CHAPTER 9

CONFIGURATION OF DISLOCATIONS IN TOPAZ CRYSTALS
## Contents

<table>
<thead>
<tr>
<th>Section</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.1</td>
<td>Introduction</td>
<td>084</td>
</tr>
<tr>
<td>9.2</td>
<td>Etching of (001) matched cleavages</td>
<td>086</td>
</tr>
<tr>
<td>9.3</td>
<td>Etching of (001) individual cleavages</td>
<td>089</td>
</tr>
<tr>
<td>9.4</td>
<td>Conclusion</td>
<td>092</td>
</tr>
</tbody>
</table>
9.1 Introduction

A dislocation is classified as a one-dimensional defect and is that type of imperfection which is supposed to run through the whole body of a crystal or form complex configurations within the body of the crystal. One of the means of direct observation of dislocations is to study etched surfaces of a crystal. Comparison of etch figures on matched cleavages gives a good deal of information about the existence and nature of dislocations. Fidelity of an etchant to reveal dislocations in crystals is proved if there is a mirror correspondence of etch pits on two matched halves and pits grow bigger and deeper on progressive etching. One to one correspondence of etch pits results when dislocations are either normal to or are all inclined at the same angle to the face. However, when dislocations branch or bend at or near the cleavage, mismatching or deviation from one to one correspondence of etch pits on two matched cleavages is to be expected. Small deviations from a perfect match for the etch patterns on the opposite cleavage halves were observed by Amelinckx (1956) in NaCl crystals. Occasionally he found two closely spaced etch pits on one face corresponding
to only one pit on the opposite cleavage face, and suggested that such a discrepancy could be explained as the branching of dislocations in the cleavage plane. Similar observations were made by Sagar and Faust (1967) in the case of $\text{Bi}_2\text{Te}_3$ and it was suggested that in addition to the possibility of branching of dislocations at the cleavage, some of the observed discrepancies could easily be due to bending of dislocations at the cleavage. Bontinek (1957) observed double rows of etch pits on $\text{CaF}_2$ crystals and attributed them to helical dislocations. Bhagavan Baju et al. (1969) also observed double rows of etch pits where the beak of one pit is connected to the that of the other pit, these rows being slightly displaced relative to each other, which is characteristic of a helical dislocation. This way they could record two helices oriented along <110> direction in NaCl crystals. Employing etching technique, Hari Babu and Bansigir (1969) have been able to record branching of dislocations and dislocation half loops occurring inside the crystal and bending of dislocations near the cleavage plane in the case of NaCl crystals. Branching and bending of dislocations in apophyllite crystals have been analysed by Joshi and Ittyachen (1968).
Since the etching technique can be successfully employed to trace the configuration of dislocations within the body of a crystal, an investigation of this kind was undertaken in the present work, and the results obtained are described and discussed in what follows.

9.2 Etching of (001) matched cleavages

Matched pairs cleaved along (001) faces were etched in KOH as described in chapter 7. Examination of etched surfaces revealed the presence of point-bottomed etch pits on them. Since the point bottom of an etch pit is considered to be the terminal end of a dislocation line, by following, at successive stages of etching, the tip of a point-bottomed etch pit it becomes possible to map out the configuration of dislocations threading through the crystal lattice. This procedure was employed to find out the path followed by dislocation lines. Etch pits obtained on cleaved matched pairs showed correspondence in their number, shape and size, and on successive etching etch pits were found to grow bigger in size and become deeper as is to be expected. A typical case of etch patterns obtained on a cleaved matched pair after 15 seconds of etch is shown in figures 71(a)
and 71(b). Comparison of etch patterns in these photomicrographs show one to one correspondence in regard to both the number and position of isolated etch pits, which means that the pits are formed at dislocation sites. On close examination of these etched faces the etch pits were found to be eccentric. The eccentricity observed in the structure of etch pits indicates that the linear defects are inclined at some angle to the cleavage face. This eccentric nature of point pyramidal pits should become more pronounced with the increase in the depth of pits. To investigate it, this matched pair was again etched for 30 seconds. Figures 72(a) and 72(b) illustrate etch patterns thus obtained. Here, all the pits have grown bigger in size and depth and are point-bottomed and eccentric. On comparing the etched regions in figures 72(a) and 72(b) with those in figures 71(a) and 71(b) the following points emerge.

(1) The cleavage lines are displaced relative to the pattern of pits. It is conjectured that displacement of cleavage lines by etching might be taking place as explained by Tolansky (1957).

