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Study of the Initial Stages of Wear by Electron Channeling Microscopy

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U. S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary
NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

Abstract

Studies of the initial stages of sliding wear are in progress. Both lubricated and unlubricated conditions will be involved, at low sliding speeds. Abrasive particles of various hardness values and sizes will be introduced into the bearing system. Selected area electron channeling patterns are being obtained from the vicinity of the wear track enabling direct determinations of the strains introduced during the wear process. Iron single and polycrystalline specimens are being studied. Wear debris particles are being collected and examined in order to determine the relationships between particle characteristics and the wear processes that occur.

1. Introduction

One of the principal causes of material degradation and device failure is wear. Most stressed, lubricated, bearing contacts are designed to accommodate a minimum amount of normal wear, depending on the materials of construction and the application. However, severe or abnormal wear will occur sometimes and can lead to unanticipated, sudden failures. Abnormal wear can involve fatigue cracking, corrosion, foreign particle abrasion, and other physical processes [1-3]. In high performance equipment, e.g., aircraft turbine engines, sudden failure of a single component by abnormal wear does occur and can lead to larger scale disaster.

There is a need to develop techniques for monitoring the wear taking place in mechanical systems, in order to anticipate sudden failure and avoid it by remedial actions. One technique currently being explored for wear monitoring involves the collection and examination of wear particle debris [4]. The wear debris can be obtained from the lubricating fluids of the mechanical system without disturbing the operating cycle. It is likely that some information on the modes of wear taking place can be obtained from examination of wear debris. The onset of abnormal or disasterous wear modes may be determined very early in the development of those processes. In order to conduct such investigations, it is essential to gain a background of information on the various mechanisms of wear, the type and rate of wear debris formation, and any other characteristics associated with different wear modes that may prove to be useful indicators. It is important to conduct such background studies on well characterized and controlled systems so that complicated, mixed-mode wearing conditions are avoided [5].

This project is concerned with the initial stages of contact wear, involving both lubricated and nonlubricated systems under low-load conditions [6]. Wear will be studied under noncontaminated conditions, involving, however, normal oxide films on the bearing surfaces, and also under conditions where abrasive particles are present in the bearing region. The abrasives will range from soft to hard, relative to the metal surfaces, and will be carefully controlled in size and density. The new technique of selected area electron channeling pattern (SACP) generation will be used to monitor the developing strains in the vicinity of the wear track [7,8]. This technique involves the channeling of incident electrons parallel to dense crystal planes to various depths in a bulk specimen. Detection of backscattered or secondary emitted electrons in the scanning electron microscope (SEM) that channel back out to the specimen surface will provide information on the strain distribution and lattice orientation in the region under study. Lateral dimensions of the region studied are as small as 10 μm and the depth can vary from 10^{-3} to 10^{-1} μm. Strains introduced into the specimen as a result of wear and plastic deformation will disturb the interplanar spacing and introduce losses into the electron channeling process. Since the SACP can be obtained in a matter of a few seconds of observation in the SEM, it is possible to monitor both the topographic, surface features as they develop during wear and the growth of deformation strains. This interim report will describe the initial results obtained on the project involving studies of iron surfaces after brief wear exposures and the determination of SACP's at various amounts of deformation.

2. Experimental

A constant velocity, sliding wear tester was constructed for use on this project. A photograph of the tester and associated equipment is shown in Fig. 1a. A strain gauge amplifier and chart recorder are used to monitor the frictional forces at the specimen surface-wear pin interface during testing. Details of the tester are shown in Fig. 1b. A constant velocity, motor driven cam causes a reciprocating velocity of 2.0 cm/sec. in a dovetail slide on which a sheet specimen is fixed. The linear motion can extend up to 2 cm in length. A pin bears on the specimen surface from above with a load determined by a dead-weight loading system. Current tests involve loads between 50g and 300g. The loading pin is fastened to an elastic beam strip containing two mounted resistance strain gauges. The driven displacement of the sheet specimen causes a frictional force on the pin and hence on the beam strain gauges, whose response can be calibrated directly in terms of force. Thus the result of changing conditions during wear testing, e.g., the addition of abrasive particles to the lubricating oit, can be followed in detail with each sliding cycle. Since a small number of cycle tests are being conducted at present, the lubricating fluid is simply placed on the sheet specimen surface initially.

