CHAPTER XII
MICRO-DISC PATTERNS ON (110) AND (111) FACES

12.1 Introduction

In this chapter, an attempt has been made to use, the fast etching techniques (as described earlier) to produce the micro-disc patterns on (110) and (111) faces of diamond.

Several researchers have reported the micro-disc pattern on natural rhombic dodecahedral faces of diamond along with the usual net-work pattern and boat shaped figures. Kucharenko (1950) has studied such features in detail and has attributed their origin to the corrosion processes in nature. J. F. H. Custers gave a detailed account of such features to a conference on diamond physics held at Oxford 1957. Pandeya and Tolansky (1961), from their interferometric study of such features, attributed their origin to the dissolution process in nature. Patel and Goswami (1962) made a careful study of these features on (120) faces of topaz and produced exactly similar features on its (001) cleavage surfaces by etching. Thus they provided indirect experimental evidence that the micro-disc pattern observed on the

dodecahedral faces of diamond by various investigators might have been formed by dissolution.

The absence of micro-disc patterns on rhombic dodecahedral faces of diamond when etched in the laboratory may be due to the long periods of etching necessary to produce etch patterns. Even if the micro-discs are produced by the protective action of micro bubbles (as reported by Pandeya and Tolansky and Patel and Goswami), they will last only as long as the bubbles afford protection. When the bubble bursts the micro-disc produced will be attacked by the etchant and will be soon destroyed. Since the bubble exists for a short time the micro-discs produced will be washed away in longer periods of etching. Patel and Goswami reported that they were successful in producing micro-disc patterns on (001) cleavage surfaces of topaz only because their etching was as short as 45 seconds. Even during such short periods they observed that the regions on the micro-disc and its edges were attacked by the etchant. They also reported that when a crystal with micro-discs was etched, the discs were completely dissolved. Therefore it seems likely that micro-discs will be formed by etching rapidly for short periods. Such experiments have been devised in the present work and micro-discs have been produced

on the natural rhombic dodecahedral faces and on octahedral cleavage faces of diamond.

12.2 Micro-disc patterns on (110) faces of diamond

The etching of (110) faces was carried out in two sets of experiments. In the first set of experiments the etching of the polished dodecahedral faces was carried out in an atmosphere of oxygen at a pressure of about 40 lbs/ $in.^2$ for two to three minutes at about 800° C. Thus figures 101 (X 400) and 103 (X 400) are the photomicrographs showing the discs produced within two minutes. As the surface, after etching became very rough and uneven, multiple beam interferometry could not be applied and so in order to confirm whether the discs are elevations or depressions, multiple light profile, was used. Figure 102 (X 1200) shows the light profile running over some discs, indicating clearly that they are elevations and not depressions. The height of one at the middle is 1.3 microns. That the micro-discs observed in figure 103 (X 400) are elevations was further confirmed by re-etching the face in potassium nitrate at 560° C in a muffle furnace. Figure 104 (X 400) represents the etch pattern thus produced. It is clearly seen that the

discs have reduced in size and the usual boat shaped pits have appeared, thus confirming that the discs are elevations and the face is a (110) face.

In the second set of experiments, a piece of carbon rod was burnt in an atmosphere of oxygen at a pressure of about 25 atmospheres. The diamond was subjected to the high temperature which produced an appreciable etching within a few seconds. The details of the experimental set up for etching have already been described in chapter II.

Figure 105 (X 650) represents some region of the dodecahedral face after etching, in which the micro-discs produced are clearly seen. It is established, as above, that the discs are elevated regions. Some of the discs are completely circular and some are of irregular shapes. It is interesting to note that in the lower part of the picture, many discs not perfectly circular are, anchored at a point and they interfere with each other. The region within the disc in some cases is not affected by etching while in others it is affected. A prominent circular disc in the upper portion of figure 105 does not seem to have been affected by the etchant at all. The micro-discs of figure 105 are similar to

those reported by Pandeya and Tolansky (1961).

12.3 Micro-disc pattern on (111) cleavage faces

Figures 106 (X 650) and 107 (X 650) represent the photomicrographs of the etch patterns produced on the (111) cleavage surface of the diamond. It is very interesting to observe the micro-discs produced even on (111) surface. Attention is drawn to the following features :

(1) The discs are of various sizes.

(2) Smaller discs are seen within the larger ones.

(3) It has been verified by light profile microscopy, that the discs are elevated regions on the surface, figure 108 (X 1200).

(4) When one disc encloses another, the inner disc is at a higher level than the outer one.

(5) The region within the discs is generally etched. In such cases etching is more pronounced outside the discs than within, figure 106 (X 650).

Figure 109 (X 2100) represents the photomicrograph taken at a higher magnification of the region of one of the

discs in figure 107 (X 650). The triangular etch pits within the disc are clearly seen. The etch pattern in the region outside the disc is out of focus because of the small depth of focus of the high power objectives. However, when the region outside was focussed, the triangular etch pits were clearly seen. The only difference between the two etch patterns was that the etch pits within the discs appeared smaller than those outside it, thereby indicating that the region within the disc might have been exposed to the etchant for a shorter period than the region outside. In figure 110 (X 900) a multiple light profile is seen running across a micro-disc on a (111) cleavage surface. The shift of the profile clearly indicates that the disc is an elevated region of height 2.1 μ on the surface. The region within the disc is unevenly etched, the effect being more at the edges than at its centre. Careful examination of the profile within the disc reveals that it does not run straight but is slightly curved, indicating that the region within the disc is not flat but has a slight convex curvature.

12.4 Conclusions

The experimental results described in this chapter support the conjecture of Pandeya and Tolansky (1961) on the origin of the micro-disc patterns. The absence of micro-discs on natural dodecahedral faces of many crystals can be explained by assuming that the dissolution in the case of such crystals might have proceeded for a long time, during which even if the micro-discs are produced they might have been washed away. The general absence of micro-disc patterns on the natural octahedral faces suggests that they might be more susceptible to etching than the dodecahedral faces. Thus, such micro-discs, even if produced on octahedral faces, will be easily removed in the process of etching.

The convex nature of the region observed within the micro-discs suggests that the size of the micro-bubble which gives rise to the protective action would have slowly decreased. In this process the protective action of the bubble will last longer at the centre than at the edges, thus producing the convex slope. The formation of the discs within the discs, and the inner discs at a higher level than the outer, can be explained by assuming that the bubble must

have either shrunk or expanded during the process of etching.

That the micro-discs are produced by the protective action of the bubble is supported by the observation that the effect of etching is smaller within the disc than at the edge. The experimental results indicate that to produce micro-discs on any face of a crystal, it is necessary to subject the crystal to some specific conditions of etching, and that it has nothing to do with either the etchant used or the crystal face selected.