JOURNAL OF MATERIALS SCIENCE 9 (1974) 1262–1264

Dislocation etching in molybdenite

M. K. AGARWAL, BABU JOSEPH
Department of Physics, Sardar Patel University, Vallabh Vidyanagar, 388120, Gujarat State, India

An etchant to reveal the non-basal dislocations on the (0001) faces of MoS_2 single crystals has been established. Evidence concerning the ability of this etchant to reveal the sites of non-basal screw dislocations is also given.

1. Introduction

Previous work using transmission electron microscopy with thin cleaved sheets of MoS_2 [1, 2] have revealed the presence of basal dislocations. It is believed that both basal dislocations [3, 4] and non-basal dislocations [5, 6] can have a significant effect on the electrical resistivity, photoconductivity and thermal conductivity of the material. As far as it is known, these dislocations have not been successfully revealed by simple etching techniques, although non-basal dislocations have been studied by an oxidation technique [7]. The purpose of this short paper is, therefore, to present evidence for the successful etching of MoS_2 using a sodium peroxide/potassium nitrate melt.

2. Experimental

Natural single crystals of MoS_2 used in this work were supplied by Dr Uyeda of Japan. They were cleaved either with a sharp razor blade or with an adhesive tape when thin specimens were needed.

It is reported that different oxidizing melts like sodium peroxide, sodium nitrate, potassium nitrate etc, reliably etch the dislocation sites on (0001) planes of the crystals like graphite [8], silicon carbide [9] etc. Attempts to etch the (0001) faces of MoS_2 with different oxidizing melts were made and it was found that only sodium peroxide gave encouraging results. In this case, although the crystals were etched the crystal surfaces became very rugged. However, a melt of equal proportions of sodium peroxide and potassium nitrate, in a platinum crucible at 400°C produced well defined dislocation etch pits on (0001) faces. The crystals after etching were washed in conc. HNO_3, distilled water and then finally in acetone. The etched surfaces were vacuum coated with silver to enhance the contrast for optical examination.

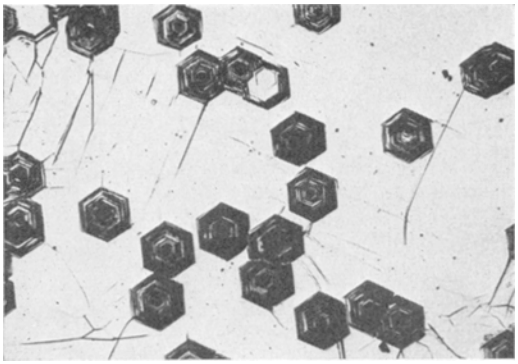

Figure 1 Typical etch pattern produced by etching in a melt of sodium peroxide and potassium nitrate. × 150.

3. Results

Fig. 1 represents the etch pattern produced on a freshly cleaved (0001) plane of MoS_2, when etched for 45 sec. It should be noted that:

1. the pits are hexagonal, the majority of them being terraced and point bottomed;
2. a number of pits are symmetrical, showing that the dislocations are parallel to the c-axis, the asymmetric pits suggest that the dislocations are at an angle to the c-axis;
3. All the pits are nearly the same size.

With prolonged etching of an already etched (0001) face, the pits become larger (Fig. 2); with some becoming flat bottomed, and some becoming point bottomed. The mechanism of formation of these flat bottomed and new point bottomed pits appears to be the same as reported by earlier workers [10].

The fact that the etch produces well defined pyramidal pits, which retain their shape while

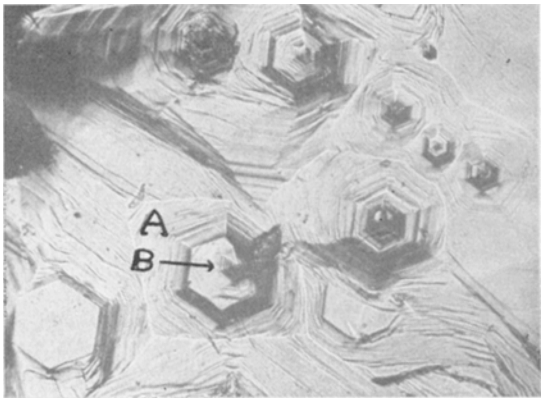

Figure 2 Etch pattern produced by prolonged etching of a cleavage face. × 300.

growing larger on further etching, strongly suggests that these pits are synonymous with the sites of non-basal edge dislocations, which are either parallel or slightly inclined to the *c*-axis. In order to ascertain whether or not this etchant reliably reveals the sites of non-basal dislocations, the following experiments were carried out (i) etching of matched cleavage faces (ii) etching of a thin crystal flake. Fig. 3a and b represent the etch patterns on a matched pair of faces, in which it can be seen that almost all the pits have a one-to-one correspondence. Similar results were obtained by etching both sides of a very thin cleavage flake.

