Stacking Faults in the Single Crystals

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The crystals of InₓMoSe₂ (0 ≤ x ≤ 1) have been grown by a direct vapour transport technique (DVT) in the laboratory. The structural characterizations of these crystals are made by XRD method. The particle size for a number of reflections has been calculated using the Scherrer’s formula. A considerable variation is shown in the deformation (α) and growth (β) fault probabilities in InₓMoSe₂ (0 ≤ x ≤ 1) single crystal due to off-stoichiometry, which possesses the stacking fault in the single crystal.

1. Introduction
The perfect crystals are neither available in nature nor fabricated in laboratory, it is an ideal concept. There are several types of defects present in a crystal e.g., point defects and stacking fault. The study of stacking fault is made either by electron microscope or by X-Ray diffraction method [1, 2]. During recent years, transition metal dichalcogenides (TMDC) of group IV-b, V-b and VI-b have received considerable attention. These compounds crystallize in quasi-two-dimensional layer structure consisting of chalcogenes, which are held together by relatively weak Van der Waal’s forces between the layers, which facilitate intercalation of foreign atoms, ions or neutral molecules to form new compounds. These crystals become superconducting when intercalated with alkali and alkaline earth metals [1-10]. The study of stacking fault is very important one, because it plays an important role in the description of defects. The conversion behaviour of a solar cell is closely related to the perfection of the electrode material and since stacking fault play a fundamental role in the description of defects structure, therefore their study is of both practical and theoretical interest [2]. The enhanced conduction of the stacking fault along the c-axis is difficult to understand because of the extreme two-dimensional characters of layered compounds of MoSe₂ and its intercalated compounds of i.e., InₓMoSe₂ (0 ≤ x ≤ 1). The only way to understand this conduction is by supposing the presence of stacking faults in these crystals. It is clear from the literature survey that research on the stacking fault of intercalated compounds of MoSe₂ is almost negligible. Hence, it was decided to work on InₓMoSe₂ (0 ≤ x ≤ 1) single crystals [1-10]. Very recently, we have reported intrinsic stacking faults of these materials [8]. Very recently, we have reported the stacking faults results of Re-doped MoSe₂ single crystals [10].

2. Experimental Details
InₓMoSe₂ (0 ≤ x ≤ 1) single crystals are grown by direct vapour transport technique. The X-ray powder diffractogram recorded with the help of Philips X-ray diffractometer using CuKα radiation. For this purpose, many small crystals from each of the groups were finely ground with the help of agate mortar and filtered through 106-micron sieve to obtain grains of nearly equal size. The values of lattice parameter ‘a’ and ‘c’, volume, and X-ray density were obtained from the diffractogram of InₓMoSe₂ (0 ≤ x ≤ 1) are shown in Table 1. The X-ray diffractograms of InₓMoSe₂ (0 ≤ x ≤ 1) single crystals are displayed in Figs. 1-4. The input parameters, which are taken from the X-Ray diffractograms of each crystal, are tabulated in Table 2, which are used in present calculation.

The formulae for deformation and growth probabilities given by Warren [11] are as follows

\[ B_{\alpha} = \frac{360 \tan \theta}{\pi} \left( \frac{d}{c} \right)^2 \left( 3 \alpha + 3 \beta \right), \quad \text{for } l \text{ even} \] (1)
Where, $B_{\beta\theta}$ denotes the full width at half the maximum intensity, $d$ is the \(hkl\) spacing, $c$ is equal to \(2d_{\text{hkl}}\), $\alpha$ and $\beta$ are the deformation fault probability and the growth probability. The presently calculated values of $\alpha$ and $\beta$ are shown in Table 2. All the calculations are performed for (102), (103) and (105) reflections.
3. Results and Discussion

The X-ray diffractograms of In$_x$MoSe$_2$ (0 ≤ x ≤ 1) in Figs. 1-4 clearly show that (002) reflection is of maximum intensity and thereby indicates strong orientation along the c-axis. The diffractograms for In$_{0.25}$MoSe$_2$, In$_{0.50}$MoSe$_2$, and InMoSe$_2$ are similar to those of MoSe$_2$. The lattice parameter 'a' remains constant for all samples, while there is a slight amount of increase in 'c' parameter which indicates that indium has been intercalating in between the layers thereby expanding the 'c' parameter. This increase is very small because the amount of indium incorporated with MoSe$_2$ is also lesser in proportion. As proportion of indium addition is increased in MoSe$_2$, its X-ray density also increases, which can be seen from the Tables 1-2. This may be because of indium atoms added to the lattice of MoSe$_2$.

From the study of Table 3, it is seen that there is a significant variation shown in the deformation fault probability (α) and growth probability (β) due to off-stoichiometry i.e., composition of Indium in the MoSe$_2$ single crystal. The variation of stacking fault i.e., both probabilities, is due to the creation of the defects in the crystal. The values of α and β are nearly of the same order. Any theoretical or experimental proof of such types of calculation is not available in the literature so that it is difficult to compare our results with them and write any strong remarks. The calculation of the stacking fault may be considered as one of the guidelines for further detailed study of defects and various properties of crystals.

