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Preparation and Characterization and Reducing Properties of MoO₃ Nano-Fibers

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1. Introduction

New methods of making new or existing inorganic materials are always interesting and challenging for materials scientists(Tenne 2006). Development of one-dimensional (1D) materials has become a focal area in nanostructured materials research, owing to their special characteristics which differ from those of respective bulk crystals(Romo-Herrera, 2007). These highly anisotropic 1D materials include elemental carbon, metals, semiconductor, alloys, sulfides, oxides, hydroxides, and so forth (Dmitruk, 2007). Among the important layered transition metal oxides and chalcogenides have been extensively investigated (Shabaev 2004).

In the course of the exploration of novel approaches for the preparation of metastable oxide materials, the main interest is focused on soft-chemical routes. Both, topochemical reactions involving ion exchange, intercalation, and pillaring as well as sol-gel, and hydrothermal reactions belong to these low-temperature methods. Especially host-guest compounds realized by the intercalation of different guest species into layered inorganic frameworks represent a new and promising class of material that can be used for the controlled preparation of complex organized structures in the nanoscale regime(Markus 2001, Xiong 2003).

MoO₃ and its derivatives are widely used in industry as catalysts, display devices, sensors, smart windows, lubricants, battery electrodes(Zhao D.W.2008, Kim Youn-Su 2007, Ghorai T.K.2007, Dillon A.C.2008) . In particular, MoO₃ has been prepared into the forms of carbon-metal-oxide nanocomposites, nanotubes, and nanorods using carbon nanotubes (CNTs) as a host material or template. The MoO₃ nanofibers obtained with this novel method are up to 15μm long with their diameters ranging from 50 to 150 nm (Ajayan 1995, Satishkumar, 2000).

An efficient method to produce nanoscopic molybdenum oxide fibers was reported. The procedure is based on the intercalation of primary amines into the layered structure of molybdic acid and subsequent transformation of the lamellar molybdenum oxide–amine intermediate into the fibrous product. Standard synthesis: In a typical procedure, molybdic acid MoO₃·2H₂O (10 mmol) was mixed with the amine in 5 ml ethanol (molar ratio 2:1). After the addition of 15 ml distilled water, the yellow suspension was stirred at room temperature for 48 hours until a white precipitate was formed. The hydrothermal reaction of this composite was performed in an autoclave at 120°C for 3 to 5 days and, after filtering

and washing with ethanol and diethyl ether, a white powder resulted. The molybdenum oxide-amine composite (2.5g) was stirred in 10 ml 33% nitric acid for 24 hours at room temperature and thereby the fibrous material was formed. The product was filtered, washed with ethanol and diethyl ether and then dried at 80°C under vacuum (10-3 mbar), (Markus,2001).

One-dimensional nanostructures of orthorhombic molybdenum trioxide (α-MoO$_3$) have been synthesized in the forms of ribbons or rods via acidification under hydrothermal conditions at 140-200°C. The reaction path has been revealed with kinetic investigations, which shows the following sequence: (i) from the starting compound $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ to (ii) formation of intermediate compound $((NH_4)_2O)_{0.0866} \cdot MoO_3 \cdot 0.231H_2O$, and then to (iii) final α-MoO$_3$ nanoribbons or nanorods in 100% phase purity. The optimal growth temperature is in the range of 170-180°C under the current experimental settings. At higher reaction temperatures, this transformation can be accelerated, but with poorer crystal morphological homogeneity. It has been found that the dimensions of these rectangular nanorods are about 50 nm in thickness, 150-300 nm (mean value at 200 nm) in width, and a few tens of micrometers in length. The crystal morphology can be further altered with inorganic salts such as NaNO$_3$, KNO$_3$, Mg(NO$_3$)$_2$, and Al(NO$_3$)$_3$. Using an H$_2$S/H$_2$ stream, the above-prepared α-MoO$_3$ nanorods can be converted completely to 2H-MoS$_2$ nanorods at 600°C. The original rodlike morphology is well-retained, although the aspect ratio of the oxide template is reduced upon the sulfidation treatment,(Xiong Wen Lou 2002).

A new whisker, α-MoO$_3$, was fabricated via molybdenum thread oxidation at 973,1023, and 1173K. Various morphologies of the whiskers, such as flakelike, platelike, and needlelike, were observed under different fabrication conditions. The lateral surfaces of the whiskers were close-packed (010) plane, and the growth direction was [001], which related to the unique layered structure of the α-MoO$_3$ crystal. Growth of the MoO$_3$ whisker was attributed to a vapor-liquid–solid (VLS) mechanism at 1023 and 1173K, whereas, at 973K, growth was attributed to a vapor-liquid(VL) mechanism (Jianqiang Li 2004).

But, for nano Mo metal powders prepared from MoO$_3$ nanorods or nanofibers have not been reported in the literatures, the principle of preparation of MoO$_3$ nanofiber prepared by hydrothermal process is not clearly, and the cross-section shape of MoO$_3$ nanofiber never characterized by electro microscopy.

Here we report the process of MoO$_3$ nanofiber prepared by hydrothermal method, and, the morphology of MoO$_3$ nanofiber was studied under different reaction conditions, and nano Mo metal powder indeed can be reduced from the resultant MoO$_3$ nanofibers through reduction conversion by using H$_2$ stream.

2. Experimental

2.1 Materials preparation

The α-MoO$_3$ nanofibers were synthesized by a hydrothermal route. Ammonium heptamolybdate tetrahydrate (AHM; $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) and nitric acid were the two starting reagents. A saturated solution of precursor compound AHM was prepared at room temperature. For each run of experiment 30.0 ml of the saturated solution was diluted with deionized water (30.0 ml) and then further acidified using the 2.2M nitric acid to a total of 70-85.0 ml in volume. The resultant solution was transparent and transferred to a Teflon-lined stainless steel autoclave and heated at 170-180°C for 40 h. The product precipitate was

filtered and rinsed with deionized water, followed by drying at 62°C for 5h. The dried sample was fibrous and pale yellowish.

The above as-grown α-MoO$_3$ crystals were further used as metal oxide precursors for reduction investigation. The reactions were carried out in a tubular quartz reactor at 400-600°C using H$_2$ stream under normal atmospheric pressure.

2.2 Materials characterization

Crystallographic information of samples including reduced ones was investigated with X-ray diffraction (XRD; Shimadzu XRD-6000, Cu Kα,λ=1.5406 Å). Crystal sizes and the morphology of crystal samples was examined before and after the reduction reactions with scanning electron microscopy (SEM, JSM-5600LV, 15 kV).

High-resolution analytical transmission electron microscopy (TEM, JEM-2010, 200 kV) was also used to examine crystalline α-MoO$_3$ and its reduced products. The specimens for TEM imaging study were prepared by suspending solid samples in acetone.

3. Results and discussion

3.1 Preparation of polymorphous molybdenum trioxide by hydrothermal process

In order to get molybdenum trioxide powder with different morphologies, especially to get molybdenum trioxide nanofiber, using saturated solution of ammonium paramolybdate and nitric acid as raw materials, molybdenum trioxide powders with different micro-morphology were successfully synthesized through changing synthetic parameters of hydrothermal synthetic process in a hydrothermal reactor.

