CHAPTER 7

DISTRIBUTION OF DISLOCATIONS AROUND INDENTATIONS
IN SrSO₄ SINGLE CRYSTALS

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Chapter 7.1 Introduction

One of the important methods of getting information regarding the slip system and the behaviour of dislocations under plastic deformation in a crystal is to study the distribution and structure of dislocation rosettes produced by indentation, a technique perfected by Gilman and Johnston\textsuperscript{1)} Patel and Sutaria\textsuperscript{2)} on MgO and several other authors\textsuperscript{3-7)} using other crystals have made detailed study of dislocation arrangements in the rosettes by repeated polishing and etching experiments.

As has been mentioned earlier all the members of the barite group crystals possess perfect ((001)) and
a nearly perfect (210) cleavages. From macroscopic observations Grigorev\(^2\) has suggested that in barite the operating slip systems are (001) [(100)] and (011) [(011)]. Patel and Koshy\(^9\) by etching, have produced rosette patterns on (001) cleavages of barite crystals, which were indented as well as subjected to dynamical impacts. Phakey and Aly\(^10\) by means of X-ray topographical studies have given a detailed account of the various possible slip directions and slip planes in BaSO\(_4\) crystals. However, it appears that no work has been reported to date on the slip system and distribution of dislocations around indentations of SrSO\(_4\) crystals. The author has therefore carried out investigations into these aspects of SrSO\(_4\) crystals, employing the technique of indentation and subsequent etching and this chapter describes these investigations.

7.2 Experimental Details

The present investigations were carried out on synthetic SrSO\(_4\) crystals grown in this laboratory since the natural crystals have high dislocation density.

Perfect (001) cleavages obtained by cleaving the synthetic crystals were indented by means of Vickers microhardness tester with a pyramidal diamond indentor.
About 10 - 20 gm of load acting for 30 sec was found sufficient to produce good rosette patterns when etched. The indented cleavages were etched in conc. H₃PO₄ containing 0.5 mg / ml. of MgO at 120° C for about an hour. The three-dimensional structure of the dislocation half loops giving rise to the rosette patterns was studied using optical microscopy in conjunction with the technique of successive polishing and etching the indented surfaces. For polishing the etched surfaces conc. H₂SO₄ at 200° C was used.

Since the etchant mentioned above was also capable of revealing dislocation sites on (210) cleavages of SrSO₄ they were indented and etched in this etchant to study the structure of rosette produced on these cleavages. These experiments helped the author in confirming the active slip planes under the action of a concentrated load.

The measurements pertaining to the shifts of the individual rows of pits on advancing into the body of the crystal and the corresponding depths were made with the help of Vickers projection microscope. A micrometer eye-piece on the microscope allowed the shifts to be measured with an estimated accuracy of ± 0.1 μm. The
measurement of depths was accomplished by measuring the thickness of the crystal at each stage. For this purpose the specimen was transferred to the microscope after each stage of etching and the thickness of the crystal was measured using a calibrated focusing adjustment. The graduated focusing knob had an accuracy of \( \pm 1 \) \( \mu \)m.

7.3 Observations and Discussion

Indentation rosettes produced on \((001)\) cleavages of \( \text{SrSO}_4 \) single crystals, a representative of which is shown in fig. 7.1(a), have two arms along \([ [100]]\). Each arm of the rosette consists of two parallel arrays of dislocation etch pits. It was observed that when the crystal faces to be indented were not kept strictly perpendicular to the load direction the two arms of the rosettes produced had different lengths.

Figures 7.1(b) to 7.1(e) show photographs of four more stages of the rosette pattern shown in fig.7.1(a) at increasing depths obtained by repeated polishing and etching. It is clearly seen from the photographs that while advancing into the body of the crystal the rosette arms become shorter. In addition, the individual dislocation arrays (which may contain one or more rows of etch pits depending upon the stress applied) of each arm of the
rosette pattern move away from each other, the outer rows disappearing first leaving behind the inner ones which therefore penetrate deeper into the crystal. This suggests that no two rows of etch pits are interconnected by dislocation half loops. Further, it is seen from fig. 7.1(c) that the dislocation pits belonging to a single row arrange themselves in pairs (see pairs marked $A$ and $B$). On further etching they coalesce into a single pit and then disappear. Figure 7.1(e) represents the final stage of etching. It shows how the tips of two pits (labelled (i) and (ii)) pertaining to a single row are inclined towards each other which clearly indicates that they nucleate at the ends of a half loop. Considering the observations described above the following inferences can be drawn:

1. The dislocation half loops do not enclose the indentation mark. This is similar to the observations made by Patel and Sutaria\(^2\) on MgO, Urusovskaya and Tyagaradzhan\(^6\) on CsI and Urusovskaya et al\(^7\) on PbS.

2. The dislocation half loops in the two arrays of the same arm of the rosette lie in two different planes which is evident from the fact that the two
arrays of pits of the same arm move away from each other.

3. The half loops which are nearer to the indentation mark penetrate deeper into the body of the crystal than those which are further away.

4. The dislocation half loops produced by indentation lie in those planes which intersect the observation plane viz. (001) along $[100]$. 

