JOURNAL OF MATERIALS SCIENCE 18 (1983) 3606–3610

# Growth and surface topography of cadmium films

AMARJIT SINGH*, C. S. HARENDRANATH
*Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Powai, Bombay 400076, India*

The growth of cadmium films deposited on amorphous glass substrate were studied using X-ray diffraction and transmission electron microscopy techniques. The analysis of the experimental data showed a very high preferred orientation of the crystallites with the crystallographic *c*-axis normal to the substrate, indicating a layer type growth with high preferred orientation of the basal planes parallel to the substrate. The films obtained at room temperature were polycrystalline in nature with large grain size. Surface features were studied using SEM, indicating a larger crystallite size for films grown on a few atomic layers of titanium on glass substrate.

## 1. Introduction

Cadmium chalcogenides forming compound semiconductors have found great technological importance because of their extensive use in electronic devices and solar cells. Incidentally, very few studies have been reported on cadmium films; one of the reasons being that it is difficult to prepare a homogeneous and continuous film of good quality. Moreover, cadmium does not readily condense on a substrate unless the vapour intensity is extremely high or it is pre-coated with atoms of low vapour pressure [1] to act as nuclei for condensation.

The studies which have been mainly reported on cadmium films in the recent past have been those on electrical properties.

The measurement of the electrical resistivity at 4.2 K was carried out on polycrystalline cadmium films by Schwarz and Luck [2], while thickness, mean grain diameter and surface quality were also varied. Later on, Hambourger and Marcus [3] studied the size dependence of the magnetoresistance of cadmium films and observed oscillations in it and in the Hall effect. The sheet resistance and Hall voltage of vacuum-evaporated cadmium films were investigated in the thickness range .5 to 25 nm by Reale [4], and the size-effect was interpreted by extending the Sondheimer theory to an anisotropic metal with non-spherical energy surfaces where the bands overlap, assuming the participation of both electrons and holes in the charge transport. Recently Ghosh *et al.* [5] reported their measurements on the Hall effect. Quasi-whisker-like structures [6] resembling stacked platelets and the dependence of the temperature coefficient of resistance and resistivity of the cadmium films on the thickness has also been investigated [7].

In the present paper, the growth of cadmium films on amorphous glass substrate is studied. Furthermore, the surface features and the influence of different substrates on the growth is also discussed.

## 2. Experimental details

### 2.1. Preparation of cadmium films

In order to obtain a high vapour intensity, high purity (99.99%) cadmium metal supplied by Light Laboratories, UK, was taken as a source material. It was placed at the bottom of a cleaned quartz tube of 10 cm length with resistance winding of nichrome wire used to heat the tube, while a carefully cleaned glass substrate was kept at a distance of 10 cm from the open end of the quartz tube. The whole apparatus was kept in a high vacuum system operated by a mechanical pump backed by

*Present address: Université Laval, Laboratoire de Physique Nucléaire, Département de Physique, Québec, Canada G1K 7P4.


0022–2461/83 $03.00 + .12 

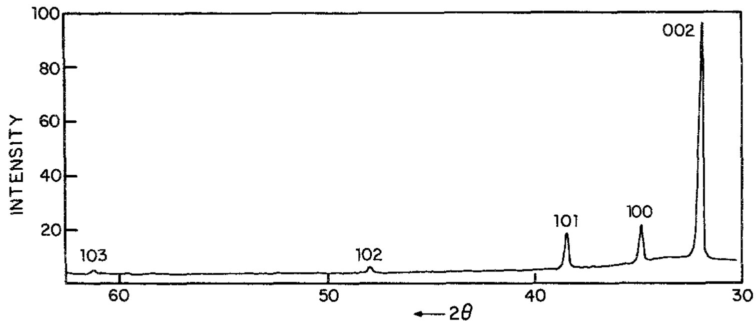


*Figure 1* X-ray diffraction pattern of a typical cadmium film of thickness 250 nm deposited on glass at room temperature. Scattering angle is $2\theta$.

an oil diffusion pump. The pressure in the coating chamber was better than $1 \times 10^{-5}$ torr.

