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

The Effects of Ion Bombardment on Molybdenite and Graphite

Kazuhiro MIHAMA
Department of Applied Physics, Nagoya University, Nagoya, Japan

The effects of ion bombardment on the specimens of layer structure such as molybdenite and graphite are studied by means of transmission electron microscopy. The ion bombardment is performed by the use of ion gun with air or argon, operated under the accelerating voltage of 5 to 10 kV. The effects of the ion bombardment observed in electron micrographs can be classified as follows: (1) Appearance of pair of "black and white spots", (2) appearance of "pair of loops", developed from the pair of spots, and (3) pinning up or tangling of dislocation lines at the positions of pair of spots or pair of loops. These productions may be due to the aggregation of point defects created by the impact of ion beam to the constituent atoms.

1. Introduction

The technique of ion bombardment has been well known since many years ago as "sputtering" and applied to several problems, such as etching and cleaning of metal surfaces. In these applications, the surface effects of ion bombardment have only been concerned, but recently theoretical and experimental studies have been done on the interactions of charged particles and matter in connection with the problem of "radiation damage", particularly the lattice defects produced in specimens[1)~5)].

In the present work, the effects of ion bombardment on the specimens of layer structure such as molybdenite and graphite are studied by means of transmission electron microscopy.

2. Experimental Results

The specimens are prepared by cleaving bulk ones by the aid of adhesive tape to the thickness of about 1000 Å.

The ion bombardment is performed by the use of ion gun[6)] with air or argon in high vacuum and operated under the accelerating voltage of 5 to 10 kV.

Ion bombardment gives the inhomogeneous sputtering over the specimen without changing the crystal orientation even in the case of severe damage of crystal surface. Fig. 1 shows a typical electron micrograph before ion bombardment of MoS_2, in which dislocation net-works and extinction contours are observed. Fig. 2 shows the intermediate stage of ion etching. Among the holes of several thousand Å in size we can find small holes of the order of 100 Å in size over the whole specimen. Fig. 3 shows the last stage of ion etching. In this stage, traces of steps existed initially on the specimen are observed and the electron diffraction pattern from the same area is the N-pattern showing that the crystal orientation is not changed. If another surface is put near the bombarded specimen, molybdenite crystals are formed on it due to the sputtered atoms (Fig. 4).

Except the etching effects, the effects of ion bombardment are characterized by the formation of the pair of black and white spots and/or the pair of loops in transmission electron microscopy (Fig. 5 and Fig. 6). These black and white spots and pair of loops appeared depending sensitively on the diffraction condition of the specimen, that is, these sizes and intensity distributions vary sensitively with the tilting of the specimen, but the positions do not change.

3. Discussions

Similar black and white spots were observed by Bollmann[7)] for neutron-irradiated graphite and also by Pashley *et al.*[4)] for ion-damaged gold. They interpreted the pair of spots as the aggregation of point defects induced by neutron or ion bombardment.

The pair of loops seem developed from the pair of spots mentioned above, and are aligned in the same crystallographic direction at least in a limited area of electron micrograph. The intensity distribution, which depends very much upon the diffraction conditions, is not always symmetrical with respect to the central line of a pair.

Sometimes, dislocation lines are pinned up to the position of pair of loops (Fig. 7). In addition, although in a few cases, the tangling

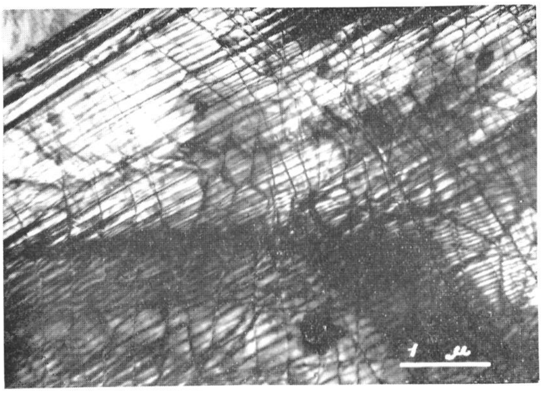

Fig. 1. MoS_2 before ion bombardment.

