

Supporting Information for

Wet-chemical Synthesis of Palladium Nanosprings

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1. Materials, Apparatus and Methods

1.1 Materials

Palladium chloride (PdCl_2) and Copper chloride ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) were purchased from Sigma-Aldrich. All commercial chemicals were used as received. Triply-distilled deionized water (Millipore, $>18.2 \text{ M}\Omega$) was used for preparation of all aqueous solutions. Au (Orotemp 24 RTU) plating solution was obtained from Technic Inc. All experiments were conducted in ambient room temperature. Commercial Anodic Aluminum Oxide templates, AnodiscTM (AAO, diameter $\approx 13 \text{ mm}$, nanochannel diameter $\approx 250 \text{ nm}$, thickness $\approx 60 \mu\text{m}$) were purchased from Whatman[®] International Ltd.

1.2 Apparatus

Electrochemical system: We performed all electrochemical experiments on an Autolab (PGSTAT12) equipped with three-electrode system (counter electrode: Pt mesh; reference electrode: Ag/AgCl (3 M KCl)).

SEM and TEM: We acquired SEM images by using JEOL JSM-7401F Field Emission Scanning Electron Microscope (FE-SEM) and TEM images by JEOL JEM2100F High Resolution Transmission Electron Microscope (HR-TEM).

1.3 Methods

(a) Assembly of electrochemical cell

The physical configuration of the cell is similar as Liu et al described¹. Briefly, an AAO disc sandwiched between a piece of glassy carbon and an insulating O-ring were assembled in a Teflon[®] cell as working electrode.

(b) Conducting layer and predeposition

The conducting layers on one side of commercial AAO templates were conveniently prepared by Au nanoparticles immobilization method previously developed by our group². In case of homemade AAO, we prepared Au conducting layer in 700 nm by thermal evaporation. Predeposition is usually applied in order to fill the branched irregular nanochannels in one side of AAO. We used to deposit Au by 2 C/cm^2 at

-0.95 V from commercial plating solution.

(c) Chemical etching: After removing AAO, Pd-Cu nanorods were usually etched by 30% (v/v) HNO₃ for 15 min.

(d) Sampling

The samples for SEM measurement were prepared on a sheet of carbon tape and heated in an 80°C oven for 10 minutes after AAO template removal by 3 M NaOH for 20 minutes and multiple rinses by deionized water. The dispersed nanorods and nanosprings were dissociated from substrates into DI water by moderate sonication. Copper TEM grids were used to carry dispersed nanostructures for TEM observation.