The shape of the end of the loading pin used so far has been either hemispherical or flat with slightly rounded edges. Small bearing balls have been used in some tests and were affixed to the pin by cementing into a conical depression. The sheet specimens are generally also cemented to the specimen platform. With the present motor speed of 23 RPM, tests from 1 cycle to 1,000 cycles duration can be conveniently conducted.

The sheet specimens and pin end surfaces are examined after testing in a commercial SEM, usually at 20 kV electron beam energies. The oil and wear debris are recovered from the sheet surface by washing prior to specimen examination. The system is designed so that tests can be interrupted for specimen examination and subsequently continued to completion. Channeling patterns are obtained in the rocking beam mode from the specimen oriented at normal electron beam incidence and operated in the emissive image mode. The minimum selected area realizable for our equipment is about 10 μm across at a working distance of about 3 mm. The angular extent of the SACP is about 18 deg. under these conditions.

3. Results and Discussion

A. Iron Single Crystal Surface

The initial studies were conducted on an iron single crystal. The crystal had been grown previously by the strain anneal method, then carefully cut and polished to provide a flat surface of about 1 cm^2 area near the (113) orientation. The specimen purity is about 99.9% Fe. Prior to testing, the surface was electropolished in a solution of 6% perchloric acid in acetic acid for about 30 minutes at 20 volts across the cell. A micrograph showing the initial smooth but wavy surface appearance is given in Fig. 2a. An enlarged SACP from this surface is shown in Fig. 2b. Considerable fine structure is present in this pattern, arising from high index channeling bands, indicating a high degree of crystal perfection.

The crystal surface was next lightly scratched in one area with a steel point. That area is shown in Fig. 3a. A typical cutting wear track was formed with considerable material displacement produced into lips adjacent to the track center. Large plastic strains would be involved in such deformation and large residual strains would be expected [9,10]. SACP's taken from the area of the scratches showed considerable pattern broadening associated with large residual strains. SACP's obtained from areas adjacent to the cutting scratches showed little evidence of broadening.

A second experiment was conducted on this crystal surface involving three sliding wear strokes taken under light ($\approx$ 25g) load, oil lubricated, with abrasive alumina particles about 1 μm diameter present. Figure 3b shows some details associated with these wear tracks. The deeper tracks extending across the field of view appear to involve small lips of material much like the larger cutting scratches above. Many of the alumina induced scratches end within this field by becoming increasingly shallow and narrow as though the load carried by the particle involved was decreasing to zero. This may indicate that the clearance between the bearing pin and surface increased locally due to surface flatness variations which could easily exceed 1 μm (40 μ-in) peak-to-peak in this sample.

A lower magnification view of this wear track is seen in Fig. 4a. The track edge appears at the right, region 1 and the track center at region 5. The amount of abrasion increases toward the track center. SACP's taken from the numbered regions are shown in Fig. 5. The loss of pattern definition and channeling line sharpness is clear as one probes into the wear track. Variations of strain in the track are also found as indicated by the pattern obtained from region 4. A further comparison of regions 1 and 3 is shown in Fig. 6. Here the SACP's are enlarged by scanning a smaller angular interval. Further, the detected electron signal along one line trace through the pattern is also shown. The increased signal contrast present in the SACP from the unworn region 1 is seen in the trace of Fig. 6a. Quantitative measurement of signal contrast is possible using this line scan technique. Calibration of the SACP signal contrast in terms of strain in the specimen through the use of predeformed specimens would permit a direct determination of local strain, the principal thrust of this project. Finally, note the details at the ends of several of the deeper wear tracks in Fig. 4b. It appears that alumina particles have suddenly ceased the abrasion action, possibly due to fragmentation or exiting from the edge of the slider pin. Some alumina particle can be seen in this field of view.

B. <u>Polycrystalline Iron Calibration Specimen</u>

An iron rod, 3 mm in diameter, purity 99.998% Fe, was cut to a length 20.3 mm. Two flat surfaces were then ground on opposite sides of the cylinder, one parallel to the rod axis, the other inclined at about 7° to the axis. This resulted in a tapered flat specimen, 1.3 mm thick at one end and 1.7 mm thick at the other end. The specimen was carefully electropolished, sealed into an evacuated capsule, annealed for 2 hours at 940°C, cooled and reexamined.