In order to determine the planes along which the dislocation half loops lie i.e. the slip planes, the shifts of the individual rows of pits and the corresponding depths through which they penetrate into the body of the crystal were measured after each of the latter three stages of etching and thus the angles contained by the observation plane and the planes containing the dislocation half loops were calculated.

From these observations the slip planes were computed. It was found within the limits of experimental error that the active slip planes in the crystal were $((011))$. These are tabulated in table No. 7.1. One can appreciate the fair agreement between the experimentally determined and theoretically calculated $\theta$ values.
Table No. 7.1

<table>
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<tr>
<th>Observation number</th>
<th>Shift in the individual row of pits in μm (S)</th>
<th>Depth below the surface in μm (D)</th>
<th>θ = tan⁻¹(D/S)</th>
<th>Crystallographic angle between the cleavage plane and (011) planes, (θ)</th>
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<tr>
<td>1 (from stage 3rd to stage 4th)</td>
<td>4.10</td>
<td>5</td>
<td>50° 39'</td>
<td></td>
</tr>
<tr>
<td>2 (from stage 4th to stage 5th)</td>
<td>7.50</td>
<td>10</td>
<td>53° 08'</td>
<td>52° 00'</td>
</tr>
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The fact that the (011) are the slip planes was further confirmed by the following experiments.

The (001) cleavage face of a crystal was first indented by means of Vickers microhardness indenter with a load of 10 gm and this crystal was then cleaved along (210) in such a way that the cleavage passed through the indentation mark. One of the (210) cleavages thus produced was then etched. Figure 7.2 shows the traces of rosette pattern obtained on this cleavage.
The complete rosette pattern produced on a (210) cleavage face is shown in fig. 7.3. Crystallographic consideration of (210) planes reveals that the arms of the rosette patterns produced on them lie along \([122]\) and \([120]\). (Individual directions along which the rosette arms lie are marked in the figures). From this it can be inferred that the traces of the rosette pattern shown in fig. 7.2. are along \([122]\) and \([122]\). Thus an indentation made on (001) cleavage plane produces dislocation rosette on both the (001) and (210) cleavages, the arms of which lie along \([100]\) and \([122]\) respectively. In other words, the dislocation etch pits along \([122]\) on (210) cleavage face must be due to the same dislocation half loops that were revealed by the etch pits along \([100]\) on (001) cleavage plane. It can be seen from the structure of SrSO₄ crystal (which is orthorhombic) that the planes which intersect the ((210)) along \([122]\) and ((001)) along \([100]\) are ((011)). These must therefore be the slip planes of SrSO₄ crystals which are the same planes as already determined.

Further, a (210) cleavage face of SrSO₄ crystal was indented with a load of 20 gm and this crystal was then cleaved along ((001)) such that the
cleavage passed through the indentation mark. The (001) as well as the (210) cleavage planes thus obtained were then etched. Figures 7.4 and 7.5 represent the traces of rosette patterns obtained on (001) and (210) cleavages respectively. It can be seen from the figures that the traces of the rosette pattern obtained on (001) face are oriented in $[\bar{1}00]$ while those obtained on (210) are in $[1\bar{2}2]$. One can now very well argue that both these traces have been produced due to the slip of the same set of planes which intersect the (001) and (210) along $[\bar{1}00]$ and $[1\bar{2}2]$ respectively. They turn out to be $(011l)$ again. Figure 7.6 schematically represents the directions along which the rosette arms produced on both the cleavage lie while fig. 7.7 represents the geometrical configuration of the dislocation loops produced on a (011) plane by indenting the (001) cleavage plane.

So far as the direction of the slip is concerned the present investigation could not lead the author to draw any conclusions. However, by analogy with BaSO$_4$ whose slip systems have been suggested by Grigorev $^8$ to be $(001l)$ [[100]] and $(011l)$ [[011]] it may be conjectured that the slip directions in SrSO$_4$ associated with $(011l)$ may also be [[011]].
However, one cannot conclude that ((011)) are the only active slip planes in SrSO₄ crystals. There may be other planes also which may act as slip planes. For example, the ((011)) planes could not account for the rosette arms along [[110]] produced on ((210)) cleavages. Probably the other slip system suggested by Grigorev for BaSO₄ may also exist in SrSO₄ and it may be able to explain the rosette arms along [[110]] on ((210)).

An attempt has also been made to determine the minimum load at which the dislocation rosettes appear on the cleavage faces and it was found that the rosettes could be produced on both the cleavage faces even at a load of one gram. By determining the microhardness on the (001) cleavage face the yield stress beneath the indentor was estimated and found to be 72.2 ± 1.6 Kg/mm². Approximately the same value was obtained for the (210) cleavage face also.

7.4 Conclusions
1. Strontium sulphate single crystals undergo plastic deformation at room temperature.
2. The slip systems in these crystals seem to be the same as those of BaSO₄ viz. ((011)) [[011]] and ((001)) [[100]].
3. The yield stress beneath the indentor is found to
be 72.2 ± 1.6 Kg/mm² for (001) cleavage faces.

References