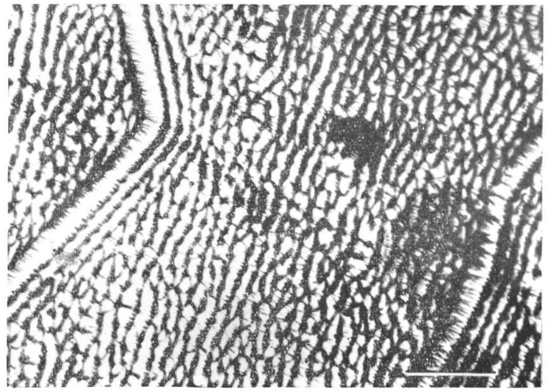

Fig. 3. Last stage of ion etching for MoS_2.

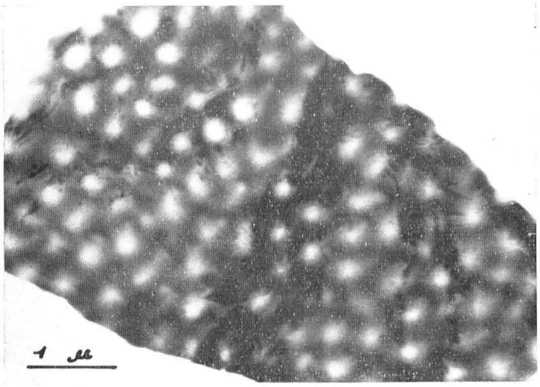

Fig. 2. Intermediate ion etching for MoS_2.

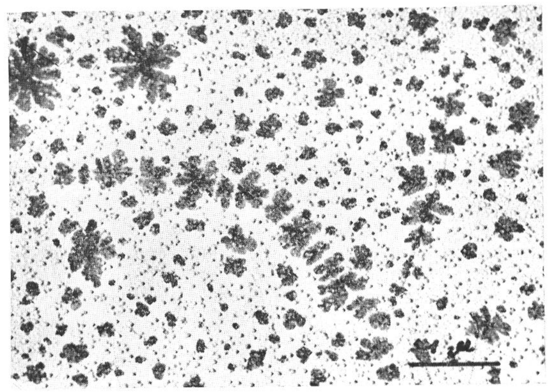

Fig. 4. Crystal growth of MoS_2 due to the sputtered atoms.

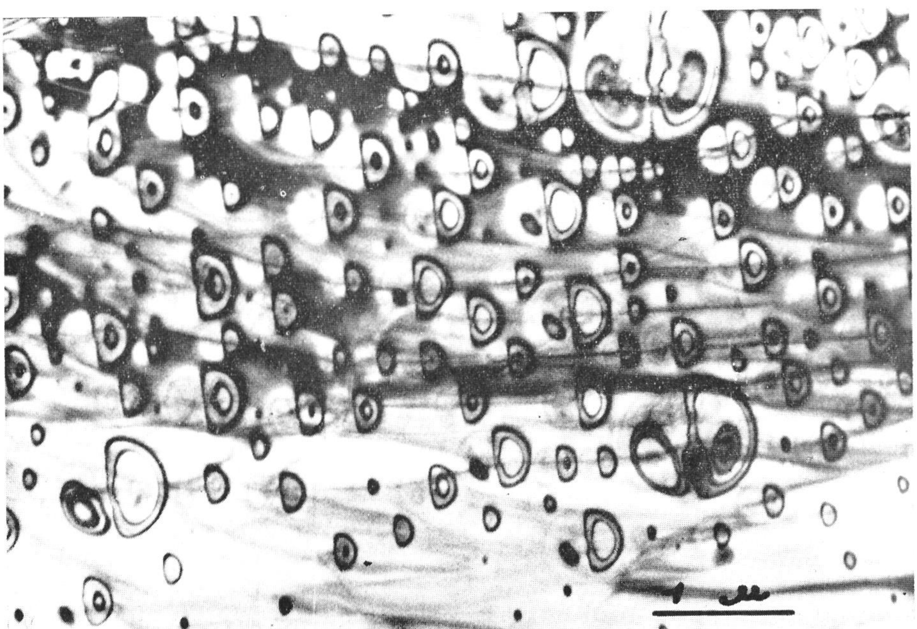

Fig. 5. Black and white spots and pair of loops induced by ion bombardment for MoS_2.