## 2.2. Preparation of samples for electron microscopy, X-ray and SEM studies

As cadmium films are resistant to dilute hydrofluoric acid, they were peeled off from the glass substrate with dilute hydrofluoric acid. This was possible because dilute hydrofluoric acid attacks preferentially the glass substrate involving the cadmium film and reacted with the glass surface. Continued reaction of dilute HF acid with the glass removed the cadmium film from the glass surface. The thickness of the films were less than 100 nm. The films thus peeled off were then mounted on copper grids and transferred to Philips transmission electron microscope model EM 301. X-ray diffraction of cadmium films of thickness 250 nm were obtained on a Philips X-ray diffractometer model PW 1051. Cu$K\alpha$ ($\lambda = 0.1542$ nm) radiation was used and the scanning range of $2\theta$ was restricted to the range 30° to 60° as no detectable peaks were observed beyond these values.

To examine the surface features of the deposited cadmium films, SEM studies were carried out using a Siemens "AUTOSCAN" scanning electron microscope at 25 kV using the secondary electron mode.

## 3. Results and discussions

Fig. 1 shows the X-ray diffraction pattern of a vapour deposited cadmium film on a glass substrate at room temperature. The scanning speed was kept $1° \mathrm{min}^{-1}$. Typical results obtained from a diffractometer trace are given in Table I. The calculated values of interplanar spacing $d$ agreed well with the National Bureau of Standards (NBS) diffraction data for cadmium powder. It was found that the reflection from the basal plane (002) gave very sharp and intense peak while the general ($hkl$) reflections were weak. These features are characteristic of layer type growth and an indication of highly preferred orientation of the basal planes parallel to the substrate.

A series of transmission electron micrographs and electron diffraction patterns were obtained to analyse the microstructural features of cadmium films deposited at room temperature. The micrograph in Fig. 2 shows the microstructure of the film. The film is found to be composed of crystallites with a high density of imperfection. The film structure consists of crystals of 156 nm grain diameter.

The electron diffraction pattern for the cadmium film is shown in Fig. 3. Films deposited on room temperature substrate gave polycrystalline diffraction patterns. The interplanar spacings were calculated from the ring radii measured accurately on a microdensitometer. The results obtained from the electron diffraction are shown in Table II.

The data relating to this particular analysis indicates that in all the films examined only ($hk0$) type reflections were seen, which further indicates a very high preferred orientation of the crystallites with the crystallographic $c$-axis normal to the substrate. Moreover, the sharp ($hk0$) rings seen indicate the presence of large crystals with their $c$-axis oriented normal to the substrate and the $a$-axis randomly oriented.

TABLE I Analysis of the X-ray diffractometer trace of 250 nm thick cadmium film deposited at room temperature

| $hkl$ | Relative intensity | Calculated $d$ spacing (nm) |
|---|---|---|
| 002 | vs | 0.2806 |
| 100 | w | 0.2578 |
| 101 | w | 0.2344 |
| 102 | vw | 0.1899 |
| 103 | vw | 0.1514 |

vs = very strong; w = weak; vw = very weak.

