It was found that the dislocation density was reduced markedly by this annealing. For example, slices with an as-grown dislocation density of $5\times 10^4/cm^2$ were found to have $5\times 10^2/cm^2$ dislocations after the anneal. This observation would suggest that the collapse of vacancy clusters into dislocation loops, and subsequent expansion of such loops, is not an important mechanism for the formation of dislocations in copper. There is some indirect evidence, which can not be presented here, that there are some small dislocation loops in these slices. Perhaps these experiments may serve to better define the limits for calculations such as Dr. Elbaum's.

Elbaum, C.: Dr. Young's experiments are very instructive, but I think that they are not necessarily inconsistent with the results that I presented, since Dr. Young apparently observes a limiting dislocation density in his crystals.

Kuhlmann-Wilsdorf, D.: One of my graduate students, Mr. R. Fabiniak, has been able to construct a furnace and temperature control apparatus which allows to grow single crystals of pure aluminum at an ambient temperature within a very few degrees of the melting point. According to x-ray evidence, these crystals are free of sub-boundaries. This is, of course, in support of the theory of Chalmers and Teghtsoonian that sub-boundaries are due to vacancy condensation.

Chalmers, B.: I agree that this experimental result does not conflict with the vacancy condensation theory. However, our own experimental results show that, under some conditions, the sub-boundaries are formed in the immediate vicinity of the solid-liquid interface.

PROCEEDINGS OF THE INTERNATIONAL CONFERENCE ON CRYSTAL LATTICE DEFECTS, 1962, CONFERENCE
JOURNAL OF THE PHYSICAL SOCIETY OF JAPAN VOL. 18, SUPPLEMENT III, 1963

Hexagonal Networks of Linear Imperfections in Single Crystals of Cadmium

J. C. CRUMP III AND J. W. MITCHELL

Department of Physics, University of Virginia, U.S.A.

A new type of hexagonal network of dislocations has been observed in thin crystals of cadmium grown by sublimation. The vacancy-type loops, observed by Price, which are introduced by bombarding the crystals with negative ions in the electron microscope disappear when they reach a certain size. After a very short delay, hexagonal networks begin to develop in the area previously occupied by the loops. These networks lie in the basal planes. The directions of the three sets of linear imperfections which form them are parallel to the $\langle\bar{1}100\rangle$ directions in the basal planes. Mobile specks which are attributed to clusters of interstitial atoms have also been observed and seen to interact with the vacancy-type dislocation loops.

Price[1),2)] first observed vacancy-type dislocation loops in thin crystals of cadmium grown by the slow distillation of cadmium in an atmosphere of argon (Coleman and Sears[3)]). We have observed the formation and growth of dislocation loops of the same kind in similarly grown thin crystals of cadmium. Our crystals which were mounted either on copper grids or between copper grids were examined in the Philips EM 100B electron microscope operated at 100KV. Fig. 1a shows a compound vacancy-type loops of this kind.

In addition we have discovered and studied a new phenomenon which was apparently not observed by Price[1),2)]. After the vacancy-type loops have exceeded a certain size they often disappear abruptly. The disappearance may take place without any apparent cause or it may occur after the interaction of the loop with a mobile dislocation or a mobile discrete speck. Following this, linear imperfections appear in the area previously occupied by the loop. These imperfections sometimes appear as single lines, as parallel pairs of lines or as parallel groups of lines. In other parts of the area of the loop, single nodes may appear from which lines making angles of 120° with each other radiate outwards. In still other areas, hexagonal elements associated with six outwardly radiating lines may appear. These linear imperfections increase in length during observation as may be seen in Figs. 1b, 1c and 1d, which were taken successively at approximately three second intervals. They eventually meet and intersect with lines lying along one of the other two directions of the networks. A segment lying between two nodes along the remaining third direction is then formed as may be seen clearly in the series of Fig. 1. When two lines meet after converging to a point at an angle of 60°, a third line expands outwards from the resulting node along the other symmetrically related direction. When three lines meet after converging into the same area, a hexagonal element is formed. The lines which radiate outwards from three of the corners of the element then continue to increase in length.

