

EFFECT OF DEPOSITION TEMPERATURE ON THE FEATURES OF MoO₃ FILMS GROWN BY MOCVD. R. Martinez Guerrero(1), J. R. Vargas Garcia(1), V. F. Santes Hernandez(2), E. Gomez Perez(3) and Ana-B Soto(4). (1)Depto. de Ingenieria Metalurgica, ESIQIE-IPN, Mexico 07300, D.F., MEXICO; (2)Instituto Mexicano del Petroleo, Eje central Lazaro Cardenas 152, Mexico 07330, D.F., MEXICO; (3)Instituto de Quimica-UNAM, Circuito Exterior, Ciudad Universitaria, Mexico 04510, D.F., MEXICO; (4)Depto. de Fisica, CINVESTAV-IPN, A.P. 14-740, C.P. 07000, Mexico D.F., MEXICO. e-mail: remague@yahoo.com

Molybdenum oxides are of great interest because of their wide variety of attractive properties useful in optical, electrical, magnetic, electrochromic and catalytic applications [1-6]. The growth of molybdenum oxide films with an appropriate control of their morphology, presence of phases and thickness is highly desirable to attain superior performance for the particular application. So far, several techniques have been employed to prepare molybdenum oxide thin films, including, thermal evaporation [7], RF sputtering [8], electron beam evaporation [9] and metal organic chemical vapor deposition (MOCVD) [10]. MOCVD, in particular, is an attractive technique for the preparation of molybdenum oxide films due to the variety in film features resulting from different deposition conditions. In this study, we report the effect of the deposition temperature on the morphology and phases of molybdenum oxide films grown by MOCVD using acetylacetonate as precursor. In a first step, molybdenum acetylacetonate was synthesized by dissolving 10 g of MoO₃ powder in 50 ml of acetylacetone (CH₃COCH₂COCH₃) under reflux for 30 h. The resulting solution was added to 250 ml of toluene (C₆H₅CH₃) leading to the precipitation of the molybdenum acetylacetonate (MoO₂(CH₃COCH₂COCH₂)₂). By filtering the solution under nitrogen atmosphere and drying the precipitate under vacuum at room temperature, a translucent red brown powder was obtained. Thermal gravimetric (TG) and differential thermal analyses (DTA) of the synthesized precursor suggested the formation of a molybdenum oxide around 430 °C. Thus, in a second step, a range of deposition temperatures from 350 to 630 °C was explored to investigate the effects on the nature of the films. Molybdenum oxide films were prepared using the synthesized molybdenum acetylacetonate onto amorphous glass substrates at 1 torr of total pressure. The precursor was evaporated at constant temperature of 200 °C and its vapors were carried by nitrogen gas at a constant flow rate of 100 cc/min. The deposition time was 30 min. Depending on the deposition conditions, the films were bluish, slightly dark or completely transparent in appearance. Figure 1 shows the XRD patterns of films prepared at deposition temperatures from 400 to 560 °C. At deposition temperature of 400 °C, small XRD lines revealed the presence of the MoO₃ phase in bluish colored films. The films prepared at 500 °C were slightly dark and exhibited a mixture of XRD lines of MoO₃ along with very small lines of Mo_{0.17}O_{0.47} phases. At 560 °C, the films became transparent and showed only well-defined XRD MoO₃ lines. At higher temperatures, different oxide phases including Mo₉O₂₆ and Mo₄O₁₁ were formed in the films. This suggests that an appropriate deposition temperature for the growth of well-crystallized and transparent MoO₃ films is about 560 °C. The SEM images, in Figure 2, illustrate the influence of the deposition temperature on morphology and crystallite size of the molybdenum oxide films. A strong dependence was observed, from like-needle shaped crystallites of about 2-3 μm in diameter at 400 °C to finer needles below 1 μm at 560 °C. Contrasting with nearly platelet shaped crystallites (~1 μm in diameter) at 500 °C.