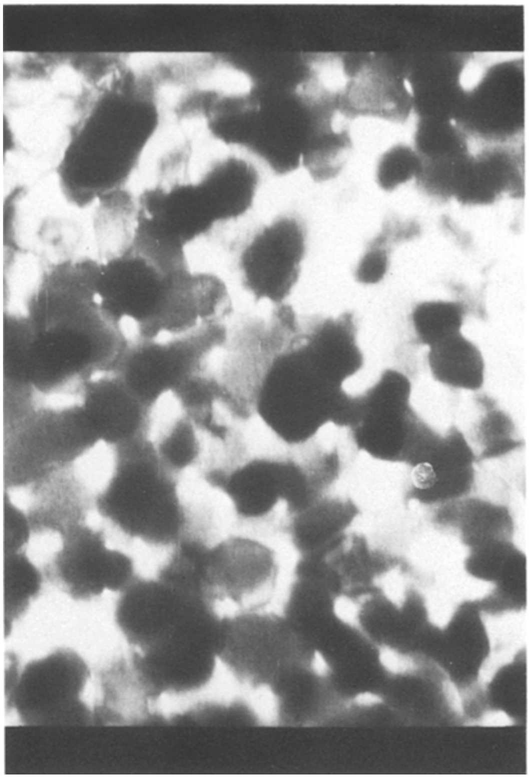

*Figure 2* Transmission electron micrograph of ~ 100 nm thick cadmium film deposited on glass substrate. Magnification (18 480 X).

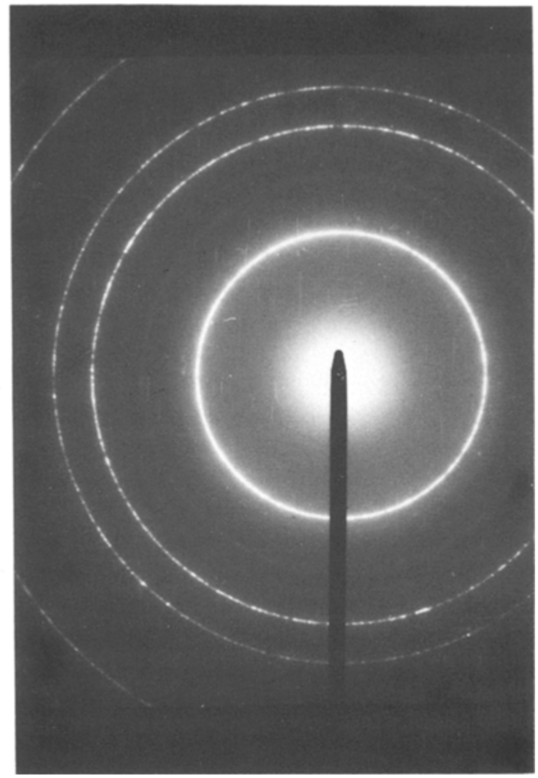

*Figure 3* Transmission electron diffraction of ~ 100 nm thick cadmium film at 80 keV.

To examine the surface topography and the influence of different substrates like glass, titanium with few atomic layers on glass, and mica on the growth of cadmium films, a series of scanning electron micrographs were obtained.

Fig. 4 shows the SEM micrographs of cadmium films deposited on different substrates. The surface topography in these micrographs illustrates the distribution of microcrystallites, their shape and growth.

Fig. 4a shows the distribution of microcrystallites of cadmium film on glass substrate. These crystallites have definite shape and its nature was found to be polycrystalline. Similar features are seen when the substrate is mica. While there is a noticeable change in the size of the crystallite for films obtained on few atomic layers of titanium on glass. These crystallite appear to have hexagonal structure. The increase in the size of the microcrystallites can be attributed to the fact that titanium has a high vapour pressure and the base nuclei helps to condense the initial incoming cadmium atoms, hence the growth is more homogeneous and larger crystallites are observed.

It is apparent that the nucleation and growth of thin film on a substrate is greatly influenced by the atomic structure of the substrate and the bond character of the material [8]. However, on an amorphous substrate, the influence of the potential field of the substrate is small and the film growth is determined by the binding forces between the condensed atoms. The freshly condensed atoms have a relatively high surface mobility and the first atomic layer is formed in a two-dimensional crystalline order corresponding to the natural arrangement of the condensing material. As a consequence, the first layer of cadmium is formed of two-dimensional platelets with the basal

TABLE II Analysis of the electron diffraction pattern of cadmium film

| Ring number | *hkl* | Calculated *d* spacing (nm) |
|---|---|---|
| 1 | 100 | 0.2585 |
| 2 | 110 | 0.1498 |
| 3 | 200 | 0.1293 |
| 4 | 210 | 0.0981 |