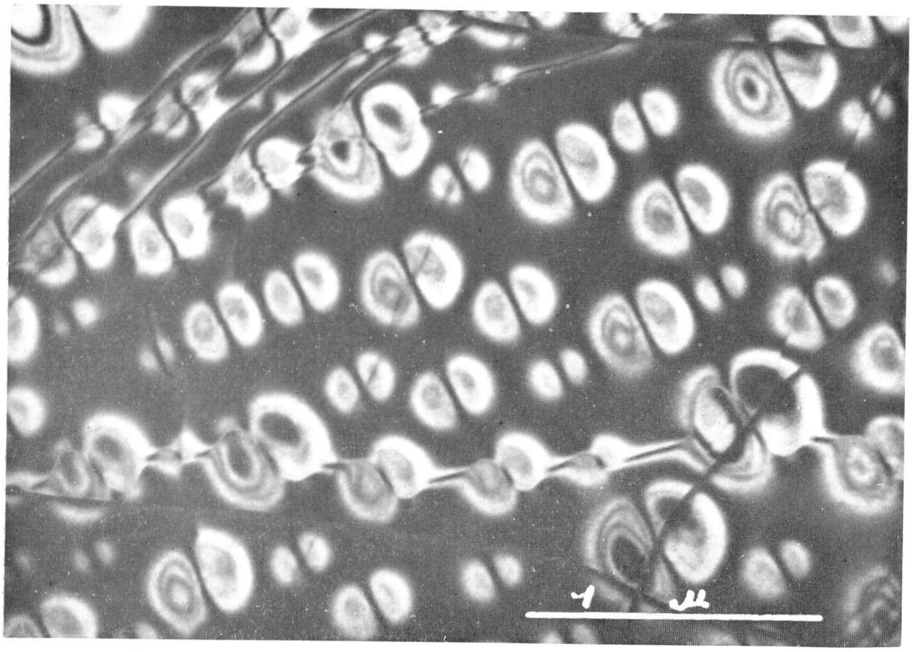

Fig. 6. Pair of loops induced by ion bombardment for MoS_2.

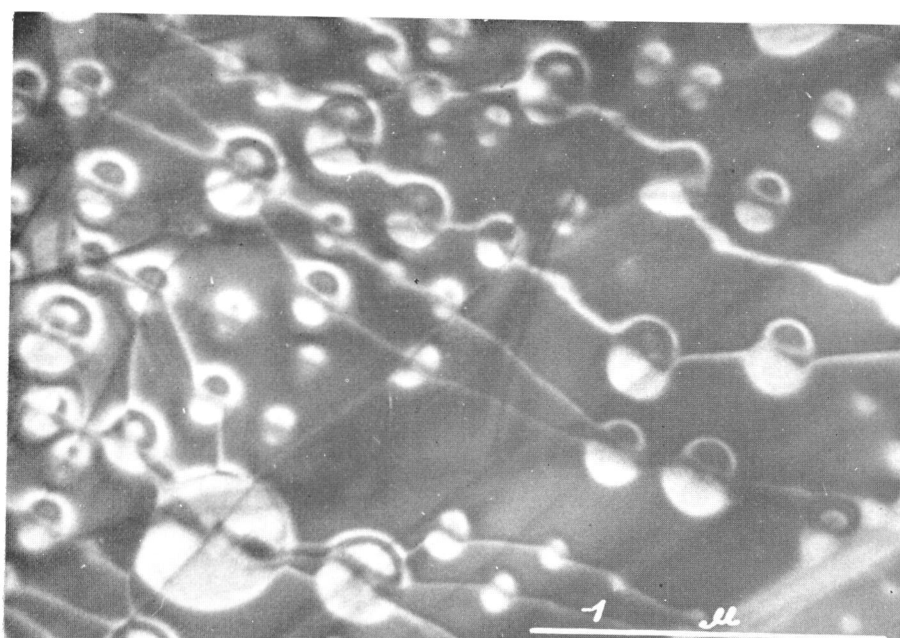

Fig. 7. Dislocation lines are pinned up to the positions of pairs of loops.