The continuation of these processes results in the formation of the hexagonal networks of dislocation lines. The elements of the networks readjust by lateral displacements

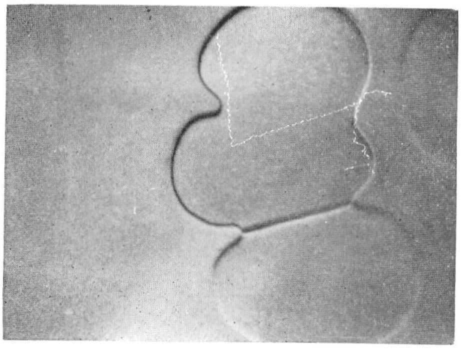


Fig. 1a. Compound vacancy-type dislocation loop formed by the fusion of three loops in a single crystal of cadmium. ×12,300.

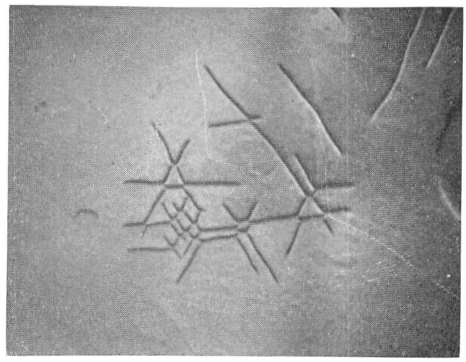


Fig. 1b. Electron micrograph showing the early stage in the formation of a hexagonal network of linear imperfections from the compound dislocation loop of Fig. 1a. ×12,300.

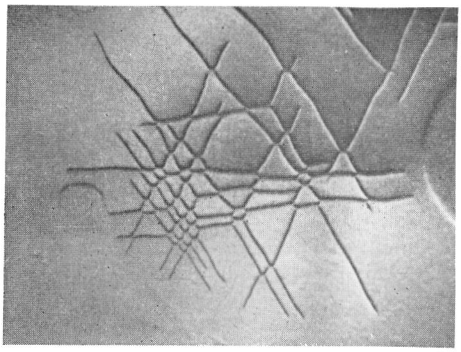


Fig. 1c. Electron micrograph taken approximately three seconds after Fig. 1b, showing the further development of the hexagonal network. Notice that the faint contrast effects which appear in Fig. 1b within the area of the loop have now faded completely. ×12,300.

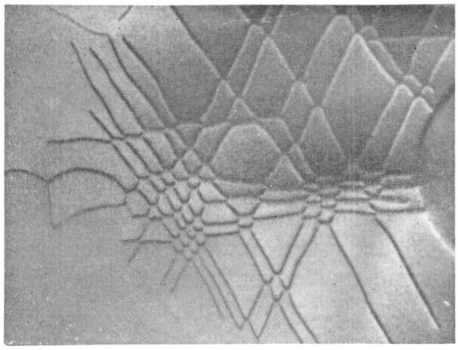
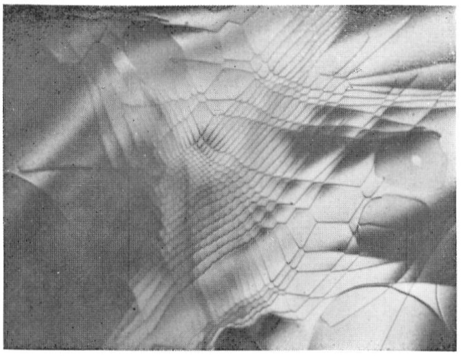
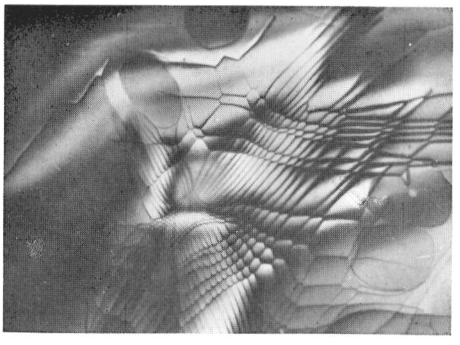


Fig. 1d. Electron micrograph taken approximately three seconds after Fig. 1c. The light and dark contrast effects in this series may arise from the presence of a bend extinction contour ×12,300



(a)



(b)

Figs. 2a and b. Successive stages in the contraction of a hexagonal network of linear impefections. ×5,400.