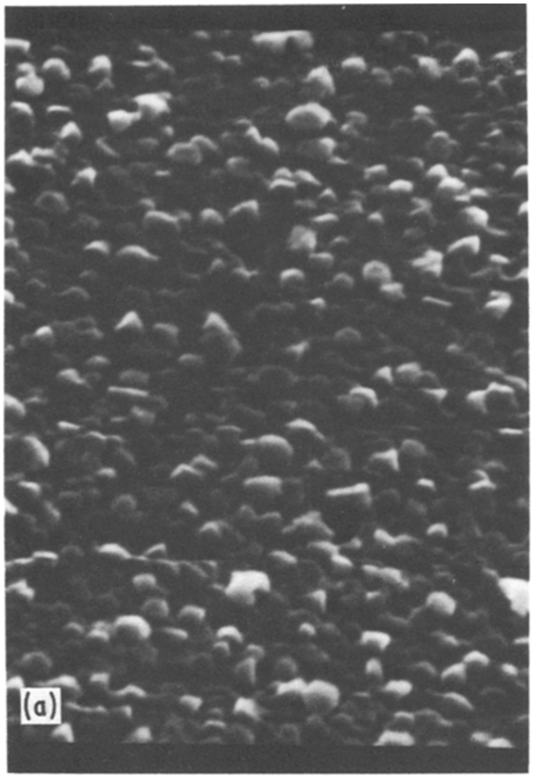




*Figure 4* Scanning electron micrographs of cadmium film grown on different substrate: (a) glass (b) few atomic layers of titanium on glass. Original magnification: (13 440 ×). (c) Mica, original magnification: (16 800 ×).

plane parallel to the substrate. These platelets form the base for crystallization of subsequently condensing layers. Thus, the film is grown with crystallites oriented with their *c*-axis normal to the substrate. Similar results have been obtained for hexagonal systems like GaSe [9] and also for tellurium films [10]. The results of surface topography reveal larger crystallites when grown on few atomic layers of titanium. This can be attributed to the fact that titanium, being a hexagonal system, will more likely help in the initial nucleation of cadmium films, which are also hexagonal systems. Moreover, cadmium having low vapour pressure will favour a more continuous growth on substrates coated with few atomic nuclei [1].

## 4. Conclusions

Cadmium films grown on room temperature substrate like glass are polycrystalline in nature, indicating a layer type growth with their basal plane parallel to the substrate, and a very high preferred orientation of the crystallites with the *c*-axis normal to the substrate. Furthermore, surface features reveal larger crystallites in size for films obtained on glass pre-coated with a few atomic layers of titanium.


## Acknowledgements

One of the authors (AS) wishes to thank NSERC, Canada, for financial assistance, and is grateful to Professor R. J. Slobodrian for the hospitality extended to him at the Université Laval, Québec, where this paper was written.


## References


1. L. HOLLAND, "Vacuum Deposition of Thin Films" (Chapman and Hall, London, 1970) p. 180.
2. H. SCHWARZ and R. LUCK, *Mater. Sci. Eng. (Netherlands)* **5** (1970) 149.
3. P. D. HAMBOURGER and J. A. MARCUS, *Phys. Rev. B* **8** (1973) 5567.
4. C. REALE, *Atti Fond Giorgio Ronchi Contribution Inst. Naz., Ottica* **15** (1973) 46.
5. C. K. GHOSH, S. K. BANDHOPADHYAY and A. K. PAL, *Thin Solid Films* **76** (1981) 313.
6. H. G. NEUMANN, *ibid.* **8** (1971) R55.
7. S. K. BANDHOPADHYAY and A. K. PAL, *Indian J. Phys.* **55A** (1981) 270.
8. G. A. BASETT, J. W. MENTER and D. W. PASHLEY, in "Structure and Properties of Thin Films", edited by C. A. Neugebauer, J. B. Newkirk and D. A. Vermilyea (Wiley, New York, 1959) p. 10.
9. M. B. THOMSON, *Thin Solid Films* **8** (1971) 273.
10. M. J. CAPERS and M. WHITE, *ibid.* **8** (1971) 353.



*Received 17 February and accepted 23 March 1983*