CHAPTER 14

SURFACE STRUCTURES AND ETCH PATTERNS ON (111) AND (100) FACES OF NaF CRYSTALS GROWN FROM AQUEOUS SOLUTION

14.1 Introduction

In chapter 11, studies made on dislocations in NaF crystals grown from melt, obtained from Harshaw Chemical Company, U. S. A., were described. Further investigations have been carried out on the crystals grown from aqueous solution in this laboratory. The present chapter has been devoted to the study of surface structures and the etch patterns on (111) habit faces and (100) basal planes of these crystals.

14.2 Experimental

A saturated solution of NaF in distilled water was prepared at room temperature in a glass beaker and heated to 100°C for 30 mins. and then cooled to room temperature. It was then left undisturbed for about 12 to 30 days. It was found that crystals were formed at the bottom of the beaker. The small crystals of size about 1 mm. (shown in figure 144) were obtained in about 12 days while it took about 28 days for the crystals of about 6 mm. to grow. It is observed that on most of the crystals only four
(111) faces developed.

14.3 **Surface Structures on (111) Habit Faces and (100) Basal Planes**

Frequently small misoriented microcrystals were observed on the (111) faces as guest crystals. One such case is shown in figure 145, where the four (111) faces of this guest crystal are easily seen. A typical example is shown in the electronmicrograph of figure 146, where the guest crystal not fully developed is seen. In some cases, growth was found to initiate from the corner of a misoriented guest crystal as shown in figure 147, in which the growth layers originating from the point 'A', the tip of a microcrystal, is clearly seen.

On examination of the basal planes of the tiny crystals four-sided, five-sided and six-sided polygonal structures as shown in figures 148 to 152 were observed. It may be mentioned that the longer sides of these basal planes were nearly parallel to <110> directions. These structures were pyramidal elevations. Figure 153 illustrates a polygonal structure in which the growth fronts are slightly modified.
14.4 Etch Patterns on (111) Habit Faces, (100) Cleavages and (110) Fractured Faces of NaF

Glacial acetic acid + distilled water (5:1) was used to etch (111), (100) and (110) faces of these crystals. Figure 154 is an electronmicrograph showing the etch pattern on a (111) habit face etched for 6 mins. It may be noted that:

1. The pits are point-bottomed as well as flat-bottomed and some of them are terraced, and they are of different sizes.
2. Pits within pits are also seen.

In order to show that the pits are nucleated at the sites of dislocations, (111) face was successively etched for 3 mins. and 12 mins. respectively. The resulting etch patterns shown in figures 155(a) and 155(b), reveal that the isolated pits grow in size on prolonged etching thus indicating that they are dislocation etch pits.

The intersecting rows of pits on (100) cleavage shown in figure 156 may be representing the low angle grain boundaries. The boat shaped pits produced on (110) fractured face (figure 157) reveal that it is indeed a (110) face.
14.5 **Low angle Grain Boundaries and Twin Boundaries**

**On (111) Habit Faces of NaF**

An electronmicrograph shown in figure 158 reveals a trisection of rows of etch pits which may be revealing low angle grain boundaries on the face.

Figure 159 is an electronmicrograph showing a typical twin boundary revealed by the pits on the two sides which are oppositely oriented.

14.6 **Discussion**

The polygonal pyramids with four, five and six-sides observed on the basal planes of few crystals are more interesting and instructive. The (111) faces seem to be developed by layer deposition by what is called the two dimensional growth mechanism. The layers on this face should be expected to be of triangular in outline as evidenced by the observations presented in this chapter. It is conjectured that the growth may first begin on the basal planes and later on, on (111) faces. Since the crystals grow on the bottom of the glass beaker as a substrate, the pyramidal faces upward develop while similar growth is not permitted on the other side.
To begin with the crystal might be growing by two-dimensional nucleation by spreading and piling up of square shaped growth layers. One should therefore expect square shaped pyramids on the basal planes.

It is suggested that the growth may take place mainly by spreading and piling up of growth sheets, probably on basal planes to begin with and later on, on the faces like (111).