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# Synthesis and characterization of indium intercalation compounds of molybdenum sulphoselenide

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The synthesis, structure and properties of indium-intercalated compounds of molybdenum sulphoselenide, $In_{1/3}MoS_xSe_{2-x}$ $(0 \le x \le 2)$ compounds were investigated. X-ray analysis shows that all these compounds possess layer-type structure with hexagonal symmetry. Studies of room-temperature magnetic susceptibility, thermoelectric power experiments and two-probe conductivity measurements in the temperature range 25–350 °C, confirmed that these are diamagnetic semiconductors with $In_{1/3}MoS_2$, $In_{1/3}MoS_{1.5}Se_{0.5}$ and $In_{1/3}MoS_xSe_{2-x}$ $(0 \le x \le 1.5)$ exhibiting n- and p-type conductivity, respectively. These results have been explained on the basis of existing band models. Thermal stability behaviour of these compounds in an air atmosphere and X-ray analysis of the oxidized product were also studied.

## 1. Introduction

The layered transition metal dichalcogenides (LTMD) $MX_2$ (M = transition metal of group IVB, VB or VIB, X = chalcogen, S, Se or Te) constitute a class of materials with unique and unusual properties based on the extreme degree of anisotropy in their structures [1–5]. They are formed by stacking "sandwiches" consisting of a layer of transition metal between the two layers of chalcogen. There exists a strong covalent bonding within the sandwiches but weak van der Waal's force between them. Within such a layer, a metal may be in trigonal prismatic or octahedral coordination depending on the relative values of the ionic ratio $r_{M^+}/r_{X^-}$ and fractional ionic character $f_i$ where, $f_i = 1 - \exp[-\frac{1}{4}(X_M - X_X)^2]$ and $X_M$ and $X_X$ are the electronegativities of the metal and chalcogen atoms, respectively [6]. These compounds exhibit a wide variation in electrical properties ranging from insulating to semiconducting, and finally metallic, with the evidence of superconductivity in some of the members of this class. It is also well established that LTMD also facilitates the process of intercalation by accommodating a wide variety of guests, e.g. metal atoms, organic and inorganic molecules in the van der Waal's gap between the layers [7–9]. The resultant changes in the structural and physical properties accompanying their intercalation process have received considerable interest over the past few decades. In many cases, the observed changes have been explained on the basis of a model which assumes that, to a first approximation, the host band structure is unchanged on intercalation and electrons are transferred from the intercalate to the conduction band of the host crystal. These LTMD and their intercalation compounds find a wide range of applications, e.g. in hydrogen storage devices [3], high-temperature–high-pressure lubricants [7], solid-state electrolyte batteries [9] in the field of catalysis [10] and in photovoltaic solar cells for solar energy conversion purposes [11].

Layer-type molybdenum dichalcogenides, being diamagnetic and semiconducting, are one such representative of this family and are most promising materials for modern uses [12–14]. These compounds form a wide range of solid solution, e.g. $MoS_xSe_{2-x}$ $(0 \le x \le 2)$, $MoS_xTe_{2-x}$ $(0 \le x \le 2)$ over the complete range of composition. These mixed crystals are isomorphous and have recently been investigated for optical and electrical studies [15–17]. Earlier studies have shown that diamagnetic semiconducting $MoS_2$ and $MoSe_2$ become metallic and superconducting when intercalated with alkali and alkaline-earth metals [18–20]. Recently, the intercalation of these compounds with the post-transition metal has also been reported by us [21–24]. These results were found to be very interesting and encouraging, and therefore we have extended our studies on indium intercalation compounds for isostructural molybdenum sulphoselenide, $In_{1/3}MoS_xSe_{2-x}$ $(0 \le x \le 2)$. Because few additional binary and ternary phases appeared with increasing indium contents we have focused our studies on the compositions mentioned earlier. These materials have been characterized by X-ray analysis for structure determination, room-temperature magnetic susceptibility and thermoelectric power experiments, two-probe electrical conductivity measurements in a temperature range 25–350 °C, thermal stability in an air atmosphere and X-ray studies of the oxidized products. A comparison of these data with those of the binary layer-type compounds $MoS_2$ and $MoSe_2$ has also been made to obtain information about the contribution of chalcogens on indium intercalation compounds of molybdenum sulphoselenide.

