# *An unusual distribution of precipitates in a diamond*

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*Summary.* The central region of a type 1 stone was found by optical and X-ray topographic examination to be densely populated with small inclusions. The X-ray studies indicated that these were precipitates formed after growth of the stone. Precipitate sizes fell into two groups, 1 $\mu$ or less, and around 5 $\mu$. Many cases of decoration of grown-in dislocations by these precipitates were observed.

IN the central region of a colourless diamond of irregular habit a certain cloudiness was noticed. A central slice about $1\frac{1}{2}$ mm thick was cut from the stone and its faces were polished so that the interior could be studied. The slice was roughly parallel to a cube plane and the section of the cloudy region cut by it was pincushion-shaped in outline. The diameter of the slice was about 5 mm and the maximum extension of the pincushion was about 3 mm along its diagonals. The stone was found to be opaque to wavelengths below 3000 Å indicating that it was type 1. Studies by optical microscopy, X-ray diffraction topography, and etching were then carried out.

## *Optical and X-ray topographic studies*

The contrast of the pincushion-shaped region was increased markedly when the slice was viewed by transmission between crossed polaroids (fig. 1). The pincushion was seen to comprise a host of small inclusions. The strain patterns of the larger of these were individually visible whereas the smallest inclusions gave rise to a general luminosity. Along the diagonals the inclusions were mostly larger than in the other regions of the pincushion. A few larger inclusions lay outside the pincushion.

The X-ray diffraction investigation was carried out by the method of projection topographs[2] and of section topographs.[3] Taking the plane of

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[2] A. R. Lang, Acta Cryst., 1959, vol. 12, p. 249.
[3] *Idem*, Acta Metallurgica, 1957, vol. 5, p. 358.

the slice as (001), the 220 reflection was obtained in symmetrical transmission, and the reflection from the ($\bar{1}$11) plane normal to (110) in unsymmetrical transmission (actually the plate was about 6° off (001) orientation). Information on the distribution of the inclusions within the depth of the slice was obtained by taking the 220 and $\bar{2}\bar{2}$0 stereo-pair

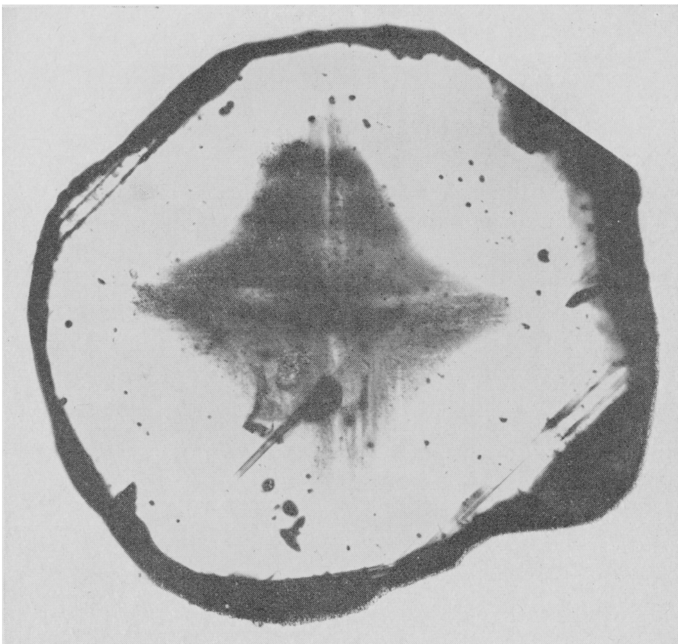


FIG. 1. Appearance of polished diamond slice between crossed polaroids (negative print). (Some saw-cut grooves still visible.)

of projection topographs, and by a set of section topographs. The 220 projection topograph (fig. 2) appears very similar to the optical picture (fig. 1) and it shows that the diagonals of the pincushion lie along the directions [110] and [1$\bar{1}$0]. In addition, the X-ray topographs show a radiating dislocation structure. The dislocation images can be traced back some distance into the pincushion, towards the centre of the stone. The topographs show no lamellar defects or growth stratifications. There are no unconformities or indications from the dislocation configuration that there has been any solution and re-growth. In fact the absence of stratifications suggests a simple history of continuous growth for the stone. The dislocation density is quite low and outside the pincushion region the crystal perfection is generally high, as is shown by the occurrence of Pendellösung fringes.[1] No cases of generation of dislocations at the small inclusions within the pincushion were observed, and there was no increase in dislocation density at the boundary of the pincushion.

