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Electron Microscopy and Electrochromic Studies of V_2O_5 Thin Films Deposited by RF Magnetron Sputtering

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Vanadium oxide is a material that shows a phase transition of semiconductor to metal when is heated around of certain critical temperature. For the V_2O_5 compound„this phase transition occur at 257±5°C. The study of vanadium compounds in thin film configuration, has received special attention in recent times because of their interesting electrochromic and thermochromic properties and potential uses as thermal sensing, optical switches, optoelectronic devices and energy saving devices with emphasis in the development of smart windows .

In this work, vanadium pentoxide (V_2O_5) thin films were deposited by RF magnetron sputtering using a ceramic V_2O_5 target. A power of 100 watts during 10 minutes was used to deposit vanadium oxide on corning glass pure and coated with a conductive layer of SnO_2:F (FTO) prepared by the spray pyrolysis technique and with an average sheet resistance of 7 Ω/sq . The V_2O_5 films were deposited on substrates kept at room temperature and 400ºC respectively. The optical and electrical properties were characterized by optical spectroscopy in the visible and ultraviolet range and the Four Points Van der Pauw method, respectively. Likewise, changes in resistance as a function of temperature were performed. The surface composition and morphological properties were followed with energy dispersive spectroscopy (EDS),scanning (SEM) and transmission electron microscopy (TEM) respectively.Cyclic voltammetry experiments were performed in a potential range: E_0= -2800mV to E= potential 2800 mV vs. a platinum reference electrode with a scanning rate of 1000 mV/s. The cyclic voltammogram exhibits the evolution of the formation of vanadium oxides until the electrochromic species be obtained. Cycling runs, were done for 1, 10 and 60 cycles respectively and the coloration and decoloration processes at different rates, were observed for all the cases. From X-rays diffraction patterns the β-V_2O_5 phase was detected and samples deposited at room temperature, present low crystallinity . HREM micrographs confirm low crystallinity in samplesjust mentioned, while in samples deposited at 400 ºC, the VO_2 phase was detected togehter with the V_2O_5 structure. From SEM micrographs obtained before and after voltammetry cycling it was observed that V_2O_5 films look regular and compact, with an uniform grain size distribution. From SEM micrographs of films deposited at room temperature and 400 ºC and after cyclic voltammetry experiments it were detected modifications in grains configuration and surface details that might be related with sample degradation and loss of electrochromic activity as a consequence of mass and charge transport during the experiment. From EDS results it was found that a noticeable reduction of V amounts occurs in the vanadium oxides films after cyclic votammetry experiments. [1]

References:

[1] The financial support of DGAPA-UNAM Project IN 105514 is appreciated and recognized. Also we thank the financial support of DGAPA–UNAM to the Posdoctoral Position of Dr. Francisco Hernández in our laboratory.

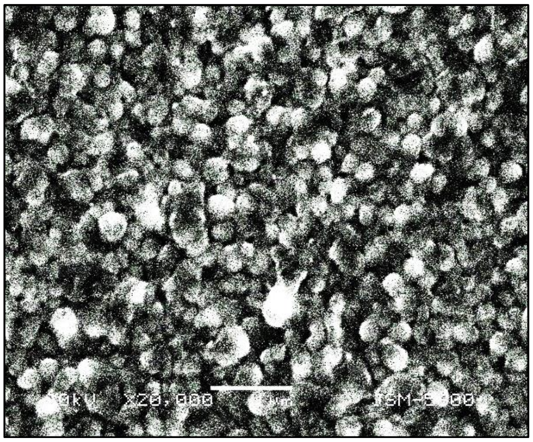

Figure 1. A SEM micrograph of a sample deposited at 400ºC.The film looks regular and compact. and grains present defined borders ana a uniform size dsitribution.

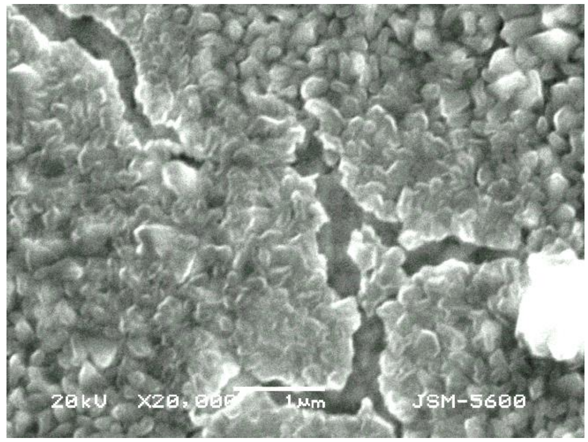

Figure 2. In this SEM micrograph the surface of films after 60 runs of cyclic voltammetry, present structural modifications : cracks and eroded grain borders are observerd frequently.