Synthesized molybdenum trioxide powders were characteristiced by XRD, SEM, and HRTEM. Results showed that molybdenum trioxide nanofibers could be got which were more than 20μm in length, 50～200 nm in diameter when the heat treatment temperature was more than 150°C, acidity coefficient (acidity coefficient is defined as ratio of number of moles of acid added in the solution with number of moles of Ammonium heptamolybdate tetrahydrate) of precursor solution was more than 15, and heat treatment time more than 20 h.

Crystal growth direction of molybdenum trioxide nanofibers was (001). Deviating of synthetic process parameters mentioned above,molybdenum trioxide powders with rodlike, platelike, and flakelike micro morphologies could be synthesized.

3.1.1 Synthesized at low acidity conditions

Low acidy conditions indicated that acidity coefficient in the reaction solution was less than 10, and the template often used in the synthesis process of nano fibers was not used in this research.

For the 25ml of AHM with 63% (wt) concentration, mixed with 2.2M HNO$_3$ solution 50ml, then, appropriate amount of milk like mixture got was put in the teflon-lined stainless steel autoclave and heated at 170°C for 20 h,the acidity coefficient is 8.6, the hexagonal short prismatic MoO$_3$ was obtained (Fig1.).

In the Fig2, the 25ml of AHM with 25% (wt) concentration, mixed with 2.2M HNO$_3$ solution 10ml, then, appropriate amount of milk like mixture got was put in the teflon-lined stainless steel autoclave and heated at 170°C for 20 h,the acidity coefficient is 4.3, the square flake MoO$_3$ was obtained.
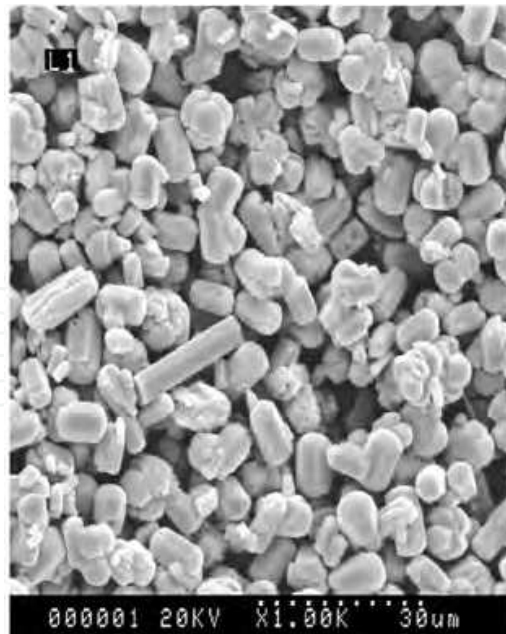
Fig. 1. MoO$_3$ powder morphology synthesized at low acidity conditions (acidity coefficient is 8.6).

From Fig2, the morphology of MoO$_3$ crystal is square flake crystal,the width and length are less than 10 micrometer, the thickness of crystal is less than 1 micrometer.
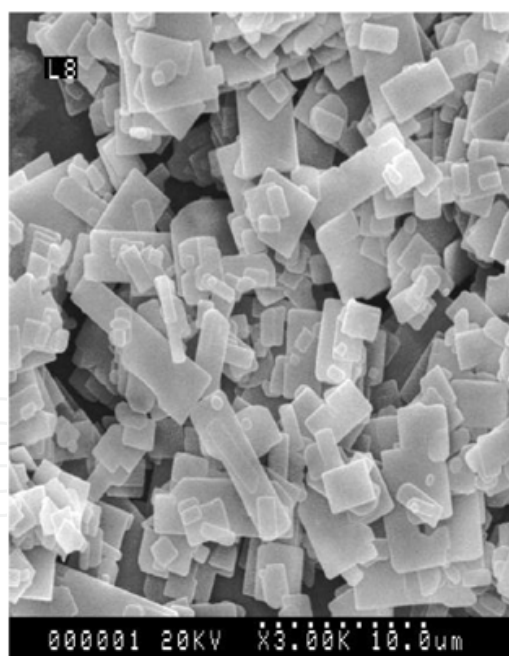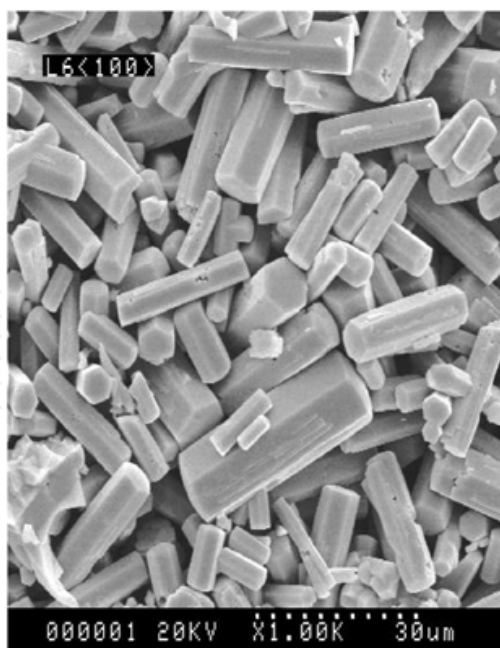

Fig. 2. MoO$_3$ powder morphology synthesized at low acidity conditions (acidity coefficient is 4.3).

In Fig 3, the 30ml of AHM with 27% (wt) concentration, mixed with 2.2M HNO$_3$ solution 10ml, then, appropriate amount of milk like mixture got was put in the teflon-lined stainless steel autoclave and heated at 170°C for 20 h,the acidity coefficient is 3.0, the hexagonal long prismatic MoO$_3$ was obtained.

Fig. 3. MoO$_3$ powder morphology synthesized at low acidity conditions (acidity coefficient is 3.0).