## 2. Experimental procedure

### 2.1. Sample preparation

In the present work, $MoS_2$, $MoSe_2$ and $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) compounds were synthesized directly from the elements (all 99.99% pure) and the method of preparation was quite similar to that reported earlier [8]. Appropriate amounts of indium powder, molybdenum powder, sulphur and selenium were accurately weighed to give a desired composition, mixed intimately to give a homogeneous mixture and placed inside the quartz ampules of 12–15 mm diameter and 100–120 mm long and vacuum sealed. For the preparation of the compounds, the samples were heated slowly ($50\,°C\,h^{-1}$) up to 400 °C inside a furnace and kept for 2 days. This heating is necessary to avoid the explosion of the tubes due to a strong exothermic reaction between molybdenum metal and sulphur. This was followed by heating of the samples at 750° C for 48 h. A loose powdered product results in having a considerably larger volume than the reacting elements. This was mixed well mechanically and subsequently placed inside the furnace and heated at 1000 °C for 40 h followed by slow cooling to room temperature.

### 2.2. Characterization

The material was studied by X-ray diffraction analysis on a Philips-1729 diffractometer using $CuK_\alpha$ radiation to examine the presence of all the possible phases. The crystallite size for each specimen was calculated using Sherrer's formula

$$t = \frac{K_\lambda}{\beta \cos \theta_0} \quad (1)$$

where $t$ is the crystallite size as measured perpendicular to the reflecting plane, $K$ is Sherrer's constant whose value is taken to be unity assuming the particles to be spherical, $\lambda$ is the wavelength of the X-radiations, $\beta$ is the half intensity width measured in radians and $\theta_0$ is the Bragg angle.

Room-temperature (25 °C) magnetic susceptibility was measured using a Gouy balance in a field strength of about 5000 g. The balance was initially calibrated using Hg $[Co(NCN)_4]$ as a standard. The type of conductivity was determined by simple thermoelectric power experiments. The two-probe electrical conductivity measurements were performed on a compressed circular pellet in a pure argon atmosphere in the temperature range 25–350 °C. Thermal analysis was carried out on a Redcroft-870 in an air atmosphere up to 1000 °C with a programmed heating rate of $10\,°C\,min^{-1}$ and sensitivity of 200 μV for DTA. The X-ray analysis of the oxidized products were performed by heating the samples at 800 °C for 2 h.

## 3. Results and discussion

All the compounds were black in colour and unlike alkali and alkaline-earth metals, intercalation compounds, e.g. $A_xMoS_2$ (A = Li, Na, K, etc.) were found to be stable on exposure to air. Such a stable behaviour is similar to that already reported for $In_xMCh_2$ (M = Nb, Ta; Ch = S, Se) where it was assumed that the guest–host bonding is fairly strong with the result that deintercalation is difficult [3].

The X-ray diffraction patterns of indium-intercalated compounds molybdenum sulphoselenide, $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) are displayed in Fig. 1. These compounds were thoroughly scanned for all the possible phases, e.g. $MoS_2$, $MoSe_2$, InS, $In_2S_3$, $In_7S_6$, InSe, $In_2Se_3$, $In_7Se_6$, $Mo_3S_4$, $Mo_3Se_4$, $In_xMo_6S_8$ [25] and $In_xMo_6Se_8$ [26], etc. It was observed that the diffraction pattern of these indium-intercalated compounds of molybdenum sulphoselenide, $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) showed a marked resemblence to that of 2H–$MoS_2$ [27] and 2H–$MoSe_2$ [28], respectively, with increasing selenium contents. It was therefore concluded that in the present case, even upon intercalation, the arrangement of Mo/W and S/Se remains virtually the same as in 2H–$MoS_2$/2H–$MoSe_2$ except for a slight expansion of van der Waal's gap due to incoming indium. It was also evident from the diffractograms that for each specimen, 0 0 2 reflections are of maximum intensity, indicating thereby a strong orientation along the $c$-axis. The intensity of the rest of the 0 0 l reflections generally increased, and broadening decreased to some extent with increasing selenium contents in $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$). This clearly suggested that the introduction of indium into molybdenum sulphoselenide has definitely introduced some layer disorder. It is also seen that with increasing amounts of selenium, the intensity of the 1 0 0 and 1 0 1 reflections continues to increase while for $hk0$ reflections it increased nearly two-fold from $In_{1/3}MoS_2$ to $In_{1/3}MoS_{1.0}Se_{1.0}$ and thereafter it reduced gradually to $In_{1/3}MoSe_2$.