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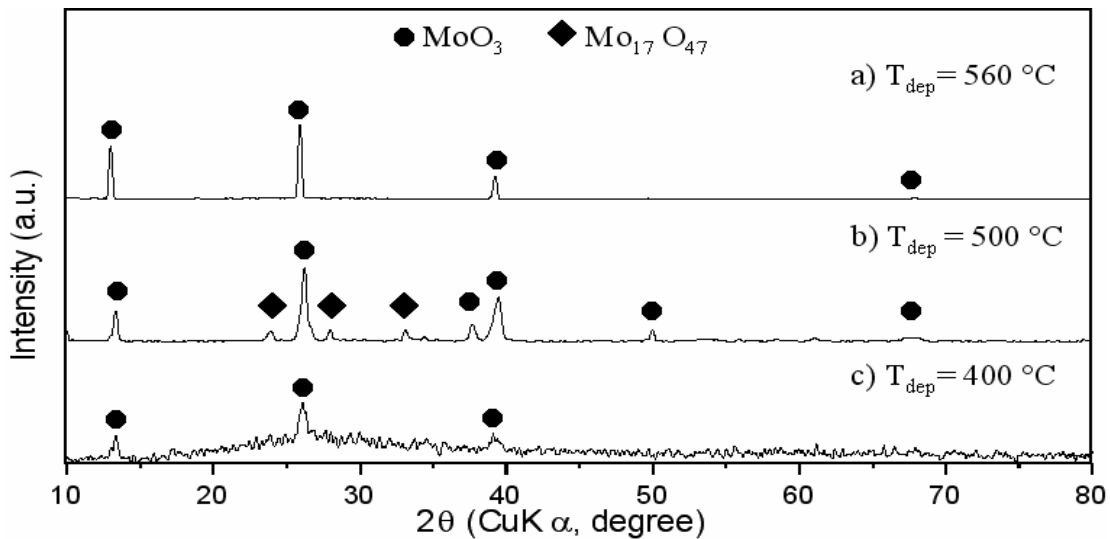


Fig.1 XRD patterns of molibdenum oxides prepared at a) 560 °C, b) 500 °C and c) 400 °C.

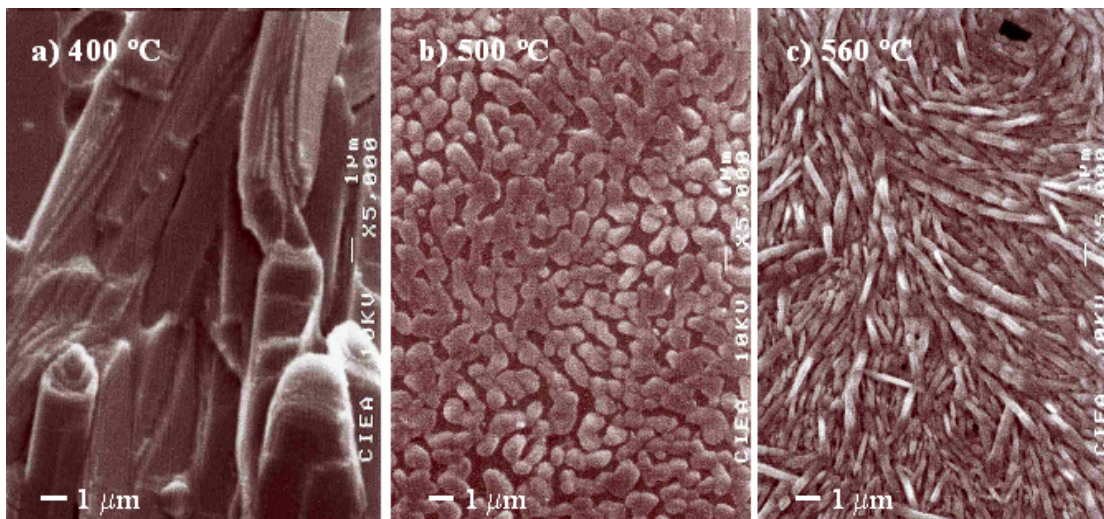


Fig. 2 SEM images of surface morphology of MoO_3 films as a function of the deposition temperature. a) 560 °C, b) 450 °C and c) 400 °C