IMPERFECTIONS IN $MoSe_2$

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Transmission electron microscopy of $MoSe_2$ has revealed the presence of dislocation loops. Using the method adopted by Hull (1969), it has been conjectured that these are of interstitial types. Dark field pictures of an isolated node and a weak beam picture of a dislocation network pattern obtained during the course of our studies have also been presented.

INTRODUCTION

COMPOUNDS of the disulphides and dichalcogenides of W, Mo, Nb and Ta have attracted considerable attention due to their layer structures and good anti-friction properties (Kalikhman & Umanskii, 1973). Some of these compounds such as $MoSe_2$ have been used in sliding electrical contacts and as thermoelectrical sources. It has been reported (Basinskii *et al.*, 1961, 1963) that the properties of materials which crystallize with a layer structure are influenced considerably by dislocations lying in the basal planes. Agarwal and Babu Joseph (1976) have recently carried out transmission electron microscopic studies of $MoSe_2$ crystals. They have reported that dislocations in $MoSe_2$ occur as (*i*) two-fold ribbons having different stacking fault widths, (*ii*) extended nodes and (*iii*) undissociated dislocations. These studies have been pursued further and the observations recorded during this study have been described in this paper.

EXPERIMENTAL

The crystals used in the present investigations were grown by the direct vapour transport method (Agarwal *et al.*, 1978).

Prior to electron microscopic studies, X-ray diffraction patterns were taken with representative crystals and it was confirmed that they were 2H-$MoSe_2$ with the trigonal prism as the basic co-ordination unit. The specimens for electron microscope studies in transmission were prepared by repeated cleavage (Agarwal & Babu Joseph, 1974).

OBSERVATIONS AND DISCUSSIONS

Dislocation Loops—In transmission electron microscopic studies of $MoSe_2$, the observation of the dissociated and undissociated dislocations is quite common (Agarwal & Babu Joseph, 1976). In addition to this, sometimes the present authors came across a striking observation of dislocation loops. These have been shown in Plate I, Fig. 1. It is clearly seen from the figure that the loops are of different types.

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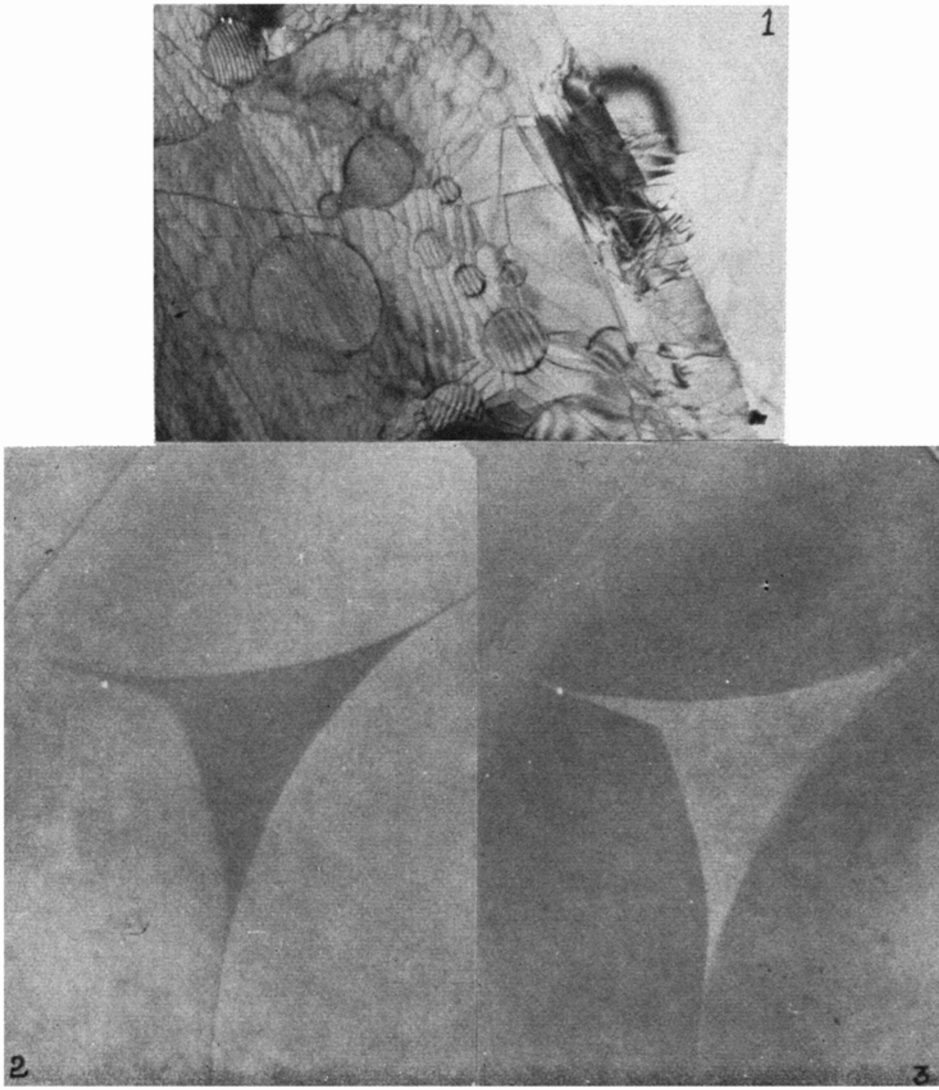