It has been shown by Amelinckx and Votava [11] and Gilman [12] that when a cleavage crack

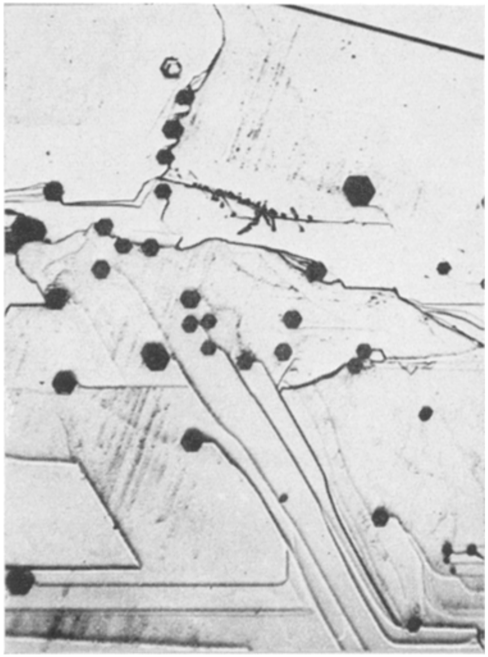

Figure 4 Etch pits developed at the ends of cleavage lines. × 100.

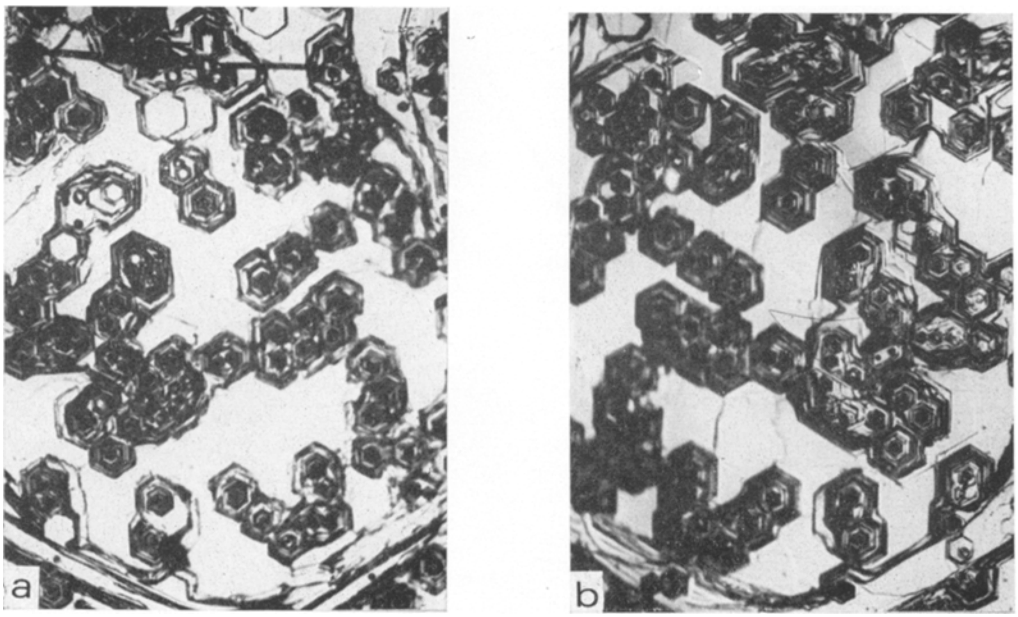

Figure 3 (a) and (b) Etch patterns on matched cleavages. × 200.