3.1.2 Synthesized with template at 170°C for 40h

To study the adding template on the morphology of molybdenum trioxide through hydrothermal process, the first use of cetyltrimethylammonium bromide(CTAB) as template agent. Because of CTAB is cationic surfactant, the particles in the AHM solution are negatively charged, so as different amount of CTAB was added, the emulsion solution easy to be formed,the heatreatment temperature is 170°C, the time is 40h, after hydrothermal reaction, the different morphologies of molybdenum trioxide crystal were obtaned (see in the Fig4-Fig5).
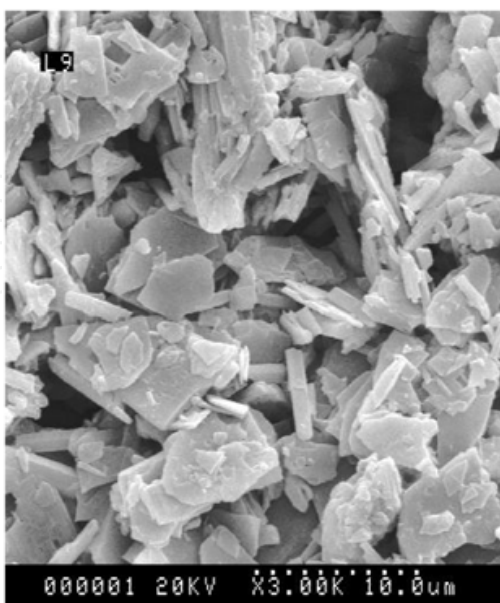
Fig. 4. MoO$_3$ morphology synthesized at different moles ratio of CTAB with AHM (CTAB / AHM =0.1).
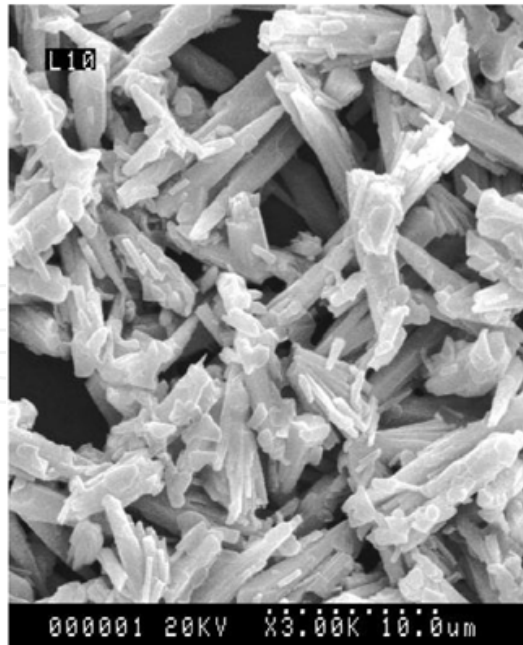
Fig. 5. MoO$_3$ morphology synthesized at different moles ratio of CTAB with AHM (CTAB / AHM =0.4).

3.1.3 Synthesized with template at 180°C for 40h

Increasing heattreatmeng temperature to 180oC, and increasing ratio of CTAB with AHM in the solution, a series of strip like MoO$_3$ crystals were obtained (in the Fig.6-Fig.8).
From Fig.6 to Fig.8, the results indicated that the size of MoO$_3$ decreaseed when moles ratio of CTAB with AHM increased, the shapes of samples were nearly strip fiber.
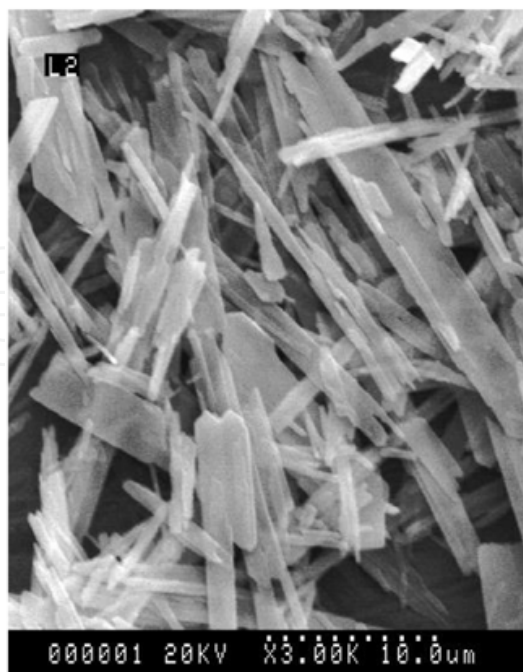

Fig. 6. MoO$_3$ morphology synthesized at 0.5 moles ratio of CTAB with AHM under 180°C for 40h.
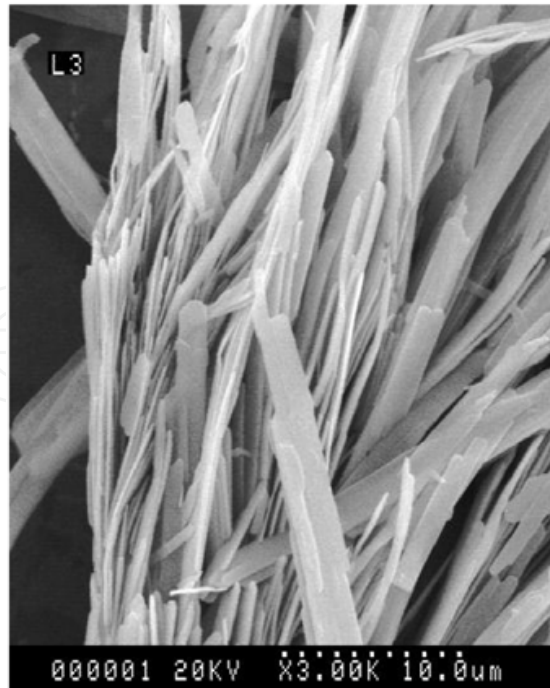
Fig. 7. MoO$_3$ morphology synthesized at 0.7 moles ratio of CTAB with AHM under 180°C for 40h.
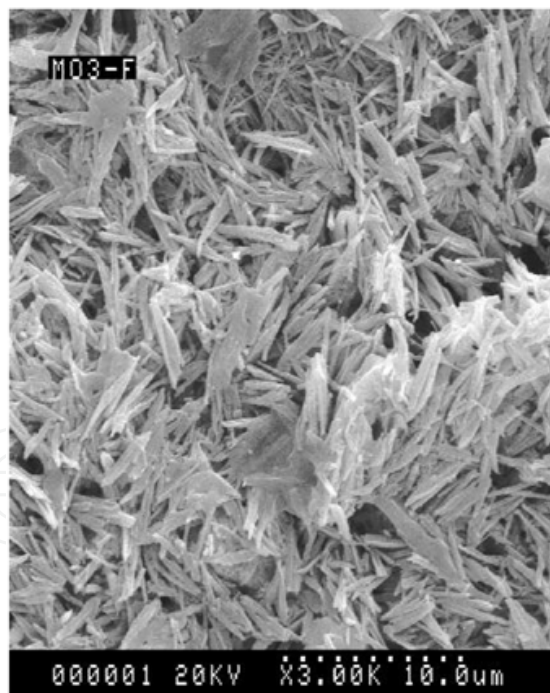
Fig. 8. MoO$_3$ morphology synthesized at 1.0 moles ratio of CTAB with AHM under 180oC for 40h.

3.1.4 Synthesized at high acidity without template at 170°C for 40h

High acidy conditions indicated that acidity coefficient in the reaction solution was more than 15, and the template CTAB was not used in this research.

