below 77°K, although the amount is small. However, the fact that we would like to emphasize is the larger increase in resistivity due to the deformation at higher temperatures, despite of the supposition that more annihilation of point defects during the plastic deformation should occur at 90°K than at 77°K. Another fact to be noted is the age-hardening followed by the almost complete recovery of the increased amount of flow stress on holding at low temperatures, as shown in Fig. 1. This fact seems to be difficult to interpret in terms of Dr. Sosin's previous view that the annihilation of close interstitial-vacancy pairs is responsible for the recovery process. With regard to Stage II, we simply assumed that the main recovery process due to interstitials was achieved around 90°K, by considering the softening process. Finally, on the subject of Stage III, there are still many arguments.

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

The Formation of Defects in Crystal Lattice by Twinning

V. I. STARTSEV

Physical Technical Institute for Low Temperatures, Kharkov, U.S.S.R.

It is shown, that the twinning of calcite crystal is accompanied by formation of dislocations, point defects and stacking faults in crystal.

According to the universally recognized point of view, crystal lattice in a twin lamella formed by the plastic deformation differs from the crystal lattice of parent crystal only in orientation. The investigation of fine structure of twinned crystal, however, shows that the twinning as well as the slip is accompanied by formation of dislocations, point defects and stacking faults in crystals.

The results of some experiments on studying the lattice defects formed in the process of twinning in crystals of calcite and other crystals are presented in this report.

The revealing of dislocations and other defects was carried out by etch pit method. The calcite crystals were etched by 2% solution of tartaric acid in water.

The investigation of twinning mechanism of calcite crystals has shown that the formation and growth of twin under mechanical loading occurs by formation and movement of twinning dislocations in the twinning plane.[1]

It was possible to observe directly the twinning dislocations and their movement and also to measure the stress necessary to begin the movement of twinning dislocation, that is, the starting stress. For calcite it seemed as low as 40–45 g/mm^2.[2] The experience shows, however, that on the twin boundary there are not only the ordinary twinning dislocations, but also dislocations of other type. The etch pits of ordinary twinning dislocations are more shallow than the etch pits of these dislocations. Obviously it shows the large lattice distortion caused by such dislocations. It appeared that these dislocations may serve as a source of formation of different defects in the body of a twin. Their appearance on the twin boundary leads to the hindrance of twinning dislocations. This is one of the causes of work-hardening during the twinning process.

Fig. 1 shows two types of dislocations on twin boundary. The deep etch pits correspond to second type dislocations. Under the action of mechanical force the dislocations of the second type do not move in composition plane, but together with twin boundary in the direction of broadening of the twin. These and other properties of the

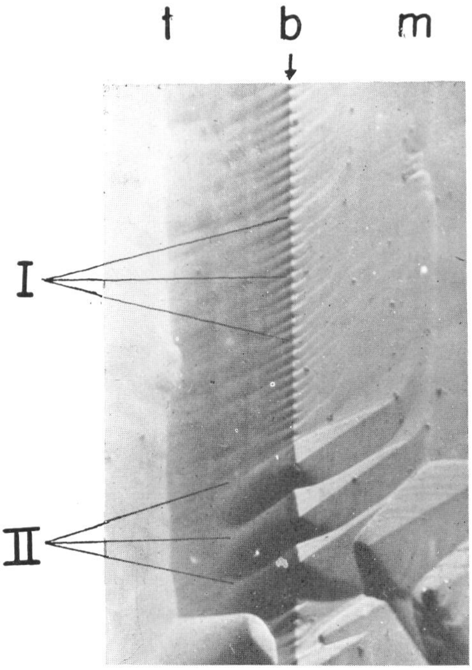

Fig. 1. t—twin, b—twin boundary, m—mother crystal, I—twinning dislocations, II—sessile dislocations. ×600.

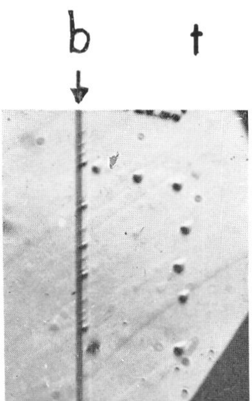

Fig. 2. t—twin, b—twin boundary. ×340.

second type dislocations find their explanation in the frames of analysis of interaction of twinning and perfect dislocations. The consideration of possible reactions between twinning and perfect dislocations on twin boundary shows that the second type dislocations may be either perfect dislocations slipping in the crystal together with the movement of twin boundary or sessile partial dislocations formed as a result of interaction of twinning and perfect dislocations.

