CHAPTER 8

INCLUSIONS IN TOPAZ CRYSTALS
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Solid, liquid or gaseous material is almost always found included in both natural and synthetic crystals. Study of such inclusions often becomes a tool to derive valuable information of the material (substance) from which crystals grew. Such inclusions vary in size and are normally found in a haphazard manner in the crystal. However, crystallographically strictly oriented inclusions are also occasionally present. In some rare cases their walls are parallel to a growing face. A variety of parameters like variation in the supersaturation of the mother liquor, composition of the solution, minor fluctuations in temperature, size of the crystal, etc., govern the formation of inclusions in crystals grown from solutions. Consequently different types of inclusions observed in different crystals may be listed as follows:

1. Relatively large layers of solution whose lateral size substantially exceeds the thickness.
2. Thin, continuous channels or chains of separate inclusions.
3. Distinct small isolated inclusions.

Buckley (1951), Amelinkx (1956), Chernov and Budurov (1964), Joshi and Vagh (1965), Brooks et al.
(1968), Belyustin and Fridman (1968), and Gross (1970) have reported inclusions in different crystals. According to Gross (loc. cit.) liquid inclusions in RDX crystals involve the trapping of solvent either interstitially followed by its migration to dislocations or even directly along dislocation lines.

Natural topaz crystals often contain many microscopic liquid inclusions. Very recently Virkkunen and Szejko (1971) have reported that fluorite (CaF₂), ilmenite (Fe TiO₃), hematite (Fe₂O₃), goethite (FeO·OH) and quartz (SiO₂) are included in topaz crystals as inclusions. These inclusions were detected by them by electron microprobe analysis. In the present chapter a variety of inclusions observed underneath (001) cleaved surfaces of topaz are described. An attempt has been made to correlate some of these inclusions with the etch pits formed at dislocation sites.

8.2 Liquid inclusions

Inclusions in topaz crystals were observed through unsilvered basal cleavages by slightly racking down the objective of the microscope focussed on the cleaved surface under examination. In most of the crystals thin as well as thick channels of liquid inclusions were commonly observed. One such case is shown
in a photomicrograph in figure 64. It is conjectured that fluctuations in the conditions of growth might have led to the aggregation of thin channels of imprisoned mother liquor. Some of the cleaved matched surfaces revealed slightly below them presence of small liquid inclusions aligned along curved lines which have exact matching on the two surfaces as illustrated in figures 65(a) and 65(b). These matched cleavages were etched in KOH for 10 seconds as described in chapter 7. The etch patterns obtained on these cleaved matched surfaces are shown in figures 66(a) and 66(b). These pits have perfect matching on the matched faces. On racking down the objective of the microscope below the surfaces the same inclusions as shown in figures 65(a) and 65(b) were observed. On further etching, these pits were found to grow bigger in size, as is to be expected. By slightly racking the microscope objective up and down it was ascertained that the pits are on the surface and the inclusions are in the body of the crystal right below the pits. All the pits are not of the same size on the surface, though the corresponding pits on the two matched halves are identical in regard to their size, shape and position. The exact correspondence of pits on the two matched halves, figures
66(a) and 66(b), indicates the presence of dislocations along these boundaries. From the correspondence between rows of pits and rows of inclusions, as seen from figures 66(a) and 65(a) or figures 66(b) and 65(b), it is suggested that some, if not all, inclusions occur at dislocation sites.