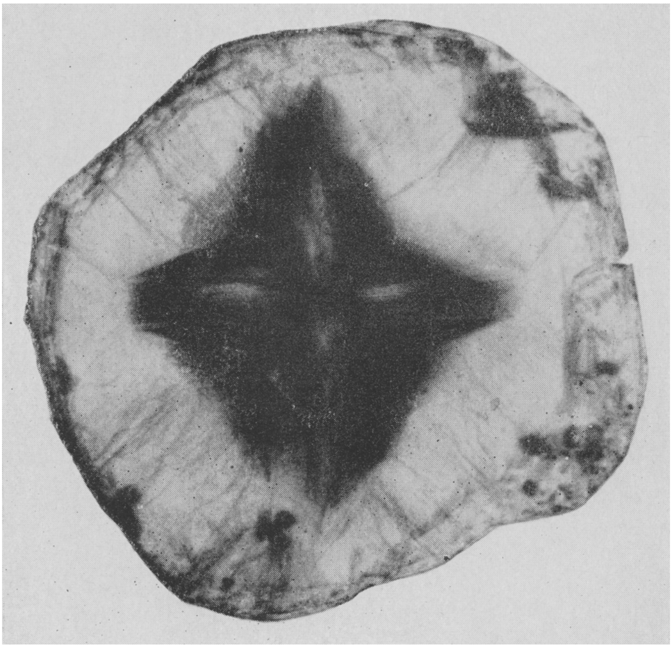


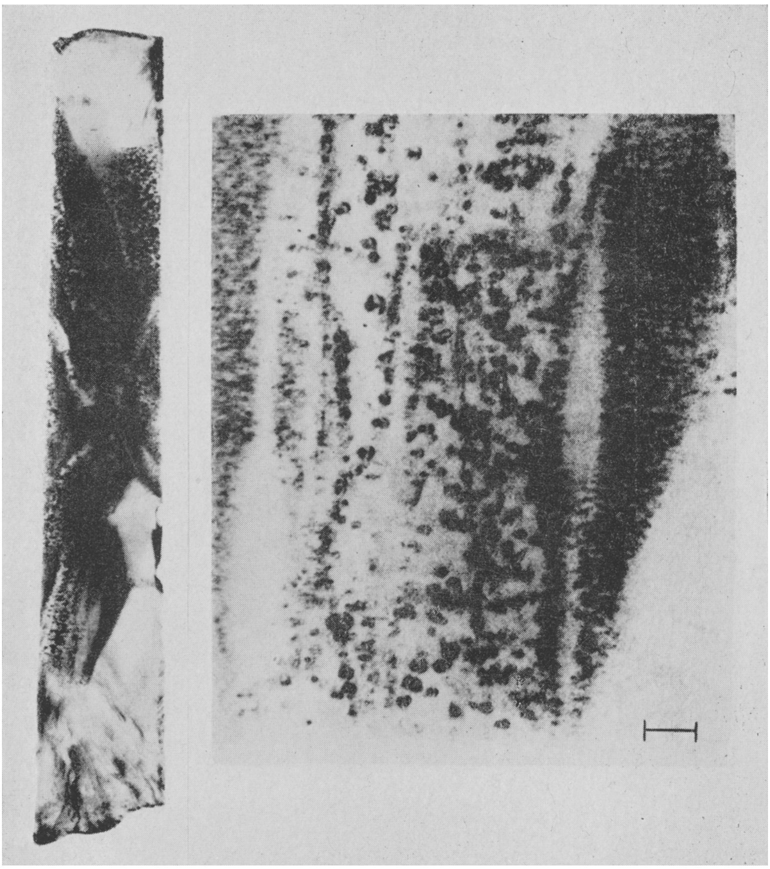
FIG. 2. X-ray projection topographs of slice, Mo-$K\alpha$ radiation, 220 reflection.

All the evidence from the X-ray topographs indicated that the inclusions were not particles mechanically included during the growth of the stone but had been formed after the stone had grown. Thus they were precipitates.

