I. INTRODUCTION

Low-dimensional structures such as nanoparticles, nanowires, and nanotubes with high aspect ratios have attracted much attention because of their high potential applications in the fabrication of electronic devices, chemical sensors, etc. [1-3]. It is well known that physical, chemical, and electronic properties of materials are strongly dependent on their shapes when their sizes are reduced to micrometer- and nanometer-scale dimensions. However, synthesis of low-dimensional structures with uniform shapes and good organization of them into ordered three-dimensional (3D) nanostructures are still challenges and require precise control over synthesis conditions, such as temperature, acidity, and redox potentials. For these reasons, direct fabrication of complex micro-/nanostructured materials is desirable and still remains a challenge in areas of materials science.

The key to producing low-dimensional materials lies in the fact that atoms or other building blocks rationally assemble into a structure with reduced dimensions [4]. Various strategies have been developed to fabricate low-dimensional materials including vapor-liquid-solid (VLS) growth method, solution-liquid-solid (SLS) growth method, oxide-assisted growth (OAG), physical evaporating process, and chemical vapor deposition (CVD) methods. Many of them were reviewed by Rao et al. [5] and Xia et al. [6]. Although these strategies have been developed for synthesis of low-dimensional materials, they typically suffer from the requirements of elevated temperature, special reaction conditions, sophisticated apparatus, tedious procedures, and catalysts or templates. Therefore, an easy and effective methodology for synthesis of low-dimensional materials would be much preferred by nanoscience researchers. Recently, low-temperature solution-phase synthesis has been proposed as an attractive approach toward low-dimensional materials of high crystalline. In particular, the wet chemistry synthesis through solution-phase reaction has been considered as one of the most promising route in terms of cost, yield, and the potential for large-scale production [7,8]. For instance, the hydrothermal fabrication, a typical wet chemistry method, shows certain noticeable advantages including moderate reaction temperature (100-300 °C), simple operating procedure, no catalyst impurities, and relatively fast reaction rate [9,10]. It also could offer a promising approach to obtain highly ordered micro- or nano-structure patterns with desirable size and shape uniformity through self assembly [11,12].

Vanadium oxides and their derivative compounds have attracted considerable interest due to their redox-activity and layered structures, which allow the insertion of various intercalation species [11,13-15]. Moreover, they also have extensive applications in many chemical processes due to their special structures, acidity, ion-exchange, separation, catalysis, and energy storage in secondary batteries [16-19]. Recently, the utiliza-
tion of vanadium pentoxide-based compounds has been a subject of intensive studies. Several kinds of low
dimensional vanadium oxides have been produced under mild hydrothermal conditions. Yu's group has reported
the hydrothermal synthesis of NaV$_6$O$_{15}$ nanowires and Na$_2$V$_6$O$_{16}$·3H$_2$O nanobelts without the use of catalyst
or templates [20,21]. Qian and his coworkers have successfully prepared the CaV$_6$O$_{16}$·3H$_2$O nanobelts via a
mild hydrothermal process [22]. Uniform β-AgVO$_3$ single crystalline nanobelts with length up to 100 µm were
synthesized via an easy hydrothermal method by Liu et al. [23]. However, the synthesis of low-dimensional
Sr-V-O systems is lacking.

In this work, low-dimensional Sr-V-O materials were synthesized via a mild hydrothermal process. The
preparation of uniform dandelion-like micro-crystallite of well-arranged β-SrV$_2$O$_6$ fibers under mild hydrother-
amal conditions, and their fundamental structural features characterized by means of powder X-ray diffrac-
tion on a Rigaku D/Max 2400 diffractometer using monochromatic Cu Kα radiation (λ=1.54 Å). The X-
ray photoelectron spectrum (XPS) analysis was acquired on a VGESCALAB MKII X-ray photoelectron
spectrometer with an excitation source Mg Kα. The morphology and microstructural features of the as-
products were studied with a field emission type scanning electron microscopy (SEM). Transmission electron
microscopy (TEM) studies were performed using a Hitachi H-9000NA microscope operating at 250 kV. Spec-
imens for TEM study were prepared by ultrasonic dispersion of the products in alcohol, and then a few
drops of this suspension containing fibers peeled from dandelion-like micro-crystallites were put on a holey
carbon film supported on a copper grid.

