Facile Synthesis and Phase Transition of V$_2$O$_3$ Nanobelts

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Experimental details and discussion

1. Synthesis of V$_2$O$_5$ nanobelts

0.75 mmol of ammonium metavanadate (NH$_4$VO$_3$), 40 mL of deionized water, and 3 mL of H$_2$O$_2$ (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 ºC for 24 h in an oven. The obtained products were centrifuged, washed with deionized water for three times, dried at 70 ºC in air and collected for further use.

2. Synthesis of V$_2$O$_3$ nanobelts

The dried V$_2$O$_5$ nanobelts were dispersed in ethanol by brief sonication and then were spin-coated onto 300 nm SiO$_2$/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 ºC with 200 sccm Ar and 50 sccm H$_2$. When the temperature reached 450 ºC, sulfur was heated by a heating belt with an individual temperature controller at ~150 ºC. After 1 h, the heating belt for sulfur was removed immediately and the furnace was cooled to room temperature. The V$_2$O$_3$ nanobelts can also be synthesized in bulk by using the as-collected V$_2$O$_5$ 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 V$_2$O$_3$ nanobelts sitting on 300 nm SiO$_2$/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 ºC 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$_2$O$_5$, there is no magnetism due to its zero magnetic moment. For VO$_2$, it was monoclinic below the transition temperature ($T_c$ ~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 $T_c$. 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 $\mu$m 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.

![Optical image of V$_2$O$_3$ nanobelts prepared by reducing V$_2$O$_5$ nanobelts with pure H$_2$.](image-url)
Fig. S2. Raman spectra of $V_2O_3$ and $V_2O_5$ nanobelts in air.

Fig. S3 XRD pattern of the commercially purchased $V_2O_3$ powder. No other VO$_x$ phase was detected.
Fig. S4 Raman spectra of V₂O₃ powders in vacuum (a) and in air (b). No Raman peak of V₂O₃ appeared in vacuum and the typical peaks of V₂O₅ were detected in air due to the oxidation of V₂O₃.

Reference