

Speedy fabrication of diameter-controlled Ag nanowires using glycerol under microwave irradiation conditions

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Experimental Section

Synthesis of Ag Nanowires

In a typical procedure, a clear solution was first formed by mixing 0.3 mmol sodium dodecyl sulfate (SDS) with 4 mL water in a 10 mL thick walled glass tube reactor. Afterwards, 0.3 mmol silver nitrate (AgNO_3) was added, and shook until the uniform turbid liquid with milk white color was formed. Then 4 mL glycerol was added dropwise to obtain colorless transparent liquid. After that, the thick walled glass tube reactor was sealed by a cap, and then was irradiated in a CEM Discover focused MW synthesis system maintaining a temperature of 100 °C (monitored by a built-in infrared sensor) for 1 min with a maximum pressure of 280 psi. The resulting precipitated Ag samples were then washed several times with water to remove organics. Similar experiments were carried out by varying the content of reactant, temperature and reaction time. Control experiment under conventional heating condition was also conducted in a stainless steel autoclave with Teflon liner, and heated in oven at 100 °C and 150 °C for 60 min, the same temperature reached in the MW system.

Evaluation of catalytic activity of as-prepared Ag samples

Catalytic reduction experiments of 4-nitrophenol were performed to evaluate the catalytic efficiency and reusability of as-prepared Ag samples. In a typical experiment, a mixture of 19.5 mL water, 0.5 mL 10 mM 4-nitrophenol, and 0.125 mol NaBH_4 was first prepared in a 40 mL vial at room temperature, and then 0.075 mol Ag samples was added with magnetic stirring. After reaction, the reaction mixture was filtered by a Millipore syringe driven filter to get clear solution for UV/Vis spectroscopy (Agilent Hewlett-Packard, 8453, USA) analysis. For comparison, the control experiment was also carried out under similar conditions without Ag samples.

The durability of the Ag nanoparticles catalytic activity was evaluated using repeated experiments of 4-nitrophenol reduction. In this experiment, the 4-nitrophenol concentration of the reacted solution was measured after 1 min of addition. In the beginning of each experimental cycle, 0.5 mL 10 mM 4-nitrophenol and 0.125 mol NaBH₄ were added into the reactor to compensate.

To investigate the reduction mechanism using glycerol, the oxidation product of glycerol was determined. After reaction, the mixture was extracted with ethyl acetate, and the products identified by gas chromatography-mass spectrometry.