II. EXPERIMENTS

The commercial chemical materials used in our synthesis experiments include Sr(NO$_3$)$_2$ (98%,
Aldrich), V$_2$O$_5$ (99.6%, Aldrich), and adipic acid (HOOC(CH$_2$)$_4$COOH), (99%, AR, China). The
starting materials are used as received without further purification process. The hydrothermal reactions
were carried out in the stainless-steel autoclaves. The dandelion-like micro-crystallites were obtained in
samples fabricated as following: 2.010 g of Sr(NO$_3$)$_2$, 0.863 g V$_2$O$_5$, and 2.775 g adipic acid were mixed
into an autoclave. After adding of 20 g deionized water, the reaction mixture with the molar ratio
(Sr(NO$_3$)$_2$·V$_2$O$_5$×adipic acid·H$_2$O=2.0:1.0:4.0:235.1) was rigorously stirred for 30 min and then treated
with ultrasound for 20 min. Subsequently, the mixture was put into reaction Teflon-lined vessels of 50 mL
capacity and sealed in stainless-steel autoclaves, and then kept in an oven at 220 °C for 60 h. The autoclave
was removed from the oven and was left to cool slowly at room temperature. The pale yellow dandelion-like
particles were isolated from the final products by filtering with deionized water. The as-products were
thoroughly washed with deionized water and dried in air at 100 °C for 2 h.

We also performed a series of similar synthesis processes by adjusting the chemical composition ratio of
the reaction mixture and the hydrothermal treating temperature. The products showed certain variations in
both morphology and microstructural features. Seven representative synthesis processes and their experimen-
tal results are selected and tabulated in Table I.

All products were analyzed by powder X-ray diffraction on a Rigaku D/Max 2400 diffractometer using
monochromatic Cu Kα radiation (λ=1.54 Å). The X-ray diffraction pattern of the as-
synthesized Sr-V-O materials. The main diffraction peaks in this XRD pattern can be well indexed by an ortho-
borhombic cell with the lattice parameter $a=9.694$ Å, $b=3.687$ Å, and $c=12.570$ Å (space group of Pnma),
which is in good agreement with the reported or-

<table>
<thead>
<tr>
<th>Composition Sr:V:AA</th>
<th>$T/{^\circ}C$</th>
<th>Mineralizer</th>
<th>Products</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0:1.0:4.0</td>
<td>220</td>
<td>Adipic acid</td>
<td>–</td>
</tr>
<tr>
<td>1.5:1.0:4.0</td>
<td>220</td>
<td>Adipic acid</td>
<td>β-SrV$_2$O$_6$</td>
</tr>
<tr>
<td>2.0:1.0:4.0</td>
<td>220</td>
<td>Adipic acid</td>
<td>β-SrV$_2$O$_6$</td>
</tr>
<tr>
<td>2.0:1.0:4.0</td>
<td>160</td>
<td>Adipic acid</td>
<td>–</td>
</tr>
<tr>
<td>2.0:1.0:4.0</td>
<td>180</td>
<td>Adipic acid</td>
<td>Belt-like β-SrV$_2$O$_6$</td>
</tr>
<tr>
<td>2.0:1.0:4.0</td>
<td>240</td>
<td>Adipic acid</td>
<td>β-SrV$_2$O$_6$+impurities</td>
</tr>
<tr>
<td>2.0:1.0</td>
<td>220</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

a: Adipic acid.
b: Dandelion-like micro-crystallites β-SrV$_2$O$_6$.
Thorhombic β-SrV$_2$O$_6$ phase (JCPDS 85-2440). The presence of impurity phase can also be recognized in most samples as illustrated by an asterisk in the XRD pattern of Fig.1. Further analysis suggests that the small amounts of impurities are likely to be the VO$_x$ as discussed in the following context based on energy dispersive X-ray spectroscopy (EDXS) data. The strong intensity of the (004) peak, in comparison with that in the standard pattern, is considered to be arising from the preferentially oriental texture of the β-SrV$_2$O$_6$ crystals in the present products. Actually, when the samples were sufficiently ground before XRD measurements, the intensity of all diffraction peaks are in good agreement with the standard pattern (JCPDS 85-2440).

B. X-ray photoelectron spectrum

The XPS measurements were carried out to detect the chemical composition and valence states of vanadium ions in the as-synthesized products. Figure 2(a) displays a survey spectrum demonstrating the presence of Sr, V, and O elements. The C1s peak actually arises from the glue which was used to fix the powder samples during measurements. Figure 2(b) shows a high-resolution XPS spectrum for V2p$_{3/2}$ and V2p$_{1/2}$ excitations demonstrating the existence of typical peaks with the binding energies of V2p$_{3/2}$ at 517.55 eV, V2p$_{1/2}$ at 525.31 eV, and O1s at 530.15 eV. These results directly suggest that the V ion typically exhibits V$^{5+}$ state in our samples [24]. Quantification analysis of the peak intensities could yield a stoichiometry for Sr:V:O as 1:2.05:6.30 in the as-prepared samples, matching well the chemical formula SrV$_2$O$_6$. Moreover, we have also performed an inductively coupled plasma analysis on the samples, yielding a Sr/V ratio of 1:2 in the products.