2. Figures

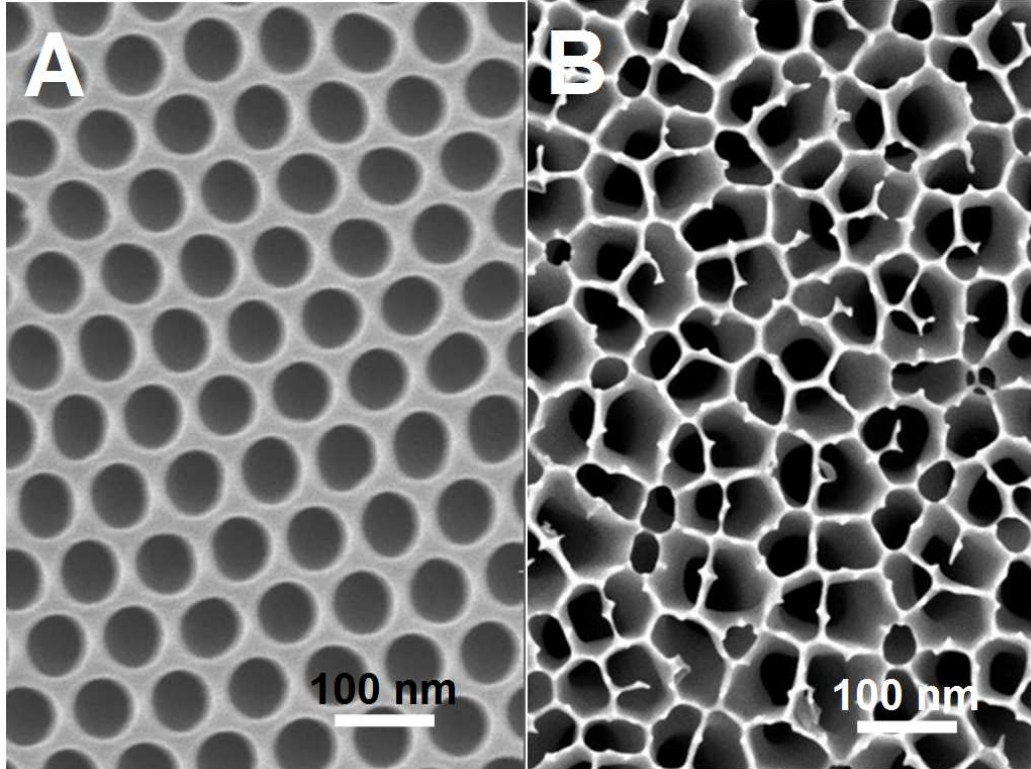


Figure S1 SEM image of a homemade AAO template, (A) top view (B) Back view.

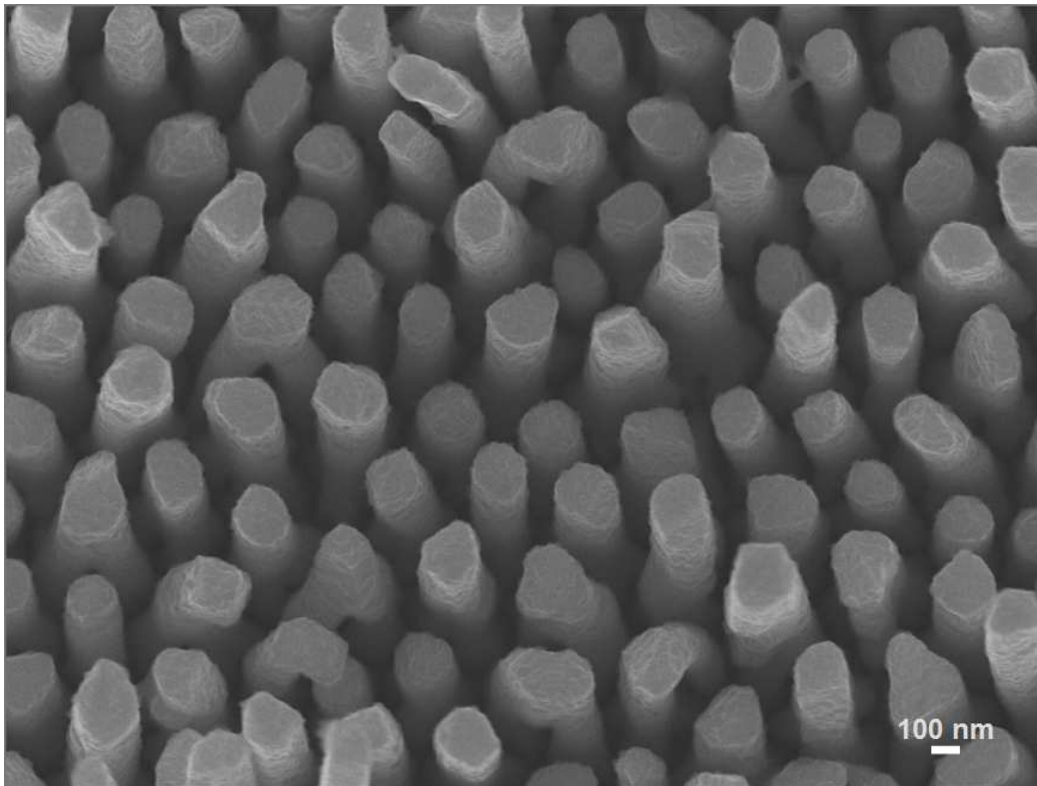


Figure S2 SEM image of Cu nanorods showing that Cu ions can be reduced under studied condition, a solution containing 20 mM $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and 0.1 M HCl and -0.1 V external electric potential.

