −1.7 V; (d) −2.0 V. Scale bar: 400 nm.

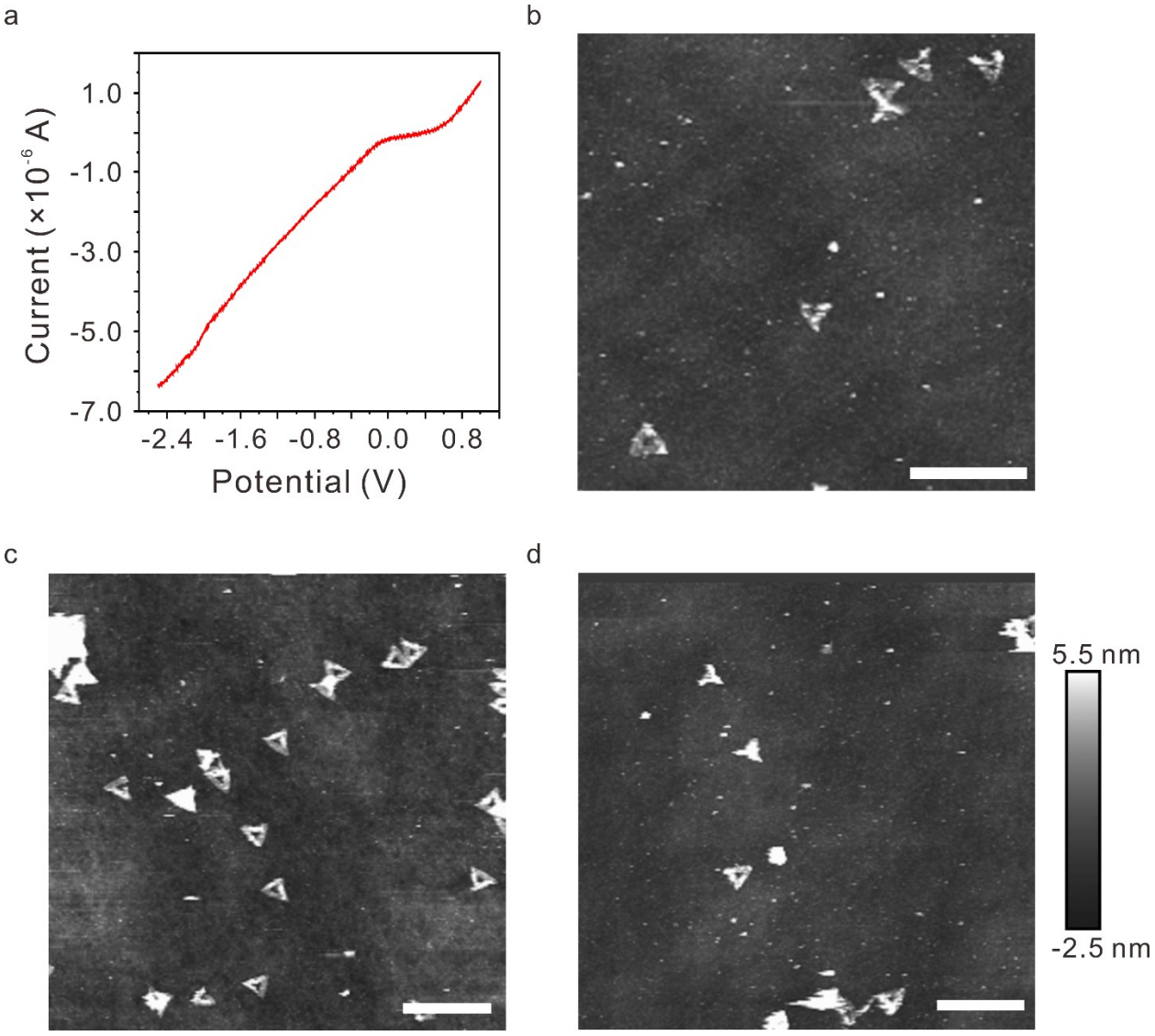

Figure S19. (a) LSV curves of the Fe^{2+} and Cu^{2+}-containing system. (b–d) Electrodeposition of Fe-Cu alloy at different potentials: (b) −1.5 V; (c) −2.0 V; (d) −2.5 V. Scale bar: 400 nm.