High quality SACP's were obtained from grains along the length of the specimen and indicated that the electropolishing and annealing treatments had left a low residual strain level. The tapered specimen was then deformed in compression between two teflon plates to a uniform thickness of 1.3 mm. Hence, one end of the specimen was reduced 0.4 mm in thickness while the opposite end remained undeformed. The distribution of calculated strain along the bar length is shown below.

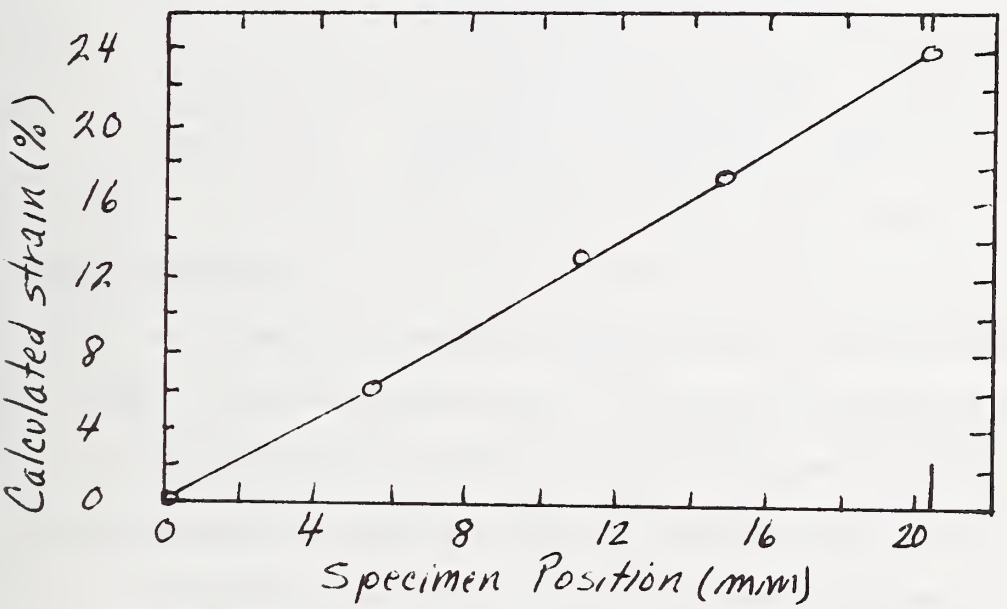

Figure 7 shows the grain structures observed at either end of the specimen and Fig. 8 shows two SACP's obtained from grains at the low strain end and the middle region of the bar. The analysis of patterns obtained from randomly selected grains along this strain gradient has not yet been completed. However, it is clear so far that regions containing strains up to about 18% produce SACP's on which measurements can be conducted. Patterns from the high strain end of the bar (ε = 24%) could not be obtained and hence represent the limit to which the SACP technique can be extended in high purity iron. The extensive plastic flow that has taken place at the high strain end of the bar is suggested by the observed grain structures (Fig. 7b).

C. Polycrystalline Iron Wear Studies

Studies have begun of the initial stages of wear on polycrystalline iron surfaces using the sliding wear tester. Experiments conducted so far have involved carefully prepared, electropolished surfaces on 99.998%Fe sheets, under clean oil-lubricated conditions and also under dry conditions. One particular test will be described here illustrating the type of experiments in progress. A steel bearing ball, 2.4 mm diameter, was loaded with 100g, bearing on the iron sheet specimen. Figure 9 indicates the worn contact area on the ball at low and high magnification. Several scratch patterns are seen on the flat portion of the sphere surface due to some interruptions of this test and reorientation of the pin relative to the direction of motion. Comparison of the unworn surface (Fig. 9b) with the flat region indicates the surface changes that have taken place. Scratch grooves are commonly found of a width approximately 0.1 μm even though considerable care was taken to exclude abrasive material contamination (note that the lubricating oil used was not subjected to additional filtering, however).