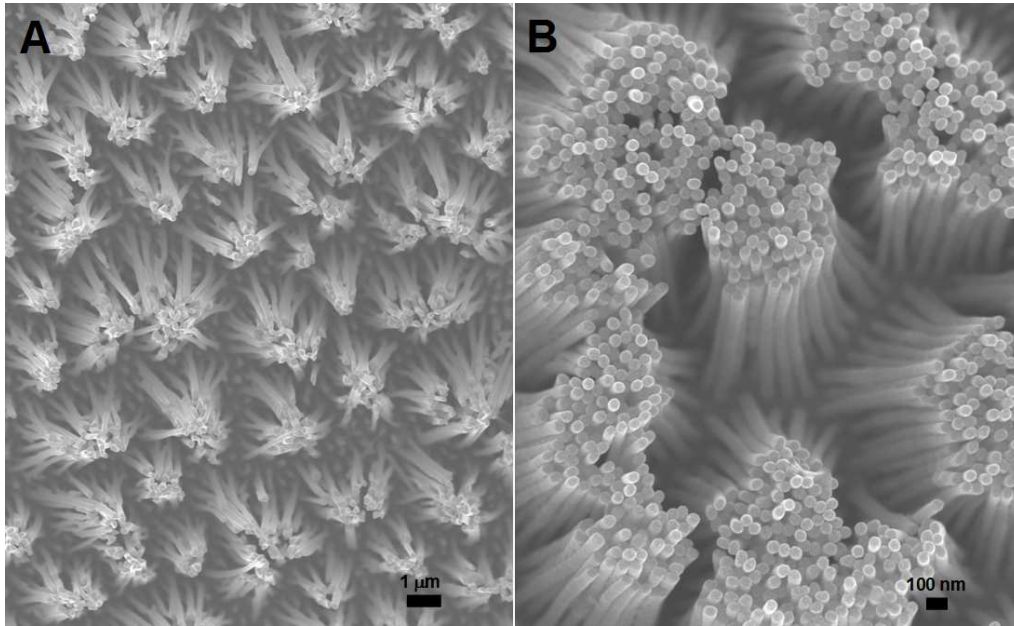


Figure S3 SEM images of homogeneous codeposited Pd-Cu nanorods (springs' precursor) from the AAO templates with varied nanochannel sizes, ~250 nm (A) and ~70 nm (B).