PLATE I

FIG. 1. Dislocation loops in $MoSe_2$($\times$ 25,400); FIG. 2. Bright field photograph of an isolated node ($\times$ 20,400); FIG. 3. Dark field photograph of the node shown in Fig. 2 ($\times$ 20,400).

A careful study of the photograph reveals that the interior of most of these loops exhibits contrast which is seen in the form of fringes. It is seen that the contrast of the loop changes with the operating reflections.

In studying dislocation loops one is primarily interested in determining whether they are formed due to vacancies or interstitials and the determination of their Burgers vector. Although the picture of loops shown above has been taken under two beam conditions, an unambiguous conclusion regarding their Burger's vector could

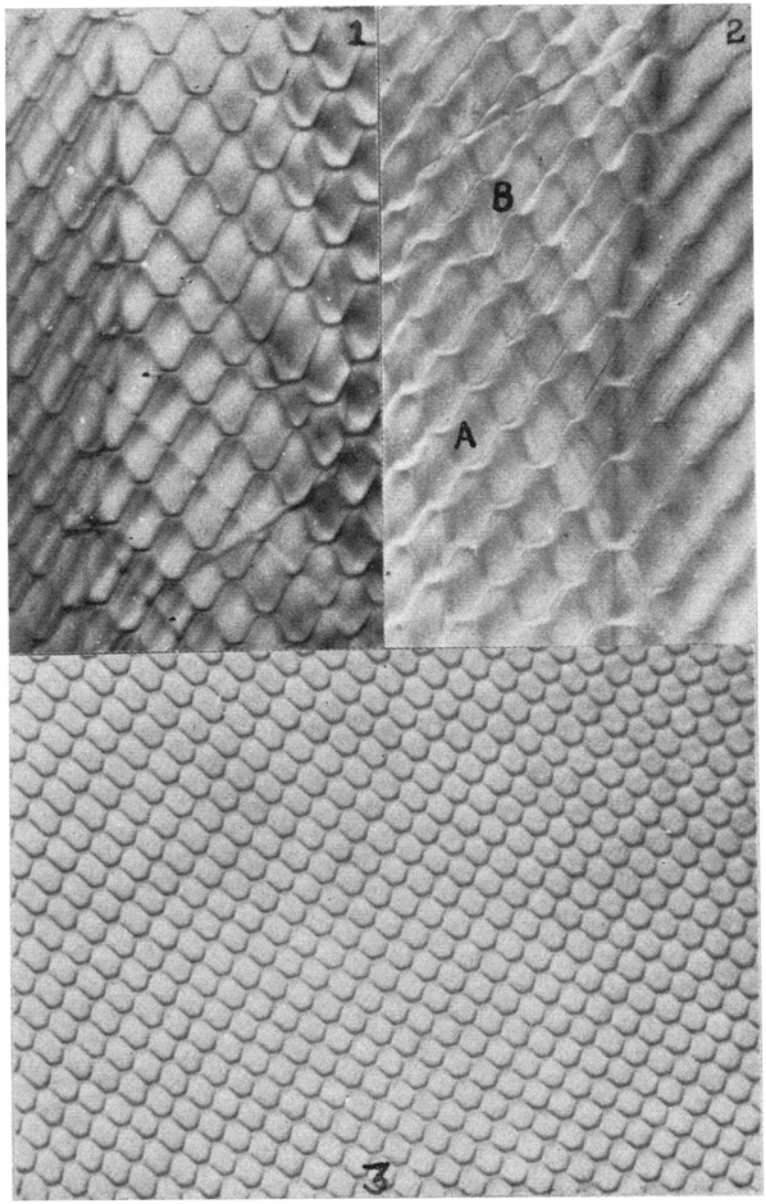

PLATE II

FIG. 1. Net work pattern in bright field ($\times$ 29,500); FIG. 2. Net work pattern in Fig. 4 in weak beam ($\times$ 29, 500); FIG. 3. Net work pattern in $MoSe_2$ ($\times$ 44,000)

not be reached. In order to decide about their nature the method adopted by Hull (1969) has been used.

The value of critical radius R_c of a loop was found to be $R_c = 3.7349 \times 10^{-4}$ cm.

Since practically all the loops observed are smaller than the critical size, it is conjectured that most of them are interstitial loops.

Now $MoSe_2$ is a layer material for which the binding within the layers is strong and that between the layers is Van der Waals bonding; it is weak enough to permit the precipitation of interstitial atoms to give rise to the formation of interstitial loops. The observation (Plate I, Fig. 1) that loops are seen as trapped between two layers supports this conjecture.

Dislocation Nodes — Sometimes it was seen that when some of our pictures taken in bright field under two-beam condition were photographed in dark field, it helped us in interpreting our pattern more easily. Plate I, Fig. 2 shows a bright field picture of an isolated node taken under the two-beam condition. Plate I, Fig. 3 shows the darkfield picture of the same region. A look at these pictures reveals that the geometry of the node pattern is clearly resolved in the dark field picture. The radii of curvature of this node is used to determine the stacking fault energy γ/μ (Whelan, 1959).