Several areas from the wear track on the electropolished iron sheet surface are shown in Figs. 10 and 11. Details typically found at the edge of the wear track are seen in Fig. 10b. Material is plastically deformed under the slider contact, moved to the edge of that contact and presumably breaks free to contribute to the wear debris. The unworn electropolished surface is shown in the lower region of Fig. 10b. The central region of the wear track after 400 cycles (Fig. 11a) still retains areas where contact has been avoided and the electropolished surface structure remains. The end region of the wear track contains a considerable amount of debris and deformed metal as seen in Fig. 11b. Since a substantial oxide film was initially present on this specimen, it seems likely that some of the debris here may be largely iron oxide. Particles whose lateral dimensions are as small as 0.5 μm are seen in this region of the wear track.

4. Summary and Plans

A sliding wear tester has been developed that permits the initial stages of wear involving selected materials to be investigated. The device can impose loads up to 300g in a pin-plate geometry, under dry or lubricated, constant velocity conditions. The design facilitates studies of single crystal and other specially prepared surfaces. Selected area electron channeling patterns have been obtained adjacent to and across a lightly loaded abrasive wear track on an iron crystal surface. The loss of pattern definition (increase in line broadening) associated with increasing residual strains due to the wear processes was observed. Calibration experiments have been conducted that will lead to a relation between SACP characteristics and wear-induced strains. Studies of wear tracks in high purity iron specimens have begun, involving observations of the surface structures, wear debris and strains produced in the first 700 cycles. Considerable surface deformation and flow has been observed. Wear debris is readily produced from lips of material formed by surface flow. The introduction of abrasive particles (e.g., alumina) leads to the development of a considerably different surface structure. However, lips of material are still present adjacent to abrasive scratches and appear to contribute some of the wear debris.

The next phase of the project will complete the determination of the SACP-residual strain relationship. From that point, strain maps will be determined around and beneath wear tracks produced under carefully controlled conditions. Oil-lubricated load-bearing contacts will be involved, including the introduction of characterized abrasive particles of various hardness and mean size values. The role of surface oxide films in the developing wear process will be examined, including the effect of oxide film thickness. Wear debris particles will be collected during these studies in order to correlate the debris characteristics with the wear modes involved.

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Figure Captions

Fig. 1 (a) View of sliding wear tester, strain gauge amplifier and chart recorder. (b) Details of constant velocity tester; cam C, push rod P, specimen S, load L, strain gauges G.

Fig. 2 (a) Electropolished surface of iron single crystal. (b) SACP of specimen, angular range 5 deg., near (113) orientation.

Fig. 3 (a) Cluster of scratches on iron single crystal surface due to steel cutting edge. (b) Scratches on iron surface from 1 μm alumina particle abrasion.

Fig. 4 (a) Area of wear track on electropolished iron single crystal surface. Region 1 is right edge of track and region 5 is approximate center of track. (b) Details of wear scratches on surface. Note differences in track-end characteristics.

Fig. 5 SACP's from numbered regions in Fig. 4a, angular range 18 deg. Regions 1 through 5 show progressive increase in deformation due to wear.

Fig. 6 (a) SACP from undeformed region of single crystal surface, angular interval 3 deg. Upper line trace shows signal intensity across SACP at location of lower, straight line trace. (b) SACP from deformed region of crystal showing loss of signal across prominent channeling bands.

