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PLASTIC DEFORMATION DURING PROPAGATION OF CLEAVAGE FRACTURE IN MgO SINGLE CRYSTALS

By Yoshito AKASHI*

The cleaved surfaces of magnesium oxide are observed utilizing X-ray topography and scanning electron microscope as well as optical microscope. At room temperature fresh dislocations are hardly introduced during cleavage. Dislocations are begun to be introduced into specimen at 347 °C. Though the number of dislocations freshly introduced is increased by noticeable value at 625 °C, it is only 15 % of that of total grown-in dislocations on the cleaved surface. The region where fresh dislocations are introduced is bounded at and near cleavage steps and dislocations are hardly introduced in flat cleaved surfaces. The load applied to a crystal to start a cleavage crack is roughly constant in the range of temperature up to 660 °C.

1. Introduction

Among fundamental studies of ceramics the understanding of fracture is a main theme to be studied [1]. Refractory oxides have been studied in greatest detail, but there are little insight into the origins of fracture and detailed processes going on during cleavage of these materials. The auther's major area of interest lies in the understanding of fracture behavior of refractory oxides, especially of cleavage fracture. And the auther has been intended to understand a cleavage fracture in LiF [2),3)] and MgO [4),5)] in the light of dislocation theory, utilizing X-ray diffraction topography. This report lies on the extention of the line investigated in these studies. In this paper are reported preliminarily the observation of the cleaved surface of MgO single crystals cleaved at up to 660 °C, utilizing X-ray topography and scanning electron microscope as well as optical microscope. Distinctive features revealed in the observation will be described in two categories, cleavage at low temperature and cleavage at higher temperature.

2. Experimental Procedures

Magnesium oxide single crystals of specified spectroscopic grade purchased from Norton Chemical Company were used. Grown-in dislocation density was approximately 10^6 dislocations/cm^2 within sub-grains of about 1 mm^2 in diameter. Magnesium oxide single crystals are easily cleaved well along the {100} planes even at relatively high temperatures. The crystal blocks were

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cleaved into specimens at room temperature by a chisel with a sharp knife edge. The size of specimens used is cited in the second to fourth column of Table 1. The specimen was placed under wedge in a device for cleaving so that the edge of wedge was along a cleavage plane and the device was, as a whole, inserted into a furnace as shown in Fig. 1. The temperature of specimen was raised by the furnace and was measured by a thermocouple inserted close to the specimen. When the temperature of the specimen was raised to a setting temperature load was added onto the wedge until a cleavage crack started in the specimen. Load was measured by the load cell through cross head. Wedge made of stainless steel was used at first, but it failed to cleave out a crystal at 525 °C. Wedge made of molybdenum was then used and it failed to cleave out a crystal at 835 °C. The temperature at which each specimen was cleaved is listed in the sixth column of Table 1

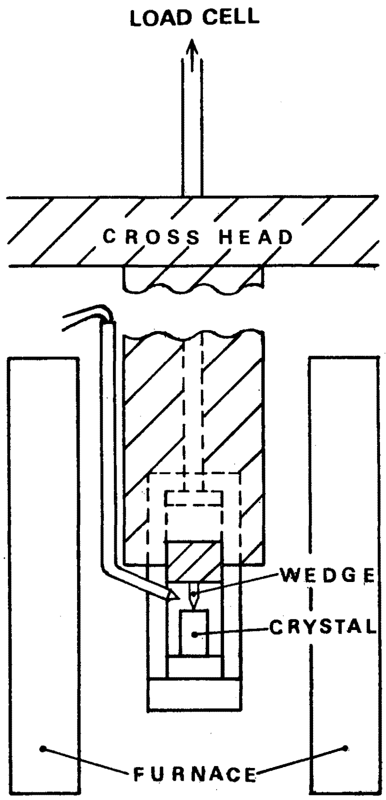

Fig. 1 Geometry for cleaving a specimen. A specimen crystal is set under a wedge. A compression is applied by traverse of cross head and the load applied is measured by load cell. Temperature of specimen is raised with furnace and thermocouple is inserted near a specimen.