(2) In regions marked A in figures 71(a) and 71(b), and figures 72(a) and 72(b) it is found that the
distance between the geometrical centres of two pits is larger in figure 72(a) than that in figure 71(a), and it is smaller in figure 72(b) than that in figure 71(b). This means that two dislocation lines associated with these pits are divergent as one goes into the interior of the crystal below the cleavage face of figure 71(a) and are convergent as one goes towards the interior of the crystal below the cleavage face of figure 71(b). In order to find out how and where these linear defects converge, the matched pair was again etched for 30 seconds. The etch patterns thus obtained are illustrated in figures 73(a) and 73(b). On comparison of etch patterns in figures 73(a) and 73(b) with those in figures 72(a) and 72(b), one finds in regions marked A of these figures that the distance between the geometrical centres of two pits in figures 73(a) is larger than that in figure 72(a), whereas on the other match face the two point-bottomed pits tend to merge together (see figure 73(b)). In the former case, the dislocation lines associated with two pits diverge inside the lattice below the region of the cleavage face of figure 73(a), whereas in the latter case the dislocation lines converge in the interior of the crystal below the region of the cleavage face of
The two pits in region marked A of figure 73(b) are flat-bottomed. A small point-bottomed pit is also seen inside a flat-bottomed pit marked X, formation of which may be attributed to the stepped nature of dislocation inside the crystal. At this stage it could be conjectured that this newly formed point-bottomed pit and also the flat-bottomed pits would grow wider and deeper and result into a single pit if further etching was carried out. In fact, etching of this match half for 30 seconds more did result into a single flat-bottomed pit. Thus the dislocation lines associated with these pits converge in the interior of the crystal below the cleavage face of figure 73(b), whereas these linear defects diverge inside the crystal below the cleavage face of figure 73(a), thereby giving rise to the formation of a dislocation half loop, schematic representation of which is given in figure 74. It may be mentioned here that in all the schematic diagrams presented here, the solid line in the centre represents the boundary separating cleavage planes and the dotted lines the three stages of etching.

9.3 **Etching (001) individual cleavages**

The above described observations
establish that dislocations are present in topaz crystals in the form of half loops. Also a series of etching experiments carried out under the same conditions (as described earlier) on basal cleavages of natural topaz crystals proved that in most of the cases the linear defects are present in the form of half loops in such crystals. A typical observation made on a basal cleavage is illustrated in figures 75(a), 75(b) 75(c) which represent the etch patterns obtained after etching for 15 seconds, 45 seconds and 75 seconds respectively. By comparing etch patterns in regions marked A of these figures, the following points are found noteworthy.

(1) A point-bottomed pit marked 1 in figure 75(a) results into a flat-bottomed pit (see figure 75(b) ) and then again becomes point-bottomed as is seen in figure 75(c). In this case it is reasonable to say that the dislocation line runs normal to the cleavage face, then as we go towards the interior it changes its direction to become almost parallel to the cleavage face and then finally bends and runs inclined to the cleavage face.

(2) Pits marked 2 and 3 in figure 75(a) are point-bottomed. On further etching, they tend to merge into
each other and become flat-bottomed (see figure 75(b)).

On still further etching, they finally result into a shallow flat-bottomed pit as is illustrated in figure 75(c). It is suggested that formation of pits in this way gives rise to a V-shaped dislocation configuration.

(3) Three pits marked 4 in figure 75(a) have correspondence with three flat-bottomed pits in the region marked 4 in figure 75(b). As these flat-bottomed pits grow bigger in size, due to their intergrowth two of the flat-bottomed pits merge into a single point-bottomed pit and this point-bottomed pit also tends to merge with the third flat-bottomed pit (see region marked 4 in figure 75(c)). Here, the formation of pits establishes that dislocations associated with these pits are present in the form of half-loops.

A schematic representation of dislocation configuration of the above mentioned three cases is given in figure 76 as marked D, E and F respectively.

One more case of etch patterns obtained on a cleavage face by etching it for 15 seconds is shown in figure 77(a). Here also all the pits are eccentric and except pits marked 3 and 4, which are quite close to each other, all pits have grown at isolated places. This cleavage face was etched for
30 seconds more and the resulting etch patterns are illustrated in figure 77(b). Here the pits have grown bigger in size and depth. It may be noted that the pit marked 1 tends to join the pit marked 2 (see figure 77(b)), which means that the dislocation line associated with the pit marked 1 is much more inclined to the cleavage face than that associated with the pit marked 2. Also, as seen from figure 77(b), the pit marked 3 has merged with the pit marked 4. At this stage it was felt that these pits may result into a single pit if further etching was carried out. To investigate it, this cleavage was again etched for 30 seconds and the etch patterns thus obtained are shown in figure 77(c). Here, as was expected, the pit marked 1 has merged with the pit marked 2 and same is the case with the pits marked 3 and 4. Development of these etch pits is thus attributed to dislocation half loops in the crystals.

9.4 Conclusion

Etching experiments carried out in the present investigation and observations reported here prove that non-parallel linear defects and dislocations in the form of half-loops are common in natural topaz crystals.