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Porous and Lamellar Molybdenum Oxide MonoCrystalline with Hexagonal Structure

F. Paraguay-Delgado[1], O. Solis-Canto[1], C. Ornelas[1,2] and J. Lara-Romero[2]

[1.] Centro de Investigación en Materiales Avanzados SC (CIMAV), Laboratorio Nacional de Nanotecnología, Chih., México.
[2.] Facultad de Ingeniería Química, Universidad Michoacana de San Nicolás de Hidalgo, Mich., Mexico.

Molybdenum is an important transition metal, both in oxide (MoO_x) and sulfurized (MoS_2) form are widely used in different applications like gas sensors, photochromic materials, recording materials, catalyst, ceramics, lubricants, etc [1]. Molybdenum sulfide (MoS_2) is usually synthesized from MoO_x by means of sulfidation process; always the morphology, shape and size of MoO_x have strong influence in the final properties and applications of sulfurized molybdenum materials. Different MoO_x morphologists and crystalline structures are synthesized in precise controlled way and thermic treatments are used to achieve phase transformation and/or crystals grow of the material [3].

In this research work Molybdenum oxide (MoO_3) was synthesized by microwave assisted hydrothermal method according to previously method reported by Paraguay-Delgado et al [3]. In order to obtain a more stable molybdenum oxide phase, a thermal treatment was performed. The Molybdenum oxide was synthesized from an aqueous solution of ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) at concentration of 0.3 M and pH controlled, this solution was heated in microwave assisted hydrothermal method at 200°C and 41 bar for 20 minutes. Later the MoO_3 samples were heated in a muffle at 450°C for 45 min in static atmosphere. After thermal treatment the materials were characterized by XRD, SEM and HRTEM.

By means of XRD the formation of Orthorhombic MoO_3 phase was confirmed. SEM characterization was perform in order to know the morphology and composition, the micrographs confirm MoO_3 and a hexagonal type bars morphology with average of 11.9 nm and 3.8 nm long and diameter respectively. They are formed by terraces (figure 1 a)); its formation in the hexagonal bars was observed in longitudinal or transverse directions. In addition, a complete HRTEM characterization of hexagonal bars with terraces was done, the figure 1 b) shows a Z contrast image in HRTEM, this micrograph shows a part of the hexagonal bar. The Z Contrast observes two different structures, the firs one is the terraces observed in SEM these are MoO_3 layered as lamellar MoO_3 with different thickness (Z1), according to with Tao Liang, et al, the Orthorhombic MoO3 form lamellar structures have van der Waals forces between layers [4]. The second structure observed by Z contrast is a porous crystalline structure join to the lamellas exposed in the outside part of the structure. Figure 2 shows the select area electron diffraction in both areas, terraces and porous structures, the results show the same crystalline structure and the same crystal orientation, perpendicular to the (-1,1,1) plane, this demonstrates the formation of molybdenum oxide porous-layered mono crystal with hexagonal structures. Another HRTEM analyses are congruent, these structures are due the physical change process in thermal treatment.

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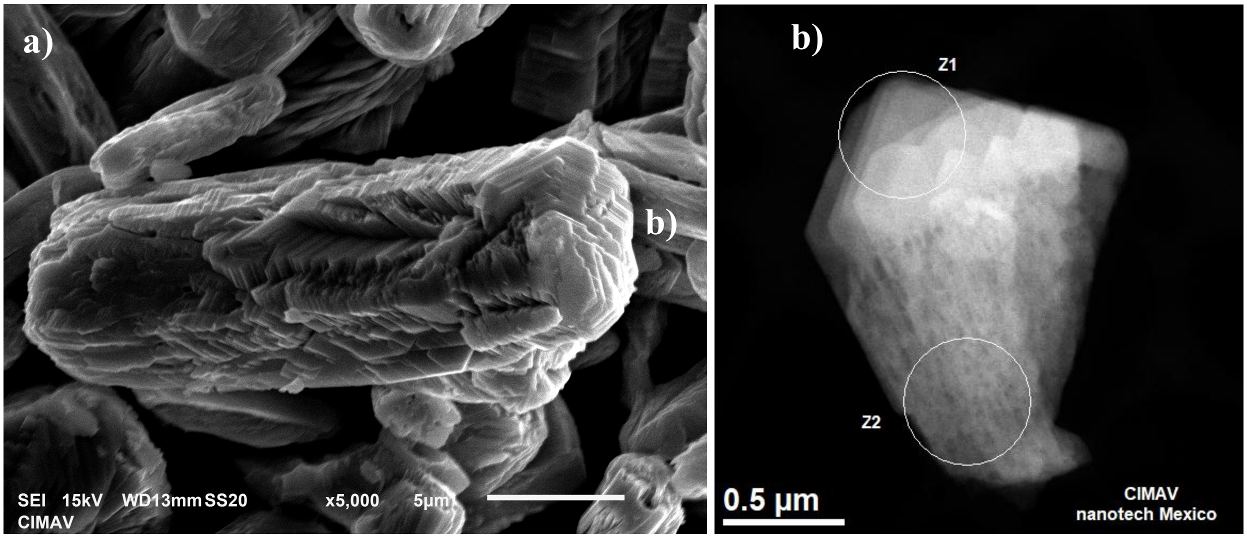
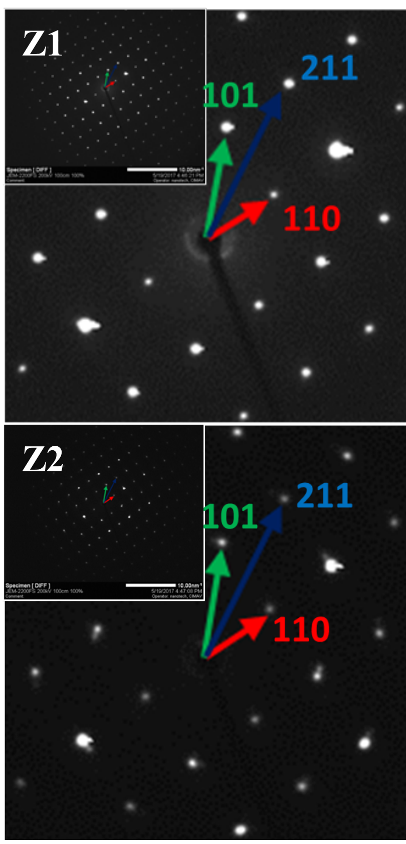


Figure 1. Molybdenum oxide characterization by a) SEM and b) HRTEM (Z-Contrast).



Orthorhombic MoO_3 01-076-2711

Vector	Plane	Theoretical distance	Experimental Measure	Theoretical Angle	Experimental Angle
1	1 1 0	3.809	4.092	--	--
2	2 1 1	1.7324	1.806	28.92	28.88
3	1 0 1	2.7032	2.7082	49.00	47.32

Orthorhombic MoO_3 01-076-2711

Vector	Plane	Theoretical distance	Experimental Measure	Theoretical Angle	Experimental Angle
1	1 1 0	3.809	4.1	--	--
2	2 1 1	1.7324	1.806	28.92	28.57
3	1 0 1	2.7032	2.772	49.00	46.99

Figure 2. SAD of molybdenum oxide (figure 1 b)) in Z1 and Z2.

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