8.3 **Square, bead-shaped and tree-like inclusions**

A number of thin flakes, each about 500 micron thick, were obtained, whose both the surfaces were basal cleavages. Observations on one such flake showed the presence of inclusions in it. These inclusions could be seen from either side of the flake. Figure 67(a) illustrates inclusions when seen through one unsilvered surface of the flake, while figure 67(b) shows the same inclusions when seen through its other unsilvered surface. It should be noted that we have three distinct types of inclusions in this case. In region marked A in figure 67(a) one sees tiny square inclusions, all of almost equal size. They are strictly oriented, are almost equally spaced and are aligned along a curved line. In region marked B in the same figure there is a row of bead-shaped inclusions. In region marked C in the right bottom corner of figure 67(a) inclusions having tree-like structure are observed. All these are seen a little
below the surface facing the objective of the microscope. This flake was then etched in KOH for 10 seconds. The resulting etch patterns as observed on the opposite surfaces of the flake are illustrated in figures 68(a) and 68(b). Etch pits in region marked A in figure 68(a) strictly correspond to the above cited square inclusions illustrated in figure 67(a). It may be noted that there is exact correspondence between pits on the opposite faces of the flake as seen in regions marked A in figures 68(a) and 68(b). Such a correspondence is indicative of formation of these pits at dislocation lines threading through the flake and cut by the opposite surfaces of the flake. Further, the correspondence between the pits in region marked A in figure 68(a) with square tiny inclusions of figure 67(a) is indicative of trapping of these inclusions at dislocation sites. In order to study the penetration of linear defects in this flake, polishing and re-etching experiments were carried out on it which are described in the following section.

8.4 Polishing and re-etching experiments

Efforts were made to polish the crystal surfaces of topaz in different chemicals, and Na₂O₂ at 510°C was found best suited for the purpose.
Thus to remove etch figures on the above-cited flake, the flake was polished in NaCl for 15 seconds. After polishing, the flake was re-examined. All the three types of inclusions in the regions marked A, B and C of figures 67(a) and of fig. 67(b) were observed through its two surfaces as before. The flake was then etched again, in the manner stated earlier, and etch patterns formed on its two opposite surfaces were examined. The pits obtained were found to be identical to those seen in regions marked A in figures 68(a) and 68(b) and also had perfect correspondence with square inclusions of figures 67(a) and 67(b). Such correspondence of pits on matched faces indicates nucleation of etch pits at dislocation sites, and the correspondence of pits with inclusions suggests trapping of these inclusions at dislocation sites.

The flake was again polished, as mentioned earlier, and then etched for 40 seconds more. The etch patterns thus obtained on the opposite faces of the flake are illustrated in figures 69(a) and 69(b) respectively. There is perfect correspondence of pits in regions marked A in figure 69(a) with those in region marked A in figure 68(a) and with the square inclusions of figure 67(a). Of course, these pits are bigger in size and are
also deeper. Correspondence is also observed in figures 89(a) and 89(b), particularly in regions marked A. It is interesting to note square tiny dots at the centre of pits in regions marked A in figures 89(a) and 89(b), which are square inclusions below the etched surface. On slightly racking down the microscope objective these inclusions were clearly visible as shown in figures 70(a) and 70(b). In these figures, inclusions in regions marked B are very much more marked than in other figures reported here. These observations endorse the above given suggestion that at least the square inclusions are trapped at dislocation sites.

8.5 Discussion

When a crystal is cleaved so as to cut dislocation lines, the terminal ends corresponding to each dislocation line will lie on the matched faces or on two opposite faces of a cleaved out thin flake, and when such a pair of matched faces or a thin flake is etched under suitable conditions of etching, the surface intercepts of dislocations become the centres of nucleation of etch pits. One to one correspondence of etch pits on matched cleavages and on opposite faces of thin flake indicates that all the etch pits reported here are nucleated at the sites of dislocations.
This is further supported by the polishing and re-etching experiments. Also, since for each pit there is a corresponding inclusion although each inclusion is not necessarily associated with a pit, at least some, if not all, inclusions are certainly trapped at or associated with dislocations.

All etch pits reported here are not all of the same size. In a particular case the inequality in the size of pits may be attributed to the different strain energies around dislocations or alternatively to the inequality in the size of inclusions if the latter are trapped at dislocation sites.

9.6 Conclusions

1. Different forms of inclusions are present in topaz crystals, some of which are crystallographically oriented.

2. Some inclusions are trapped at dislocation sites.

3. Etching technique is useful for proving the possibility of trapping of inclusions at dislocation sites.