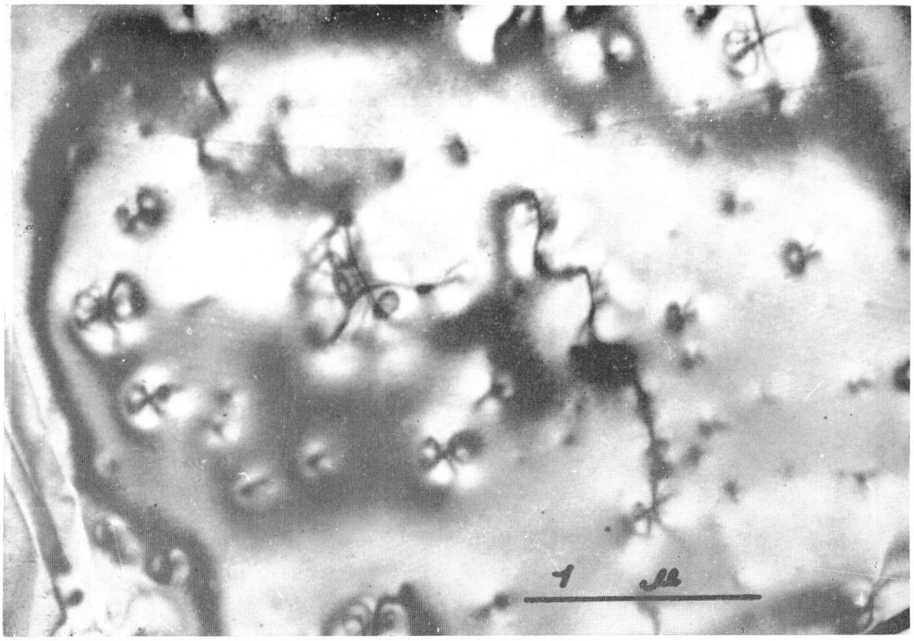

Fig. 8. Tangling of dislocation lines at the positions of pairs of loops.

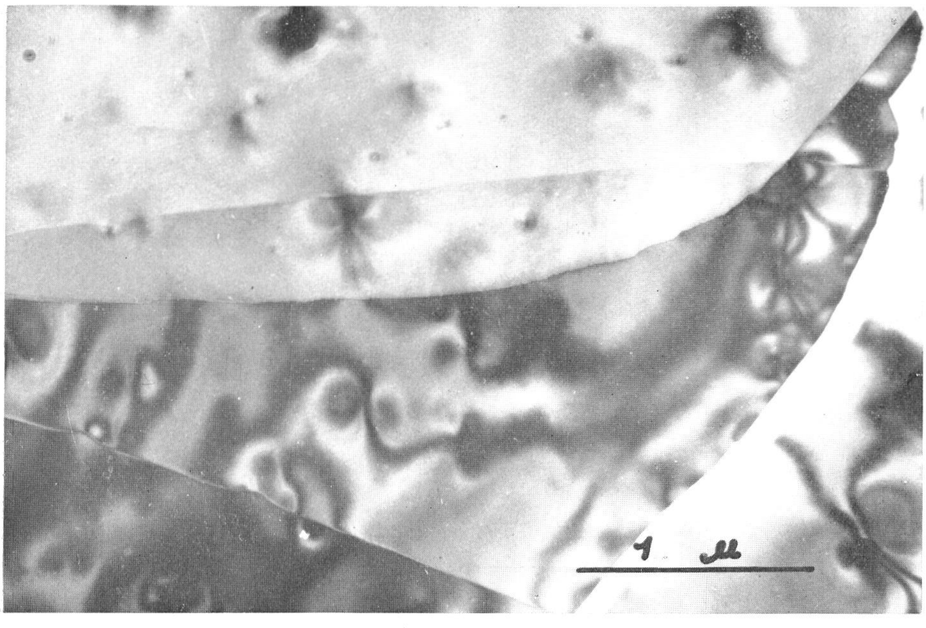

Fig. 9. Pair of loops for graphite.