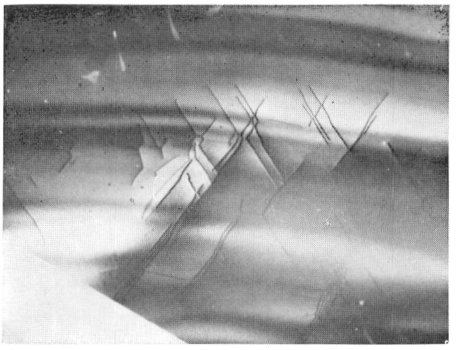


Fig. 3. Electron micrograph taken at the edge of the specimen showing that the linear imperfections lie along the directions of p-vectors. The edge of the crystal which appears in the bottom left-hand corner is parallel to an a-vector. ×3,300.

and changes in length and groups of reasonably regular hexagonal networks form in the plane previously occupied by the dislocation loop. The networks, which appear to lie in a basal plane of the cadmium crystal, then shrink in a quite remarkable way by the inward displacement of the hexagonal elements towards areas of maximum surface density of elements. This process is illustrated in Figs. 2a and 2b. The general arrangement of the nodes remains essentially unaltered during this contraction. The hexagonal elements of the networks simply shrink in size as they drift inwards towards the central area. They can finally no longer be seen in this area and a circle of slow fading then spreads outwards from it. No trace of the system of lines can ultimately be seen by diffraction contrast observations.

We have established by electron diffraction observations that the lines lie along the $\langle\bar{1}100\rangle$ directions in the basal planes. The edge of the crystal which lies along the direction of an a-vector is shown in Fig. 3. It can be seen that the linear imperfections formed by the collapse of a number of small vacancy-type dislocation loops lie generally along the directions of the p-vectors. The most striking feature of the linear imperfections has always been that they appear to end within the crystals. In networks of the type shown in Fig. 1, we have not been able to observe any segments linking the ends of the dislocation lines with the surface of the crystal. If this is indeed true, the linear imperfections cannot be ordinary dislocation lines. It might appear that the loops which collapse are so close to the surface that segments linking the networks with the surface would not be observed even if they were present. The observations of the stacking fault contrast of the loops showed, however, that some of the loops were located near the surface but that others were located at considerable depths below the surface. The collapse of loops at various depths below the surface was studied but no essential difference in the phenomena was observed. The conclusion that the linear imperfections may terminate in the basal plane appears to be confirmed by observations made of the interaction between them and other dislocation loops which had not yet collapsed.

On the basis of these and other observations, we propose that the lines which we have observed are formed by narrow ribbons of stacking fault bounded by imperfect dislocations with Burgers vectors of the type $\frac{1}{2}c+p$. The lines lie generally along the directions of the p-components of their Burgers vectors and they must move laterally by conservative climb processes. Their elongation results from the addition of vacancies to their ends according to a whisker type of growth mechanism. The arrangement which we propose for the imperfect dislocations in two adjacent nodes of a network is shown in Fig. 4. The nodes have the same structure as that which has been discussed by Berghezan *et al.*[4] for the interaction of three approximately circular vacancy-type loops in zinc and by Price[2] for the similar interaction in cadmium. This is the only model with which we have so far

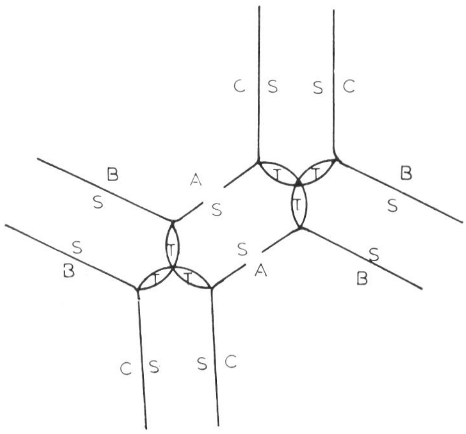


Fig. 4. The arrangement of the imperfect dislocations in two adjacent nodes in an intersecting system of ribbons of stacking fault. (See Berghezan *et al.*, 1961 and Price 1961a for the notation).

been able to work out a satisfactory description of the vector geometry of the interactions between the linear imperfections and between the linear imperfections and the vacancy-type dislocation loops. We have, however, encountered a number of other difficulties in the detailed interpretation of the observations. Several of these have not yet been fully resolved and our proposed interpretation has only the status of a tentative working hypothesis.