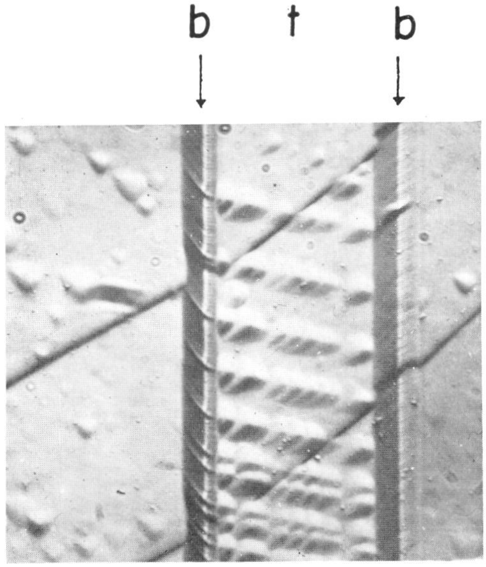

Fig. 3. t—twin, b—twin boundary. ×306.

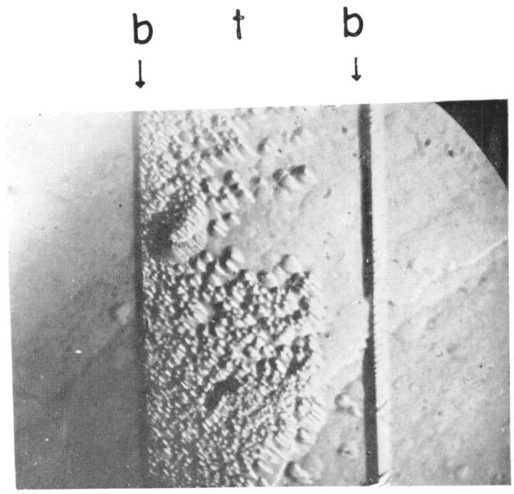

Fig. 4. Formation of dislocations in twin. t—twin, b—twin boundary. ×340.

The availability of such dislocations on the twin boundary is often accompanied by appearance of perfect dislocations in the body of twin. In the next figure (Fig. 2) we see perfect dislocations in the body of twin, that synonymously correspond to less mobile dislocations in the boundary. A number of experiments shows that the pairs of etch pits correspond to the going out of two branches of one dislocation loop.

The less mobile dislocations often lead to the slip in the twin. The next figure (Fig. 3) shows the slip lines in twin, arrived at less mobile dislocations on the twin boundary. These slip lines are marked off by rows of etch pits. Perfect dislocations in a twin may form on a mass scale. Thus, Fig. 4 shows a lot of perfect dislocations formed in a twin in the process of twinning. There are slip dislocations among these dislocations but the sessible dislocations are also available. The theoretical considerations show that the sessible dislocations may have Burgers vector along [110] direction and even lie in (111) plane, which is not the plane of easy glide in calcite.

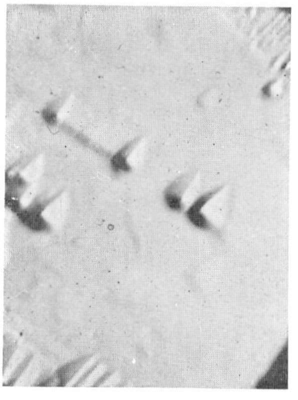

Fig. 5. Stacking fault in twin of calcite crystal. $\times 600$.

The dislocations, that lie in (111) plane, may suffer splitting in partial dislocations, forming a stacking fault in (111) plane between them. Fig. 5 shows the etch pattern that appears in this case. There are seen two etch pits, connected by etch groove, orientated along $[1\bar{1}0]$. Deep etching and the analysis of etch pattern on two adjacent faces of crystal show that this defect is in (111) plane.

