doi:10.1017/S1431927617008893
Microsc. Microanal. 23 (Suppl 1), 2017

Molybdenum Oxide Structures Synthetized by Microwave Technique and Its Phase Transformation by Thermal Treatment

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

[1.] Centro de Investigación en Materiales Avanzados SC (CIMAV), Laboratorio Nacional de Nanotecnología, Miguel de Cervantes No.120, Chihuahua, Chih., México.
[2.] Facultad de Ingeniería Química, Universidad Michoacana de San Nicolás de Hidalgo, Morelia Mich., Mexico.

The use of microwave radiation for the materials synthesis presents great benefits: energy saving, short processing times, increased performance, economical and environmentally friendly procedures, etc. In a microwave-assisted synthesis, process there is an inverse temperature profile compared to conventional methods: heating occurs by conversion rather than by energy transfer, so the temperature distribution in the synthesis is uniform and highly controlled, which allows rapid synthesis, efficient and with better results than traditional heating [1]. This discovery has allowed the synthesis of new materials in more controlled morphologies [1-3], additionally a phase transformation can be achieve by thermal treatment [3], and the morphological changes are due to the crystalline structures obtained.

Molybdenum oxide (MoO_3) materials were synthesized according to previously method reported by Paraguay-Delgado *et al* [3], for which an aqueous solution of ammonium heptamolybdate ($(NH_{46}Mo_7O_{24} \cdot 4H_2O$) at concentration of 0.3 M and controlling the pH was treated in microwave assisted hydrothermal method at 200°C for 20 minutes, achieving pressures around 41 bar. The microwave-assisted hydrothermal method allows the synthesis two samples of MoO_3 with hexagonal bar morphology structures as showed by scanning electron microscopy (SEM) in image 1 and hexagonal MoO_3 phase according to X ray diffraction technique (XRD) in figure 2 whit a clear difference in (100) and (210) patterns intensity directions. A complete statistical study made to both samples with pressure variation during the synthesis show DCLML2 MoO_3 sample with hexagonal bars 8% shorter and 7% thinner than DCLM1 MoO_3 sample. The pressure was for DCLM1 and DCLM2 41 and 38.5 bar respectively.

In order to made a phase transformation the MoO_3 samples were treated in a muffle at 450°C for 45 min in static atmosphere, the materials after treatment are characterized by SEM, the results are shown in figure 3 and the XRD results are show in figure 2. X ray diffraction show a complete phase transformation in both materials to orthorhombic MoO_3 structures and by SEM and high resolution transmission electron microscopy (HRTEM) the morphological changes in the hexagonal bar structures are studied. Deformation in the morphology hexagonal bars structures of MoO_3 samples are observed and transformation in the sheets type within the bars was observed, these sheets are observed both horizontal and vertical with respect to the original bar, the transformation process is studied by means of HRTEM and its morphological changes is due to crystalline phase transformation process.

References:

[1] J. Prado-Gonjal and E. An. Quím. **107(2)** (2011) p. 129.
[2] M. Santos-Beltran *et al*, J. Alloys Compd. **648** (2015) p. 445.
[3] M. Santos-Beltran *et al*, J Mater Sci: Mater Electron **28(I3)** (2017), p. 2935.