Table 1 Specimen sizes are listed with condition of cleavage. On cleaving stainless steel wedge was used in specimens numbered with star and molybdenum one in specimens without star.

Speci-mens	Specimen Size (in mm)			Height/Width Ratio	Specimen Tempera-ture (in °C)	Load At Cleavage (in kg/mm)	Traverse Speed (in mm/min)
	Width	Height	Thick-ness				
1	4.5	6.3	3.9	1.4	25		50
2*	4.0	5.9	3.2	1.5	81	12.2	0.5
3	5.0	4.9	3.9	1.0	105	10.0	0.5
4	4.9	4.8	3.8	1.0	107	8.9	0.5
5*	4.5	6.7	4.1	1.5	157	10.1	0.5
6*	4.1	6.3	4.6	1.5	168		0.5
7	3.5	6.3	4.1	1.8	187	17.4	100
8*	4.0	6.5	3.7	1.6	220	7.6	0.5
9*	4.1	6.6	2.9	1.6	265	18.1	0.5
10	4.8	4.8	3.9	1.0	272	16.5	50
11	5.0	5.0	3.7	1.0	345	22.6	50
12*	4.6	6.5	4.1	1.4	347	15.7	0.5
13*	4.1	6.4	4.0	1.6	404	14.0	0.5
14	4.3	6.6	3.7	1.5	420	14.9	50
15	4.0	6.4	4.1	1.6	450		50
16*	4.2	6.6	4.0	1.6	525	NO CLEAVAGE	0.5
17	4.9	6.5	4.0	1.3	625	11.6	50
18	4.1	6.5	3.9	1.6	660	16.6	50
19	4.8	6.4	4.2	1.3	835	NO CLEAVAGE	50

and the load at which a cleavage crack started in a crystal is listed in the seventh column. In the eighth column is cited the traversing speed of the cross head.

The cleaved face of specimens was observed through optical microscope and by X-ray topography of limited projection type, of which details were described in another paper[2]. The $MoK\alpha_1$ radiation operated at 60 kV and 10 mA with effective focal size 100 $\mu m\phi$ was employed. The diffracting planes used were (200) and (020). In this experimental condition μt is about 2 where μ is linear absorption coefficient of magnesium oxide and t is thickness of specimen. Exposure time of 20 min/mm was enough with Sakura nuclear plate El (emulsion thickness 50 μm). After the X-ray observation specimens were etched to obtain etch pit patterns due to dislocations using the etchant of three kinds: (1) 1 pt. H_2SO_4, 1 pt. H_2O and 5 pt. NH_4Cl, (2) 10 % H_2SO_4 with 30 % HCl and (3) 1 pt. H_2SO_4 and 1 pt. NH_4Cl (etching time was 20～30 min at room temperature). Gold film was evaporated onto the cleaved surface for the observation with scanning electron microscope. Scanning electron microscope (SEM), Hitachi-Akashi MINI-SEM, was used to observe the regions with very dense dislocations. Optical microscopic observation was also carried out to make a whole map on specimens.

3. Results and Discussion

From characteristic features observed it is suitable for us to report two typical mode of fracture. Cleavage at low temperature and at higher temperature will be mentioned.

3.1. Cleavage at low temperature

An optical micrograph of the one of as-cleaved surfaces cleaved at room temperature is shown in Photo 1. The wedge was inserted from the upper edge of the crystal and from the cleavage steps observed it is noticed that a crack started at the left upper corner. Along the left edge in the Photo is shown a feature indicating as if this part of crystal were torn off. Micrograph of the other cleaved surface shows the pattern of Photo 1 (a) in the mirror. In Photo 2 limited projection topographs of the crystal are shown. Photo (a) and (b) are (200) and (020) topographs respectively. Slip lines started at left upper and lower corners are noticed in Photo 2 (a) as well as the region, along the left edge of the crystal, of strong intensity showing production of plastic deformation. Slip lines overlapped with the image showing elastic strain are also observed at the lower edge. In Photo 2 (b) are noticed large regions showing existence of elastic strain at the upper one third and at the lower edge.

