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Study of the Molybdenum Oxide Rods Sulfidation Process

C. Ornelas[1,3]*, K.G. Guerrero−Hernández[2], J. Lara-Romero[1], C. Leyva-Porras[3] and F. Paraguay-Delgado[3].

[1.] Facultad de Ingeniería Química, Universidad Michoacana de San Nicolás de Hidalgo, Morelia Mich., 58000, México.
[2]Área Electromecánica industrial, Universidad Tecnológica de Tulancingo, Camino a Ahuehuetitla 301, C. Las Presas, C.P. 43642, Tulancingo, Hgo, México
[3.] Centro de Investigación en Materiales Avanzados SC (CIMAV), Laboratorio Nacional de Nanotecnología, Miguel de Cervantes No.120, C.P. 31136, Chihuahua, Chih., México.
* Corresponding author: carlos.ornelas@cimav.edu.mx

Molybdenum sulfide (MoS_2) is widely used as a catalyst, semiconductor, and tribology. MoS_2 has a laminar crystalline structure with Van der Waals forces between S-Mo-S layers in (002) direction. Different molybdenum compounds synthesis methods and morphologies have been reported [1, 2]. Some of these get molybdenum morphologies as oxides, following sulfidation process to obtain MoS_2 [1]. The stability of oxide structures and the formation of intermediate suboxides hinder the sulfidation process of the material [3]. Different conditions were studied to achieve complete molybdenum sulfidation.

Molybdenum oxide (MoO3) was synthesized by the microwave-assisted hydrothermal method (MAHM) previously reported by Paraguay-Delgado et al [4]. To synthesize MoO_3, an aqueous solution of ammonium heptamolybdate 0.3 M was heated in NAHM at 200°C and 41 bar for 20 minutes. To obtain MoS_2, the MoO_3 was treated in a tubular furnace in the sulfur atmosphere. Sulfidation conditions like heating rate, temperature, atmosphere and time of treatment were adjusted to obtain complete MoS_2. The materials were characterized by TGA-DTA, XRD, SEM, EDS and HRTEM techniques.

Figure 1 shows the XRD of KC obtained by NAHM and MoO_3 after sulfidation process. The MAHM synthesis gives a hexagonal MoO_3 and no other phases were detected. Figure 2 a) shows the SEM images of KC, which present hexagonal rod morphology with 11.9 µm long and 3.3 µm diameter averages. Traditionally sulfidation conditions (KM-500°C-H_2S/H_2) resulted in low sulfurized molybdenum oxide with 7.4% of MoS_2 according to EDS analyses, high-temperature sulfidation (700°C) in the same conditions (KM-700°C-H_2S/H_2) give just 26.6% of MoS_2. According to XRD traditionally synthesis at 500°C and 700°C samples have phase change to MoO_2 (figure 2), complete reduction of MoO_3 to MoO_2 was done. Complete sulfuración was achieved at 700°C with H_2S/N_2 flow (KM-700- H_2S/H_2) showed in figure 2 d).

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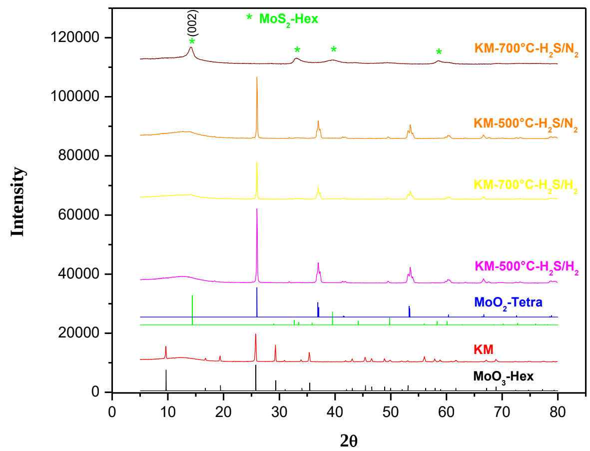

Figure 1. Molybdenum oxide X-ray diffraction.

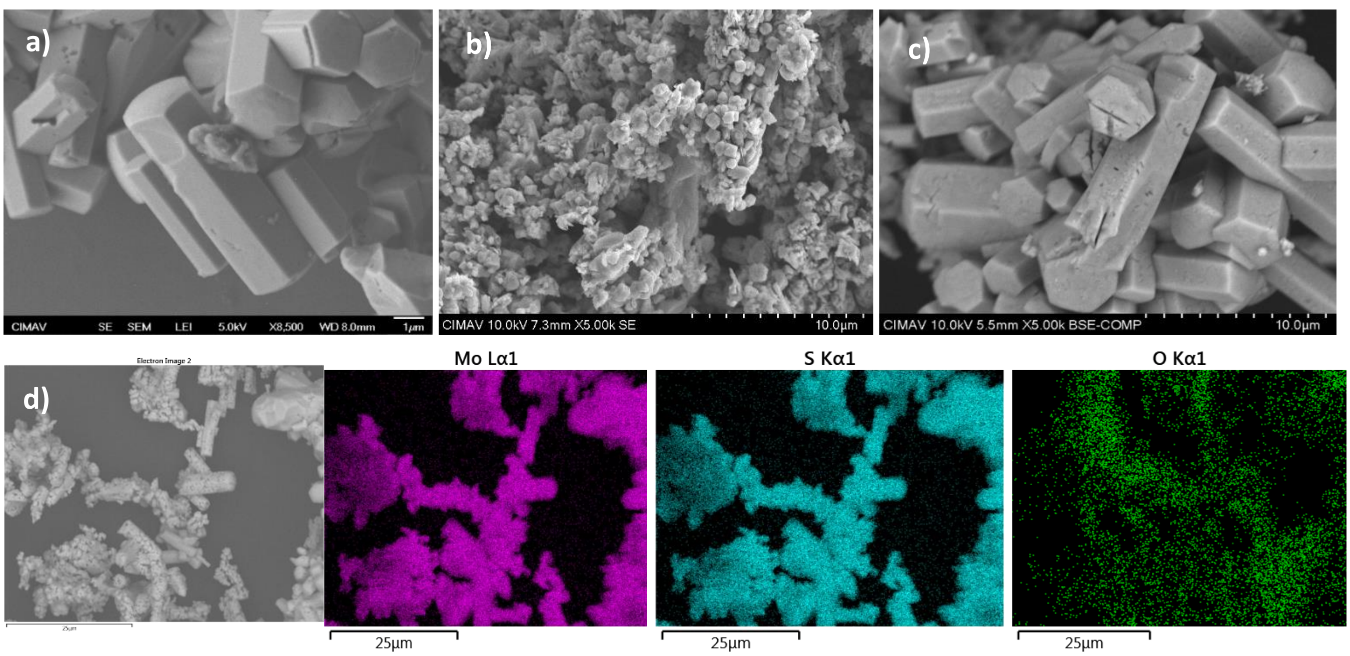

Figure 2. SEM micrographs of a) KM b) KM-700°C-H_2S/H_2 c) KM-700°C-H_2S/N_2 and d) KM-700°C-H_2S/N_2 elemental mapping.

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