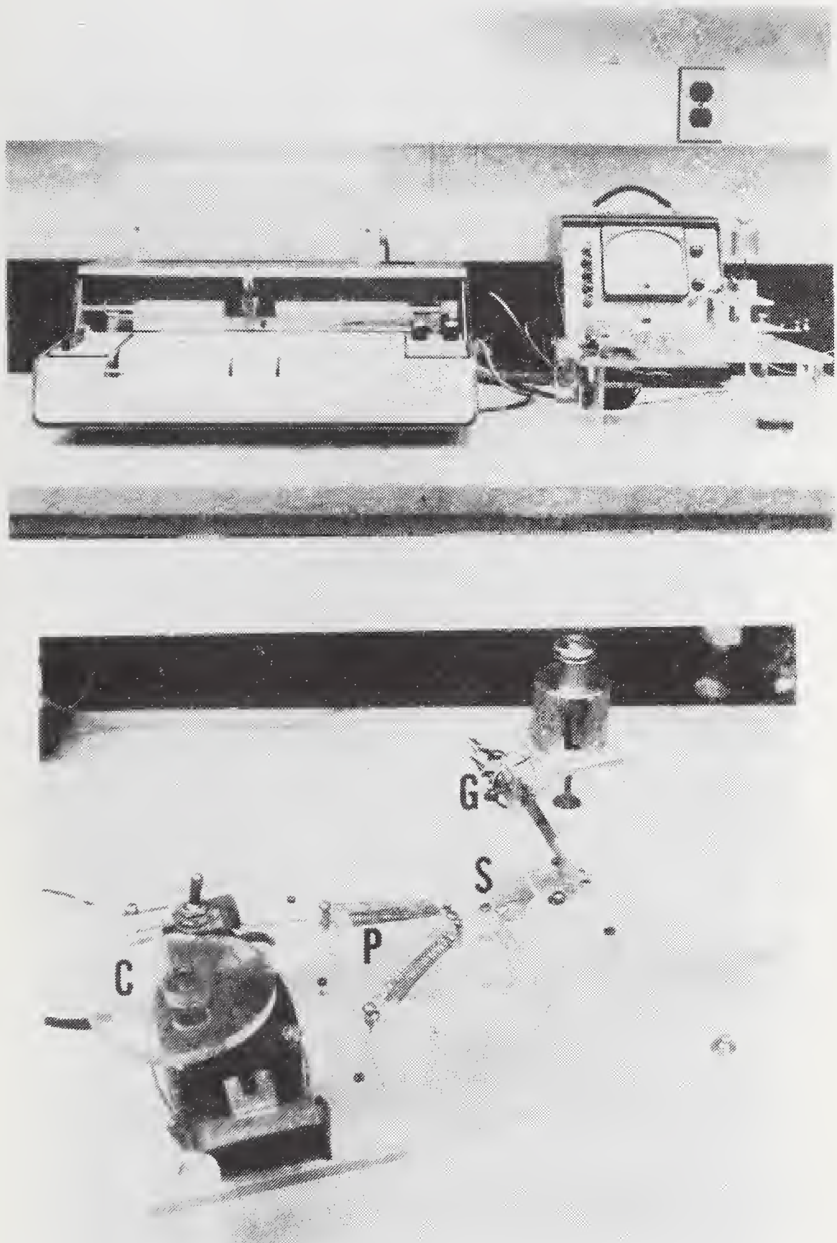
Fig. 7 (a) Polycrystalline iron bar specimen, low strain end, after deformation. (b) High strain end showing surface flow and grain structure.

Fig. 8 (a) SACP from grain at low strain (<1%) location in iron bar specimen. Note contrast at 110 band marked B. (b) SACP from another grain at midstrain (≃13%) location. Note contrast at 110 band marked.

Fig. 9 (a) Worn region on 52100 steel bearing ball after 700 cycle wear test on iron sheet specimen. (b) Detail of worn region.

Fig. 10 (a) Wear track on high purity electropolished iron sheet specimen after 400 cycles under oil. (b) Details at track edge.

Fig. 11 (a) Central region of wear track showing small unworn areas surrounded by deformed areas. (b) End of slider wear track showing displaced material (oxides?) and wear debris.