Photo 1 (b) is an optical micrograph of the crystal taken after etching and evaporation of gold film. Slip lines, a region of plastic deformation along a side of crystal and a damage at lower edge are also noticed as well as grown-in sub-grain boundaries. Isolated dislocations are not revealed in these photographs and they are revealed employing SEM as shown in Photo 3. Photo 3 (b) is observed in the region which looks like being torn off. The surface of crystal shows no flat feature like Photo (a) and suggests that this part of crystal may be separated after a cleavage crack separated the other part of the crystal. The large step in Photo (b) correspond to a whity region near the left edge in Photo 1 (b). Photo 3 (a) is a typical micrograph of cleaved surface of this specimen. White lines in Photo is small cleavage steps and only grown-in dislocations are noticed.

When a crystal is cleaved by applying a shy blow onto a wedge and the cleaved surfaces are observed, the slip lines starting from the upper and lower edge, the damage at lower edge and the region looks like being torn off have never been observed. As typically shown in Photo 2 (b) elastic strain is introduced owing to a compression applied through wedge until a cleavage crack start. The compression may also cause the slip lines and the damage at lower edge. Characteristic feature in this category is that fresh dislocations are hardly produced during the propagation of a cleavage crack.

3.2. Cleavage at higher temperature

A typical example will be mentioned on specimen No. 18. Optical photograph of the one of as-cleaved surfaces of specimen are shown in Photo 4. In Photo 5 are shown optical micrographs of cleaved surfaces after etching and evaporation of gold. It is noticed that surface patterns in a pair of photographs are quite different. Many small flakes of the crystal are scattered over cleaved surface in Photo 4. On upper one quarter surfaces complicated patterns showing a production of large plastic deformation are appeared. An exact comparison of these surface patterns shows that concave and convex on these surfaces are in relation to complete each other, and that some parts of crystal is lost. Limited projection topographs are shown in Photo 6, where (a) and (b) show (200) and (020) topographs of the crystal shown in Photo 4. On the upper one third image showing elastic strain is observed in (200) topograph and this image is observed on the upper half in (020) topograph. The extent of this image is wider than that in Photo 2. Slip lines started at the lower edge reached to side edges in these topographs and three times longer than that in Photo 2. Many cleavage steps, especialy wide one in the central part, are observed in these topographs, though they are never observed in topographs of crystals cleaved at room temperature. This indicate that many dislocations must be introduced at cleavage steps. With the criterion for disappearance of dislocations, $\mathbf{g}\cdot\mathbf{b}=0$, where $\mathbf{g}$ is the diffraction vector used and $\mathbf{b}$ the Burgers vector, some of the dislocations at cleavage steps are characterized. Parallel lines, for example, denoted by **X** in Photo 6 (b) are edge dislocations.

In Photo 5 slip lines running obliquely are noticed as well as grown-in sub-grain boundaries. Circular patterns like a stain of water drop is caused by unfaithful evaporation of gold film and is not concerned to freshly induced defects on cleaved surface. Most of the flakes and some parts of the cleavage steps, such denoted by **A**∼**C** in Photo 4, are disappeared in these photographs. Freshly introduced dislocations are concentrated at or near cleavage steps as shown in Photo 7. Photo (a) shows a region near relatively small step. Many dislocations are cocentrated at step as expected from X-ray topographs. At a remarkably large step fresh dislocations are concentrated at and under a step as shown in Photo (b). While no fresh dislocations are observed at flat surfaces as shown in Photo (d). In Photo (c) more dominant concentration on dislocations at terrace of step is observed. The extent of these dislocation-concentrated region is so wide that they are noticed in topographs as a band-shaped image.

Characteristics in this category is that a great number of dislocations are intently introduced at or near cleavage steps though dislocations are scarcely introduced in the other region on the surface of crystal. The area where fresh dislocations are introduced is noticeably larger than that in the specimens other than No. 17 and 18.

A large cleavage step shown in a central part in Photo 5 may be produced after both sides of the step have been cleaved because of following reasons. In a part of this step parallel array of edge dislocations running perpendicular to the propagation direction of main crack is observed. As the most of fresh edge dislocations introduced during the propagation of crack are apt to line up along the propagation direction [2], it is reasonable to consider that local propagation direction of cleavage crack in this part is perpendicular to the propagation direction of main crack. Some parts adjacent to this step are missing. Main crack in this crystal may leave the part of step behind like the region of plastic deformation observed previously [4].