The diffractograms of $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) also indicated that new $d$-lines (0.318, 0.237, 0.221 nm), though of relatively very low intensity ($< 5$), appeared in a few cases. These may be due to some poorly developed phases, the identity of which, however, could not be ascertained. Table I records the interplanar distance $d$ for $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) compounds.

The lattice parameters for $MoS_2$, $MoSe_2$ and their indium-intercalated molybdenum sulphoselenide compounds are presented in Table II. It was noted that indium intercalation produced an increase in $c$-parameter when compared to the corresponding parent layered host crystals [17]. However, this increase in $c$-parameter is not as large as that observed in alkali and alkaline-earth metal intercalates of $MoS_2$ and $MoSe_2$ [17–19]. Our findings were found to be in good agreement with other workers on $Cu_xNbS_2$ [26], $Cu_xNbSe_2$ [27], $Al_xTaS_2$, $Zn_xTaS_2$ [28], $Li_xYCl$, $Li_xGdCl$ [29] and $Li_xNiPS_3$ [30] which showed a small or no expansion in the $c$-parameter. This led to the conclusion that the process of intercalation is not necessarily accompanied by an increase in

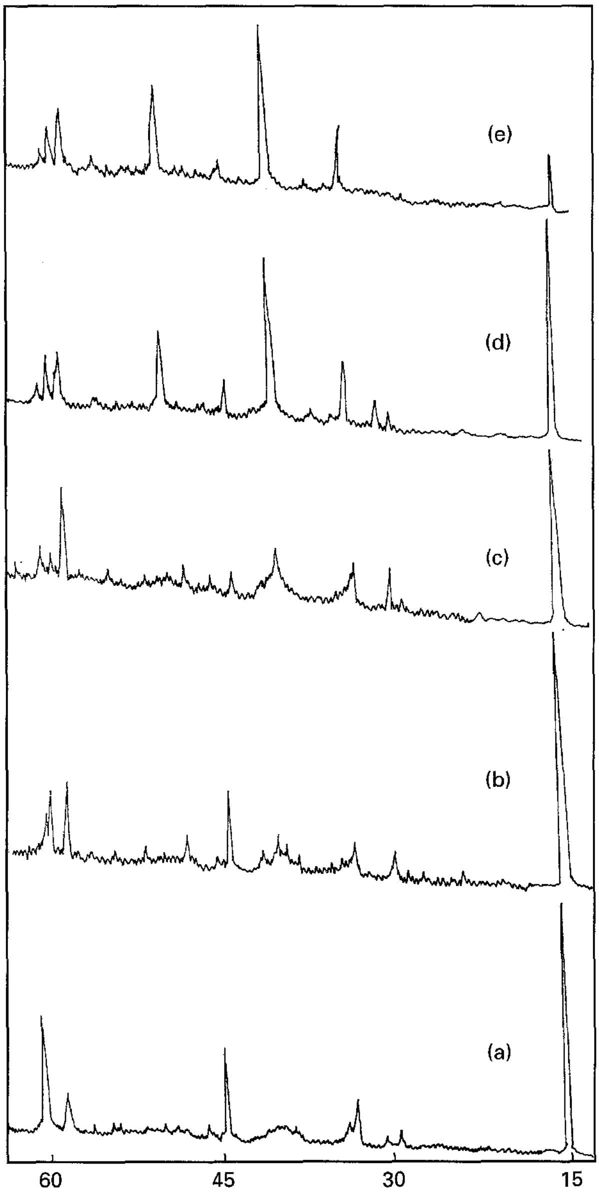

*Figure 1* X-ray diffractograms of (a) $In_{1/3}MoS_2$, (b) $In_{1/3}MoS_{1.5}Se_{0.5}$, (c) $In_{1/3}MoS_{1.0}Se_{1.0}$, (d) $In_{1/3}MoS_{0.5}Se_{1.5}$, (e) $In_{1/3}MoSe_2$.