For different concentration of AHM(wt%), 25 %, 12.5%, 26% respectively with different acidity coefficient 15, 20, 22, the precursor solutions were all transparent, after heattreatment at 170°C for 40h, fibers of MoO_3 were obtained (see in the Fig.9- Fig.11).
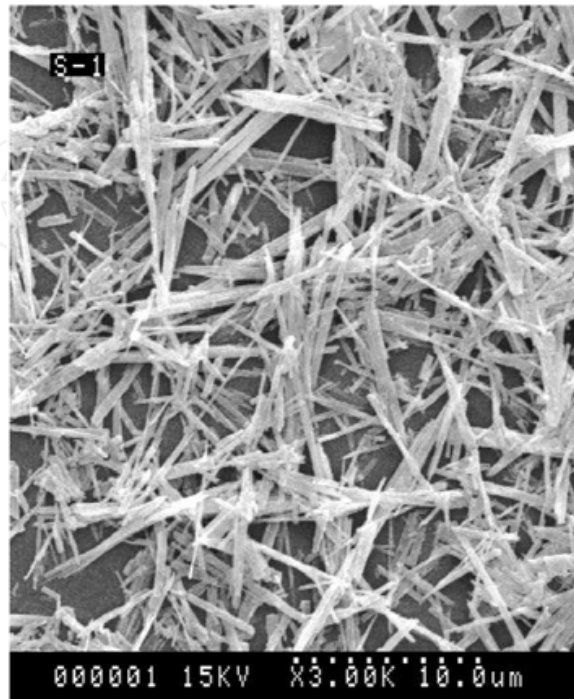
Fig. 9. MoO_3 morphology of precursor of 25% AHM at acidity coefficient 15(180°C for 40h).
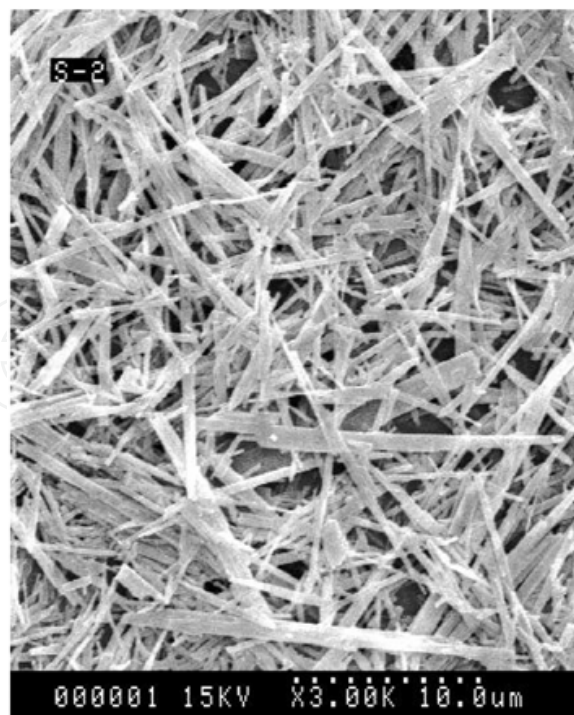

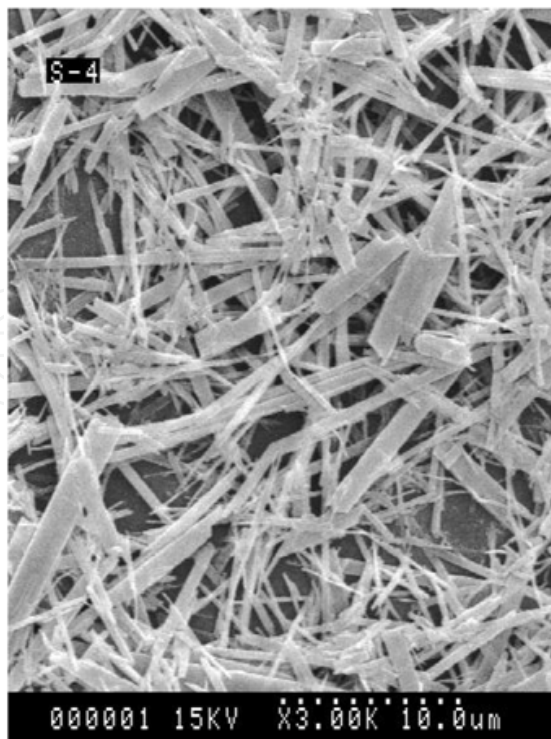Fig. 10. MoO_3 morphology of precursor of 12.5% AHM at acidity coefficient 20(180°C for 40h).

Fig. 11. MoO₃ morphology of precursor of 26% AHM at acidity coefficient 22 (180ºC for 40h).

3.1.5 Optimization of synthesis process parameters

A SEM image of a typical product showed the sample as an entirely fiber-like structure after optimized synthesis process parameters(Fig.12),the nanofiber length is more than several tens of microns.
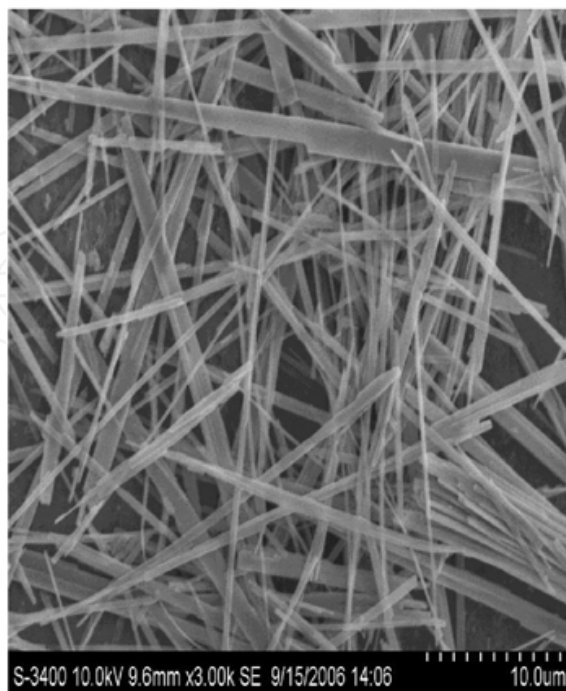

Fig. 12. Typical morphology of MoO₃ nano fibers synthesized under optimized conditions.

3.2 Characterization of MoO$_3$ nano-fibers
3.2.1 Characterized by HRTEM and FSEM

The result of typical MoO$_3$ nanofibers characterized by HRTEM is shown in fig.13, the nanofiber cross- section is not round, some nanofibers is not single fiber, combined by two single fiber. Most fibers are with width of about 50 to 200 nm, and length of about a few tens microns.

Nearly rectangle-like cross section of the nanofibers could be seen from the image of Fig.14. HRTEM image of an individual nanofiber (Fig.15) provided further insight into the structure of these products. HRTEM images recorded perpendicular to the growth axis of the single nanofibers could be attributed to the [010] of orthorhombic MoO$_3$, and suggested that the nanofibers grew along the [001] direction.
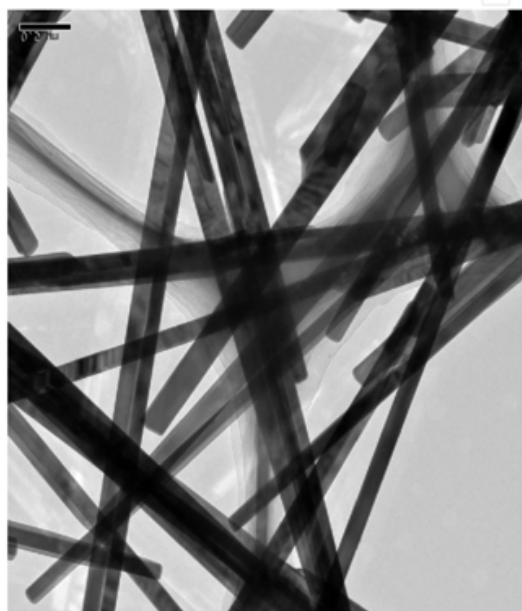