3. 3. Plastic deformation during cleavage

Experimental results obtained over 19 samples will be summarized. Seven specimens are omitted because of the following reasons. Specimens No. 3, 4, 10 and 11 did not contain flat cleavage plane probably because wedge is easily get out of cleavage plane caused by its shape. Specimens No. 16 and 19 were failed in cleaving. Specimen No. 7 also did not contain flat cleavage plane probably because wedge might turn towards wrong direction. The results on other specimens are listed in Table 2. The second column in the table shows the correspondence of the defect patterns on a pair of cleaved flat surfaces. The notation 'Good' means a good correspondence on a pair of cleaved surface through optical microscope and 'Disturbed' means some disagreements in pattern like a pair in Photo 5. The number of slip lines which was observed on X-ray topography is listed in third column. In the last column in the table is listed the ratio of freshly introduced dislocations to total grown-in dislocations over cleaved surface. The specimens on which precise description is made are No. 1 in section 3.1. and No. 18 in section 3.2.

Table 2 Characteristic features in observation are summarized.

Specimens	Correspondence in Surface Pattern	Slip Lines on Topograph	Fresh Dislocations/Grown -in Dislocations Ratio
1	Good	No	
2	Good	No	
5	Good	No	
6	Good	No	
8	Good	No	
9	Good	No	
12	Good	2	0.02
13	Disturbed	9	0.1
14	Disturbed	7	0.01
15	Disturbed	10	0.01
17	Disturbed		11.9
18	Disturbed	18	14.8

As the temperature at which the specimens were cleaved increase, dislocations were begun to be introduced into the specimen at 347 °C. The place where dislocations were introduced is bounded at and near cleavage steps. This is quite different, though this was predicted in a previous paper[4], from the case of LiF where dislocations are introduced forming deformation zones[6]. At 625 °C the number of dislocations freshly induced by cleavage increased abruptly. The reason why cleavage steps were observed in X-ray topographs is that many fresh dislocations, as revealed by SEM observations, were introduced at these cleavage steps. The area where fresh dislocations were introduced is remarkably wide in specimens No. 17 and 18, which were cleaved at 625 °C and 660 °C, comparing with other specimens. Though even in these specimens the number of fresh dislocations is only 12~15 % of that of grown-in dislocations on a cleaved surface. The load which was applied to a crystal through wedge when a cleavage crack started, is roughly constant in the range of temperature up to 660° C as noticed in the seventh column of Table 1.