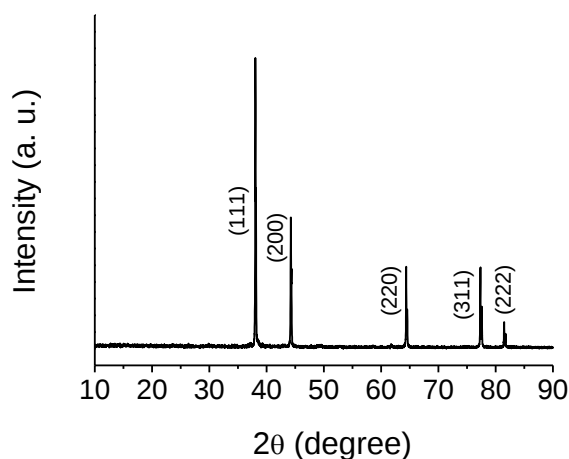


Fig. S1. XRD pattern of as-prepared Ag Nanowires

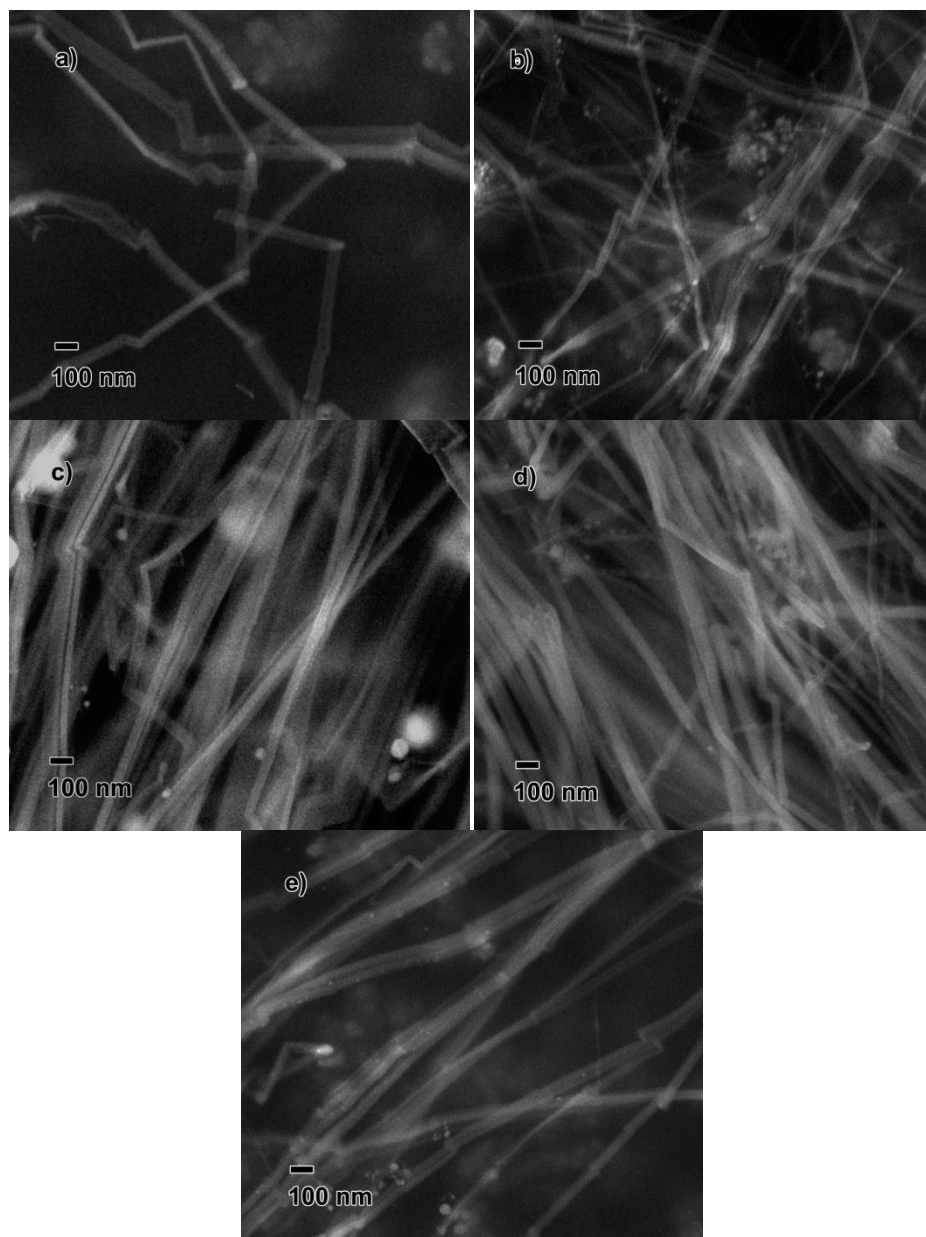


Fig. S2. SEM images of the as-prepared Ag samples. Reaction condition: a) 0.3 mmol AgNO_3 , 0.6 mmol SDS; 4 mL glycerol and 4 ml water; b) 0.1 mmol AgNO_3 , 0.3 mmol SDS, 4 mL glycerol and 4 ml water; c) 0.3 mmol AgNO_3 , 0.3 mmol SDS, 2 mL glycerol and 6 ml water; d) 0.3 mmol AgNO_3 ; 0.3 mmol SDS; 6 mL glycerol and 2 ml water; e) 0.3mmol AgNO_3 ; 0.3 mmol SDS; 7 mL glycerol and 1 ml water.

