RF sputtered and sol-gel prepared comparison of MOO_3-TIO_2 microstructure and gas sensing properties

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Semiconducting molybdenum oxide and titanium oxide (MoO3-TiO2) binary compound thin films have been fabricated onto alumina and silicon substrates using r.f.sputtering and sol-gel techniques. By researching the same compositional material with different fabrication techniques a better understanding of the sensing material could be achieved. The MoO3-TiO2 novel binary compound system overcomes several limitations that MoO3 single metal oxide films intrinsically possess. MoO3 has promising O2, CO and NO2 sensing capabilities [1-3], however, due to its low evaporation temperature the thin films cannot be reliably operated above 300°C nor thermally treated for use as gas sensors beyond 500°C. MoO3 films also have a low conductivity making then impractical as gas sensors. The introduction of TiO2 improves on these concerns. Sputtering depositions were performed by an RFS plant (Kenotec) operated at 50 W RF power; the target was made of a Titanium and Molybdenum alloy (25% and 75% in weight). The deposition was executed under a reactive atmosphere of argon and oxygen at 2×10-3 mbar partial pressure for each gas while the substrate was kept at a temperature of 300°C. The MoO3-TiO2 sol-gel films prepared from Mo and W metal alkoxide precursors were deposited by the spin coating technique onto alumina substrate transducers, incorporating finger electrodes and a heater on the backside. Samples were annealed at temperatures between 450°C-800°C.

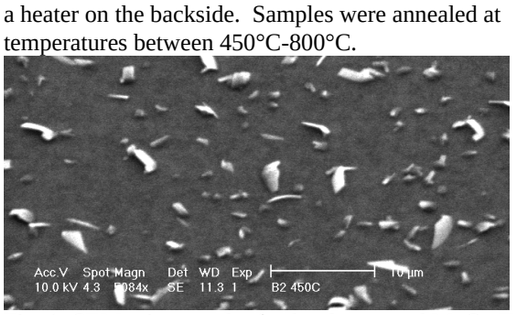

Fig. 1: SEM micrograph of sol-gel prepared MoO_3-TiO_2 (75-25 at. %) prepared thin film on Si substrate annealed at 450°C for 1 hour.

Fig. 1 shows an SEM micrograph of the sol-gel prepared thin film. Large grown up crystallised particles reaching a length of 1 _m exist. The particles have been identified to be MoO3 from EDX analysis. Fig. 2 reports the bright field (B.F.) image of MoO3 (75%)-TiO2 (25%) sol-gel thin film sample annealed at 450ºC. The grain size distribution is broad, peaking at 8 nm. The selected area diffraction patterns (SADP) revealed the presence of a predominant MoO3 orthorhombic phase and of a mixture of anatase and brookite TiO2 phases. Gas sensing performance of the MoO3-TiO2 r.f.sputtered thin films show that the sensors have a fast response of 32 seconds to 100 ppm of CO and 36 seconds to 3 ppm of NO2 while operating at 300°C. The best sensing performance of the r.f.sputtered samples was achieved with the as-deposited and 450°C annealed samples as shown in Fig. 3.

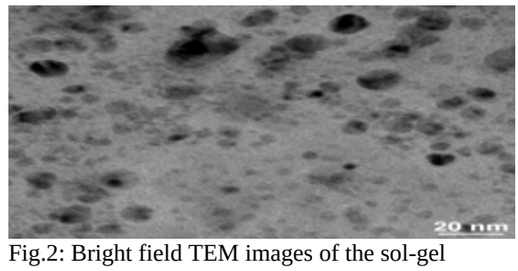

Fig.2: Bright field TEM images of the sol-gel MoO_3(76%)- TiO_2(24%) film annealed at 450°C.

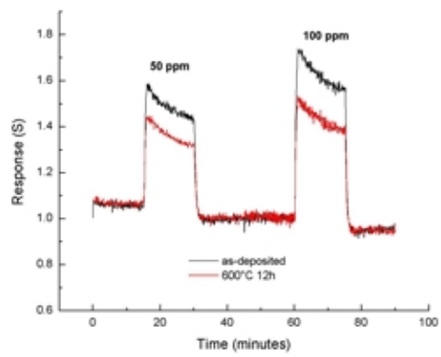

Fig. 3: R.f. sputtered CO response of MoO_3-TiO_2 thin film gas sensors prepared by different thermal treatments operating at 300°C.

The sol-gel samples annealed at 450°C showed better response to CO than that fabricated from r.f.sputtering. A response of 2 towards 25 ppm of CO and 2.6 to 2ppm of NO2 was obtained for the sol-gel sample at an operating temperature of 300°C as shown in Fig. 4. Such a sensor would be suited to a sensor array functioning as an electronic nose.

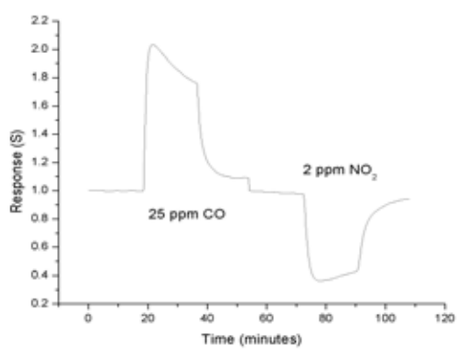

Fig. 4: CO and NO_2 response of sol-gel prepared MoO_3-TiO_2 (75-25 at. %) thin film gas sensors operating at 300°C.

Reference:

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[3] Y. X. Li, K. Galatsis, M. K. Ghantasala, et al., AVS 46th, Oct. 22-6, 1999. Washington, USA