*c*-parameter. It may be stated here that the increase in *c*-parameter, maximum stoichiometry, change in interlayer stacking mode and coordination geometry of the host metal ions, are all correlated with the type and electronic nature of the host lattice, ionicity of M–X bonding, and size and concentration of the guest intercalate.

Table III records the crystallite size for each specimen as measured perpendicular to the various planes. As the thickness measured to different planes is more or less the same, there exists an isotropy towards the crystallite size.

Room-temperature magnetic susceptibility measurements and thermoelectric power experiments suggest that, like the parent host 2H–$MoS_2$ and 2H–$MoSe_2$, all indium-intercalated compounds of molybdenum selphoselenide, $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$), are diamagnetic with $In_{1/3}MoS_2$, $In_{1/3}MoS_{1.5}Se_{0.5}$ and $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 1.5$) exhibiting n- and p-type conductivity, respectively (Table II).

The two-probe electrical conductivity data for all these compounds in the temperature range 25–350 °C are presented in Fig. 2. This indicated that all the compounds are semiconducting. Such a behaviour is contrary to the usual paramagnetic and metallic character of alkali and alkaline-earth metal intercalates of $MoS_2$ and $MoSe_2$ [12]. Thus it may be concluded that the charge-transfer band model, which assumes that electrons are transferred from the intercalate to the conduction band of the host crystal, does not seem to be applicable for all the intercalated layered transition metal dichaleogenides [1]. A behaviour similar to our case has also been observed by other workers on some other transition metal dichaleogenides, e.g. $A_xZrS_2$ (A = Fe, Co, Ni) [31], $A_xZrS_2$ and $A_xHfS_2$ (A = Cu, Fe for $x \leq 0.22$) [32, 33] where the host crystal remained semiconducting even upon intercalation. Rouxel [34] suggested that the electrons given by the intercalate may become trapped in certain sites rather than being delocalized in the conduction band of the host crystal. Yacobi *et al.* [32, 33] proposed that the intercalate atoms might be regarded as deep-level impurity leading to an additional energy level near the top of the valency band or alternatively a modification of the host crystal might occur due to the interaction between the intercalate and chalcogen atoms.

Table II records the room-temperature conductivity, $\sigma$, data for $MoS_2$, $MoSe_2$ and $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) compounds. It shows that indium intercalation in $MoS_2$ and $MoSe_2$ produced a relatively sharp increase in conductivity, while in $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$), it increased initially with increasing selenium contents, became steady over

TABLE I X-ray powder diffraction data for $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$)

| $In_{1/3}$ $MoS_2$ *d*(nm) | $I/I_1$ | $In_{1/3}MoS_{1.5}$ $Se_{0.5}$ *d*(nm) | $I/I_1$ | $In_{1/3}MoS_{1.0}$ $Se_{1.0}$ *d*(nm) | $I/I_1$ | $In_{1/3}MoS_{0.5}$ $Se_{1.0}$ *d*(nm) | $I/I_1$ | $In_{1/3}$ $MoSe_2$ *d*(nm) | $I/I_1$ |
|---|---|---|---|---|---|---|---|---|---|
| 0.6162 | 100 | 0.6258 | 100 | 0.6266 | 100 | 0.6366 | 100 | 0.6451 | 28 |
| 0.3078 | 5 | 0.3063 | 5 | 0.31 | 22 | 0.3186 | 7 | 0.3235 | 5 |
| 0.2058 | 17 | 0.2074 | 14 | 0.2105 | 15 | 0.2133 | 22 | 0.2154 | 14 |
| 0.1542 | 8 | 0.1560 | 12 | 0.1580 | 17 | 0.1600 | 24 | 0.1615 | 26 |
| 0.2737 | 11 | 0.2726 | 6 | 0.2771 | 24 | 0.2814 | 28 | 0.2840 | 37 |
| 0.1581 | 22 | 0.1594 | 12 | 0.1606 | 50 | 0.1623 | 30 | 0.1646 | 37 |
| – | – | – | – | – | – | 0.2575 | 8 | – | – |
| 0.2271 | 5 | 0.2283 | 6 | 0.2321 | 28 | 0.2349 | 72 | 0.2374 | 100 |
| 0.1832 | 5 | 0.1850 | 5 | 0.1871 | 10 | 0.1895 | 37 | 0.1914 | 50 |
| – | – | – | – | 0.1555 | 20 | 0.1574 | 10 | 0.1591 | 8 |