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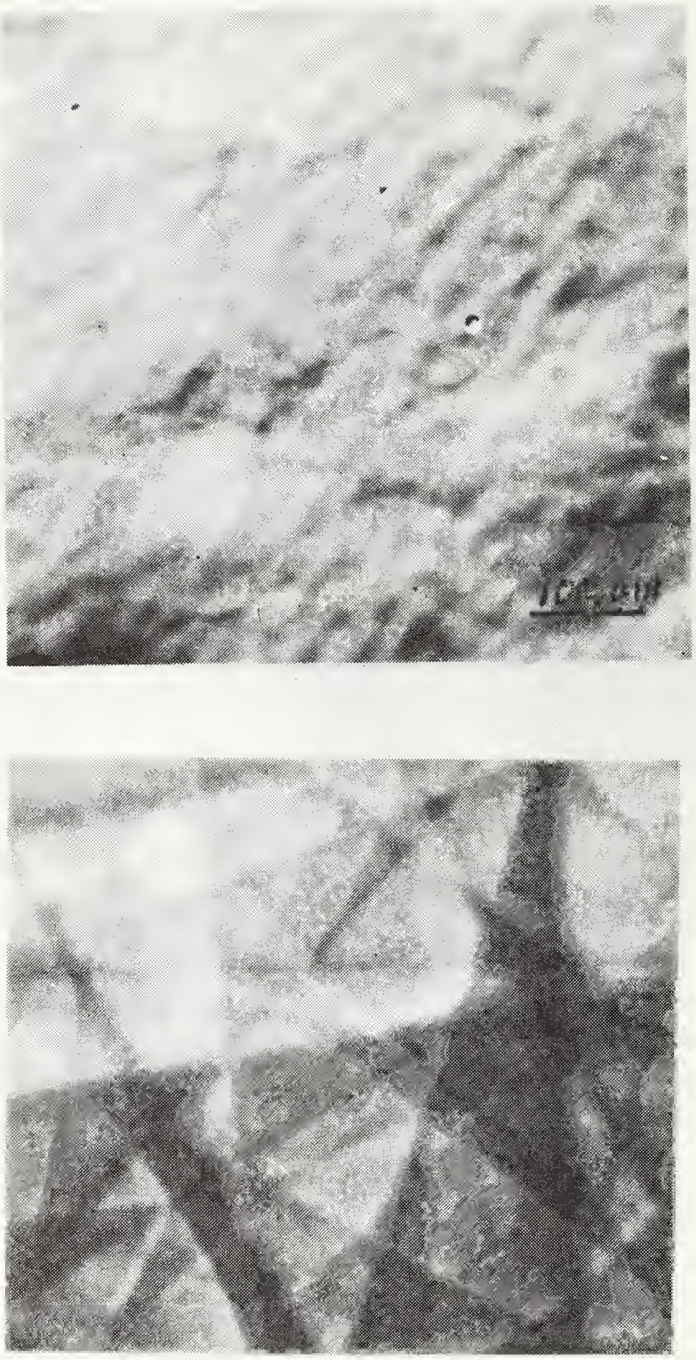

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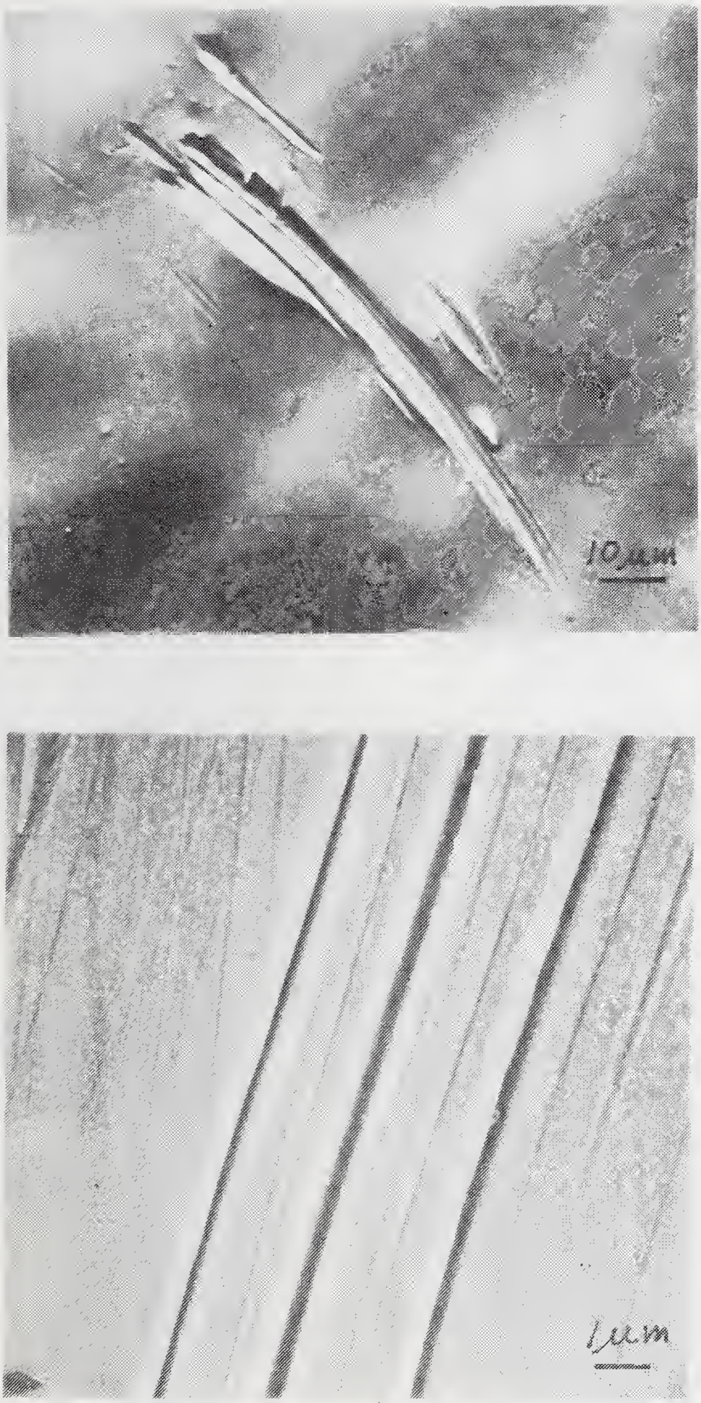

