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X-RAY STUDY OF THE EFFECT OF HYDROSTATIC PRESSURE ON THE DISLOCATION STRUCTURE OF SILICON SINGLE CRYSTALS

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X-ray examinations of the effect of hydrostatic pressure on the structure of diamond-type single crystals, especially silicon, have revealed nucleation of new dislocations after the material is submitted to a pressure of 10 kilobars. The aim of the present study is to examine these effects more closely in the case of various pressures. After the crystal was submitted to a pressure of 20 kilobars, the Lang method was used to observe the formation of line defects, which may be interpreted as systems of dislocations having a Burgers vector in the $[01\bar{1}]$ direction lying under a large angle with respect to the crystal surface.

Experimental studies of the effects caused by the action of hydrostatic pressure on various types of crystals performed hitherto have shown that changes occur in the dislocation structure of crystals after an appropriately high pressure has been applied. Among other things, it was noticed that new dislocations nucleate in policrystals of cubic metals, while the use of etch pitting methods gives evidence of an increased dislocation density in NaCl ionic crystals (Evans, Redfern, Wroński 1970). *X*-ray examination of the effect of pressure on single crystals of diamond structure, silicon above all, have revealed the nucleation of new dislocations (Auleytner *et al.* 1966). Observations were made after a pressure of the order of 10 kbars was applied to the crystals. The purpose of the present work is to study these effects closely in the case of various different pressures applied. The silicon crystal described in this paper was submitted to three successive stages of hydrostatic pressuration to values of 5,10 and 20 kbars. The silicon sample used had the shape of a plane-parallel plate with the surface orientation almost parallel to the (111) plane. It was cut perpendicularly to the direction of crystal growth obtained by the Czochralski method. Such sample orientation enables an interpretation of the dislocation structure in the crystal to be made relatively uniquely, both as regards the direction of the Burgers vector and the spatial location of the defects. Sample shape and overall picture of its interior is given

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in Fig. 1, whereas its stereographic projection is shown in Fig. 2. The sample was chracterized by regions of varied dislocation density, contained within the limits from 10^2 cm^{-2} to 10^5 cm^{-2}. The plate was approximately 0.4 mm thick. Lang's method of transmission topography permitting an image of the examined material's interior to be obtained was used in the investigation. After each stage of pressuration a series of *X*-ray photographs

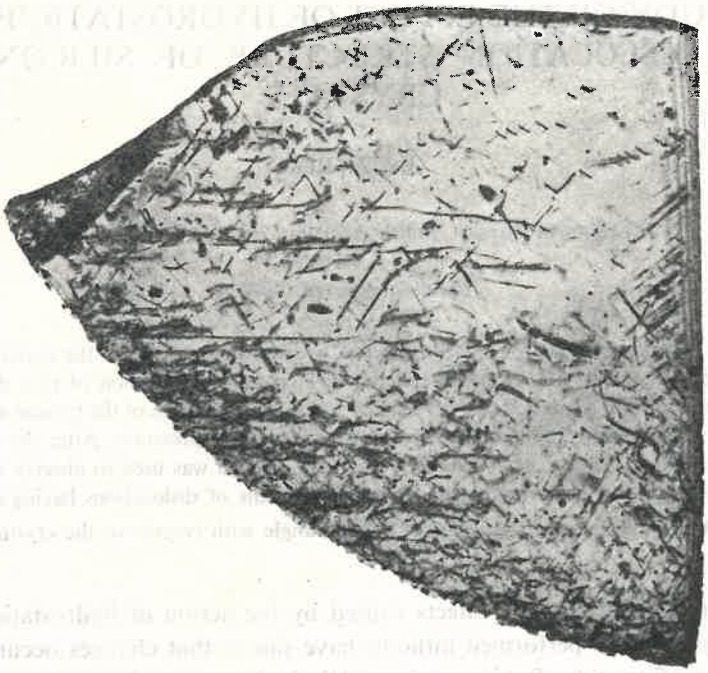

Fig. 1. Image of crystal interior obtained by Lang method for $(\bar{2}\bar{2}4)$ reflection; K_{α_1} Mo radiation, magnification 10 times