TABLE II Unit cell dimensions and other data for $MoS_2$, $MoSe_2$ and $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) compounds

| Compounds | Lattice parameters | | | | Magnetic susceptibility $-\chi_g (10^{-6}$ cgs units | Conductivity type | Conductivity, $\sigma_{300\ K}$ ($\Omega^{-1}cm^{-1}$) | Activation energy (eV) |
|---|---|---|---|---|---|---|---|---|
| | $a$ (nm) | $c$ (nm) | $c/a$ | $V$ ($nm^3$) | | | | |
| $MoS_2$ | 0.3160 | 1.2295 | 0.3890 | 0.1063 | 0.136 | n | $8.3 \times 10^{-6}$ | 0.38 |
| $In_{1/3}MoS_2$ | 0.3158 | 1.2322 | 0.3900 | 0.1064 | 0.140 | n | $1.4 \times 10^{-4}$ | 0.34 |
| $In_{1/3}MoS_{1.5}Se_{0.5}$ | 0.3171 | 1.2462 | 0.3931 | 0.1084 | 0.406 | n | $1.9 \times 10^{-4}$ | 0.31 |
| $In_{1/3}MoS_{1.0}Se_{1.0}$ | 0.3212 | 1.2652 | 0.3941 | 0.1129 | 0.285 | p | $9.2 \times 10^{-4}$ | 0.25 |
| $In_{1/3}MoS_{0.5}Se_{1.5}$ | 0.3242 | 1.2811 | 0.3954 | 0.1164 | 0.367 | p | $8.3 \times 10^{-4}$ | 0.19 |
| $In_{1/3}MoSe_2$ | 0.3285 | 1.2930 | 0.3940 | 0.1208 | 0.352 | p | $9.2 \times 10^{-3}$ | 0.15 |
| $MoSe_2$ | 0.3288 | 1.2891 | 0.3932 | 0.1210 | 0.250 | p | $2.9 \times 10^{-6}$ | 0.40 |

TABLE III Particle size distribution (nm) in $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$)

| $hkl$ | $x$ | | | | |
|---|---|---|---|---|---|
| | 0 | 0.5 | 1.0 | 1.5 | 2.0 |
| 006 | 38.2 | 47.6 | 27.1 | 23.7 | 52.4 |
| 008 | 34.1 | 33.9 | 33.7 | 25.2 | 48.2 |
| 110 | 33.7 | 33.6 | 32.1 | 20.4 | 33.1 |
| 103 | 28.4 | 31.3 | 21.3 | 26.7 | 42.4 |
| 105 | 26.2 | 29.1 | 27.3 | 24.2 | 32.1 |

a certain range and then further increased with increasing selenium contents. Because $\sigma$ is proportional to the mobility and carrier concentration, such a change in $\sigma$ with composition is therefore more likely due to either of these two, or both.

Results on thermal stability of $MoS_2$ and $MoSe_2$ in an air atmosphere showed that the behaviour is not significantly altered and a common trend is always

