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Highly dispersed Ag nanoparticles embedded in alumina nanobelts as excellent surface-enhanced Raman scattering substrates

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Experimental Section

Materials Synthesis. The one-dimensional Ag/Al₂O₃ nanocomposites as surfaceenhanced Raman scattering substrates were fabricated by a multistep method. All the reagents used were AR grade and were not purified further. DI water was used throughout the experiment. Firstly, 5 mL silver nitrate aqueous solution (45 mM) was poured slowly into 50 mL melamine aqueous solution (15 mM) under magnetic stirring to obtain milky intermediate products at room temperature. The clean glass substrates were drop-coated with the as-prepared intermediate products to form uniform thin liquid membranes. The above glass substrates, after drying in 45 °C oven, were put into deposition chamber of atomic layer deposition (ALD) to deposit aluminum oxide layers on the surface of intermediate products. The final products were produced by heating as-prepared ALD products in nitrogen atmosphere at 400 °C for 2 h with a ramping rate of 2 °C/min. The control experiment without undergoing ALD pretreatment was conducted under the same conditions except skipping above ALD step.

ALD Procedure. The ALD process was carried out in a hot-wall flow-type ALD reactor. The trimethylaluminum (TMA, Al(CH₃)₃) and deionized water as precursors were alternatively pumped into the reaction chamber (maintained at 160 °C) using high purity N₂. Each substrate was deposited 10 cycles. Typically, one complete reaction cycle took ~40 s, and consisted of four steps: (1) TMA reactant exposure time, 30 ms; (2) N₂ gas purging time, 20 s; (3) water vapor exposure time, 70 ms; and (4) N₂ gas purging time, 20 s.

SERS Measurements. SERS experiments were performed on Renishaw Invia Reflex Laser Raman spectrometer with 514 nm laser excitation based on R6G as a probe molecule. SERS spectra were acquired with a ×50 objective (NA = 0.75), exposure time 10 s and one cumulation. Laser power was set between 0.01 and 0.1 mW to maximize signal while reducing influence of dye photobleaching. A series of R6G standard solutions with different concentrations from 1.0×10^{-1} M to 1.0×10^{-9} M were prepared with stepwise dilution method. SERS mapping were conducted under the conditions of ×50 objective (NA = 0.75), exposure time 1 s and laser power 0.05 mW. The 611 cm⁻¹ Raman peak was selected basing on signal intensities to baseline analytical method.

Materials Characterization. The morphologies and microstructures of the products were examined by a field-emission scanning electron microscope (FESEM; HITACHI, S4800, operated at 10 kV) and a high-resolution transmission electron microscope (HRTEM; JEOL, JEM-2100, 200 kV). Thermogravimetry analysis (TGA) was carried out on samples placed in corundum crucibles with a heating rate of 5

K/min (TA, Q600 apparatus, USA).



Fig. S1 Low-magnified SEM image of AgMA CPs









Fig. S3 TG and DTG curves of the as-prepared AgMA CPs calcined in nitrogen

Fig. S4 (a) TEM image and (b) the Ag nanoparticles size (*D*) distribution statistic analysis graph of the as-prepared Ag/Al_2O_3 nanobelts; (c) SEM image of the products without ALD pretreatment.



Fig. S5 Raman spectra of R6G molecules with different concentrations on the products without ALD pretreatment.