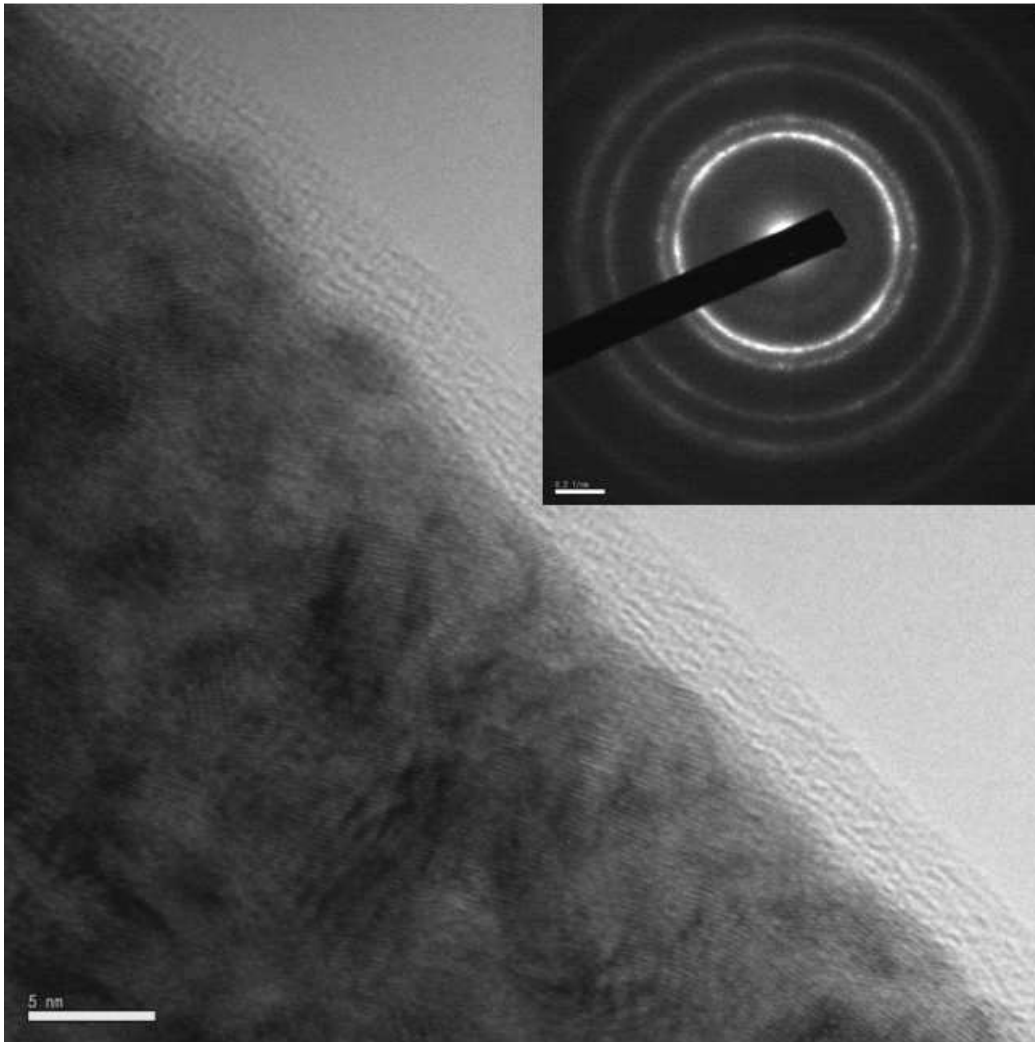


Figure S4 HR-TEM image of Pd-Cu nanorod and SAED pattern (inset) showing multicrystalline property.

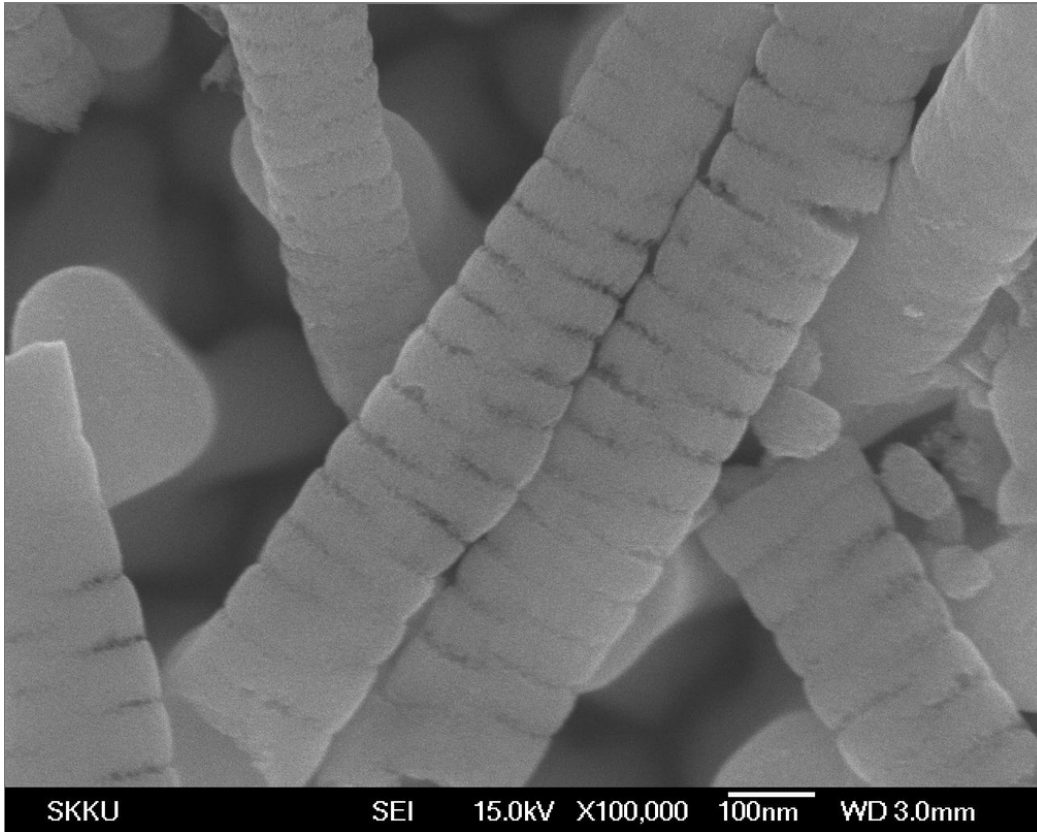


Figure S5 SEM image of Pd residual nanorods after etching Cu component. Experimental condition for Pd-Cu deposition: 40 mM PdCl₂, 20 mM CuCl₂•2H₂O and 0.1 M HCl and -0.1 V external electric potential.