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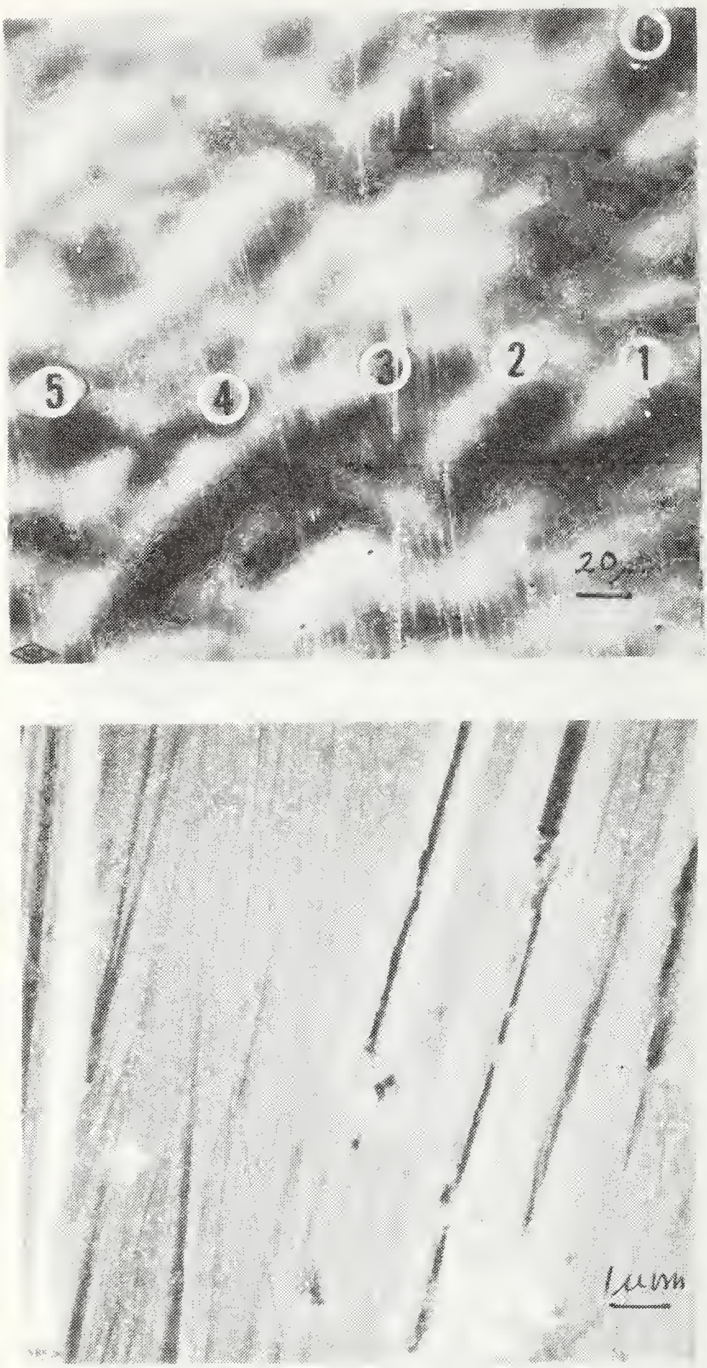