The value of γ/μ in the present case comes out to be 0.528×10^{-12} cm.

A weak-beam picture of network patterns — It is now well known that the weak beam technique provides a method of increasing the resolution of the features which can be obtained in a relatively simple way. Since our specimens were too thin to permit the appearance of kikuchi patterns in the electron diffraction patterns, we first of all get the crystal to as near two-beam condition as possible by maximising the brightness of one of the diffraction spots, while simultaneously minimising the brightness of the other spots. The weak spots in the diffraction pattern were then used in turn to form a dark field image.

Plate II, Fig. 2 shows a weak beam image of the same network pattern as is shown in Plate II, Fig. 1. The bright field image of the network pattern in $MoSe_2$ crystal was taken under the two beam condition with $g = 1\bar{2}10$. The diffraction vector used to form the weak beam image was $1\bar{1}00$ with $g = 1\bar{2}\bar{1}0$ almost exactly excited. One can easily see that :

1. The network in Plate II, Fig. 2 is exactly similar to the network pattern shown in Plate II, Fig. 3. This pattern has been interpreted in the past as arising due to two families of dislocations lying in separate planes and their interaction being only an electron optical effect (Pashley & Presland, 1960). But the weak beam picture of Plate II, Fig. 2 shows how false one's interpretation can be. The same sets of dislocations to interact with each other and form the familiar node pattern (Marked 'A' in Plate II, Fig. 1) reported earlier by Agarwal & Babu Joseph (1976).
2. There are three dislocation lines (Marked 'B' in Plate II, Fig. 2) running from top to bottom in the weak beam picture, which are absent in the bright field picture.

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