8.1 Introduction:

Circular elevated disks have been observed on dodecahedral faces of diamond by several investigators. The first description of these patterns, as found in literature, are the two pencil drawings of dodecahedral faces made by Sutton (1928). The circular disk patterns described by Sutton and the patterns observed in the present investigation are more or less similar. Kucharenko (1950), from his studies on Ural diamonds, has also described and discussed such features. He has attributed the origin of the patterns to the corrosion process in nature. These features occur exclusively on dodecahedroid faces of diamond which usually exhibit the signs of corrosion. J.F.H. Custers gave a detailed account of such features at a diamond conference held at Oxford, 1957. In 1958 at a diamond conference in Cambridge, Tolansky and Pandeya gave detailed description of these features and more information is given by Pandeya (1959). Pandeya and Tolansky (1961) have described such features on a number of dodecahedral faces of diamonds. They have thoroughly investigated the microtopography of these features both by microscopy and by multiple beam interferometry. The interferometric study has shown
that the disks are slight elevations on the surface. They, therefore, conjectured that they may have been formed due to the protective action of micro-bubbles, during the dissolution process in nature. Patel and Goswami (1962) have reported similar features on (120) natural faces of topaz and produced identical features on (001) cleavage faces of topaz by etching them in the laboratory. The micro-disks are randomly distributed and are of different sizes. Joshi and Vagh (1964) have reported similar micro-disk patterns on prism faces of natural and cultured quartz crystals. Patel and Goswami (loc cit) have reported that they could not establish correspondence in the number and position of these disks, produced on matched faces of topaz cleavages. They therefore could not conjecture the cause of formation of the micro-bubbles which give rise to the micro-disks.

In the present investigation, similar circular disk patterns have been produced on the (111) cleavage faces of calcium fluoride and the cause of formation of the micro-bubbles is discussed. It may be mentioned that in earlier experiments on etching of the calcium fluoride cleavage faces, the micro-disks were never produced. It was thought that this may be due either to: (1) the comparatively long period required for producing suitable etch patterns as reported by Patel and Goswami (loc cit)
or/^{(ii)} unsuitable etchant to produce the micro-bubbles. Number of etchants were therefore tried and ultimately the micro-disk patterns were successfully produced, when cleavages were etched in a mixture of perchloric acid and aluminium chloride. With this etchant appreciable etching is produced within 20 seconds. The investigations have been carried out in two stages:-

1. By studying the circular disk patterns produced on (111) cleavage faces.

2. By studying the disk patterns on the matched cleavage faces.

8.2 Micro-Disk Patterns On (111) Cleavage Faces :

Fig. 68 (X 125) represents the micro-disk pattern on a (111) cleavage face of calcium fluoride, produced by etching it in a mixture of perchloric acid and aluminium chloride in the proportion of 3:1 for 15 seconds at 90°C. Fig. 69 (X 140) illustrates another region in which typical micro-disks are clearly seen. A careful study of these photomicrographs reveals the following:-

1. The etch patterns consist of the micro-disks, areas resistant to etch and large number of crowded triangular pits arranged in linear rows in \(\langle 110\rangle\) direction.
2. The disks are of various sizes and many of them interfere with each other.

3. The micro-disks, more or less circular, are randomly distributed and are of different sizes.

4. When the areas resistant to etch or the linear rows of crowded pits cross the micro-disks, they maintain their character within the disks.

5. The amount of etching produced within the disks is less than that outside the disks.

6. Disks are observed within the disks. In these cases, the inner disks are always raised compared to the outer disks and the amount of etching within the inner disks is always less than that in the outer disks.

7. Disks within the disks, anchored at a point, are also observed.

To decide whether these features are elevations or depressions, they were examined by a light profile microscope. Thus Fig. 70 (X 175) represents the multiple light profile running across a micro-disk which indicates that it is an elevation of height 2.10 microns.

Fig. 71 (X 175) represents a positive low phase contrast photomicrograph showing a typical variety of disks within disks anchored at a point. Fig. 72 (X 100)
is another fine example of the micro-disks.

8.3 Micro-Disks On Matched Cleavage Faces:

In order to investigate the origin of the disk pattern, matched cleavage faces of calcium fluoride were selected and etched simultaneously. Thus Figs. 73(a) and 73(b) (X 125) represent the etch patterns produced on the matched faces, etched simultaneously, very carefully, for 20 seconds in a mixture of perchloric acid and aluminium chloride in the proportion of 3:1 at 90° C.

The following points are noteworthy:

1. The micro-disks are produced on both the matched faces.

2. The number of micro-disks in Fig. 73(b) is more than that in Fig. 73(a). This is clearly illustrated in Figs. 74(a) and 74(b) (X 200) which are magnified photographs of Figs. 73(a) and 73(b).

3. Black lines running across these photomicrographs are the cleavage lines. It appears as if there is no correlation whatsoever in the position of micro-disks on the two matched faces, with reference to the cleavage lines.

Patel and Tolansky (1957) reported that the cleavage lines on the matched faces move in the opposite direction, during the process of etching. A careful comparison of the micro-disks was therefore made on the
matched faces, not with reference to the cleavage lines but with reference to the dislocation etch pits which generally do not move during etching. Thus the correspondence in the position of three micro-disks on the matched faces has been established. These have been shown by marking A, B, C and A', B', C' in Figs. 73(a) and 73(b). It may be noted that: (i) the micro-disks match only in position but not in sizes and (ii) large number of micro-disks have no correlation on the matched faces.

8.4 Discussion:

The present investigation supports the findings of Patel & Goswami (1962), that the micro-disks can only be produced with a selective etchant under suitable conditions. That the amount of etching within the disks is less than that outside the disks, supports the conjecture of Pandeya and Tolansky (1961), that the disks may be the result of the protective action of some micro-bubbles formed on the crystal face. The micro-bubbles will save the region within the bubbles from the action of the etchant, till they burst. Thus the region outside the micro-disks will be exposed to the etchant for a longer period compared to the region within the disks. The amount of etching will thus be more outside than within the disks as observed. It is indeed very interesting to find the correlation in the position of some micro-disks on matched faces. This suggests that the formation of the micro-bubbles, the protective action of which
produces the micro-disks, may in some way be related to the crystal imperfections. These imperfections may be deposition of some impurity atoms during the growth of the crystal. When the crystal is cleaved, the cleavage may divide these impurity atoms on the two matched faces. When the cleavages are etched in a suitable etchant one of the product of the reaction of the etchant with the impurity atoms may be gaseous. This gas will naturally form micro-bubbles at the position of the impurity atoms and will thus protect the region within the bubbles from the action of the etchant till they burst. The correlation in the position of the micro-disks will thus be produced. The division of the impurity atoms on the two matched faces may not be equal. Thus the micro-bubbles formed on the matched faces may not be of equal sizes. In some cases, it is likely that, the cleavage may leave all impurity atoms on one face. In these cases there will not be any correlation in the micro-disks formed on the matched faces, even if the correlation exists in the micro-bubbles formed on matched faces, some bubbles may burst much earlier on one face than on the other. The micro-disks formed at the position of those bubbles which burst earlier will be completely washed out as reported by Patel and Goswami (loc cit). This will also make unfavourable conditions for the correlation of the micro-disks on matched faces.

The disk within the disk observed may be explained
by assuming that the micro-bubbles may increase or decrease in size due to various reasons. It is therefore conjectured that the existence of micro-disks on crystal faces indicates the presence of some impurity atoms deposited in the crystal during the growth.