Acknowledgments

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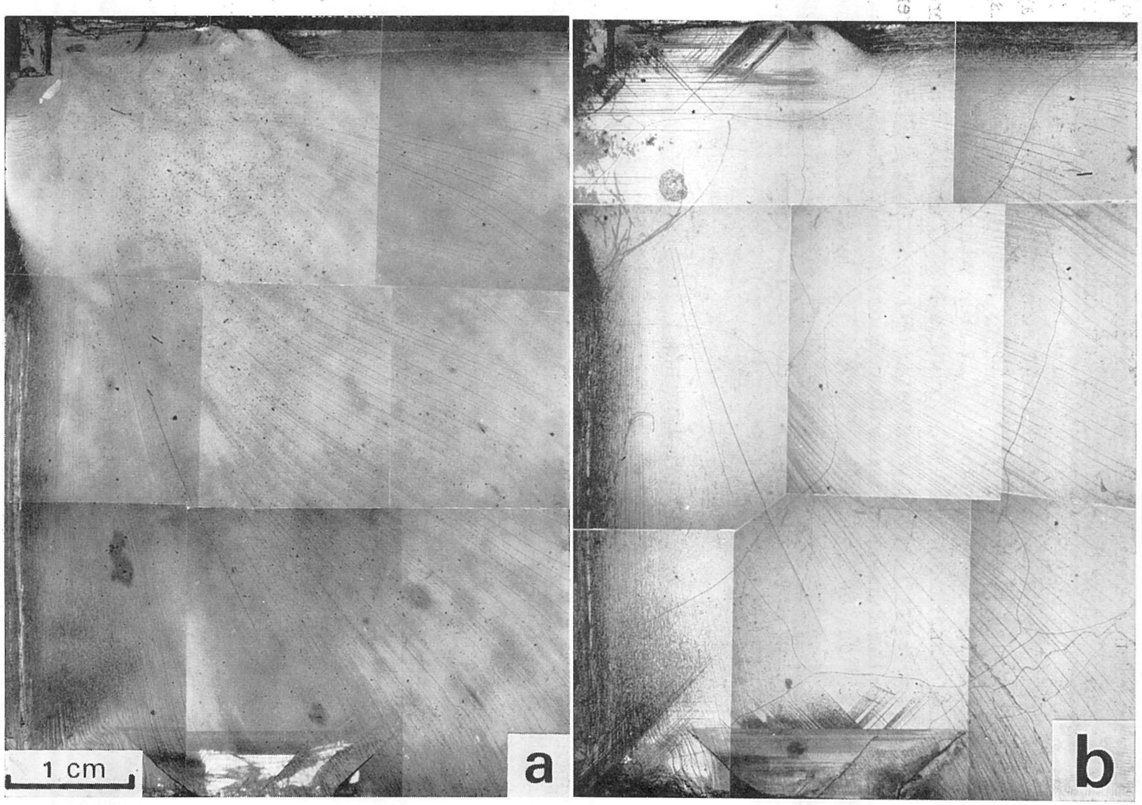

Photo 1 An optical micrograph of as-cleaved surfaces (a), and an optical micrograph of etched and evaporated surfaces (b). Specimen (No. 1) was cleaved at room temperature.