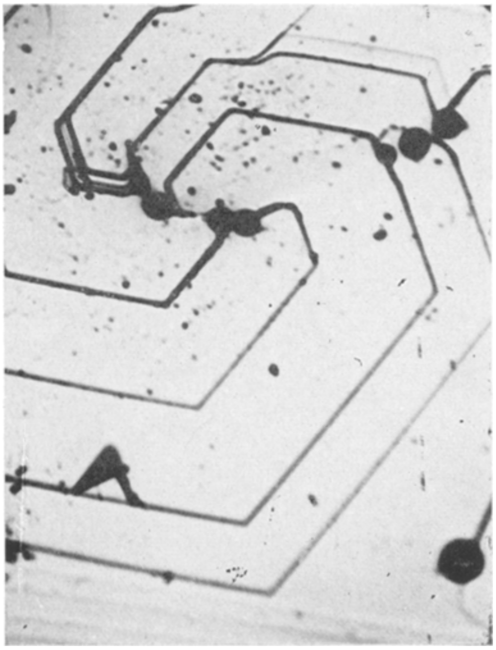

Figure 5 Etch pits developed at the apex of growth spirals. × 150.

propagates in the basal plane and intersects a screw dislocation it creates a jog in the crack-front, and with the further advancement of the crack, the jog leaves behind a cleavage step. The termination of such a step should, therefore, indicate the site of the core of a screw dislocation. If such a crystal face containing cleavage steps is etched, pits should invariably develop at the end of these steps, thus revealing the core of the screw dislocations. Fig. 4 confirms this fact, where the development of etch pits at the end of the cleavage lines can be clearly seen.

In order to further confirm that the above etchant reliably reveals the sites of screw dislocations the basal face of a synthetic MoS_2 crystal containing a number of growth spirals was etched; Fig. 5 reveals that the etch pits are invariably formed at the apex of the growth spirals, where screw dislocations are known to be present.

It may, however, be mentioned that these observations could not be confirmed on the available natural MoS_2 crystals since none of these contained growth spirals.

4. Discussion

The fact that the etch pits retain their shape on successive etching, and the excellent correspondence in the etch pattern on matched cleavage faces and on the opposite sides of a thin flake, suggests that non-basal dislocations in MoS_2 are revealed by etching the crystals in a melt of sodium peroxide and potassium nitrate.

The pit marked A in Fig. 2 becomes flat bottomed on successive etching and a new point bottomed pit, B, appears within it. This suggests the presence of a dislocation loop inside the body of the crystal. Further, the production of the etch patterns at the end of the cleavage steps and the development of the etch pits at the apex of the growth spirals shows that the etchant reported in this paper is capable of revealing the sites of non-basal screw dislocations.

Acknowledgements

The authors would like to express their gratitude to Professor A. R. Patel for his keen interest in this work. One of us (B.J.) wishes to acknowledge the financial support given by the University Grants Commission, Government of India, New Delhi.

References

1. D. W. PASHLEY and A. E. B. PRESLAND, Proceedings of the European Regional Conference on Electron Microscopy, Delft (1960).
2. S. AMELINCKX and P. DELAVIGNETTE, "Direct observation of imperfections in crystals" (edited by J. B. Newkirk and J. H. Wernick) (Interscience, New York, 1962) p. 295.
3. Z. S. BASINSKI, D. B. DOVE and E. MOOSER, *Helv. Phys. Acta* **34** (1961) 373
4. *Idem, J. Appl. Phys.* **34** (1963) 469.
5. C. ROSCOE and J. M. THOMAS, *Proc. Roy Soc.* **A297** (1967) 397.
6. J. M. THOMAS and E. L. EVANS, *Nature (London)* **214** (1967) 167.
7. O. P. BAHL, E. L. EVANS and J. M. THOMAS, *Proc. Roy. Soc.* **A306** (1968) 53.
8. O. P. BAHL, Ph.D. Thesis, Sardar Patel University (1966).
9. K. JOHN MATHAI, Ph.D. Thesis, Sardar Patel University (1970).
10. AMRIT SAGAR and J. W. FAUST, *J. Appl. Phys.* **38** (1967) 482.
11. S. AMELINCKX and E. VOTAVA, *Naturwissenschaften* **18** (1954) 422.
12. J. J. GILMAN, "Progress in Ceramic Science", Vol. 1 (edited by J. E. Burke) (Pergamon Press, New York, 1961) p. 194.

Received 14 January and accepted 22 February 1974.