

**Supporting Information**

**Simple-cubic microcubes assembled by palladium nanocubes**

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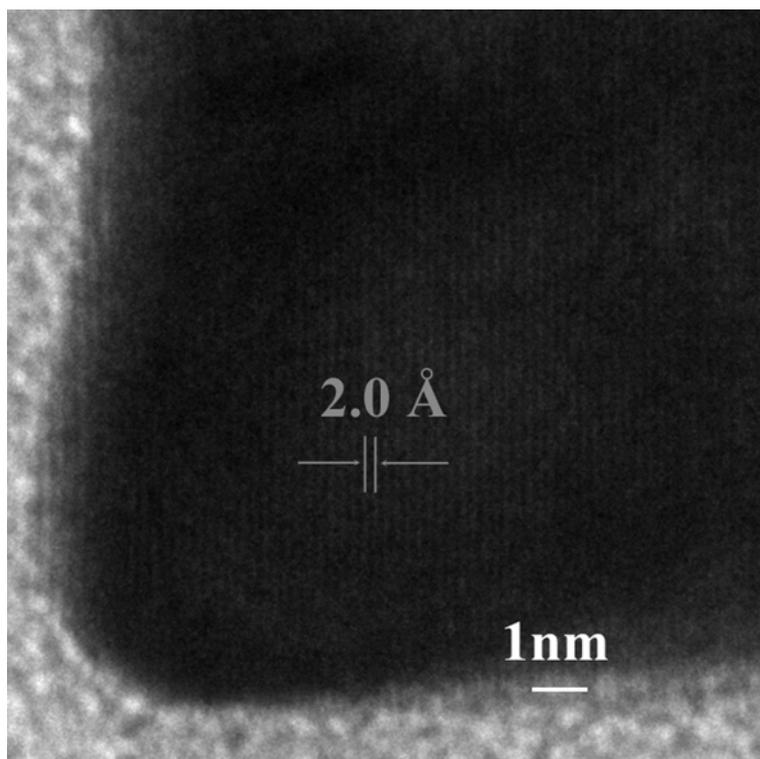
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### **Experimental details:**

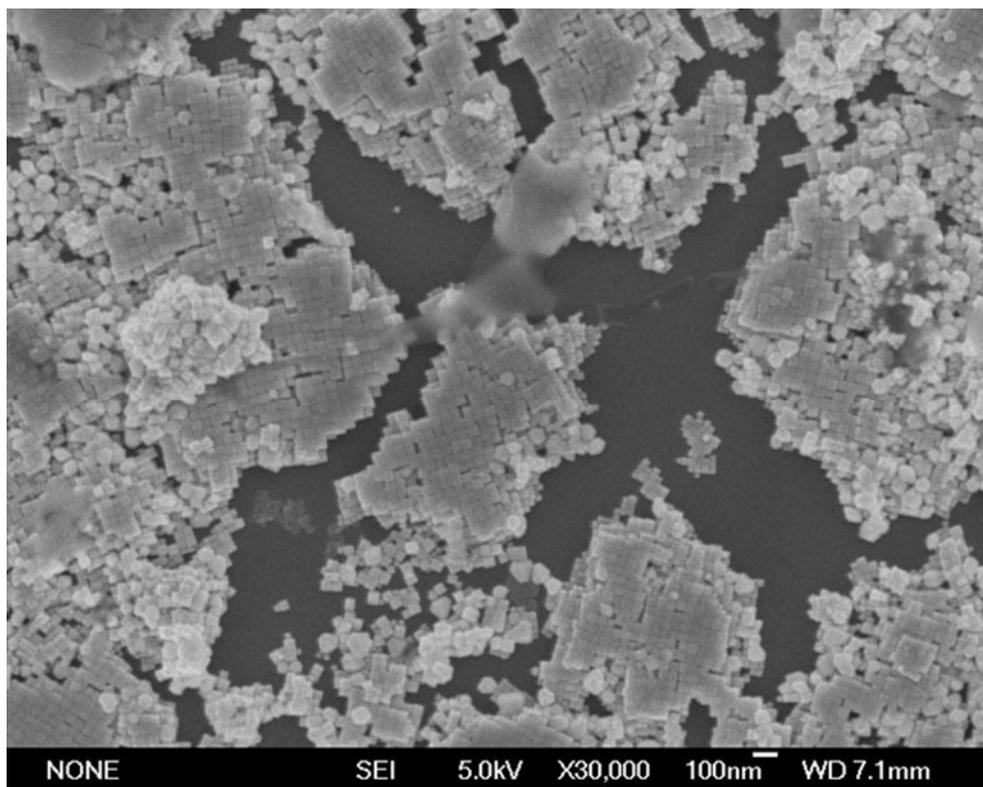
**Materials.** Palladium nitrate dihydrate ( $\text{PdNO}_3 \cdot 2\text{H}_2\text{O}$ ), sodium ascorbate (99%), ethylene glycol (EG, A.R.) and cetyltrimethylammonium bromide (CTAB, 99%) were obtained from Sinopharm Chemical Reagent Co. Ltd. All the chemicals were introduced as purchased without further purification. All aqueous solutions of  $\text{PdNO}_3$ , CTAB, and sodium ascorbate were freshly prepared before use.