The images of the linear imperfections sometimes show light and dark contrast which would be characteristic of simple dislocations rather than of dislocation dipoles and this, at first sight, is difficult to reconcile with the hypothesis. In the same areas, however, the dislocation loops which enclose areas of stacking fault also show the same light and dark contrast at opposite ends of a corresponding diameter. It is difficult to mount the thin ribbons without introducing slight curvatures and these contrast effects appear to be associated with bend extinction contours. We have also frequently observed an abrupt change in contrast across the linear imperfections, the area on one side being light and the area on the other side dark. Tilting experiments have shown that the crystal is kinked through a small angle at the imperfection. Strong curvatures develop during the later stages of this dislocation phenomenon as can be clearly seen in Figs. 2a and 2b.

Our observations appear to indicate that the vacancy-type loops become unstable as they increase in size and there may be two reasons for this. Vacancies and interstitials are being produced continuously by ion bombardment during the observation of the phenomena in the electron microscope. The vacancies will associate preferentially with the outer perimeter of the loop to increase its size while the interstitials will associate with the central area of the loop. The free energy of the loop will be increased if interstitials become trapped in the plane of the loop as a distribution of mobile atoms. A second reason for instability may arise from the fact that the elastic strain energy can be reduced if a large loop with which a single p-vector is associated is transformed into a dislocation configuration in which all three p-vectors are symmetrically involved.

The hypothesis that the linear imperfections are formed by narrow ribbons of stacking fault bounded by imperfect dislocations is the only one which appears to be consistent with the interactions between linear imperfections and vacancy-type loops which we have observed. It is, however, very difficult to understand how the ribbons can remain of uniform width as they elongate and move laterally by conservative climb processes. It would appear that the ribbons should transform spontaneously by climb processes into circular discs of equivalent area. The stability of the ribbons against dissociation into vacancies which would diffuse to the free surface or to other sinks for them must be determined by the presence of a supersaturation of vacancies in the system. The outwardly directed chemical forces then oppose the inwardly directed forces due to the attractive interaction between the imperfect dislocations bounding the stacking fault and the tendency for the area of the stacking fault to decrease. The energy per unit length of a narrow ribbon of stacking fault formed by the condensation of vacancies may, moreover, not decrease steadily with decreasing width. At a certain critical width the ribbon of stacking fault must transform into a system of parallel rows of associated vacancies with an increase in the energy per unit length of the system. In this case, although the imperfect dislocations bounding the ribbons of stacking fault exert a long range attraction, they should experience short range repulsion. In these circumstances, a minimum would appear in the curve for the energy per unit length as a function of the width of the stacking fault, provided that the supersaturation in vacancies exceeded a certain critical value.

The shrinking of the networks and their final disappearance is attributed to the diffusion of the equivalent number of vacancies to the surface of the specimen or to sinks associated with the high density of dislocations present at this stage. The pattern of nodes is preserved while the dimensions of the networks decrease.

Much more experimental work will be required before all the aspects of this system can be fully understood but we believe that the results are of sufficient interest to justify the presentation of a preliminary account of the work.

Acknowledgement

This experimental work has been supported by the United States Atomic Energy Commission under Contract No. AT-(40-1)-2488 and by the Office of Naval Research under Contract No. NONR 474-05. This support is greatly appreciated.

References

1 P. B. Price: Phys. Rev. Letters **6** (1961) 615.
2 P. B. Price: General Electric Research Laboratory Report, No. 61-RL-2794M (1961).
3 R. V. Coleman and G. W. Sears: Acta Met. **5** (1957) 131,
4 A. Berghezan, A. Fourdeux and S. Amelinckx: Acta Met. **9** (1961) 464.