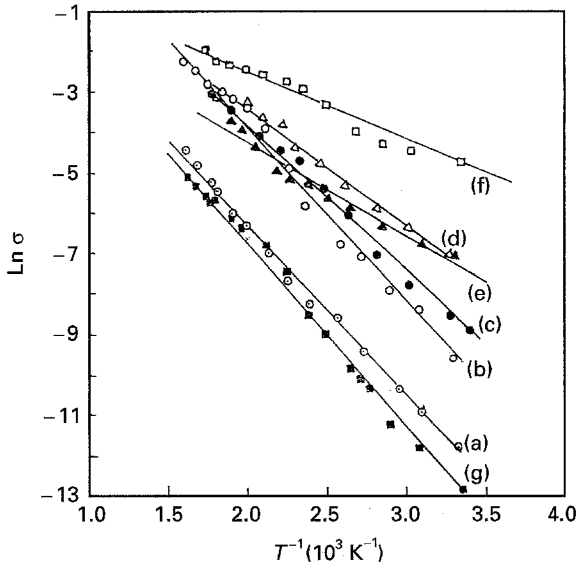

*Figure 2* Temperature dependence of electrical conductivity for (a) $MoS_2$, (b) $In_{1/3}MoS_2$, (c) $In_{1/3}MoS_{1.5}Se_{0.5}$, (d) $In_{1/3}MoS_{1.0}Se_{1.0}$, (e) $In_{1/3}MoS_{0.5}Se_{1.5}$, (f) $In_{1/3}MoSe_2$, (g) $MoSe_2$.

TABLE IV X-ray data for the oxidized product of $In_{1/3}MoS_xSe_{2-x}$ ($0 \leq x \leq 2$) when heated in air at 800 °C for 2 h

| $In_{1/3}$ $MoS_2$ $d$(nm) | $I/I_1$ | $In_{1/3}MoS_{1.5}$ $Se_{0.5}$ $d$(nm) | $I/I_1$ | $In_{1/3}$ $MoS_{1.0}$ $d$(nm) | $I/I_1$ | $Se_{1.0}$ $In_{1/3}MoS_{0.5}Se_{1.5}$ $d$(nm) | $I/I_1$ | $In_{1/3}$ $MoSe_2$ $d$(nm) | $I/I_1$ |
|---|---|---|---|---|---|---|---|---|---|
| 0.6976 | 95 | 0.6970 | 100 | 0.6970 | 100 | – | – | – | – |
| 0.6026 | 10 | 0.6026 | 37 | 0.6026 | 18 | – | – | – | – |
| 0.4745 | 30 | 0.4745 | 95 | 0.4745 | 70 | 0.4745 | 87 | 0.4720 | 100 |
| 0.4529 | 29 | 0.4529 | 29 | 0.4529 | 18 | 0.4522 | 28 | 0.4506 | 26 |
| 0.4250 | 16 | 0.4250 | 46 | 0.4264 | 37 | 0.4250 | 43 | 0.4230 | 52 |
| 0.4017 | 27 | 0.4022 | 88 | 0.4029 | 67 | 0.4095 | 68 | 0.4004 | 94 |
| 0.3818 | 28 | 0.3818 | 22 | 0.3813 | 23 | 0.4047 | 70 | 0.3801 | 17 |
| 0.3708 | 10 | 0.3708 | 32 | 0.3708 | 29 | 0.3708 | 21 | 0.3693 | 37 |
| 0.3590 | 19 | 0.3590 | 54 | 0.3600 | 44 | 0.3604 | 51 | 0.3590 | 63 |
| 0.3539 | 100 | 0.3479 | 49 | 0.3479 | 23 | – | – | 0.3453 | 13 |
| 0.3368 | 30 | 0.3376 | 87 | 0.3376 | 70 | 0.3376 | 79 | 0.3363 | 98 |
| 0.3326 | 12 | 0.3314 | 37 | 0.3326 | 29 | 0.3314 | 34 | 0.3314 | 42 |
| 0.3267 | 26 | 0.3266 | 38 | 0.3267 | 28 | 0.3267 | 17 | 0.3255 | 28 |
| 0.3079 | 11 | 0.3068 | 34 | 0.3079 | 28 | 0.3079 | 37 | 0.3068 | 35 |
| 0.2763 | 11 | 0.2959 | 40 | 0.2954 | 30 | 0.2956 | 35 | 0.2949 | 37 |
| 0.2763 | 14 | 0.2755 | 50 | 0.2758 | 37 | 0.2763 | 53 | 0.2755 | 51 |
| 0.2730 | 29 | 0.2730 | 94 | 0.2730 | 75 | 0.2730 | 100 | 0.2730 | 95 |
| – | – | 0.2495 | 9 | – | – | – | – | 0.2488 | 11 |
| 0.2309 | 60 | 0.2309 | 26 | 0.2315 | 12 | 0.2368 | 12 | – | – |
| 0.2270 | 59 | 0.2287 | 16 | 0.2291 | 12 | 0.2292 | 15 | 0.2287 | 18 |
| – | – | 0.2099 | 21 | – | – | 0.2103 | 21 | 0.2094 | 16 |
| 0.1785 | 11 | 0.1787 | 43 | 0.1787 | 34 | 0.1791 | 42 | 0.1784 | 43 |
| – | – | 0.1653 | 10 | – | – | 0.1686 | 14 | 0.1684 | 14 |
| 0.1631 | 5 | 0.1629 | 13 | 0.1626 | 9 | – | – | 0.1626 | 12 |
| 0.1570 | 8 | 0.1588 | 11 | – | – | – | – | – | – |