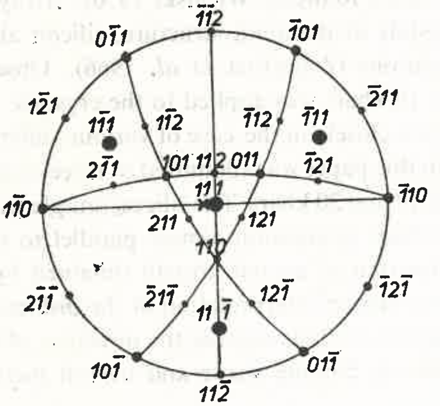

Fig. 2. Stereographic projection of examined crystal. × denotes projection of vector perpendicular to crystal surface

was taken, for three reflections of the {220} type, three {422} reflections and three {111} reflections. Examination of the initial crystal revealed that it had many different types of dislocations lying along various directions and with all ⟨110⟩ type Burgers vectors possible for this lattice, but there was a majority of 60° dislocations with ⟨110⟩ line direction.

The first changes in the internal crystal structure, namely, the nucleation of single defects of the dislocation type, were observed only after a pressure of the order of 10 kbars was applied. Examination of the crystal after submitting it to a pressure of 20 kbars showed

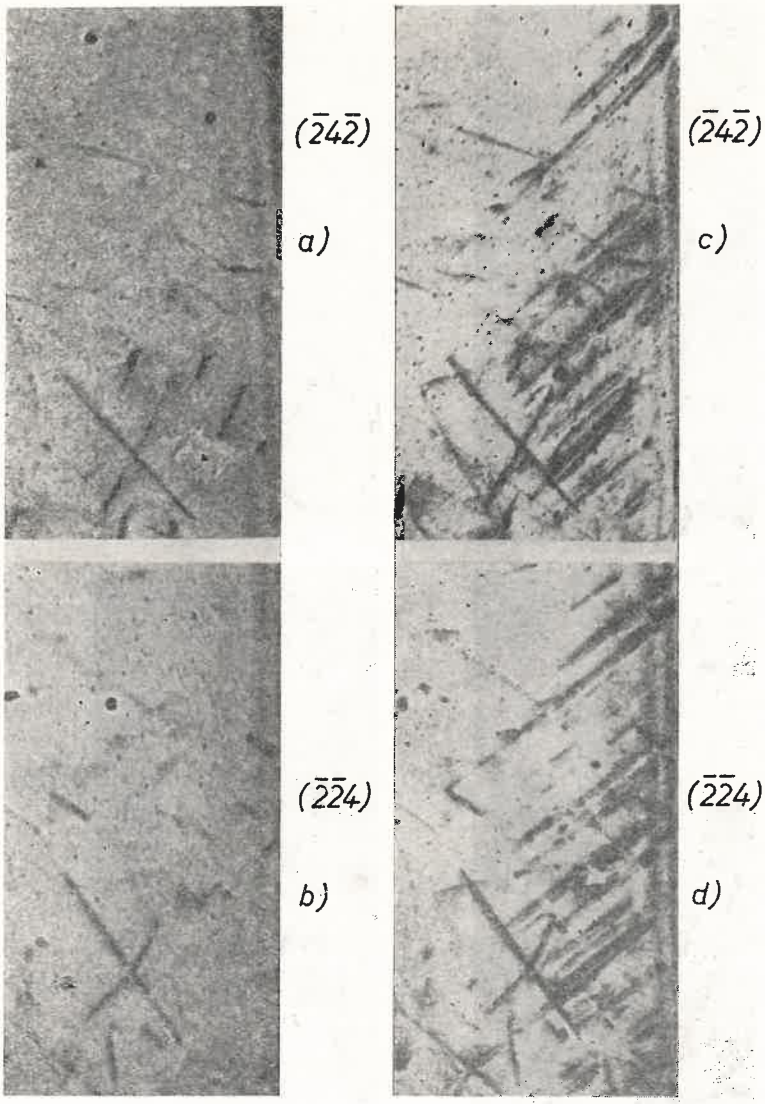

Fig. 3. View of the same crystal region: a, b — prior to pressuration to 20 kbars, c, d — after pressuration to 20 kbars. Magnification 30 times