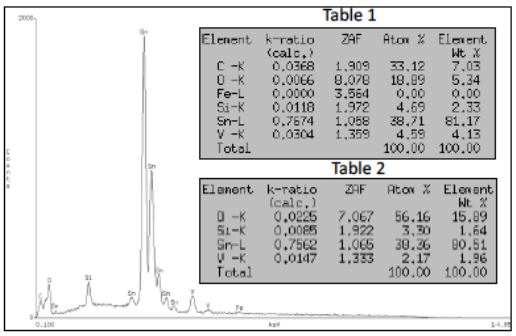

Table 1

Element	k-ratio (calc.)	ZAF	Atom %	Element Wt %
C -K	0.0368	1.909	33.12	7.03
O -K	0.0066	8.078	18.89	5.34
Fe-L	0.0000	3.564	0.00	0.00
Si-K	0.0118	1.972	4.69	2.33
Sn-L	0.7674	1.058	38.71	81.17
V -K	0.0304	1.359	4.59	4.13
Total			100.00	100.00

Table 2

Element	k-ratio (calc.)	ZAF	Atom %	Element Wt %
O -K	0.0225	7.067	56.16	15.89
Si-K	0.0085	1.922	3.30	1.64
Sn-L	0.7562	1.065	38.36	80.51
V -K	0.0147	1.333	2.17	1.96
Total			100.00	100.00

Figure 3. The EDS displayed comes from an as-deposited film. The tables attached correspond to a sample before (T1) and after (T2) 60 runs of cyclic voltammetry

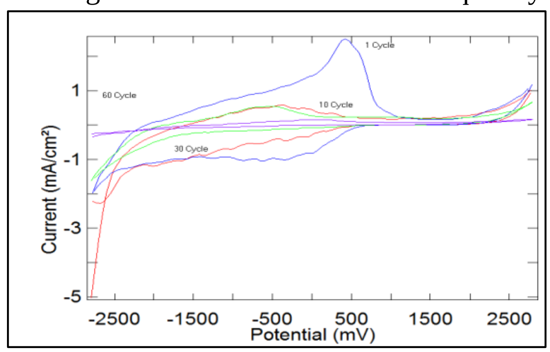

Figure 4. Cyclic voltammograms graphics corresponding to 1,10,30 and 60 cycles of a sample deposited at 400 ºC.The variation in height of cathodic and anodic peaks with the number of cycles can be observed.

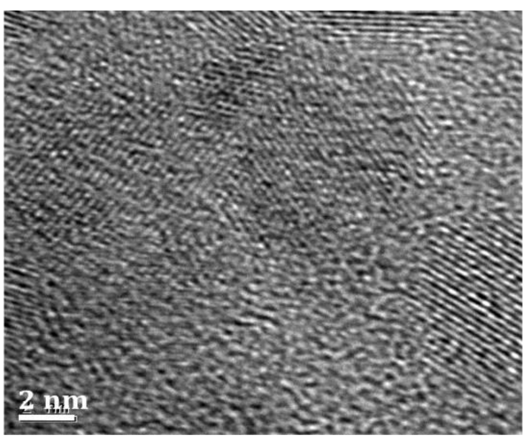

Figure 5. A typical HREM micrograph of a film deposited at room temperature. V_2O_5 grains with different orientations and amorphous zones can be observed in the image

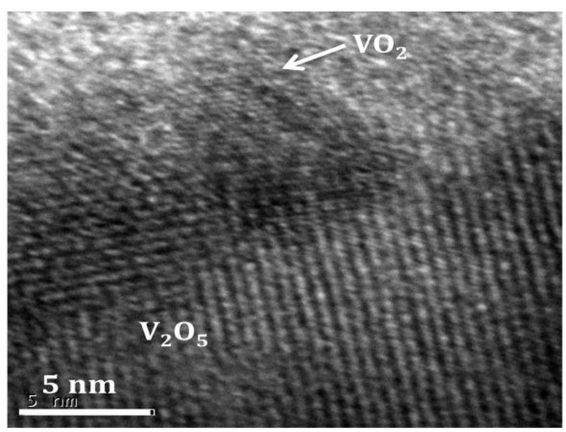

Figure 6. The coexistence of VO_2 and V_2O_5 phases derived from optical diffraction analysis, is detected in this HREM micrograph of a film deposited at 400 ºC.

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