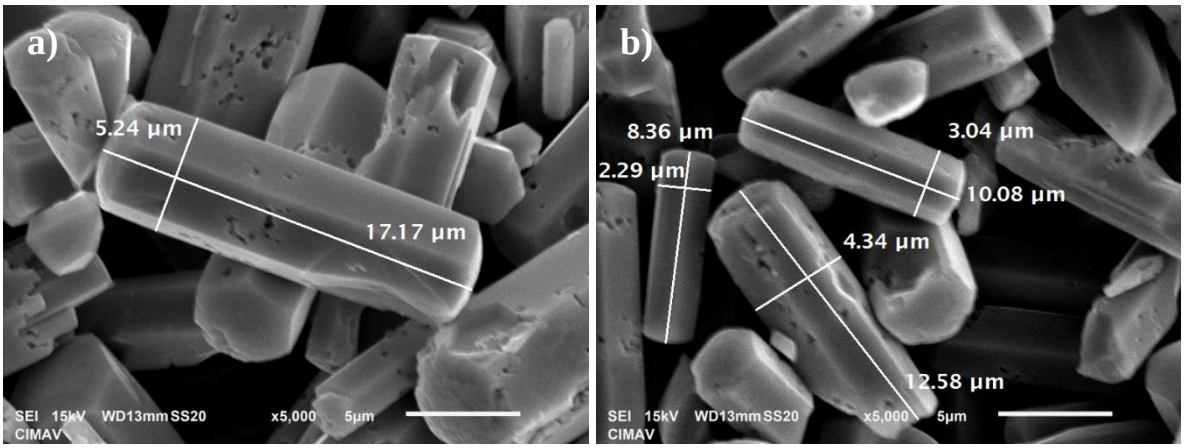

Figure 1. Molybdenum oxide by microwave-assisted hydrothermal method a) DCML1 and b) DCML2

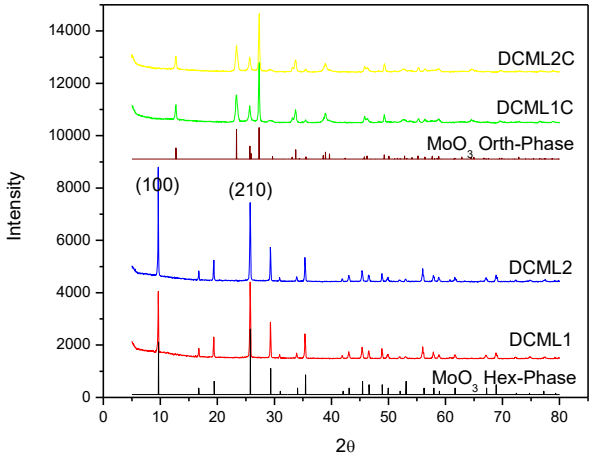

Figure 2. Molybdenum oxide. X ray diffraction

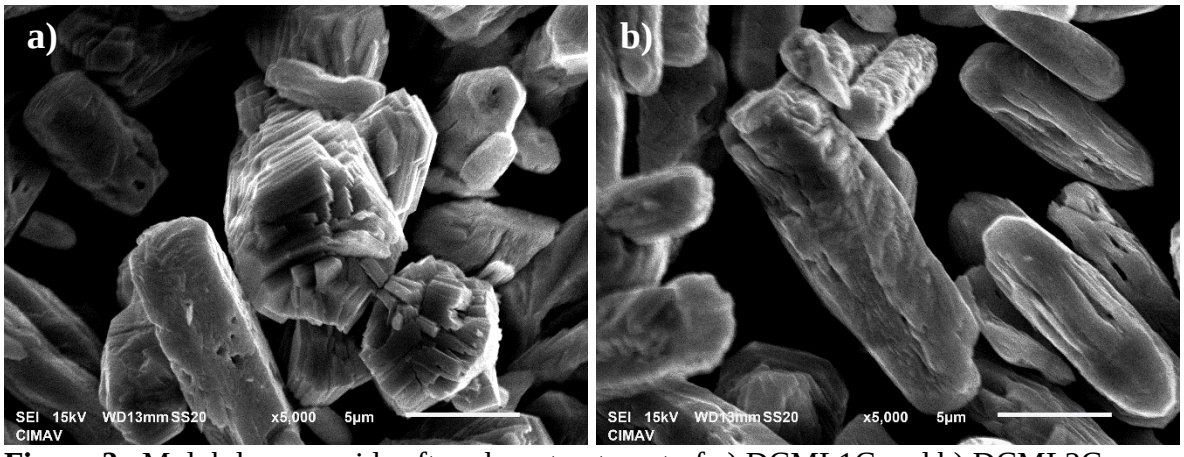

Figure 3. Molybdenum oxide after phase treatment of a) DCML1C and b) DCML2C.

https://doi.org/10.1017/S1431927617008893 Published online by Cambridge University Press