Table S1. Detailed DNA sequences for triangular DNA origami templates:

Name	Sequence (5' to 3')
A01	AAAAAAAAAAAAAAACGGGGTTTCCTCAAGAGAAGGATTTTGAATTA
A02	AAAAAAAAAAAAAAAAGCGTCATGTCTCTGAATTTACCGACTACCTT
A03	AAAAAAAAAAAAAAATTCATAATCCCCTTATTAGCGTTTTTCTTACC
A04	AAAAAAAAAAAAAAAATGGTTTATGTCACAATCAATAGATATTAAAC
A05	AAAAAAAAAAAAAAATTTGATGATTAAGAGGCTGAGACTTGCTCAGTACCAGGCG
A06	AAAAAAAAAAAAAAACCGGAACCCAGAATGGAAAGCGCAACATGGCT
A07	AAAAAAAAAAAAAAAAAAGACAACATTTTCGGTCATAGCCAAAATCA
A08	AAAAAAAAAAAAAAAGACGGGAGAATTAACTCGGAATAAGTTTATTTCCAGCGCC
A09	AAAAAAAAAAAAAAAGATAAGTGCCGTCGAGCTGAAACATGAAAGTATACAGGAG
A10	AAAAAAAAAAAAAAATGTACTGGAAATCCTCATTAAAGCAGAGCCAC
A11	AAAAAAAAAAAAAAACACCGGAAAGCGCGTTTTCATCGGAAGGGCGA
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A14	AAAAAAAAAAAAAAACTCAGAGCATATTCACAAACAAATTAATAAGT
A15	AAAAAAAAAAAAAAAGGAGGGAATTTAGCGTCAGACTGTCCGCCTCC
A16	AAAAAAAAAAAAAAAGTCAGAGGGTAATTGATGGCAACATATAAAAGCGATTGAG
A17	AAAAAAAAAAAAAAATAGCCCGGAATAGGTGAATGCCCCCTGCCTATGGTCAGTG
A18	AAAAAAAAAAAAAAACCTTGAGTCAGACGATTGGCCTTGCGCCACCC
A19	AAAAAAAAAAAAAAATCAGAACCCAGAATCAAGTTTGCCGGTAAATA
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A21	AAAAAAAAAAAAAAACAGAGCCAGGAGGTTGAGGCAGGTAACAGTGCCCG
A22	AAAAAAAAAAAAAAAATTAAAGGCCGTAATCAGTAGCGAGCCACCCT
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A36	AAAAAAAAAAAAAAACAAGTACCTCATTCCAAGAACGGGAAATTCAT
A37	AAAAAAAAAAAAAAAAGAGAATAACATAAAAACAGGGAAGCGCATTA

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B55	AAAAAAAAAAAAAAAAACAGCTTGCTTTGAGGACTAAAGCGATTATA
B56	AAAAAAAAAAAAAAACCAAGCGCAGGCGCATAGGCTGGCAGAACTGGCTCATTAT
B57	AAAAAAAAAAAAAAACGAGGTGAGGCTCCAAAAGGAGCC
B58	AAAAAAAAAAAAAAAACCCCCAGACTTTTTCATGAGGAACTTGCTTT
B59	AAAAAAAAAAAAAAAACCTTATGCGATTTTATGACCTTCATCAAGAGCATCTTTG
B60	AAAAAAAAAAAAAAACGGTTTATCAGGTTTCCATTAAACGGGAATACACT
B61	AAAAAAAAAAAAAAAAAAACACTTAATCTTGACAAGAACTTAATCATTGTGAATT
B62	AAAAAAAAAAAAAAAGGCAAAAGTAAAATACGTAATGCC
B63	AAAAAAAAAAAAAAATGGTTTAATTTCAACTCGGATATTCATTACCCACGAAAGA
B64	AAAAAAAAAAAAAAAACCAACCTAAAAAATCAACGTAACAAATAAATTGGGCTTGAGA
B65	AAAAAAAAAAAAAAACCTGACGAGAAACACCAGAACGAGTAGGCTGCTCATTCAGTGA

Other sequences can be found in the sequences list for the triangle origami.[1]

References

1. S. Pal, Z. Deng, H. Wang, S. Zou, Y. Liu and H. Yan, *J. Am. Chem. Soc.*, 2011, **133**, 17606-17609.