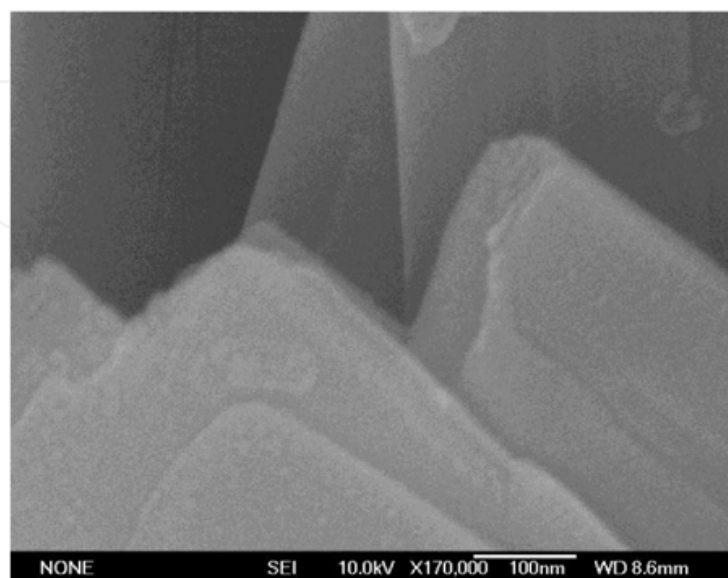Fig. 13. Typical image of MoO$_3$ nano fibers characterized by HRTEM.

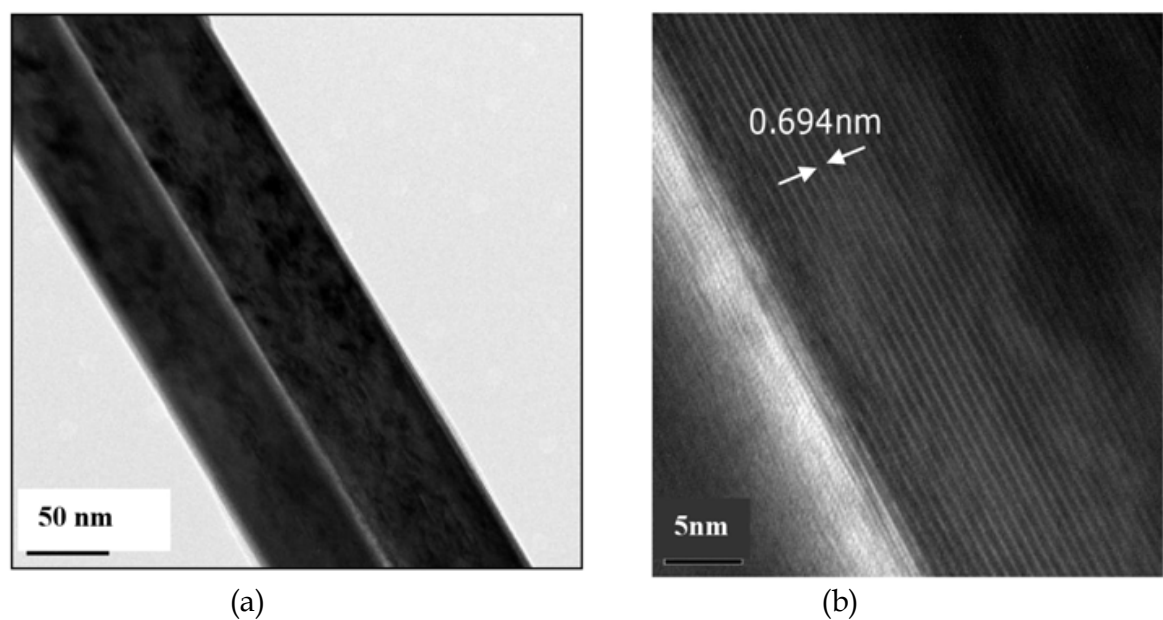Fig. 14. Typical image of cross-section of MoO3 nano fibers characterized by FSEM.

(a) (b)

Fig. 15. HRTEM image of an individual nanofiber combined by two single fiber(a); and the lattice image of MoO$_3$ nanofiber crystal (b).

3.2.2 Characterized by XRD

X-ray diffraction showed that samples consisted only strongly oriented orthorhombic α-MoO$_3$ (a = 1.385 nm, b = 0.3696 nm, c = 0.3966 nm, JCPDS 89-7112). It can be observed in Fig.16., that(020)，(040)，(060) are strong peak respectivily, that means all the strong diffraction peaks correspond to (0h0) reflections. (110),(021),(002) are weak peaks, which shows that crystal growth is slow in these direction, causing the crystal diffraction line intensity decreased.
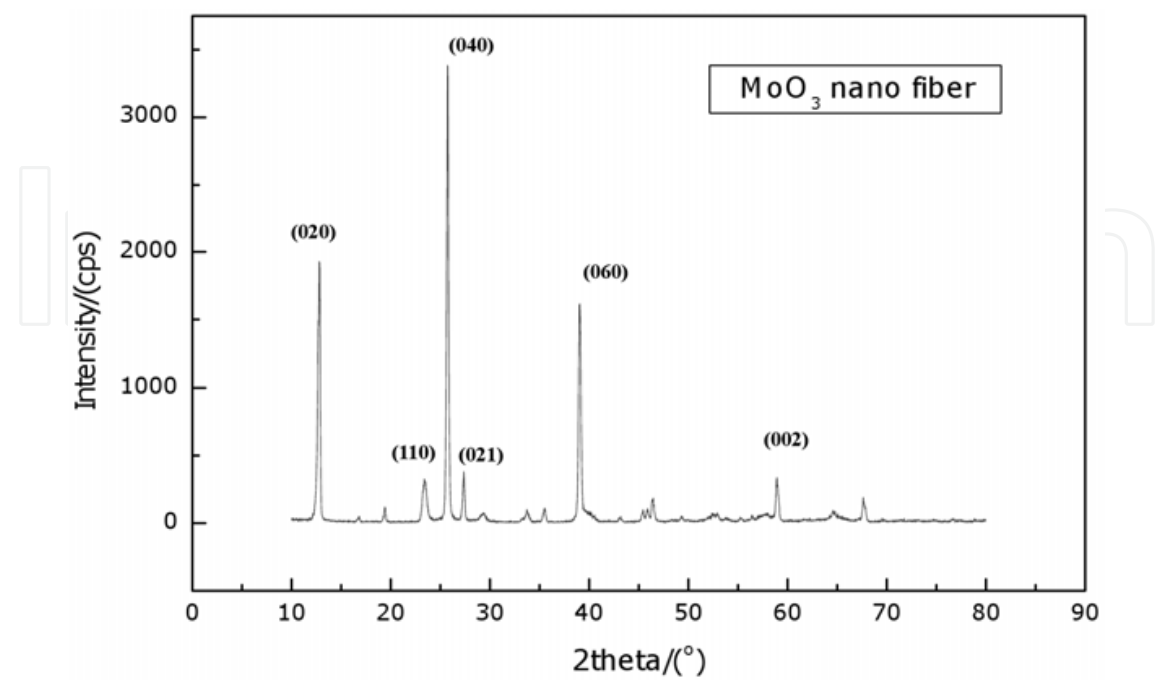

Fig. 16. The XRD patterns of MoO$_3$ nano fibers.