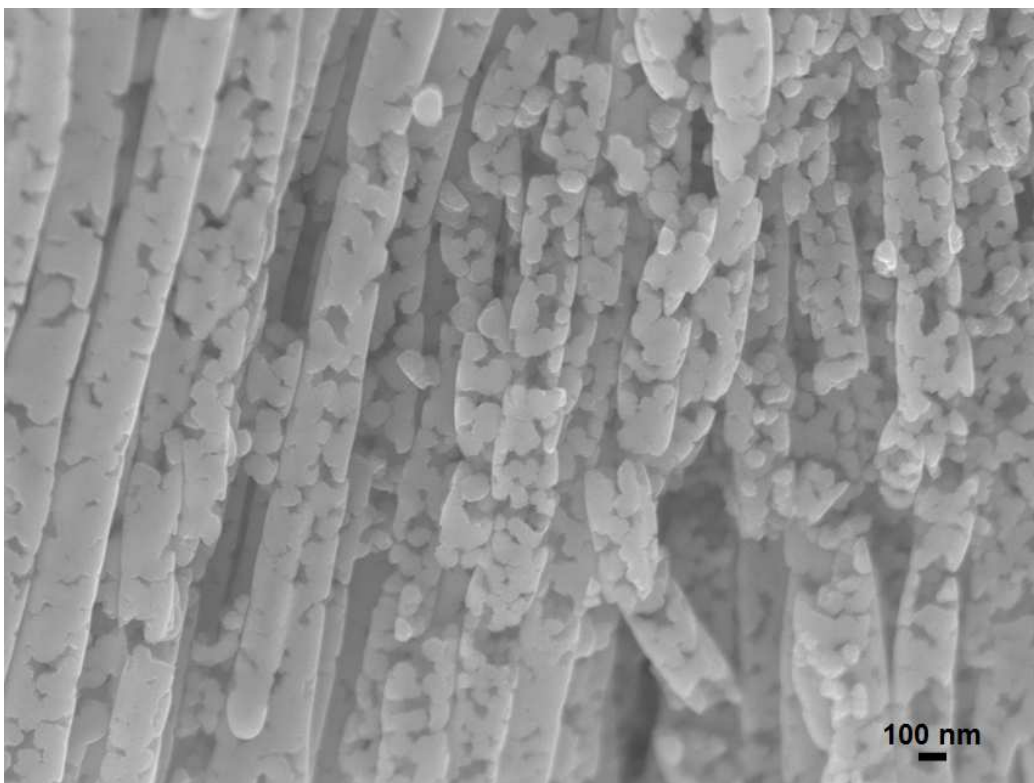


Figure S6 SEM image of Pd-Cu nanodeposits. Experimental condition for Pd-Cu deposition: 25 mM PdCl₂, 40 mM CuCl₂•2H₂O and 0.1 M HCl and -0.1 V external electric potential.

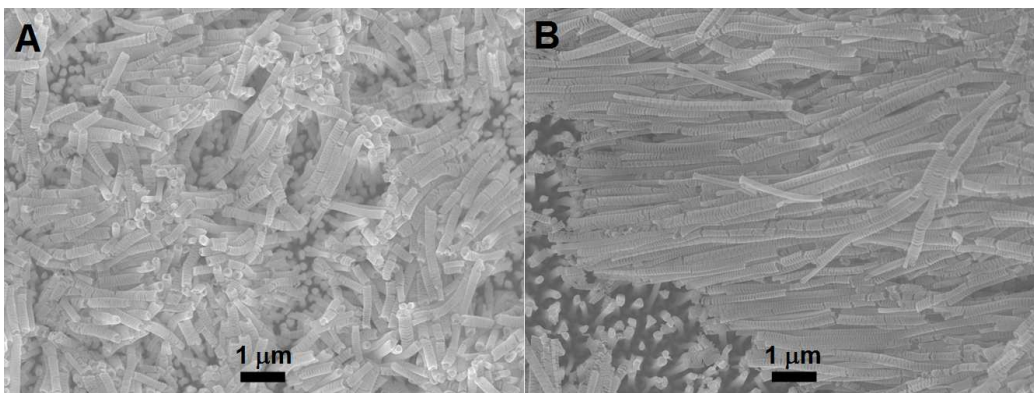


Figure S7 SEM images of length controllable Pd nanosprings, ~1.5 μm for 1 C/cm² deposition and ~6 μm for 4 C/cm² deposition.

