

COMMUNICATION

Facile Synthesis and Phase Transition of V₂O₃ Nanobelts

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Experimental details and discussion

1. Synthesis of V₂O₅ nanobelts

0.75 mmol of ammonium metavanadate (NH₄VO₃), 40 mL of deionized water, and 3 mL of H₂O₂ (30 wt %) were mixed and stirred to dissolve the solid powder completely. The obtained solution was sealed in a 100 mL Teflon vessel and heated at 220 °C for 24 h in an oven. The obtained products were centrifuged, washed with deionized water for three times, dried at 70 °C in air and collected for further use.

2. Synthesis of V₂O₃ nanobelts

The dried V₂O₅ nanobelts were dispersed in ethanol by brief sonication and then were spin-coated onto 300 nm SiO₂/Si substrates at 3000 r.p.m. for 1 min. Then the substrates were loaded into a home-built chemical vapor deposition (CVD) furnace with 1 inch quartz tube. After purging the system with Ar for 20 min, the furnace was heated up to 450 °C with 200 sccm Ar and 50 sccm H₂. When the temperature reached 450 °C, sulfur was heated by a heating belt with an individual temperature controller at ~150 °C. After 1 h, the heating belt for sulfur was removed immediately and the furnace was cooled to room temperature. The V₂O₃ nanobelts can also be synthesized in bulk by using the as-collected V₂O₅ nanobelts in powders as the starting materials under the same conditions as mentioned above.

3. Device fabrication and variable- temperature electrical and magnetic measurements

Source and drain electrodes were fabricated on V₂O₃ nanobelts sitting on 300 nm SiO₂/Si substrates by electron-beam lithography followed by electron-beam evaporation of 5 nm Ti/50 nm Au. After the lift-off, the obtained devices were annealed in vacuum at 400 °C for 30 min. The variable-temperature electrical measurements were carried out in a cryogenic probe station (Janis ST-500) at a pressure of ~10⁻⁶ mbar with Agilent B1500A semiconductor parameter analyzer. VT-magnetic susceptibility was measured by

MPMS (SQUID) VSM with an applied magnetic field of 1T. The range of temperature was from 80 to 300 K.

4. The magnetic properties of other vanadium oxide phases

For V_2O_5 , there is no magnetism due to its zero magnetic moment. For VO_2 , it was monoclinic below the transition temperature ($T_c \sim 340$ K) and the V^{+4} ions tend to be V-V pairs and form covalent-type bonds. The V-V pairs shared the d electrons and therefore VO_2 was diamagnetic below T_c . At above T_c , VO_2 was metallic and the V-V pairs were replaced by V-O pairs and form metallic bonds which may release the available d electrons.¹ So VO_2 was paramagnetic above T_c .² For VO, the crystal structure of VO was like rock salt and had a large number of cation and anion vacancies. The stoichiometric ratio of VO was wide, extending over $0.75 \leq x \leq 1.30$.³ The magnetic properties is related to the cation vacancies and temperature.⁴ VO_x might be antiferromagnetic when $x \geq 1.147$ and paramagnetic between 77 and 300 K.⁵

5. Variable-temperature Raman spectroscopy measurements

VT-Raman spectra were collected with Horiba-Jobin-Yvon Raman system using 514 nm excitation with a laser power of 4 mW and the integration time was 20 s with 5 times accumulation. Raman mapping images were taken with a step of 0.8 μm and the integration time was 0.8 s at each spot. The Si peak at 520 cm^{-1} was used for calibration in the experiments.

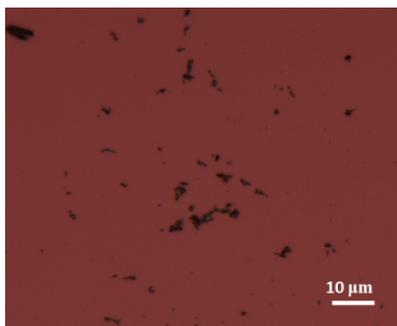


Fig. S1. Optical image of V_2O_3 nanobelts prepared by reducing V_2O_5 nanobelts with pure H_2 .

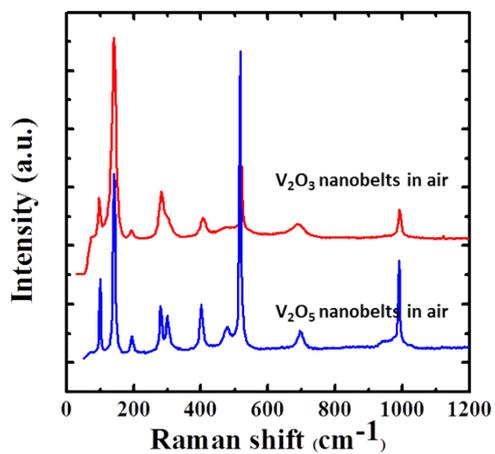


Fig. S2. Raman spectra of V₂O₃ and V₂O₅ nanobelts in air.

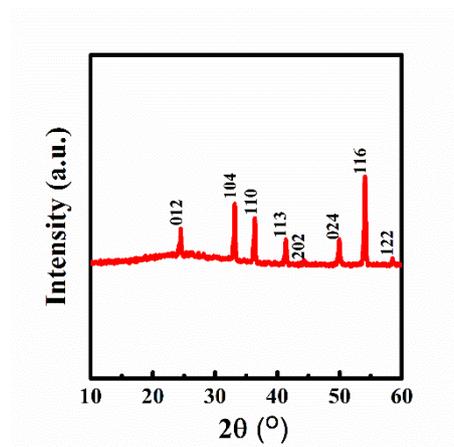


Fig. S3 XRD pattern of the commercially purchased V₂O₃ powder. No other VO_x phase was detected.

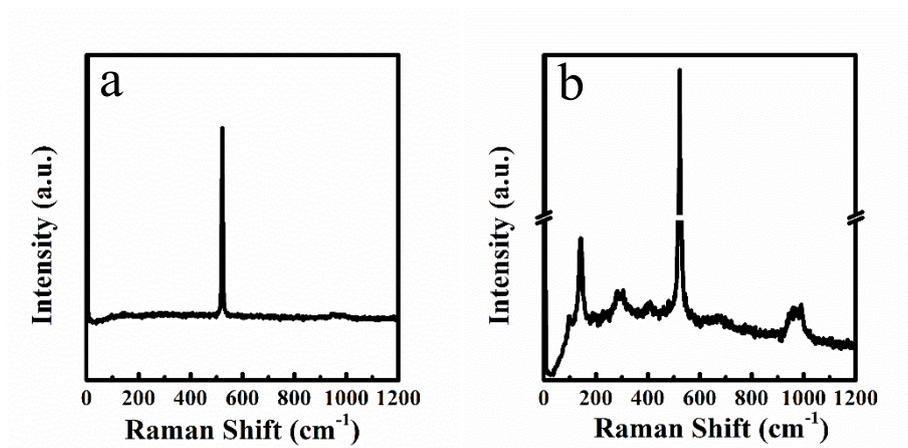


Fig. S4 Raman spectra of V_2O_3 powders in vacuum (a) and in air (b). No Raman peak of V_2O_3 appeared in vacuum and the typical peaks of V_2O_5 were detected in air due to the oxidation of V_2O_3 .

Reference

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