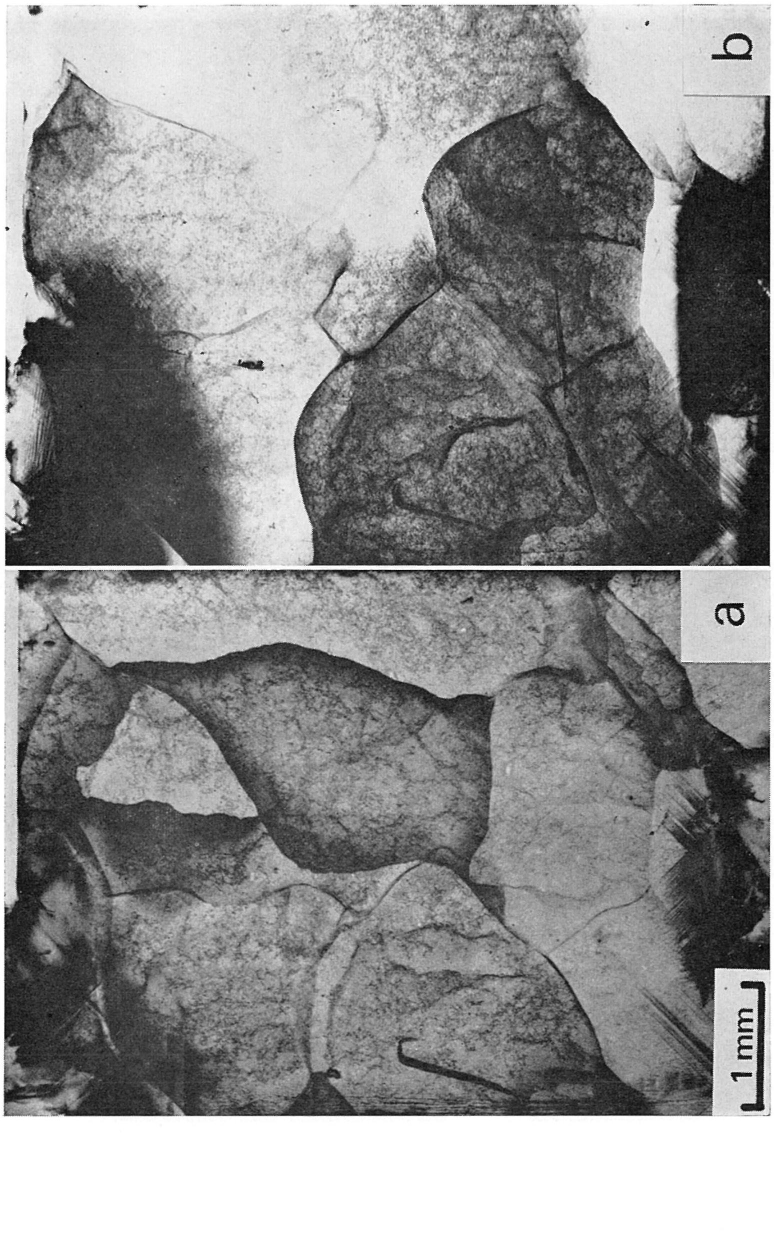

Photo 2 Limited projection topographs of specimen (No. 1).
(a) is (200) topograph and (b) is (020) topograph.

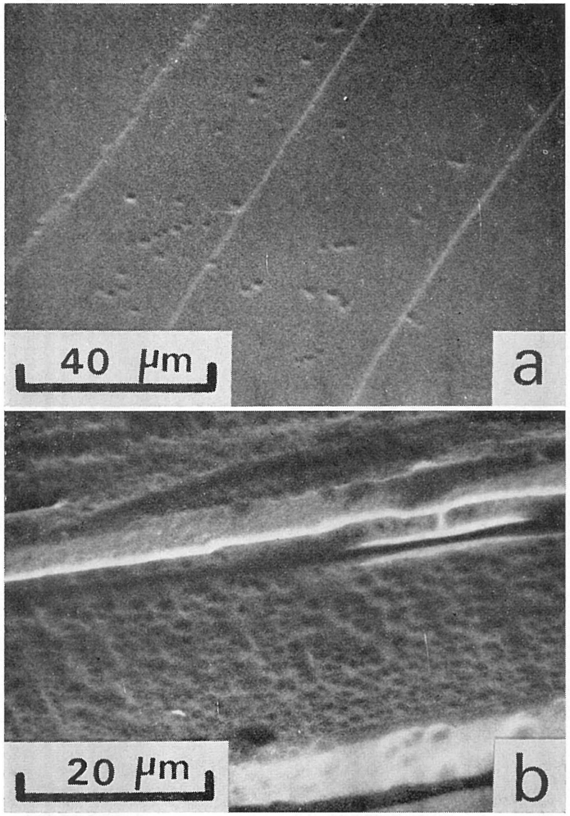

Photo 3 Scanning electron micrographs of specimen No. 1.

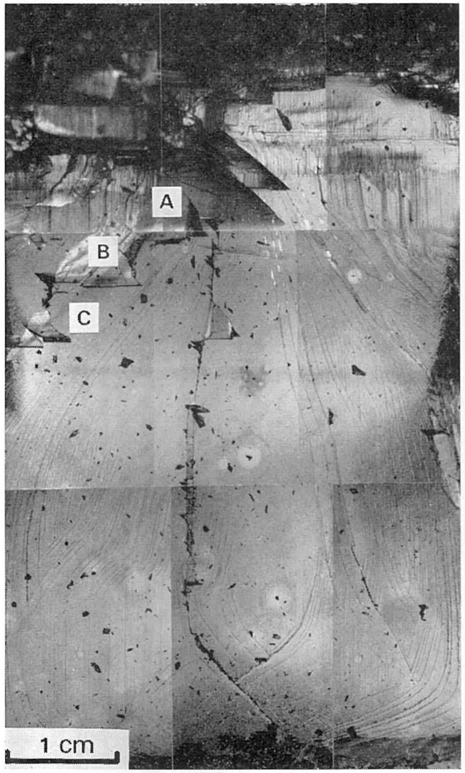

Photo 4 An optical micrograph of as-cleaved surfaces. Specimen (No. 18) was cleaved at 660 ℃.