On X-ray section topographs the boundary of the pincushion appeared quite sharp, and a bimodal size distribution of precipitates was apparent (see fig. 3). Along the pincushion diagonals, [110] and [$1\bar{1}0$], finger-like regions containing respectively large precipitates, small precipitates, and apparently no precipitates lay side-by-side. Elsewhere there was

[1] N. Kato and A. R. Lang, Acta Cryst., 1959, vol. 12, p. 787.

some tendency for the regions containing smaller precipitates to predominate at the boundaries of the pincushion. It is difficult to estimate from the X-ray topographs the actual size and shape of the precipitates



FIGS. 3 and 4. Fig. 3 (left). X-ray section topograph of slice, Mo-$K\alpha$ radiation, $\bar{2}\bar{2}0$ reflection. Section slightly right of centre of fig. 2. Fig. 4 (right). Part of X-ray section topograph, Cu-$K\alpha$ radiation, $\bar{1}11$ reflection, [110] vertical. Scale mark 50 $\mu$.

since it is their strain field that causes the local intensification of diffracting power. Individual spots of extra blackening can be observed down to a size of 1 $\mu$, and this is the resolution limit of the method using Cu-$K\alpha$ radiation. The average size of the smaller precipitates seen is probably about 1 $\mu$. The larger precipitates usually give a double crescent type of diffraction image (such figures are given also by precipitates and inclusions in calcite and quartz). Sometimes an anchor-like figure is observed, the anchor shaft lying perpendicular to the Bragg plane. The average size of the larger precipitates is estimated to be 5 $\mu$. Within the pincushion there was X-ray evidence of some general warping of the lattice, covering an angular range of perhaps 20″ of arc. Probably this was due to the irregular density distribution of the precipitates.

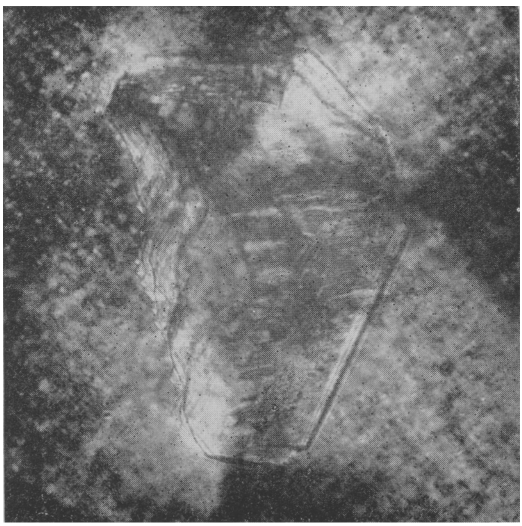


FIG. 5. Appearance of a large inclusion between crossed polaroids, maximum dimension about 0·4 mm.

A notable feature revealed by the X-ray topographs was the decoration of dislocations. In most cases when the image of a dislocation was traced back into the pincushion its course was found to be studded with precipitates. (Dislocations beaded by precipitates have also been observed in X-ray topographs of calcite and quartz.) The beading of dislocations was especially noticeable among the larger precipitates lying along the [110] and [$1\bar{1}0$] directions. An example is shown in fig. 4: this is part of a $\bar{1}11$ section topograph taken with Cu-$K\alpha$ radiation. However, the finger-like development of precipitate regions along [110] and [$1\bar{1}0$] does not seem to be attributable to dislocations running especially along these directions, for dislocations appear to radiate in all directions from the centre of the stone. This decoration of the dislocations is clear evidence that the precipitates were formed later than the dislocations, hence that they were formed after growth of the crystal.

It is reasonable to assume that the present distribution of precipitates corresponds to the distribution of some impurity incorporated during growth. This latter distribution would depend upon the habit of growth. In pure octahedral growth one would expect impurity to be concentrated where faces meet, i.e. on cube planes, with an extra concentration on the cube axes, along which four octahedral faces meet. Fingers of precipitates parallel to $\langle 110 \rangle$, as observed in the present crystal, might arise if the crystal grew in its early stages essentially as a cubo-octahedron. Lines radiating from the crystal centre to crystal corners would then lie along $\langle 110 \rangle$ directions.

### *Etching and polishing studies*

Strain birefringence and X-ray topography revealed a few larger inclusions both within and outside the pincushion. These may not be related at all to the small precipitates. One of these larger inclusions was cut by the surface of the slice. It appeared to be a diamond in a different orientation from the matrix since its polishing direction was different from that of the rest of the stone. Fig. 5 shows this inclusion in transmission between crossed polaroids: its maximum dimension is about 0·4 mm. Another inclusion appeared to be an octahedral diamond.

The slice was etched in a flux of potassium nitrate at 560° C for $1\frac{1}{2}$ hours. Square and rectangular pits were formed, with edges parallel to [110] and $[1\bar{1}0]$. They could be divided into isolated, large pits, and minute, barely resolvable pits. The density of large pits was greater in the pincushion region.

Nothing can be said yet about the chemical nature of the precipitates. It is hoped to perform further experiments on the stone.

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