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Nanobeam Electron Diffraction Study of MoO_3 Nanorods and Nanobelts

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The aim of the present study is concerned with the synthesis and characterization of MoO_3 microstructure with different shapes. A detailed study of the individual MoO_3 structure is important since this material is used in the oil industry as catalyst for sulfur removal [1]. The materials were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The structure was studied using nanobeam electron diffraction (NBD) technique, using a JEM-2200 FS, with a spherical aberration corrector in the probe mode.

Materials synthesis was performed following the hydrothermal method [2]. A saturated solution of ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) was acidified using a 2 M solution of HNO_3 until a final pH of 5 was reached. Then the acidified solution was hermetically stored for one week for aging. In order to produce the nanostructures, 5 to 10 ml of the aged solution were mixed with ionized water (0 to 15 ml) and further acidified with 5-10 ml of 2 M solution of HNO_3. The resultant solution was transferred to a Teflon-lined stainless steel autoclave and heated at 140-200 °C for 24 h.

Figure 1 shows a SEM image where the different types of morphology are not easily distinguished but the lengths of all shapes are more than 200 microns. However, according to the EDS analysis all of them contain Mo and O. This was verified by XRD where it shows a typical polycrystalline diffraction pattern in Figure 2. In the other hand, Figure 3 shows a diffraction pattern acquired using the NBD technique. Each type of morphology shows an orthorhombic α-MoO_3 phase, but each pattern shows changes in the specific symmetric spot intensities. This means that the preferential growing of certain planes characteristic for different type morphology. In the bright field TEM image (Figure 4) can be distinguished four different shapes, among them can be notice details for each one. The type 1 shape has defined edges with around 50 nm of thickness. Type 2 shapes are part of a belt with 20 nm of thickness. In the case of type 3 shapes, it looks as a very thin belt, which can be folded. Type 4 shapes seem to be a solid rod with 90 nm in diameter and it can be bending. The relation between the different shapes and the intensity of the spots in the diffraction pattern will be discussed in detail.

References:

[1] M. A. Albiter, R Huirache-Acuña, F Paraguay-Delgado, J L Rico and G Alonso-Nuñez, *Nanotechnology,* **17,** 3473-3481 (2006).

[2] F. Paraguay-Delgado1, M. A. Albiter, R. Huirache-Acuña, Y. Verde, and G. Alonso-Nuñez, *J. Nanosci. Nanotechnol*. **7**, 3677–3683 (2007).

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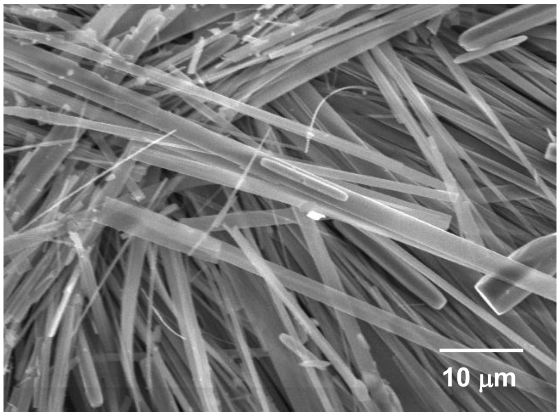

Figure 1. SEM image shows elongated MoO_3 with different morphologies.

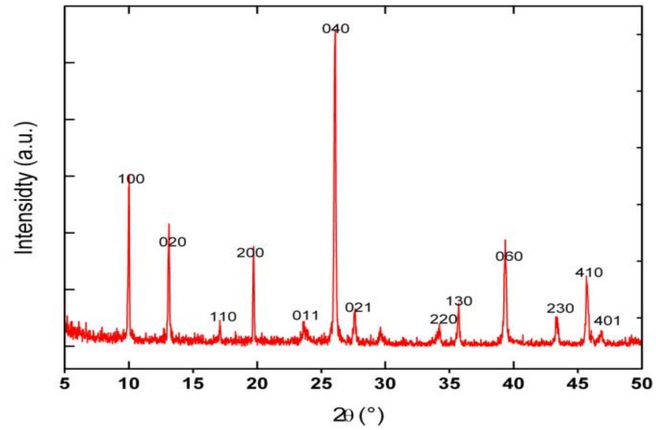

Figure 2. XRD pattern shows a typical polycrystalline material.

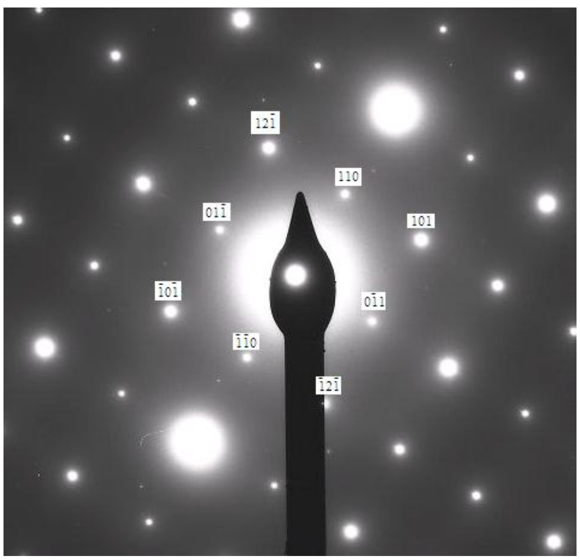

Figure 3. NBD pattern showing intensity changes between symmetric spots.

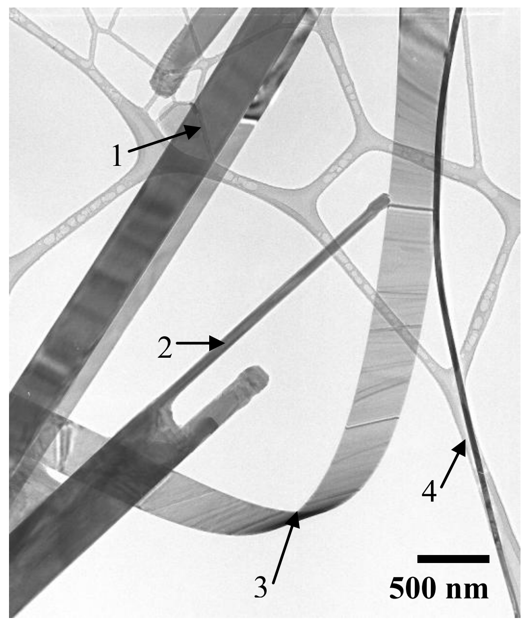

Figure 4. TEM image showing four different types shapes of MoO3.