that in the region which had apparently been without perturbances an entire system of defects characterized by the same type of contrast was formed (Figs 3 and 4). Since a change in intensity of the contrast on defects was observed to depend on the type of reflecting plane, it may be supposed that these are probably dislocations or, perhaps, systems of dislocations. For some reflections the observed long lines (the $(\bar{1}11)$ and $(\bar{4}22)$ reflections, see Fig. 4a, b) split up into systems of oblong spots, the centers of which are aligned along a direction with azimuth heading in the $[\bar{2}11]$ direction. The structure of the long lines for these reflections indicates that the spots are images of the various dislocations lying at

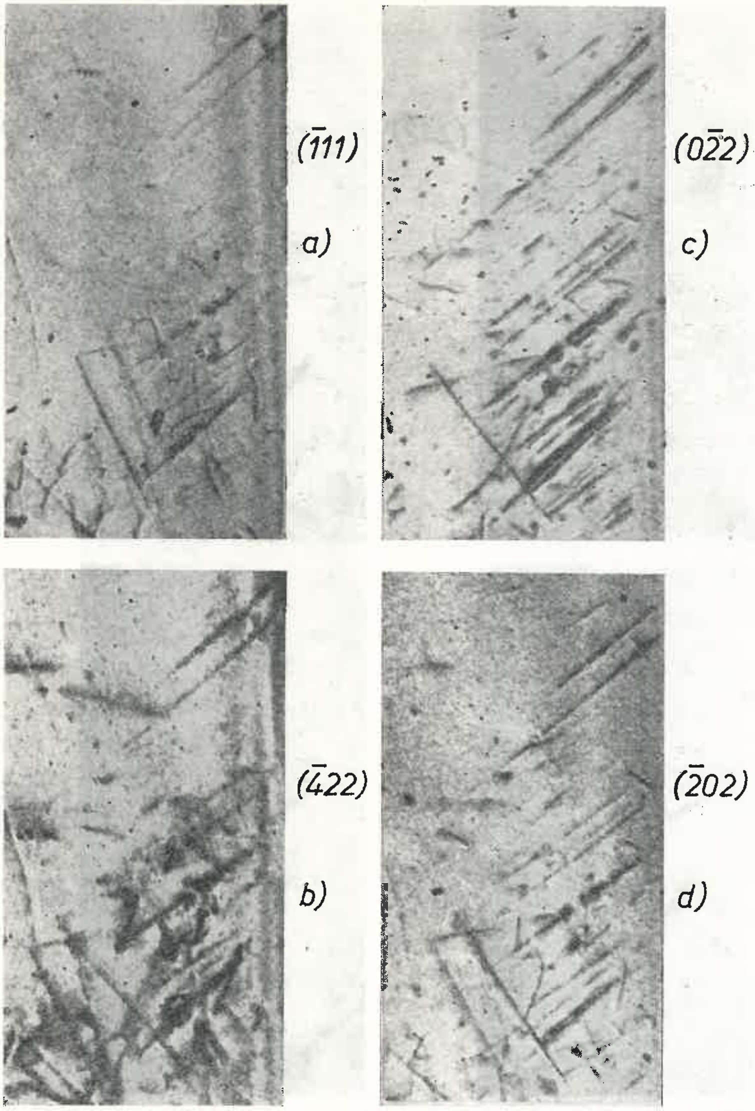

Fig. 4. Views of crystal region in which defects arose after pressuration to 20 kbars obtained for reflections off various crystal planes. Magnification 30 times
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a large angle to the surface, whereas the long lines are an image of a system of parallel and closely aligned dislocation lines. The behaviour of the contrast of the long lines for the various reflections, that is, considerable weakening for the $(\bar{1}11)$ and $(\bar{4}22)$ reflections (Fig. 4a, b) and strengthening for the $(\bar{2}4\bar{2})$, $(\bar{2}\bar{2}4)$ and $(0\bar{2}2)$ reflections (Figs 3c, d and 4c, respectively), implies that the probable direction of the Burgers vector of the occurring dislocations is $[0\bar{1}1]$. As this vector must lie in a slip plane, there thus two possible slip planes in which it may be, *viz.* (111) and $(\bar{1}11)$. If the slip occurred in the (111) plane, the various dislocations would have to be almost parallel to the sample plane. This is not in conformity with the observed picture of the whole region with defects revealed. There remains therefore the possibility of slip along the $(\bar{1}11)$ plane. Thus, the most probable representation of the long defects is as follows. It is a system of dislocation lines lying at a large angle to the surface, the components of which are characterized by a Burgers vector of direction $[0\bar{1}1]$ and slip plane $(\bar{1}11)$. These may be dislocations proceeding along a spatial direction, perhaps [101], [110], or [211]. Owing to the large angle that the dislocation lines make with the surface, however, these are probably dislocations along the [211] direction. Hence, the occurring defects may be interpreted as being [211] edge dislocations having a $[0\bar{1}1]$ Burgers vector and $(\bar{1}11)$ slip plane.