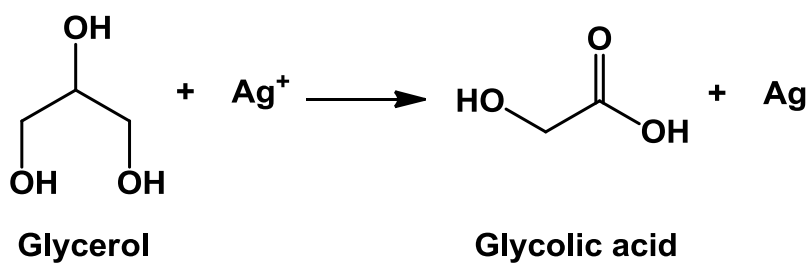


Fig. S3. The plausible reduction of silver salts during the the formation of Ag nanowires

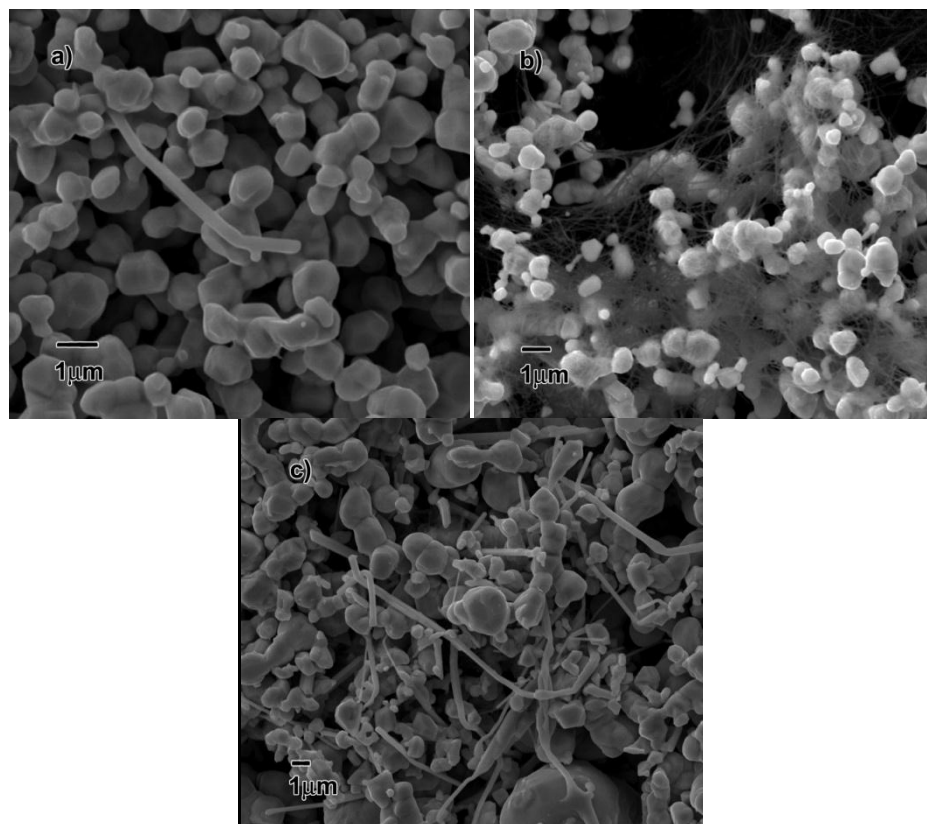


Fig. S4. SEM images of prepared Ag samples. a) 180 °C, 5 min, no SDS; b) 180 °C, 5 min, 0.3 mmol SDS; c) 100 °C, 60 min, 0.3 mmol SDS