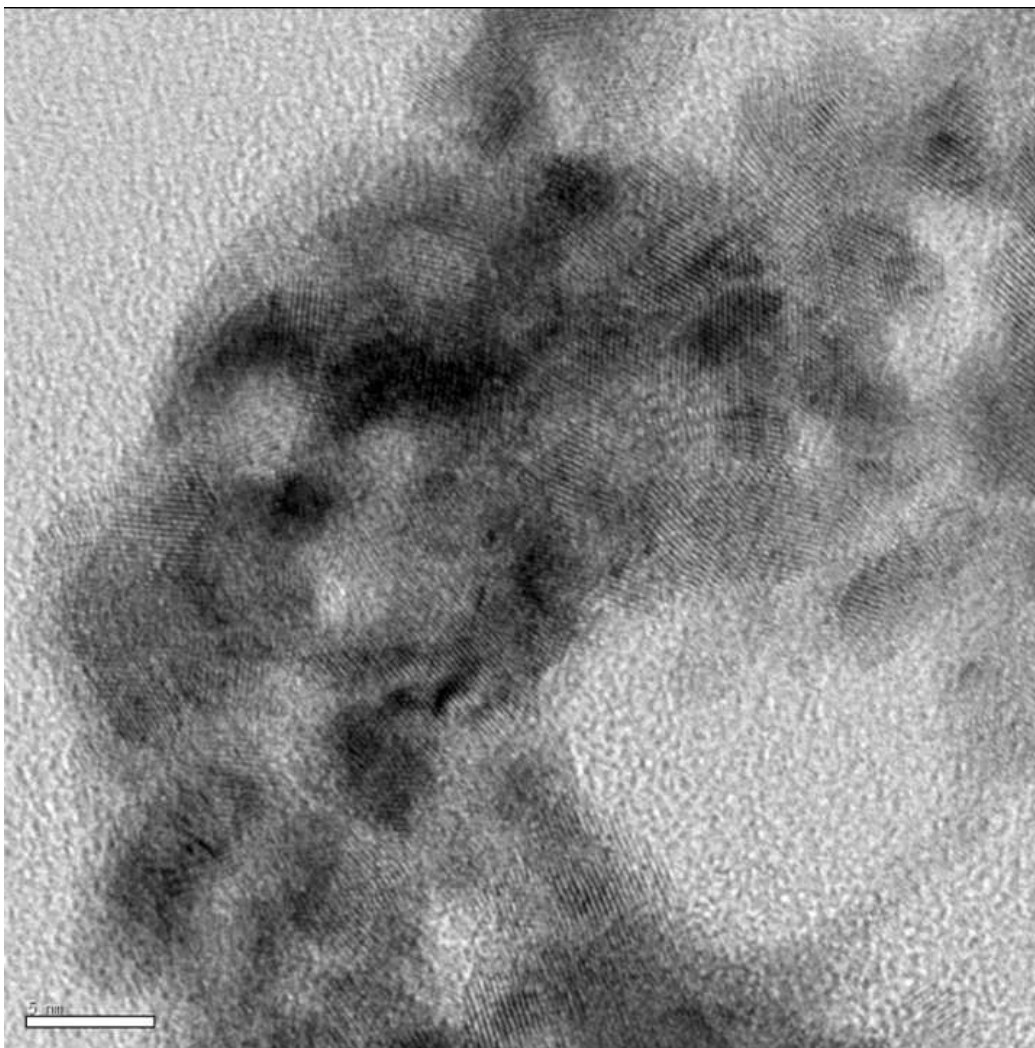


Figure S8 HR-TEM image of a turning corner of Pd nanospring showing clear grains and multicrystallinity.

Reference:

- (1) Liu, L. F.; Pippel, E.; Scholz, R.; Gosele, U., Nanoporous Pt–Co Alloy Nanowires: Fabrication, Characterization, and Electrocatalytic Properties. *Nano Letters* **2009**, *9* (12), 4352-4358.
- (2) Yoo, S.-H.; Liu, L.; Park, S., Nanoparticle films as a conducting layer for anodic aluminum oxide template-assisted nanorod synthesis. *Journal of Colloid and Interface Science* **2009**, *339* (1), 183-186.