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Preparation and characterization of thin films of molybdenum sulphide and selenide by a chemical deposition technique

P. PRAMANIK*, S. BHATTACHARYA
Department of Chemistry, Indian Institute of Technology, Kharagpur 721 302, India

Recently, considerable attention has been directed to the layered dichalcogenides of the group IVB and VB transition elements for device application. Generally, the thin films of these have been prepared by sputtering [1, 2] and the electrodeposition method [3]. We have attempted to deposit MoS_2 and $MoSe_2$ thin films by the chemical deposition technique which can be applicable for deposits on conducting and insulating substrates. The present letter describes our successful attempt to deposit MoS_2 and $MoSe_2$ thin films by the chemical deposition technique. No chemical method for the deposition of MoS_2 or $MoSe_2$ has yet been reported.

For the deposition of MoS_2 thin films, 10 ml 5% ammonium molybdate solution was taken in a 50 ml beaker and to it 15 ml 14 M ammonia solution and 10 ml 80% hydrazine hydrate were added successively, with constant stirring in order to form a clear homogeneous solution. To this 15 ml 1 M thioacetamide solution was added and mixed. The solution was then poured into another beaker containing a scrupulously cleaned glass slide, clamped vertically. Finally, the mixture was heated at 100° C with constant stirring for $\frac{1}{2}$ h. After this time the beaker was taken out and was kept at room temperature for 12 h.

For the deposition of $MoSe_2$ thin films, 10 ml 5% ammonium molybdate was taken in a 50 ml beaker. Then 15 ml ammonium acetate, 3 ml acetic acid, and 10 ml hydrazine hydrate were added successively. The solution was stirred well and 10 ml 0.4 M sodium selenosulphate solution was added to it. In a similar way as for molybdenum disulphide, the solution containing a glass slide was heated at 100° C with constant stirring for 1 h. It was then kept at room temperature for 12 h.

The basic chemical reactions that lead to the formation of MoS_2 and $MoSe_2$ are as follows
For sulphide

$$CH_3\text{-}\underset{\underset{S}{\|}}{C}\text{-}NH_2 + OH^- \rightarrow CH_3\text{-}\underset{\underset{O}{\|}}{C}\text{-}NH_2 + S^{2-} \quad (1)$$

$$MoO_4^{2-} + NH_2NH_2 + S^{2-} \rightarrow MoS_2 + H_2O + N_2 \quad (2)$$

For selenide

$$Na_2SeSO_3 + 2OH^- \rightarrow Na_2SO_4 + Se^{2-} + H_2O \quad (3)$$

$$Se^{2-} + NH_2NH_2 + MoO_4^{2-} \rightarrow MoSe_2 + N_2 + H_2O \quad (4)$$

After 12 h, both slides which were covered with a brown deposit, were taken out, washed with distilled water and dried in a desiccator. The thickness of the MoS_2 films weas in the range 0.5 to 0.65 μm and for $MoSe_2$ films 0.4 to 0.5 μm. The thickness was measured using a Taylor Hobson Talystep instrument.

X-ray diffraction data revealed that the films of MoS_2 prepared by the present method are amorphous

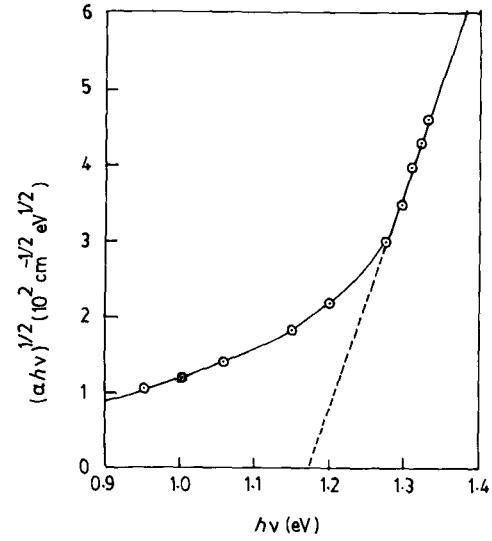

Figure 1 Optical absorption of MoS_2 thin films.

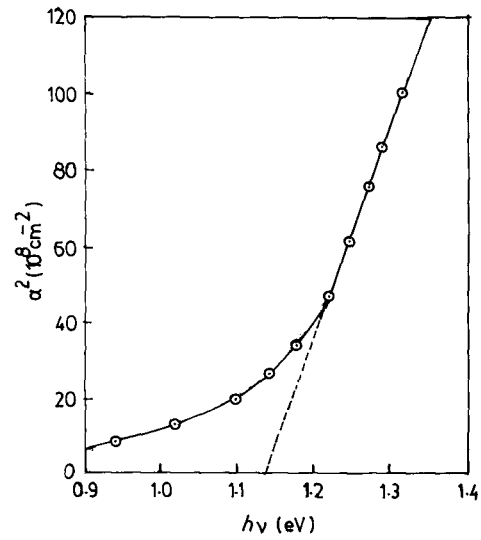

Figure 2 Optical absorption of $MoSe_2$ thin films.

*Author to whom all correspondence should be addressed.

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TABLE I X-ray data of MoS_2 films prepared by the present method

Observed d values for MoS_2 (nm)	Possible identification with standard d (nm)
0.615	0.615 (002)
0.274	0.2737 (100)
0.227	0.2277 (103)
0.205	0.2049 (006)
0.182	0.1830 (105)
0.159	0.1581 (110)
0.154	0.1538 (008)

TABLE II X-ray data of $MoSe_2$ films prepared by the present method

Observed d values for MoS_2 (nm)	Possible identification with standard d (nm)
0.645	0.646 (003)
0.340	0.343 (006)
0.215	0.216 (009)
0.165	0.1643 (110)
0.160	0.1615 (0012)

in nature because the X-ray diffractograph did not show any sharp peaks; however, after crystallization in the above reagent at 300° C in a high-pressure autoclave under an inert atmosphere, sharp peaks of MoS_2 were observed. The observed d values of MoS_2 (crystallized) and $MoSe_2$ are in good agreement with the standard d values, taken from the ASTM Diffraction Data File [4, 5], which are shown in Tables I and II. The films of $MoSe_2$ are polycrystalline in nature. The chemical analyses are also consistent with the chemical formulae of MoS_2, $MoSe_2$.

The optical density of MoS_2 and $MoSe_2$ thin films measured at various wavelengths by a Cary 17-D spectrophotometer, were used to determine the absorption coefficient, α, for the samples at various photon energies. A plot of $\alpha h\nu$ against $h\nu$ for MoS_2 films is shown in Fig. 1 and a plot of α^2 against $h\nu$ for $MoSe_2$ films is shown in Fig. 2. In both cases the linear portion of the curve to $\alpha^2 = 0$ or $\alpha h\nu = 0$ gives the optical band gap, which is 1.17 eV for MoS_2 and 1.14 eV for $MoSe_2$. These values are in good agreement with the reported gap [5, 6].

Using a thermoelectric probe it was observed that the present films are n-type in nature. Their conductivities lie in the region of $10^{-4}\,s\,cm^{-1}$, as measured by the four-probe method at room temperature (30° C). In all cases, graphite paint was used as an electrical contact. More detailed studies are in progress.

References

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