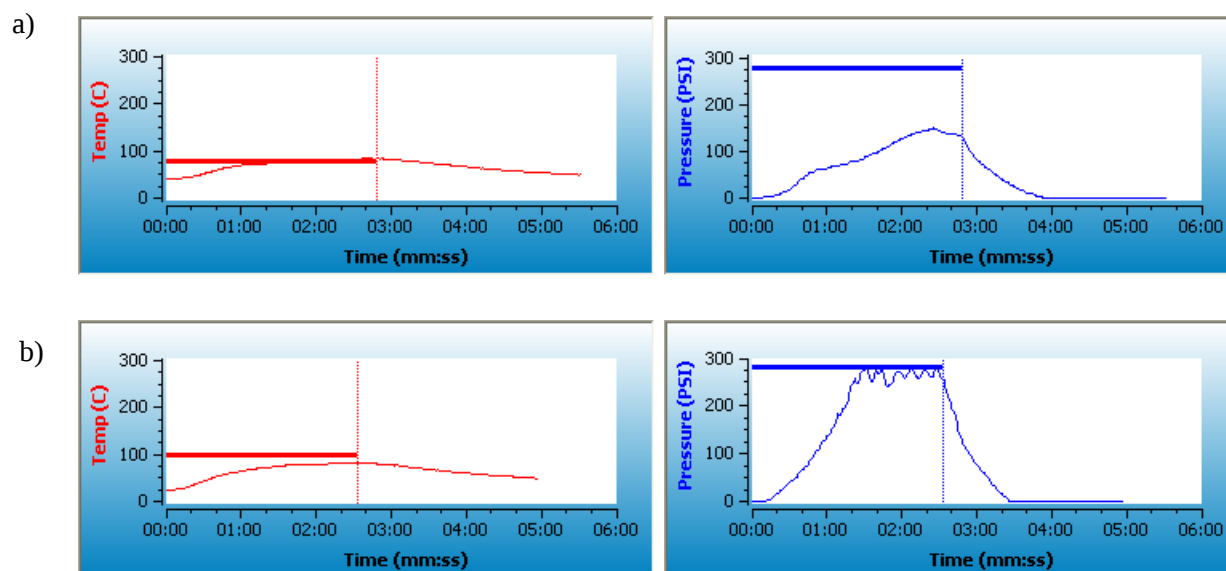


Fig. S5. Reaction profile of the system irradiated by 120 W MW at 100 °C for 1 min. a) with and b) without stirring.