It was shown by Cockyne et al. [12] that significant improvement in the resolution of the structure of lattice defects could be obtained from dark field electron micrographs taken in weakly diffracted beams. Ray and Cockyne [13], using the weak beam technique, directly observed the splitting of dislocations into partials of Si. Since then several investigators [14-18] and most recently Mao and Knowles [19] have observed dissociation of lattice dislocations into partials. The presence of stacking faults has been recently shown in WS$_2$ single crystals by Agarwal et al. [20]. All these investigators have used the spacing between partials to estimate the stacking fault energy. Gross and Teichler [21] formulated a real space method, Kenway [22] used atomic lattice stimulation and Xiliang et al. [23] developed a method based on improved embedded-atom for theoretical estimation of stacking fault energies in different materials. All these estimations, when compared with SFE measurements made using weak beam techniques, show a favourable agreement.

The low values of stacking fault probabilities allows for easy gliding on the basal plane of In$_x$MoSe$_2$ (0 ≤ x ≤ 1) layers thus leading to easy creation of stacking faults and its excellent properties as solid lubricating agent [20].

4. Conclusion

X-ray diffractograms have clearly demonstrated the difference in In$_x$MoSe$_2$ (0 ≤ x ≤ 1) single crystals due to off-stoichiometry. The analysis of deformation fault probability (α) and growth probability (β) of In$_x$MoSe$_2$ (0 ≤ x ≤ 1) single crystals has shown that indium intercalation affects the stacking fault probabilities. The experimental proof is not available in the literature, but the present investigation provides an important set of data for In$_x$MoSe$_2$ (0 ≤ x ≤ 1) single crystals, which can be very useful for further comparison either with theory or experiment. Such study on the stacking fault of other single crystals is in progress.

References


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Table 1: Structural data of $\text{In}_x\text{MoSe}_2$ ($0 \leq x \leq 1$).

<table>
<thead>
<tr>
<th>Parameters</th>
<th>$\text{MoSe}_2$</th>
<th>$\text{In}_{0.25}\text{MoSe}_2$</th>
<th>$\text{In}_{0.50}\text{MoSe}_2$</th>
<th>$\text{In}_{0.75}\text{MoSe}_2$</th>
<th>$\text{InMoSe}_2$</th>
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<tr>
<td>$a$ (Å)</td>
<td>3.287</td>
<td>3.287</td>
<td>3.287</td>
<td>3.287</td>
<td>3.287</td>
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<tr>
<td>$c$ (Å)</td>
<td>12.921</td>
<td>12.924</td>
<td>12.925</td>
<td>12.928</td>
<td>12.930</td>
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<td>Volume (Å$^3$)</td>
<td>120.90</td>
<td>120.92</td>
<td>120.94</td>
<td>120.96</td>
<td>120.97</td>
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<td>X-ray density (gm/cm$^3$)</td>
<td>6.972</td>
<td>7.2218</td>
<td>8.5449</td>
<td>9.3313</td>
<td>10.1184</td>
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Table 2: Input parameters, which are used in present calculation.

<table>
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<tr>
<th>$hkl$ values</th>
<th>$\text{In}_{0.25}\text{MoSe}_2$</th>
<th>$\text{In}_{0.50}\text{MoSe}_2$</th>
<th>$\text{In}_{0.75}\text{MoSe}_2$</th>
<th>$\text{InMoSe}_2$</th>
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<tr>
<td>$d$ - values (Å)</td>
<td>$\text{Peak Intensity counts (}\beta_{2\theta}\text{)}$</td>
<td>$\text{Angle }\theta$ (2$\theta$)</td>
<td>$d$ - values (Å)</td>
<td>$\text{Peak Intensity counts (}\beta_{2\theta}\text{)}$</td>
</tr>
<tr>
<td>102</td>
<td>2.6158</td>
<td>0.080</td>
<td>34.255</td>
<td>2.6111</td>
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<td>103</td>
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<td>37.810</td>
<td>2.3756</td>
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<td>105</td>
<td>1.9152</td>
<td>0.100</td>
<td>47.430</td>
<td>1.9112</td>
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Table 3: Presently calculated values of stacking fault probabilities.

<table>
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<tr>
<th>Stacking fault probability</th>
<th>$\text{In}_{0.25}\text{MoSe}_2$</th>
<th>$\text{In}_{0.50}\text{MoSe}_2$</th>
<th>$\text{In}_{0.75}\text{MoSe}_2$</th>
<th>$\text{InMoSe}_2$</th>
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<tr>
<td>$\alpha$</td>
<td>0.0025037</td>
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<td>0.0023574</td>
<td>0.0033694</td>
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<td>$\beta$</td>
<td>0.0025299</td>
<td>0.0027837</td>
<td>0.0022524</td>
<td>0.0032857</td>
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