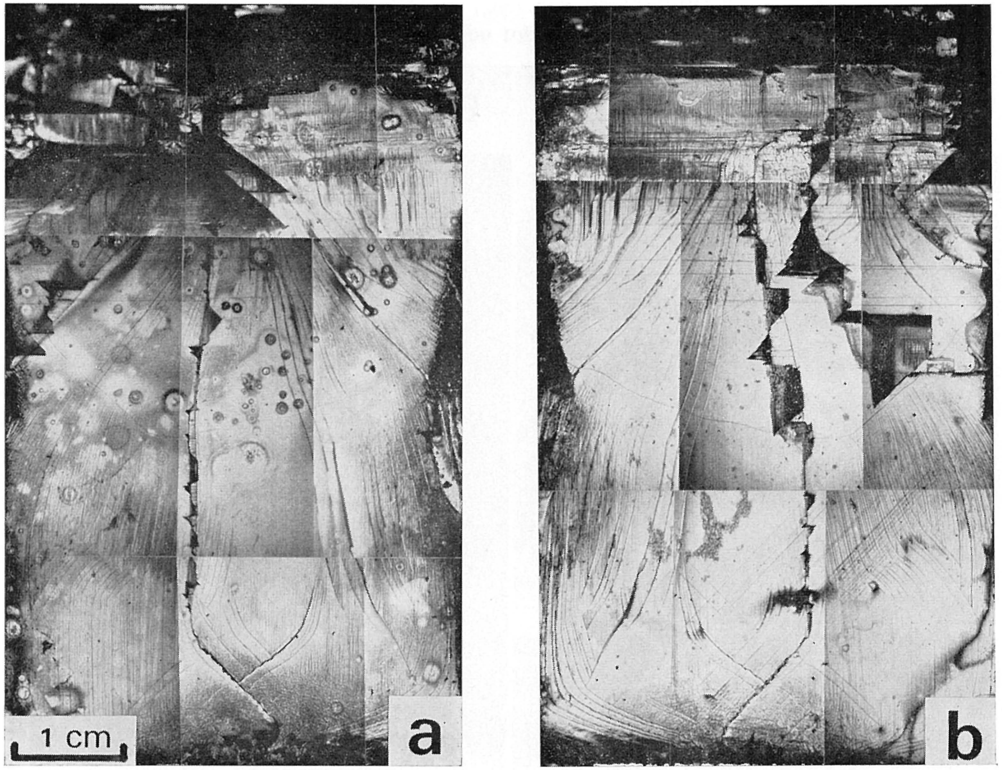

Photo 5 A pair of optical micrograph of etched and evaporated surfaces (No. 18).