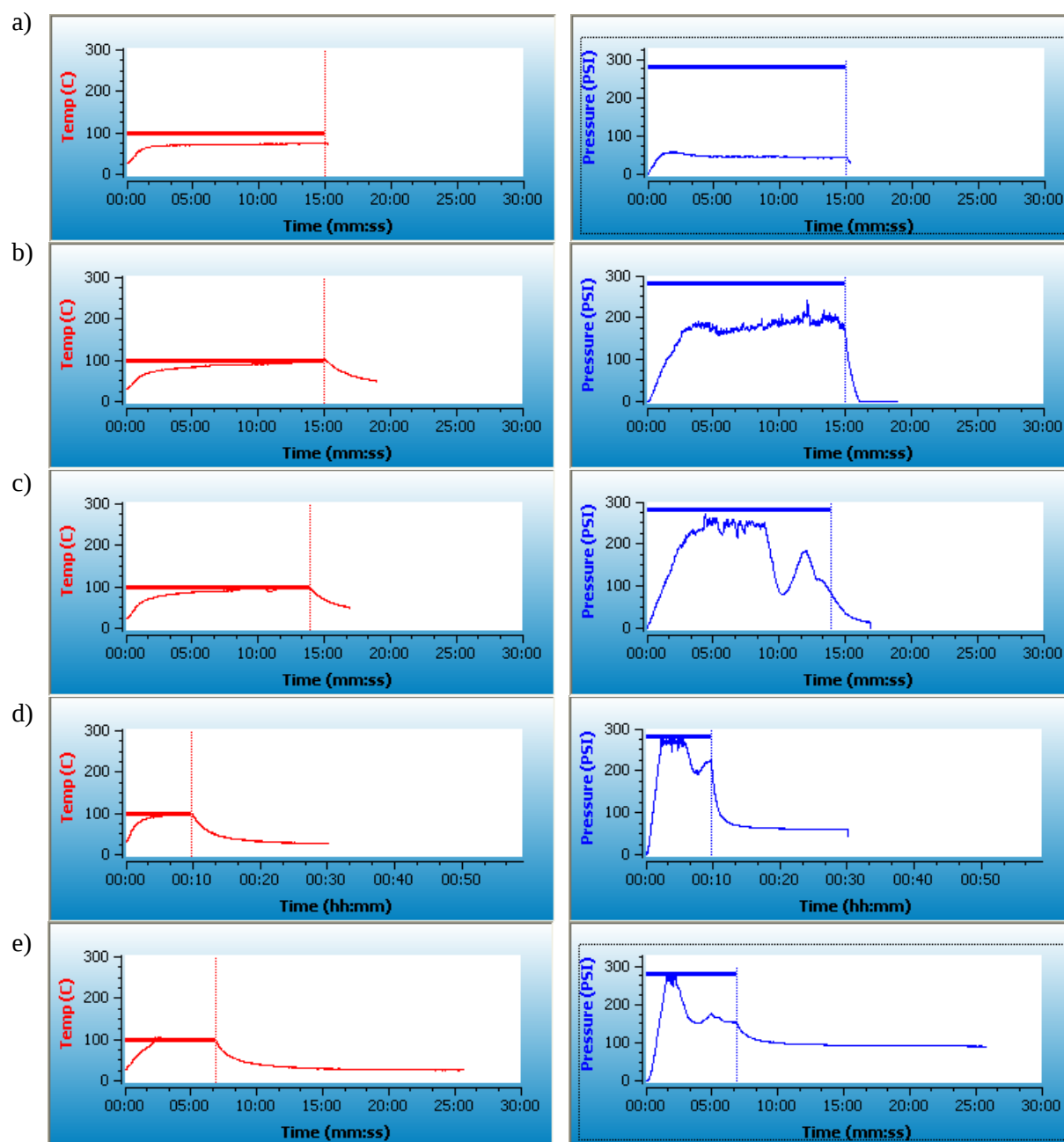


Fig. S6. Reaction profile of system with different AgNO_3 amount irradiated by 100 W MW. a) 0.06 mmol ; b) 0.1 mmol; c) 0.3 mmol; d) 0.6 mmol; e) 1.2 mmol.

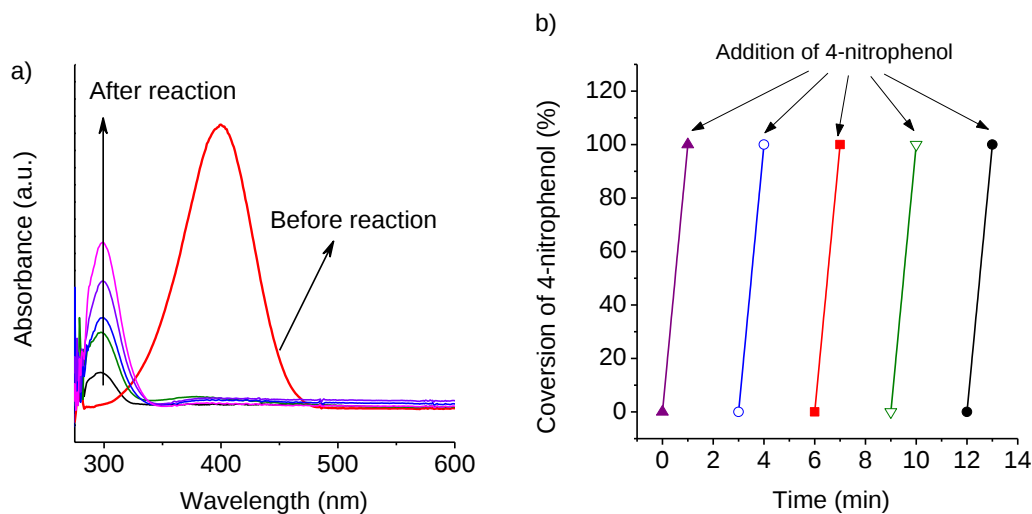


Fig. S7. Catalytic reduction of 4-nitrophenol. a) UV-vis spectra of solutions of reaction mixture in the presence of as-prepared Ag nanowires; b) catalytic activity and recyclability of as-prepared Ag nanowires.

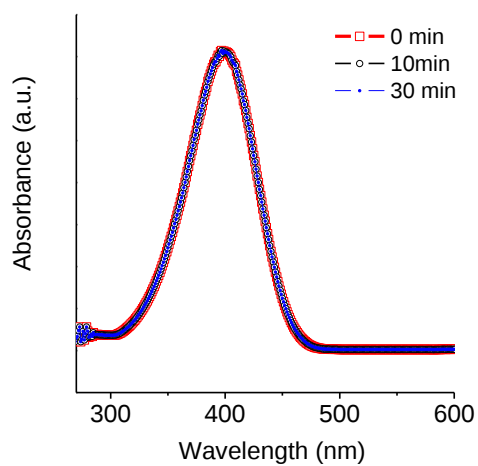


Fig. S8. UV-vis spectra of 4-nitrophenol solutions without catalyst.