Chinese Chemical Letters Vol. 16, No. 2, pp 249-252, 2005
http://www.imm.ac.cn/journal/ccl.html

Preparation of Single Crystalline V_6O_{13} Nanobelts

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Abstract: Ultralong beltlike nanostructures was successfully synthesized for V_6O_{13} crystal by a hydrothermal route. The products are characterized by means of X-ray powder diffraction, transmission electron microscopy and high-resolution transmission electron microscopy. The experimental results give the evidence that the V_6O_{13} nanobelts are pure, structurally uniform and single crystalline, with typical widths of 50 to 300 nm and lengths of up to a few millimeters.

Keyword: V_6O_{13} crystal, nanobelt, hydrothermal route.

V_6O_{13} crystal is now widely used as cathode active material[1~4], self-switching electrochemical cells[5~6] and uncooled infrared sensor[7]. The current studies of V_6O_{13} crystal have been focused on two-dimensional films and zero-dimensional particles, which can be readily synthesized with various well-established techniques such as sputtering[8~9], thermal evaporation[10], PLD[11~12], PECVD[13~14] (for films), and laser pyrolysis[15], thermal decomposition[1] (for particles). In contrast, Investigations of wirelike V_6O_{13} nanostructures are cumbersome because of the unavailability of nanowire structures. Recently, a group of distinctly different semi-conducting oxide nanostructures was reported[16] which has a rectangular cross section, in correspondence to a belt-like morphology. The as-prepared oxides with the nanobelt morphology cover cations with different valence states and materials with different crystallographic structures, and it seems to be a common structural characteristic for the family of semiconducting oxides. The synthesis of the semi-conducting oxides with belt-like morphology is based on thermal evaporation of the corresponding oxide powders under controlled conditions without the presence of catalyst. Here, we report a novel V_6O_{13} crystal with a belt-like morphology by a hydrothermal route.

Experimental

Analytically pure V_2O_5 (10mmol) and KI (3.3 mmol) were put into a 200 mL teflon liner autoclave, then distilled water was used to fill the tank to 80% of the total volume. The autoclave was maintained at 250°C for 24 h and then allowed to cool to room temperature.

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The product was filtrated and washed with distilled water and absolute ethanol several times. After drying in vacuum at 80°C for 4 h, the corresponding product was obtained.

The phases and the crystallographic structure of the product was determined by X-ray powder diffraction (XRD), using Japan Rigaku D / max-B X-ray diffraction with Cu Kα radiation (λ=1.5418 Å). To examine the morphology and particle size of the product, the transmission electron microscope (TEM) image was taken on a Hitachi model H-800, using an accelerating voltage of 200 KV. The direct observation of a single nanobelt was carried out by high-resolution transmission electron microscopy (HRTEM) on a JEOL-2010 transmission electron microscope.

Results and Discussion

Figure 1 shows the XRD patterns of the as-prepared product. All peaks could be indexed to the monoclinic structured V_6O_{13} crystal with lattice constants of a = 11.928Å, b = 3.701 Å and c = 10.135Å, which is close to the reported data (JCPDS cards 43-1050).

Figure 2a shows that the morphologies of V_6O_{13} crystal are the nanobelts with straight and twisted shape. Each nanobelt has a uniform width along its entire length, and the typical widths of the nanobelts are in the range of 50 to 300 nm. No particle was observed at the ends of the nanobelts. Meanwhile, the ripplelike contrast was observed in the TEM image (as indicated by arrows), which is similar to the literature reported[16]. It was thought that is due to strain resulting from the bending of the belt. **Figure 2b** shows the TEM image of a single V_6O_{13} nanobelt with twisted shape. It reveals that the typical width and width-to-thickness ratios of the nanobelt are 150 nm and 6, respectively. The ED pattern of single V_6O_{13} nanobelt shows single crystallite character (**Figure 2c**). In this pattern, the diffraction spots correspond to the (200), (005) and (020) lattice planes, respectively, of monoclinic structured V_6O_{13} crystal with an interplanar spacing of about 0.549 nm, 0.201 nm and 0.186 nm.

However, the strongest evidence is the direct observation of a single V_6O_{13} nanobelt with the ripplelike contrast by high-resolution transmission electron microscopy as shown in **Figure 3a**. The HRTEM images under higher magnification of selected area are shown in **Figure 3b** and **3c**, they reveal the fabrication of the V_6O_{13} nanobelt. **Figure 3b** shows HRTEM image verifies that the surface of the V_6O_{13} nanobelt consists mainly of domains of single crystal, since the straight lattice arrangement is observed. This image reveals that the lattice fringes with lattice spacing around 0.37 nm are parallel to the axis of the nanobelt. This lattice arrangement maybe corresponds to (010) lattice plane, since the corresponding lattice spacing (0.37 nm) is close to the twofold interplanar spacing of (020) lattice plane (as indicated in **Figure 1**, d = 0.184 nm). **Figure 3c** shows the lattice arrangement of the ripplelike point is similar to one of surface of belt, there is only a straight lattice arrangement with interplanar spacing around 0.37 nm is observed, and is not found anther lattice plane occurs, which verifies that the ripplelike contrast is due to strain resulting from the bending of the belt.