3.3 Principle of MoO₃ nano-fibers crystal growth

The crystal growth process of MoO₃ under hydrothermal conditions could be explained according literatures, and the MoO₃ nanofiber crystal growth modle is shown in the Fig.17. (Xiao Lin Li 2002).
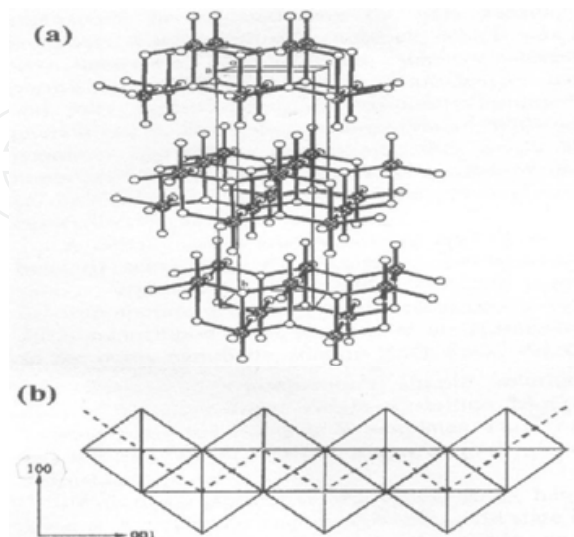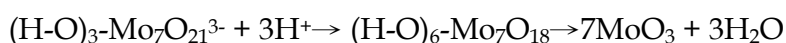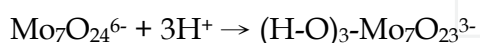

Fig. 17. The atom structure of MoO₃ (a), and schematic diagram of the crystal growth direction(b).

From Fig.17(a), It can be seen, that molybdenum trioxide is a typical orthogonal layered structure. Layered structure constitutes the smallest unit is MoO6 octahedron. Fig.17 (a) shows that the crystal chemistry cell of molybdenum trioxide is selected in three layers of MoO6 octahedron, where a, c values are relatively small, namely 3.966 and 3.693Å, while the b value is relatively large 13.858Å (JCPDS05-0508).

Each layer of molybdenum trioxide crystal is formed by MoO6 octahedrals twist to share angle connectivity in the a axis direction, and in the c-axis direction, MoO6 octahedrals connect by a sharing edge way, this sharing edge way twists and turns along acording the Z-shaped.

From Figure 17 (b) , It can be seen, along the [100] direction and [001] direction ,the same chemical environment is not same, which led to the [001] and [100] direction connection requires different chemical activation energy. If along the [001] direction is necessary to form two Mo-O bond, while along the [100] only the formation of one Mo-O bond, so MoO6 octahedron cenection along [001] will release more energy, so the crystal growth along [001] preferred orientation, especially in the presence of H⁺, the occurrence of the following reaction:

$$Mo_7O_{24}^{6-} + 3H^+ \rightarrow (H\text{-}O)_3\text{-}Mo_7O_{23}^{3-}$$

$$(H\text{-}O)_3\text{-}Mo_7O_{21}^{3-} + 3H^+ \rightarrow (H\text{-}O)_6\text{-}Mo_7O_{18} \rightarrow 7MoO_3 + 3H_2O$$

As can be seen from the above reaction, increased the concentration of AHM and nitric acid, were very favorable reaction towards the right, which is the main reason to synthesis of nano-fibers with high concentration of molybdenum trioxide.

Secondly, in order to ensure that the oxygen ions prolapse from AHM to form water molecules, excessive acid concentration to be necessary, the minimum theoretical acidity coeffcent is of 7, the actual value in the this research increases to 15 or more,so synthesized the MoO₃ nano fibrous structures, that the high acidity is also needed.

Molybdenum trioxide crystal is the vertex connected along the [100] direction, the combination of atoms along [010] direction is van der Waals force, resulting in fiber diameter direction, the atoms combination is not close, so the double-fiber structure is formed.

3.4 Reducing of MoO₃ nano-fibers

Molybdenum and molybdenum alloys have good electrical conductivity, thermal conductivity, high temperature strength and high temperature hardness, corrosion resistance and good performance, therefore, widely used in chemical engineering, metallurgy and aerospace industry and other fields. With the rapid development of high-tech, material properties of molybdenum and its alloys in many aspects of the request beyond the traditional performance. For getting high performance molybdenum metal materials, ultra-fine powders of molybdenum is its first step (Tuominen 1980).

The methods of ultrafine Mo powder preparation are traditional MoO₃ reduction method, hydroxyl thermal decomposition method, molybdenum chloride vapor method, molybdenum chloride pyrolysis method, laser flash method, and microwave plasma method, etc.(Malikov 1997, Futaki Shoj 1992, Chow 1991, Liu Binghai 1999, Gonzalez 2001)).

However, all of these methods, to achieve large-scale industrial production of fine metal powders of molybdenum, the traditional reduction method still has a certain appeal. It is possible that ultrafine Mo powder could be produced by reducing industrialized nano molybdenum oxide.

3.4.1 MoO₃ nanofibers microstructure before H₂ reduction

The purpose of reduction test of nano-fibers of molybdenum trioxide synthesized by hydrothermal process, is to prepare ultrafine Mo powder. Reduction experiment of molybdenum trioxide nanofibers was performed in the hydrogen reduction furnace by using of the traditional hydrogen reduction method.

The XRD pattern of molybdenum oxide nanofibers befor reduction is shown in Fig.16,and nanofibers of molybdenum trioxide SEM photograph is shown in Fig.18.
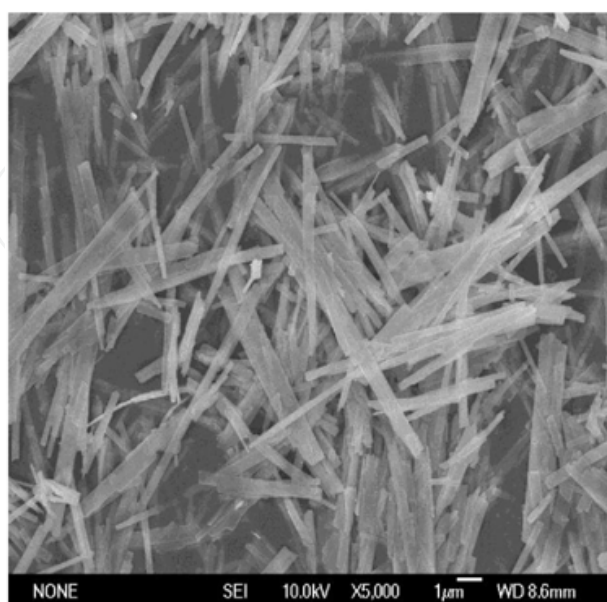

Fig. 18. FSEM image of MoO₃ nanofiber before reduction.

In fig.18 FSEM photo, the nano-fiber length ranging is 10-30 microns, width of it is about 50-200 nm. The MoO_3 nano-fibers has better dispersion and smooth surface. The nano-fiber size is uniform befor hydrogen reduction.

3.4.2 Reduction result of MoO_3 nanofibers

The MoO_3 nanofibers were reduced at different temperature under H_2 stream atmosphere. From fig19, the XRD patterns indicated that MoO_3 nano fibers could be reduced as MoO_2 at 530°C for 1 hour, the materials had a pure phase of MoO_2. That means the MoO_3 nanofibers have been reduced as MoO_2 completely at 530°C. Corresponding to the fig 18, the morphology of the MoO_3 nanofibers have been changed, the fibers-like morphology was partial retained in the MoO_2 sample (Fig.20).