The formation of perfect dislocations in crystals in the process of twinning is due to relaxation of stresses at the boundaries of twins, arising from piling up of twinning dislocations on the boundaries. The process of formation of perfect dislocations is dependent on the temperature and velocity of movement of twin boundary. When the process of twinning goes intermittently, that is, the twin boundary is stopped for some time in the process of twinning, and then moves again, perfect dislocations form at the place of stoppage of the boundary. Fig. 6 shows etch pattern of crystal, which was subjected to such twinning. At the places of stoppage of the boundary by the twinning, vertical rows of perfect dislocations were formed. The true position of the boundary is marked off by vertical etch groove.

In twinning, side by side with dislocations and stacking faults, there occur clusters of point defects. They come to light by etching as thinnest grooves. Such grooves often trail as traces after less mobile dislocations, that are in the twin boundary. Sometimes they finish on perfect dislocations within twin. There are found crystallographically orientated grooves (along [100], [110] directions) and also the grooves of arbitrary

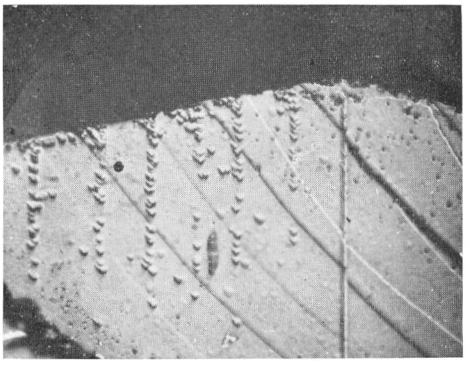

Fig. 6. Rows of dislocations at the places of stoppage of the twin boundary. $\times 340$.

Fig. 7. Clusters of point defects in the twin. $\times 600$.

orientation. In the next figure (Fig. 7) is given the etch pattern of a part of a twin which contains above mentioned clusters of point defects. There are seen the etch grooves, one of which finishes at a dislocation etch pit.

The deep etching method shows that there are not only linear but also two-dimensional clusters of point defects. In the last case the groove remains by deep etching. Fig. 8 shows etch patterns, corresponding to different depths of etching (a—2μ, b—15μ, c—25μ). There an etch pattern of stacking fault is given for comparison. It is seen that the deep etching reveals also the dislocation loops which do not go out on the surface of crystal and are not disclosed at short time etching.

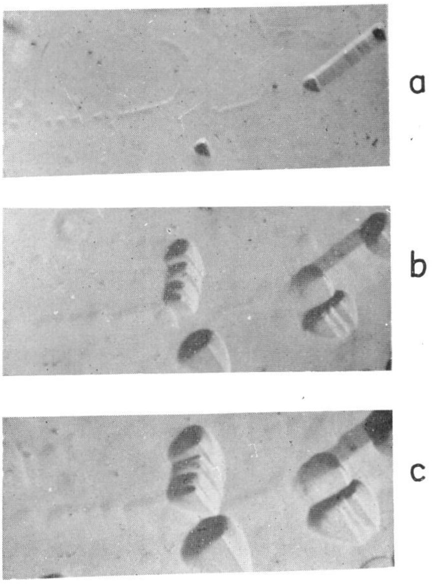

Fig. 8. Deep etching. a—2μ, b—15μ, c—25μ. $\times 600$.

The study of thermal stability of described defects shows that the low temperature (500–550°C) annealing from one to three hours leads to vanishing of some crystallographically orientated defects, while many arbitrarily orientated defects do not disappear (it is verified by repeated etching after annealing).

In the next figure (Fig. 9a, b) it is shown that the etch groove, corresponding to the cluster of point defects, became flat-bottomed as a result of repeated etching after annealing. That is, the cluster disappeared because of annealing.

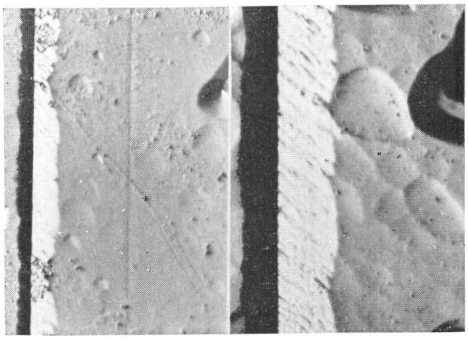

Fig. 9. a—crystal before annealing. b—crystal after annealing. $\times 600$.