C. Scanning electron microscopy

The microstructure and local chemical compositions of the samples were systematically studied with a SEM equipped with EDXS. Figure 3(a) shows a typical SEM image taken from a sample consisting of the dandelion-like micro-crystallites. It is clearly recognizable that these dandelion-like micro-crystallites have an average diameter of about 0.5 mm and are made up of well-organized microrod bundles. Figure 3 (b) displays a larger magnification image of a micro-crystallite, showing the closely packed structure and the uniform distribution of the microrods. These microrods in general have diameters ranging from 1 µm to 5 µm. Figure 3(c) shows a cross-section image of dandelion-like samples, illustrating an overview of the core structure and the microrod bundles. The figure clearly shows that all the rods are bound at one end and grow towards the outside of the cluster. The length of the rods is about several hundreds of micrometers. The average length/diameter ratio of the rods is estimated to be larger than 100. Figure 3 (e) and (f) display two EDXS spectra taken from the micro-rods and the core part, respectively, indicating that the core region consists mainly of V and O in sharp contrast with the results obtained for the rods. The noticeable differences in chemical compositions will be discussed in connection with the growth mechanism in the following text.

D. Transmission electron microscopy

The structure and growth direction of the single-crystal rod were further analyzed by TEM investiga-
tion. Figure 4 (a) and (b) show the bright field TEM images for two typical microrods and their corresponding electron diffraction patterns (insets), indicating that those microrods are highly crystallite in nature and can be well indexed by the known $\beta$-SrV$_2$O$_6$ orthorhombic phase. Moreover, based on the analysis of the electron diffraction, we obtain that the preferential growth direction of the microrods is along the $<010>$ crystallographic axis.

HREM observation was performed to reveal the microstructural features of the $\beta$-SrV$_2$O$_6$ microrods. Figure 4(c) shows an experimental HREM image taken along the [001] zone, illustrating the atomic structure at the end of a microrod. It is recognizable that the growth front goes along the $<010>$ direction, further confirming the results obtained from electron diffraction. The inset shows a Fast-Fourier Transformations (FFT) pattern from this image, which has the same symmetric properties as the pattern in Fig.4(b).

E. Crystallite growth mechanism

In order to investigate the effects of hydrothermal treating temperature and chemical concentration of reaction mixtures on structural properties of the products, we carried out a series of synthesis processes under slightly difference reaction conditions by changing reaction temperature, chemical composition ratio, and mineralizer, as shown in Table I. It was found that the dandelion-like micro-crystallite could be only achieved within a typical temperature range and under certain chemical compositions. By changing the chemical composition ratio of Sr/V from 1:1 to 2:1, the experimental results shows that an excess amount of Sr(NO$_3$)$_2$ is required for the formation of the pure $\beta$-SrV$_2$O$_6$ phase in final products, and the deficiency of Sr concentration in the reaction mixture often causes unknown phases of impurities. Reaction temperature also plays an important role in controlling morphology by means of controlling nucleation processes. By varying hydrothermal treating temperature from 160 °C to 240 °C, the experimental results show that at lower reaction temperature ($\sim$160 °C), we failed to obtain the pure phase of $\beta$-SrV$_2$O$_6$ crystallite. Increasing reaction temperature ($\sim$180 °C) leads to the formation of $\beta$-SrV$_2$O$_6$ nanoribbon as shown in Fig.3(d), which was taken from a product prepared at the temperature of $\sim$180 °C for 60 h. These long belt-like nanostructures around 100 nm in width exhibit visible twist and waving in shapes, showing high flexibility in nature. Careful TEM examination also showed that these nano-ribbons are actually made up of well-organized thinner fibers along the same
the range 4.5-6.0). In another way, it also similarly serves as a morphological directing agent to template the formation of the micro-pattern of $\beta$-$\text{SrV}_2\text{O}_6$ crystallite.