Fig.19 showed that the MoO_3 nanofibers had been reduced as Mo powders completely when the reduced temperature was increased to 600°C, corresponding to the fig21, the morphology of Mo powders is not fibers-like, most of metal Mo powders are irregular shape, neither the ball-like, nor fibers-like.
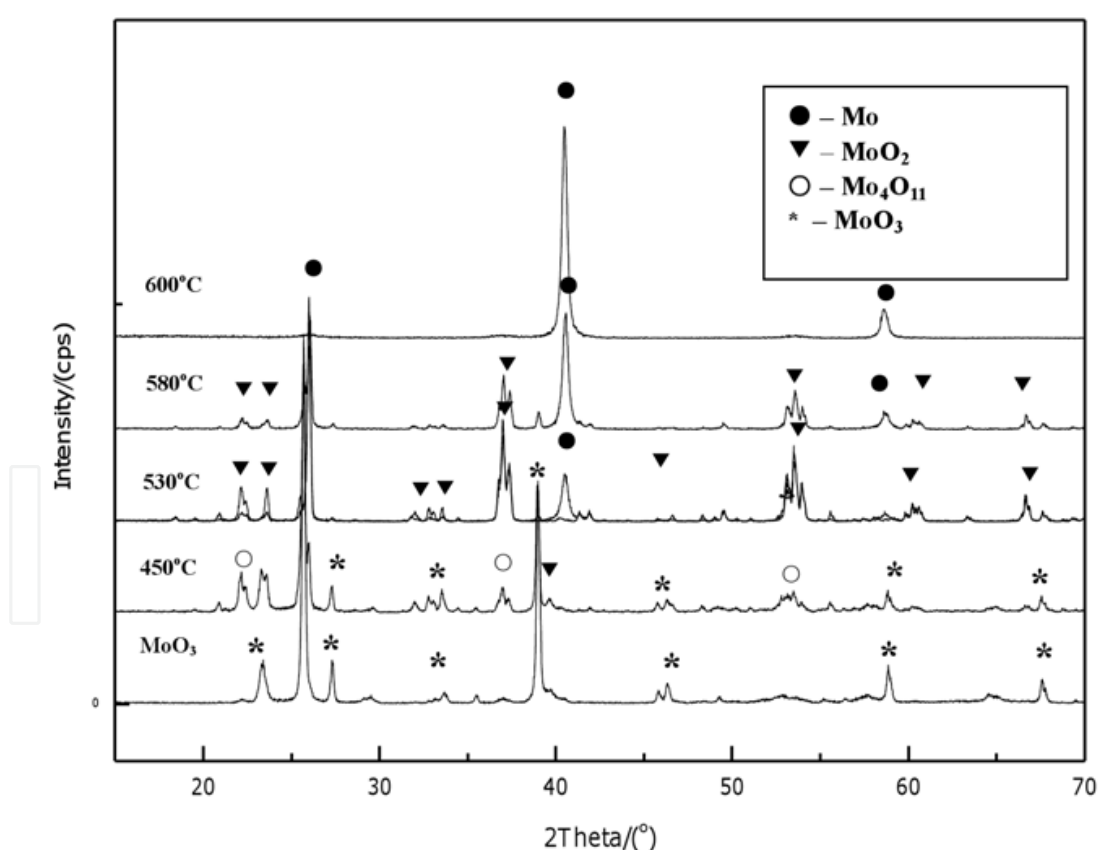

Fig. 19. The XRD patterns of MoO_3 nano fibers reduced at different temperatures by using H_2.
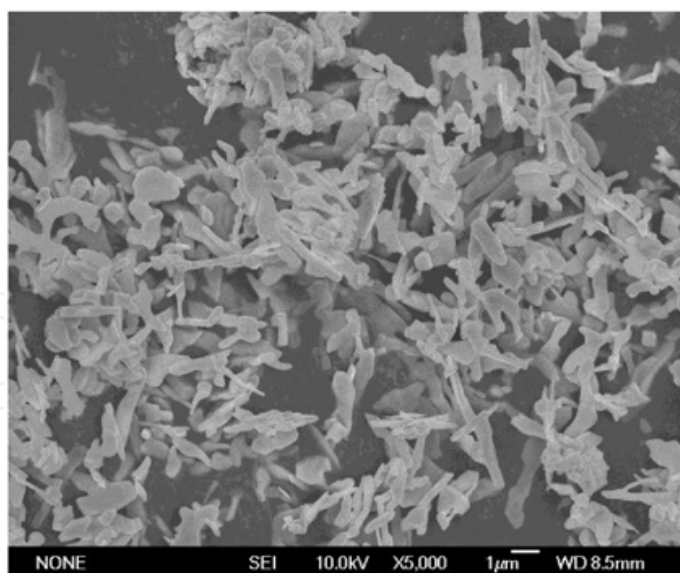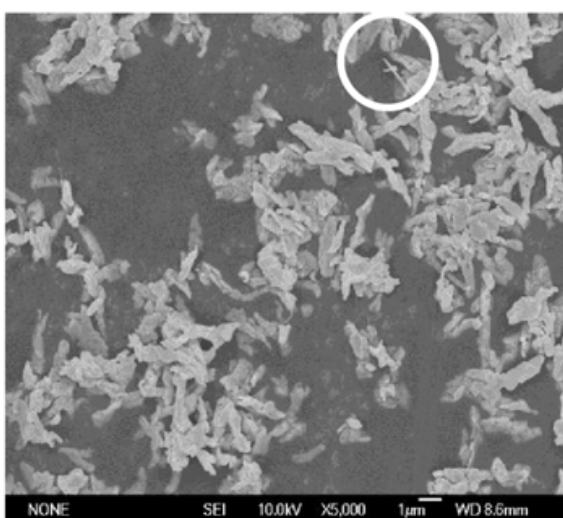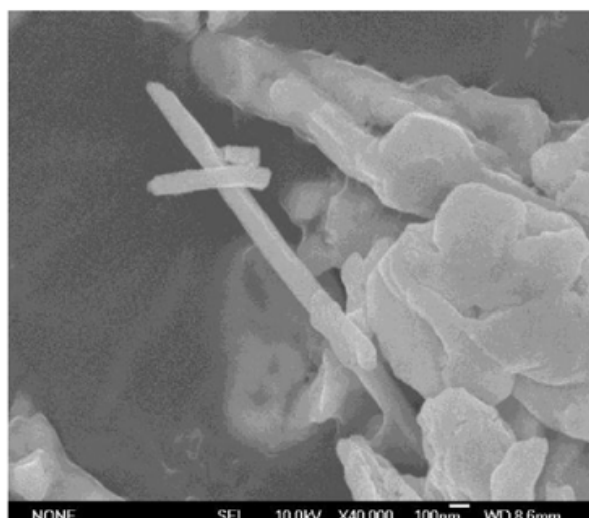
Fig. 20. The SEM image of nanofibers molybdenum trioxide reduction at 530°C for 1h.