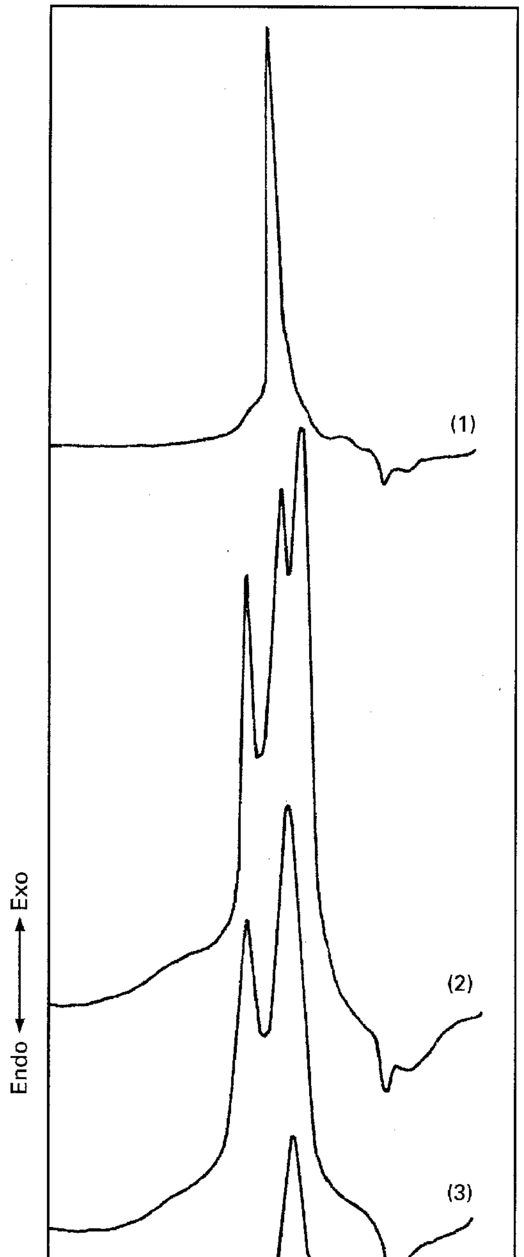

*Figure 3* (a) DTA and (b) TGA, for $In_{1/3}MoS_xSe_{2-x}$ $(0 \leq x \leq 2)$ compounds. (1) $In_{1/3}MoS_2$, (2) $In_{1/3}MoS_{1.5}Se_{0.5}$, (3) $In_{1/3}MoS_{1.0}Se_{1.0}$, (4) $In_{1/3}MoS_{0.5}Se_{1.5}$, (5) $In_{1/3}MoSe_2$, and (6) $MoSe_2$.

maintained. It showed the presence of a highly exothermic peak at 500 and 450 °C. Thermogravimetry (TG) showed the corresponding weight loss due to oxidation of these compounds to $MoO_3$, the presence of which was also confirmed by X-ray analysis of the oxidized product [35] (Table IV). Another sharp endothermic peak appeared at 790 °C due to melting of $MoO_3$ and no weight change was observed in its neighbourhood. At even higher temperature, a rapid weight loss in TG was noted due to continuous evaporation of $MoO_3$. These observations were found to be in good agreement with the reported data [36].