The crystallization mechanism of hydrothermal reaction in general is very complex, due to the coexistence of the gel, the solution and the dissolution-precipitation equilibrium [25]. During hydrothermal process, the utilization of a solvent could accelerate the mixing process of several chemical elements, and leads to the formation of homogeneous products. This technique also offers a potential possibility for controlling the crystal growth into particles with desired morphologies. The initial properties of the crystal nuclei formed in the earlier stage play an important role for the particle morphologies in products. Actually, these nuclei change accordingly with the reaction temperature and chemical composition. Based on an extensive analysis on the $\beta$-$\text{SrV}_2\text{O}_6$ system, we propose a possible mechanism for the formation of dandelion-like micro-crystallite as follows: the starting materials $\text{Sr(NO}_3)_2$ (Sr source), $\text{V}_2\text{O}_5$ (V source) and the adipic acid (mineralizer solvent) were mixed and dissolved in the deionized water, vanadium pentoxide was firstly dissolved and hydrolyzed to form various species such as $\text{VO}^2^-$, $\text{VO}_4^{3-}$, $\text{VO}_3^{3-}$, and $\text{H}_2\text{VO}_3$ [21]. The presence of adipic acid provided an acid reaction environment, in which these species condensed and polymerized to form polyvanadates chains, which condensed by sharing edges or corners to form 3D framework structure [25]. The Sr ions could be accommodated into the framework tunnel resulting in the preferential growth of microrods as seen in our products. In the $\beta$-$\text{SrV}_2\text{O}_6$ system, due to the high reaction temperature, the polymerized polyvanadate particles are expected to form first, then a small amount of $\beta$-$\text{SrV}_2\text{O}_6$ nuclei are formed around each polymerized polyvanadate particle under hydrothermal conditions. The nuclei should serve as seeds [26] and subsequently grow up in a highly anisotropic way. The one-dimensional morphology of the microrod is also determined by its high anisotropic properties in crystal structure. Figure 4(d) shows a structural model schematically illustrating the anisotropic features of $\beta$-$\text{SrV}_2\text{O}_6$. This compound is composed of isolated ($\text{V}_2\text{O}_5$)$_n$ double chains which extend along the b-axis, and interstitial Sr ions that occupy trigonal prisms of oxygen atoms pertaining to three different chains and cement the structure [27,28]. This proposed mechanism can be further supported by our EDXS results as shown in Fig. 3(e) and (f), which show that the core part mainly consists of V and O elements, while the microrod contains a reasonable amount of Sr element.

In recent studies, we have also extended investigation to the synthesis of other kinds of new low-dimensional vanadium oxides by using hydrothermal method. We used $\text{KVO}_3$ and KOH as precursors in the presence of mineralizer adipic acid and have successfully obtained uniform $\text{K}_{0.5+z}\text{V}_2\text{O}_5$ crystalline nanobelts in size of
FIG. 5 A typical bright-field TEM image of a potassium-vanadium oxide shows the microstructure feature of nanobelts, the average width is estimated to be around 50 nm.

around 50-100 nm. Figure 5 displays a typical bright-field TEM image of the as grown K$_{0.5+x}$V$_2$O$_5$ material, showing the fundamental microstructure feature of the crystalline nanobelts. Further investigation on the potassium-vanadium oxide nanobelts is still under progress.

IV. CONCLUSION

In summary, under mild hydrothermal reaction conditions and in the presence of mineralizer adipic acid, we obtained dandelion-like micro-crystallites and belt-like nanostructures of $\beta$-SrV$_2$O$_6$ materials. Their crystallographic structure properties and microstructural features were characterized by means of powder XRD and electron microscopy diffraction measurements, respectively. The powder XRD data determined that these micro-/nanometer materials belong to an orthorhombic structure with the lattice parameters $a$=9.694 Å, $b$=3.687 Å, and $c$=12.570 Å (space group of Pnma) as reported for $\beta$-SrV$_2$O$_6$ phase. Electron microscopy studies revealed that the dandelion-like micro-crystallites in general consist of well-arranged microrods with the average length of a few hundred micrometers and the diameters ranging 1 µm to 5 µm, while the long belt-like nanostructures have a diameter of about 100 nm with visible twist and waving shapes. The electron diffraction patterns also indicated the micro-/nanometer $\beta$-SrV$_2$O$_6$ preferentially grow along the $\langle 010 \rangle$ crystallographic direction. To obtain the optimal reaction conditions for the synthesis of micro-/nanometer $\beta$-SrV$_2$O$_6$, the effects of the hydrothermal treating temperature and chemical compositions ratio of Sr to V on the morphology of the final products were extensively investigated. We also extended the hydrothermal process to the synthesis of other low dimensional metal-vanadium oxides.

V. ACKNOWLEDGMENTS

The work was supported by the National Natural Science Foundation of China. We would like to thank Mr. Y. Q. Zhou and Mrs. Y. Li for their assistance in preparing the TEM specimen and Dr. R. I. Walton for fruitful discussion and help for manuscript preparation.