(a)   (b)

Fig. 21. SEM images of Mo powder reduced at 600°C(a), and (b) is enlargement of the white circle in (a).

But in some case, the nanofibers-like shape of MoO_3 was retained, the result could be seen in the fig21. From fig.21(b), it could be found that needle-like metal Mo powder was formed. The reason of forming needle-like metal Mo powder could be explained that the MoO_3 nanofiber was restricted in a free space, no other MoO_3 nanofiber contact with each other, although orthorhombic crystal structure of MoO_3 changed as FCC crystal structure of metal Mo, no other metal Mo particles to combine with, so the nanofibers-like shape was retained.

3.4.3 Optimization of reduction result of MoO₃ nanofibers

According to the reason mentioned above, we using small bulk (about 2-3mm) MoO_3 nanofiber as raw materials instead of the MoO_3 nanofibers powders. The small bulk MoO_3

was reduced at 600°C for 1h, XRD indicated that MoO_3 had been completely changed as metal Mo powder. SEM result shown in fig22. Much more needle-like metal Mo powders were formed. It means nanofiber-like MoO_3 could be reduced as needle-like metal Mo powder in some special case.
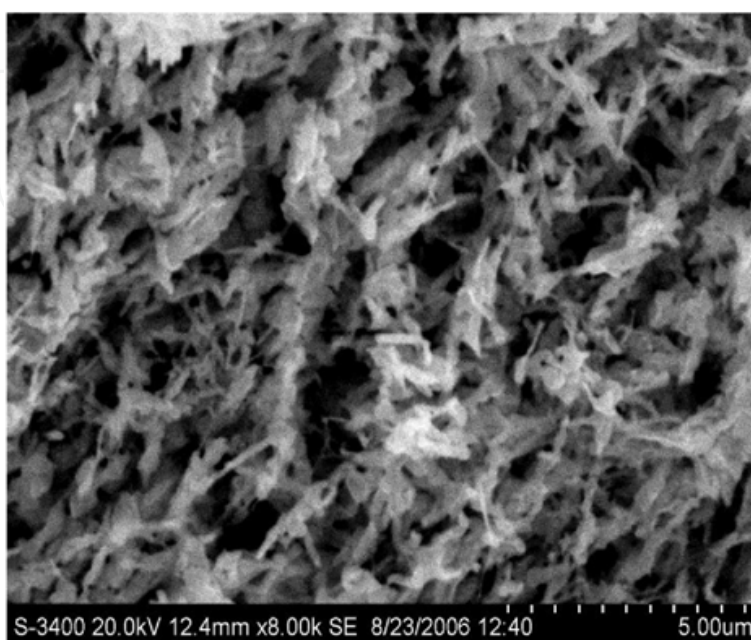

Fig. 22. SEM image of needle-like metal Mo powder.

3.5 Principle of MoO₃ nano-fibers reduced by using H₂

Through analysis the reduction process of nanofibers molybdenum trioxide, in general, the fibrous structure of molybdenum trioxide is difficult to maintain or inherited. Just because of molybdenum trioxide is orthogonal layered structure, and molybdenum dioxide is rutile structure.As we know, the reduction process of nanofibers molybdenum trioxide is a very complicated local chemical processes, including following steps (Tuominen 1980, Thorsten 2001):

1.   adsorption of reduction gas
2.   the chemical reaction between molybdenum oxide with the hydrogen molecules
3.   changes in crystal chemistry and the desorption of gas in the reaction products

The first step of reduction process of molybdenum trioxide is mainly broken of crystal at 450°C or more, because of the molybdenum dioxide is gradually began to be formed on the surface of molybdenum trioxide and the layer of molybdenum dioxide could be peeled off, and the crystal of molybdenum trioxide could be broken or cracked.

As the reaction further, the layer thickness of molybdenum dioxide is more than half the thickness of the crystal of molybdenum trioxide, molybdenum dioxide peeling process could be stopped, the molybdenum trioxide completely transformed into molybdenum dioxide, the shape of crystal was changed generally.

Delamination of molybdenum dioxide should be occurred in the weak place of crystal surface of molybdenum trioxide, due to anisotropy of chemical bonding strength, so it is broken easily in the [001] direction of molybdenum trioxide. Therefore, when molybdenum trioxide nanofiber is reduced as molybdenum oxide, the crystal size is shorter (Fig.23).
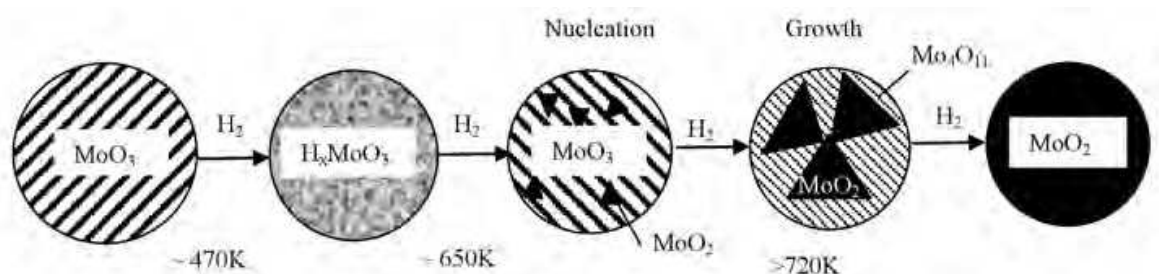
Fig. 23. Schematic diagram of the reduced process of MoO$_3$ to MoO$_2$.

4. Conclusion

One-dimensional nanostructures of orthorhombic molybdenum trioxide have been synthesized in the forms of fibers under hydrothermal conditions. The dimensions of the fibers are about 50nm in thickness, 100-200nm in width and a few tens of micrometers in length under our experimental settings.

The MoO$_3$ fibers prepared can be converted completely to Mo metal powders at 600°C by ussing H$_2$ stream. The original fiber-like morphology is not well-retained, but in some specials cases, fiber-like morphology can be partial retained, although the aspect ratio of the oxied fibers is reduced upon the reduction treatment.

5. Acknowledgment

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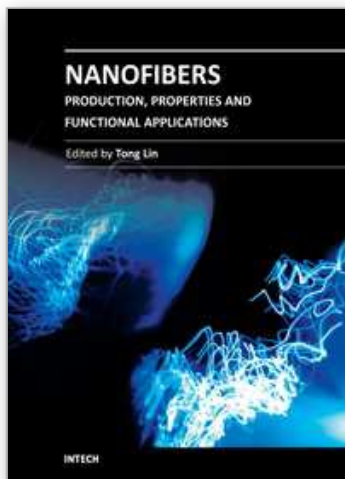
Nanofibers - Production, Properties and Functional Applications

Edited by Dr. Tong Lin

As an important one-dimensional nanomaterial, nanofibers have extremely high specific surface area because of their small diameters, and nanofiber membranes are highly porous with excellent pore interconnectivity. These unique characteristics plus the functionalities from the materials themselves impart nanofibers with a number of novel properties for advanced applications. This book is a compilation of contributions made by experts who specialize in nanofibers. It provides an up-to-date coverage of in nanofiber preparation, properties and functional applications. I am deeply appreciative of all the authors and have no doubt that their contribution will be a useful resource for anyone associated with the discipline of nanofibers.

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