It was also seen from the thermograms of $In_{1/3}MoS_xSe_{2-x}$ $(0 \leq x \leq 2)$ compounds in Fig. 3 that an exothermic peak appears at 430 °C which becomes sharper with increasing sulphur content and without a concomitant weight loss. Such a change is more likely due to some phase transitions of the products originating from the oxidative degradation of these compounds. However, this peak was absent in $In_{1/3}MoS_2$. In addition, a strong exothermic peak appeared in each case at $\approx 560$ °C with the only exception of $In_{1/3}MoS_{1.5}Se_{0.5}$ where it was immediately followed by another exothermic peak at $\approx 610$ °C. As a result, a weight loss in TG was observed due to oxidation of these compounds. The thermograms of all indium-intercalated compounds $In_{1/3}MoS_xSe_{2-x}$ $(0 \leq x \leq 2)$ are also characterized by the presence of a very small endothermic peak at $\approx 770$ °C, unlike in $MoS_2$ and $MoSe_2$ where it appeared at 790 °C due to melting of $MoO_3$. Subsequently, a rapid weight loss was observed due to the volatilization of the oxidized

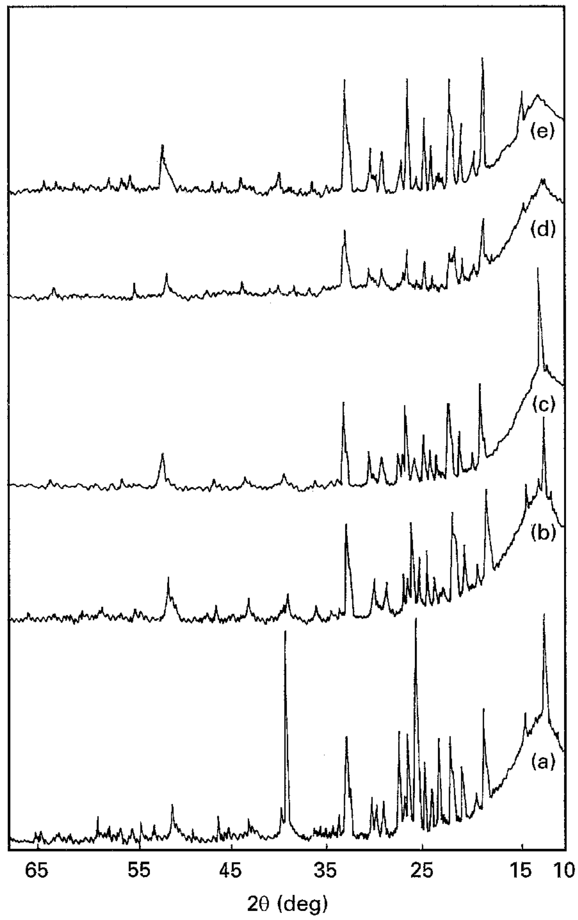

*Figure 4* X-ray diffractograms for the oxidized products of (a) $In_{1/3}MoS_2$, (b) $In_{1/3}MoS_{1.5}Se_{0.5}$, (c) $In_{1/3}MoS_{1.0}Se_{1.0}$, (d) $In_{1/3}MoS_{0.5}Se_{1.5}$, (e) $In_{1/3}MoSe_2$.

products. Fig. 4 records the X-ray diffractograms of the oxidation products of $In_{1/3}MoS_xSe_{2-x}$ $(0 \leq x \leq 2)$ when heated in air at 800 °C for 2 h and their respective interplanar distance *d* recorded in Table IV. It was interesting to note that there exists a good resemblance in all the diffractograms except for some variations in the intensity corresponding to various reflections for each specimen. These were thoroughly scanned for all the possible phases, e.g. InO, $In_2O_3$, $MoO_3$, $In_2MoO_6$, $In_2(MoO_4)_3$, etc. This suggested that the few lines in the diffractogram of $In_{1/3}MoS_2$ and $In_{1/3}MoS_{1.5}Se_{0.5}$ matched, to some extent, with $MoO_3$ which, however, were nearly absent with further increasing selenium contents in $In_{1/3}MoS_xSe_{2-x}$ $(0 \leq x \leq 2)$ compounds. The presence of a common endothermic peak at $\approx 770\,°C$, as noted earlier (Fig. 3), for all $In_{1/3}MoS_xSe_{2-x}$ $(0 \leq x \leq 2)$ compounds, thus clearly suggested that this, in all probability, is not due to the melting of $MoO_3$ and is more likely to be due to some peritectic transformation taking place at this temperature under the prevailing experimental conditions.

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