The type of contrast which forms at the defects in mention is very interesting. Namely, for some reflections there occurs a uniform white line surrounded by two black irregular lines (Figs 3c, d, 4c), for others a weakening of contrast and a division of the line into irregular spots (Fig. 4a, b) and, finally, for some a single line (Fig. 4d). The general picture of the contrast may be taken as yet another confirmation of the interpretation stating that the defects are not single dislocations. The formation of a strong kinematic contrast for the dislocation pattern may be regarded as proof that both the Burgers vector and the vector normal to the diffracting planes have approximately the same directions. Hence, the occurrence of a distinctly double contrast for the $(0\bar{2}2)$, $(\bar{2}\bar{2}4)$ and $(\bar{2}4\bar{2})$ reflections corroborate the $[0\bar{1}1]$ direction of the Burgers vector. The regularity of the thin white line shows that the dislocations in this system are arranged exactly parallelly along the $(0\bar{1}1)$ plane. On the other hand, the irregularity of the black contrast may be proof that this system is not in periodic order, *i. e.* the defects do not lie at constant mutual spacings. The appearance of such a system should provide some hints regarding the way in which these defects arise. The grouping of dislocations in the $(0\bar{1}1)$ plane perpendicular to the $(\bar{1}11)$ slip plane implies that the dislocation systems become formed under the influence of a force acting perpendicularly to this slip plane. From simple reasoning based on the Peach-Koehler formula. (Read 1953)

$$\boldsymbol{F} = \boldsymbol{b}\boldsymbol{P} \times \boldsymbol{dl}$$

where

$$P = \begin{pmatrix} -p & 0 & 0 \\ 0 & -p & 0 \\ 0 & 0 & -p \end{pmatrix}$$

for the force acting on an element $\boldsymbol{dl}$ of the dislocation with Burgers vector $\boldsymbol{b}$ set in an external stress field described by a tensor ofdiagonal form P(p being the pressure applied).

and originating from triaxial hydrostatic pressuration, it follows that this force acts on the edge dislocation component only along the direction perpendicular to its slip plane. The formed dislocation systems imply that they were formed by this kind of force acting on the crystal which, before pressuration, may have had some microdefects in certain places which become the nucleus of the system brought about by the pressuration. The grouping of dislocations in the $(0\bar{1}1)$ plane also corroborates the fact that $\{110\}$ planes in diamond-type structures are also slip planes of the material.

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