The different thermal stability of point defects clusters is apparently due to their different electrical nature. The neutral clusters, obviously, are thermally more stable than charged ones. In this sense the behavior of point defects in calcite is identical to their behavior in other ionic crystals, for example in alkali halide crystals. The appearance of point defects clusters by twinning may be connected with the movement of screw dislocations containing jogs or with the movement of less mobile dislocations that are in the twin boundary.

By detwinning there are the same processes as by the direct twinning, though a part of defects here is removed from the crystal. In the next figure (Fig. 10) we see a picture of repeated etching of calcite crystal after detwinning. The etch pits, which correspond to dislocations in crystal before detwinning, became flat-bottomed. Flat-bottomed became also etch grooves on the

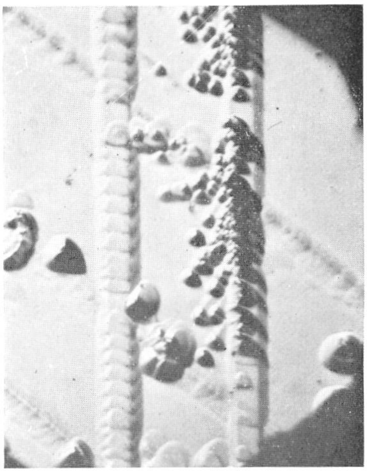

Fig. 10. Crystal after detwinning. $\times 340$.

boundaries of the twin. However, there appeared more new dislocations, stacking faults and so on, which were not present in crystal before detwinning. Thus, the crystal that had been subjected to twinning contains much more defects of crystal lattice than at the origin.

It is not out of place to mention that near the surface of crystal the number of dislocations, formed by twinning, is essentially more than in its depth. It may be connected with lower energy of formation of lattice defects near the surface of crystal than in the depth of it.

The phenomena described in this report were also observed by us in sodium nitrate crystals, which is a crystallographical analogue of calcite, and in metal crystals.

References

1 V. I. Startsev, V. S. Bengoose, F. F. Lavrentjev, and L. M. Soifer: Crystallographia **5, 3** (1960) 441-445; **5, 5** (1960) 737-743 (in Russian)

2 V. S. Bengoose, S. N. Komnik, and V. I. Startsev: *Dokladji.*, Academiji Nauk, U. S. S. R. **141, V3** (1961) p. 607-610 (in Russian).

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

Point Defect Clustering in Nickel*

I. G. GREENFIELD AND H. G. F. WILSDORF
The Franklin Institute Laboratories, Philadelphia 3 Pennsylvania, U.S.A.

Point defects were introduced into nickel by neutron bombardment and quenching. Irradiations of 1×10^{18} nvt (total fast flux) were carried out at 70°C and 200°C, and the quenching treatments were varied by quenching to temperatures ranging from −10°C to above the Curie temperature. From the results obtained, it has been concluded that, because of local regions of damage which act as nucleation sites, prismatic dislocation loops are more easily formed in irradiated nickel than in quenched nickel.

Two methods of introducing point defects into a metal crystal are (1) by radiation with high energy particles (2) by quenching. Radiation produces both vacancies and interstitials in local regions of each encounter, whereas rapid quenching from high temperatures will result in a supersaturation of vacancies in a random distribution. Depending upon the temperature, dispersion of point defects, and the activation energy to be surmounted for the formation of various aggregates of point defects, the point defects will either remain dispersed, annihilate opposites, or form clusters of their own species. This investigation contrasts the point defect behavior in nickel produced by irradiation with high energy neutrons and by quenching. The major experimental technique is dependent upon the observations made with the electron microscope. Although single vacancies or interstitials have not been identified by electron microscopy, structures such as prismatic dislocation loops which are directly attributable to a supersaturation of point defects, have been observed in quenched and irradiated metals (see refs. 1–4). Since the energy of a small void below a critical size would be less than that of a prismatic dislocation loop[4], it is probable that voids exist before prismatic dislocations are formed. It is estimated that voids could be seen by electron microscopy because of a mass thickness contrast mechanism only when the void is greater than ten percent of the thickness of the foil. The experiments described are designed to attempt to make visible the structures which result from various neutron irradiation treatments and

* This research was supported by the U. S. Atomic Energy Commission.