of dislocation lines at loops (Fig. 8) is observed, like the observation of Wilsdorf[8] for the specimen of nickel irradiated by neutrons. Interactions between loops and dislocation lines, as well as the similarity of Bollmann's observations to the present ones, suggest that the effects of ion bombardment and neutrons irradiation are very similar in physical nature. However, it must be noted here that these loops are not observed homogeneously over the whole bombarded specimen and also, in a very few cases, the loops were observed in the specimen without bombardment, although the number of loops increased definitely after bombardment. For the specimen of graphite, the effects of ion bombardment are quite similar to those of molybdenite, characterized by the formation of black and white spots and pair of loops (Fig. 9).

Similar loops were observed by Hibi *et al.*[9] in the electron microscopes of KCl which were bombarded by electrons and they interpreted the loops as voids created in the specimen. Previously, Pernoux[10], Möllenstedt[11] and Rang[12] also observed similar loop images, and interpreted them as lens-shaped voids. In these cases, however, three pairs of loops are always superposed, corresponding to three eqivalent net planes. Therefore, the present cases are rather hard to be interpreted by the same model as in the cases of Pernoux, Möllenstedt and Rang. On the physical nature of the defects, which are observed as the pair of spots or pair of loops in electron micrographs, will be discussed in the near future.

This work was carried out partly at C.N.R.S. of Bellevue, France. The author is grateful to Professor J. J. Trillat and also to Professor N. Kato for their many helpful discussions.

References

1 F. Seitz and J. S. Koehler: Solid State Physics **2** (1956) 305.
2 F. Keywell: Phys. Rev. **97** (1955) 1611.
3 E. Gillam: J. Phys. Chem. Solids **11** (1959) 55.
4 D. W. Pashley and A. E. B. Presland: Phil. Mag. **6** (1961) 1003.
5 R. Castaing and B. Jouffret: Compt. rend. **252** (1961) 2696.
6 J. J. Trillat, N. Terao, L. Tertian and H. Gervais: J. Phys. Soc. Japan **11** (1956) 456.
7 W. Bollmann: J. Appl. Phys. **32** (1961) 869.
8 H.G.F. Wilsdorf: Phys. Rev. Letters **3** (1959) 172.
9 T. Hibi and K. Yada: J. Phys. Soc. Japan **17** Suppl. **B-II** (1962) (*Proc. Inter. Conf. Magnetism and Crystallography, 1961*) p. 212.
10 E. Pernoux: Thèse de l'Universite de Paris, (1953).
11 G. Möllenstedt: Optik **10** (1953) 72.
12 O. Rang: Optik **10** (1953) 90.

DISCUSSION

Brandon, D. G.: Precisely similar contrast effects have been reported by Bowden and myself for ion-bombarded gold* and interpreted in terms of the strain field around a dislocation loop perpendicular to the foil surface. This interpretation has been confirmed by Ashby and Brown** who have shown that a comparison of the bright field and dark field images together with a knowledge of the sign of the reciprocal lattice vector giving rise to contrast can lead to an unambiguous derivation of the sign of the loops, *i.e.*, whether they are vacancy or interstitial.

* D. G. Brandon and P. B. Bowden: Phil. Mag. **6** (1961) 707.

** M. F. Ashby and M. Brown: To be published.

Amelinckx, S.: I wonder whether you have considered the possibility that the contrast effects you observed are due to lenticular cavities formed on electron irradiation in the microscope? This takes place, for example, in mica.

Mihama, K.: As to the origin of loop-images, we have not yet any adequate model, but I think there is some difficulty in interpreting them as the lens-shaped cavities.