**Synthesis of Pd Nanocubes.** CTAB (0.1820 g) and sodium ascorbate (0.0099 g) were dissolved in 15 mL of deionized water in a 50-mL vial. The vial was put in a 50 °C water bath under magnetic stirring.  $\text{PdNO}_3 \cdot 2\text{H}_2\text{O}$  (0.0108 g) was dissolved in 5 mL of deionized water and the solution was rapidly added into the vial. Allow the mixed solution to react for 30 min at 50 °C under magnetic stirring. Then the product was collected by centrifugation.

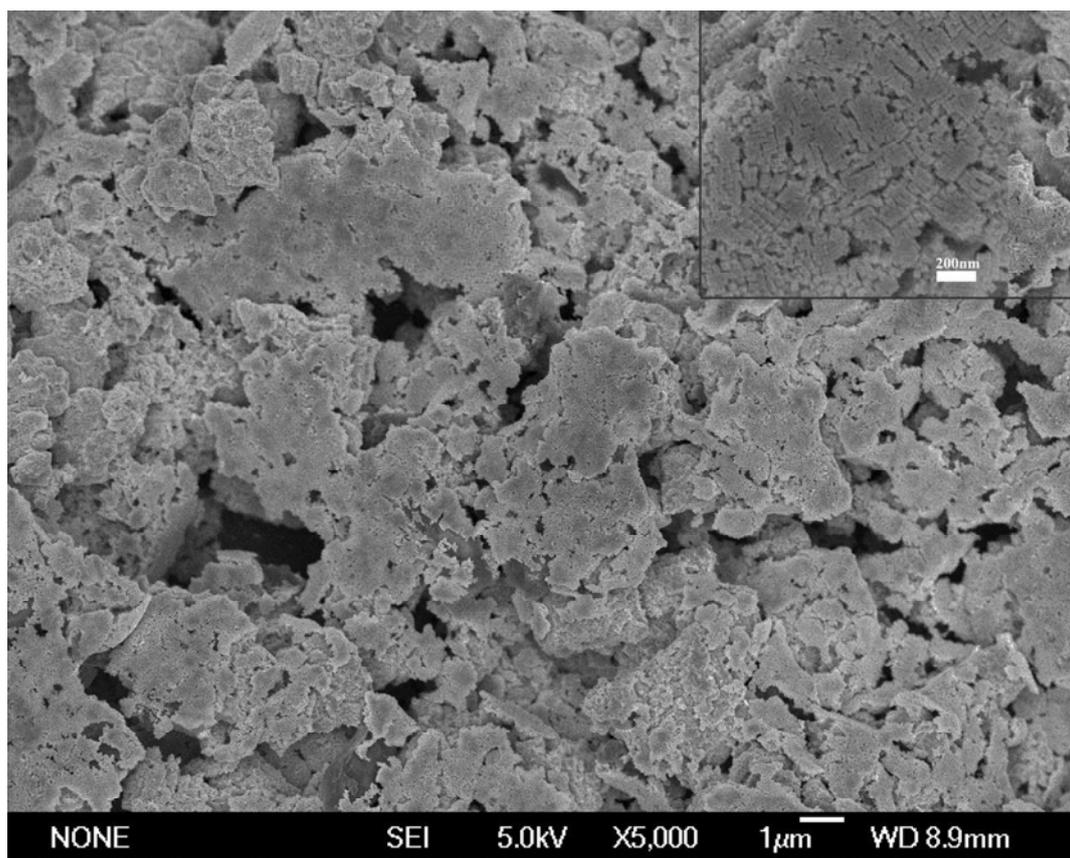
**Self-assembly of Pd Nanocubes.** 1 mL of the reaction mixture was centrifuged at 16000 rpm for 15 min. The supernatant solution was discarded. Then, the solid residue was re-dispersed in 50  $\mu\text{L}$  of 25 mM aqueous CTAB solution at 50 °C by sonication. The concentration of Pd nanocubes was about  $2 \times 10^{13} \text{ mL}^{-1}$ . 25  $\mu\text{L}$  of this solution was dropped on a silicon wafer ( $4 \times 4 \text{ mm}^2$ ), which was put at the bottom of a 5 mL vial in advance. The vial was capped with a coverslip and kept at 25 °C for about 12 h to completely evaporate the solvent. Finally, the residual CTAB on the silicon wafer was removed by immersing the substrate in 5 ml of ethanol before the sample was characterized by scanning electron microscopy (SEM) and optical microscopy.



**Fig. S1.** HRTEM image of one of the corners of a Pd nanocube along the [100] zone axis.



**Fig. S2.** SEM image of the sample prepared at 50 °C. Quasi 2D square arrays with a few haphazard aggregates are obtained.



**Fig. S3.** SEM image of the sample prepared with 100 mM CTAB.