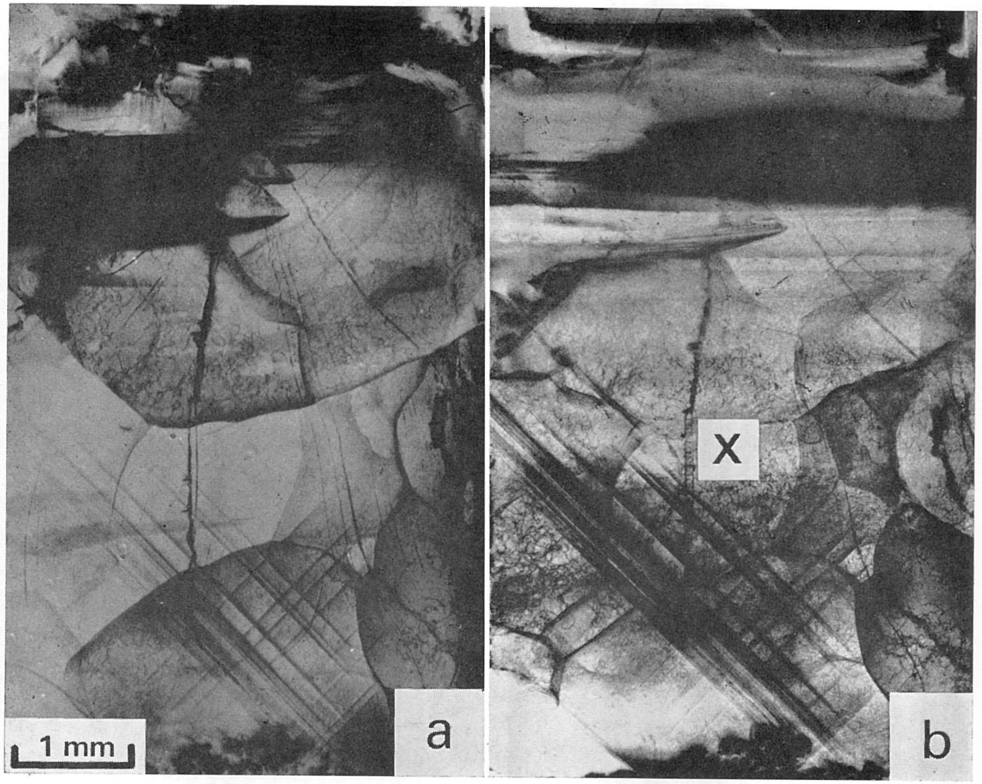

Photo 6 Limited projection topographs of specimen (No. 18).
(a) and (b) are (200) and (020) topograph.

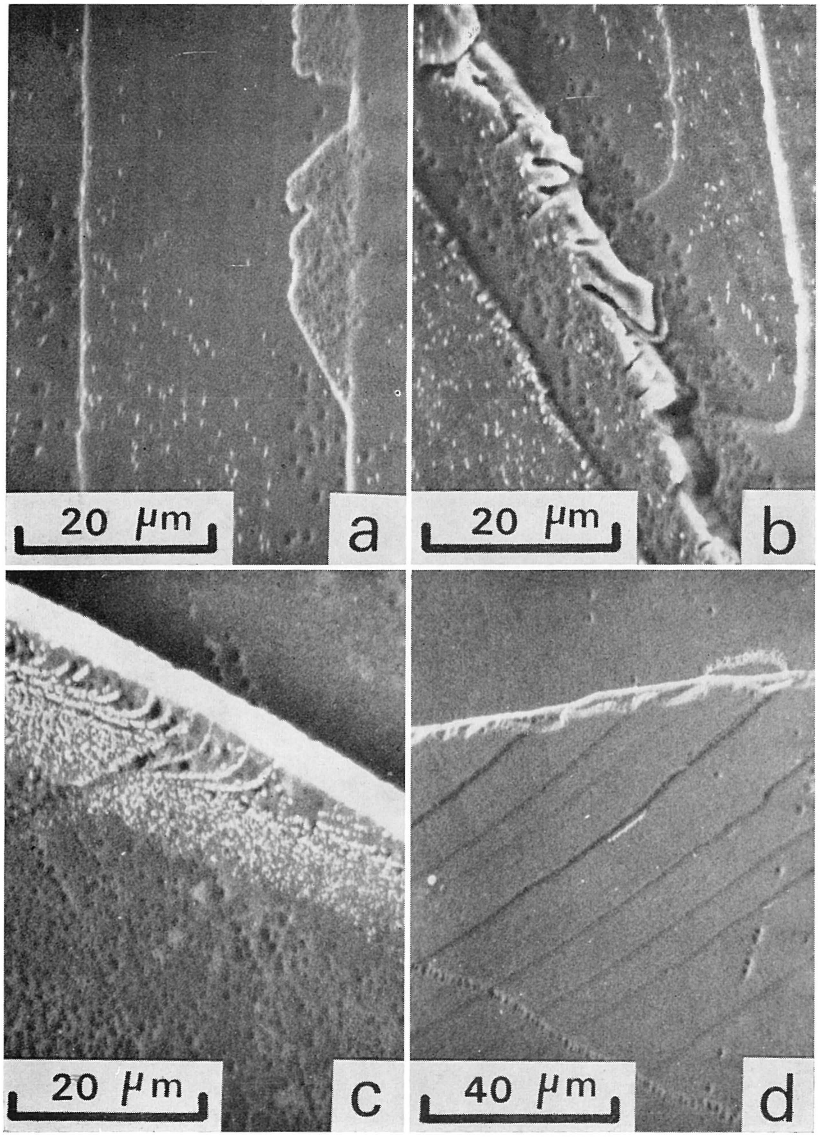

Photo 7 Scanning electron micrographs of specimen No. 18.