Figure 1 The X-ray diffraction patterns of the as-prepared product

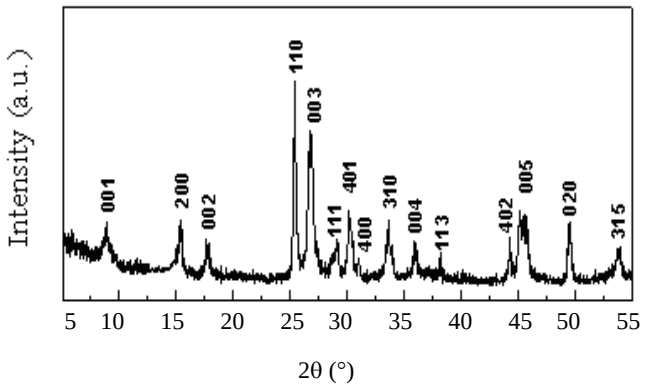


Figure 2 **(**a), (b) TEM images and (c) ED image of as-prepared product

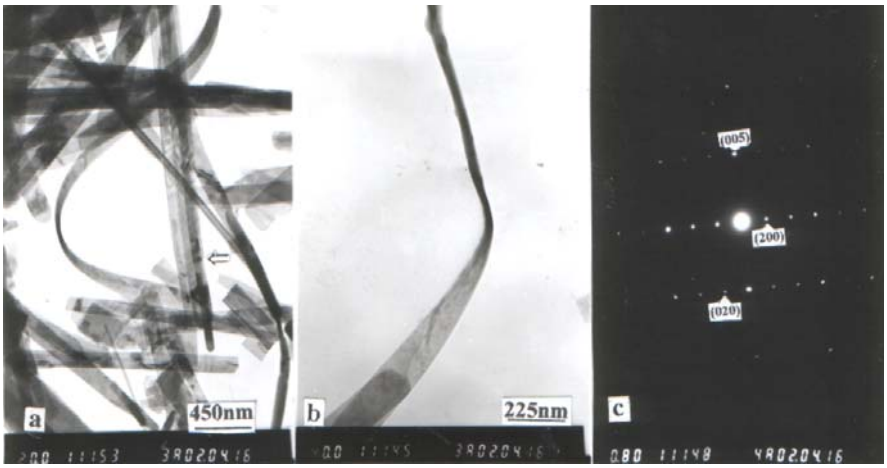
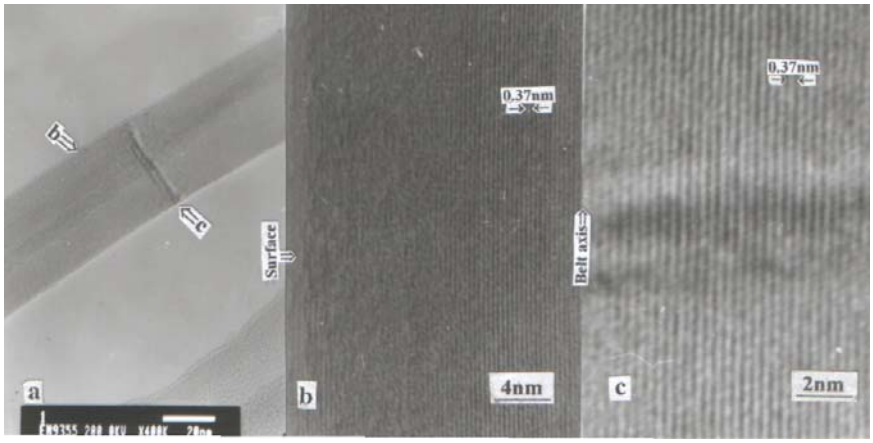


Figure 3 HRTEM images of a single nanobelt with the ripplelike contrast



Conclusion

In conclusion, ultralong beltlike nanostructures was successfully synthesized for V_6O_{13} crystal by a hydrothermal route. The as-prepared V_6O_{13} nanobelts are pure, structurally uniform and single crystalline, with typical widths of 50 to 300 nm and lengths of up to a few millimeters. Meanwhile, hydrothermal conversion perhaps affords a simple route to synthesize the single crystalline V_6O_{13} nanobelts.

Acknowledgment

This work was supported by the National Natural Science Foundation of China (No 50072028).

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Received 18 November, 2003