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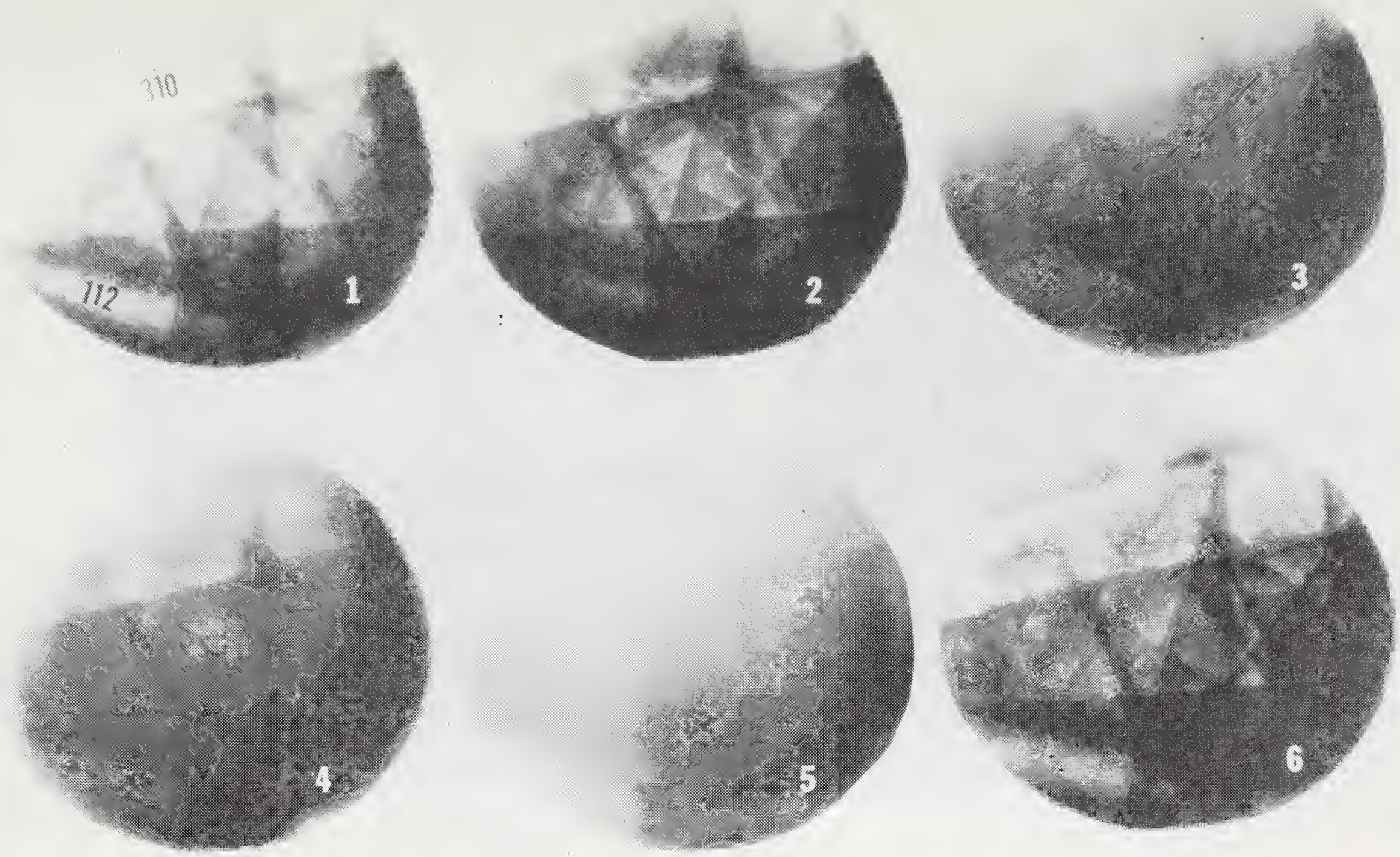

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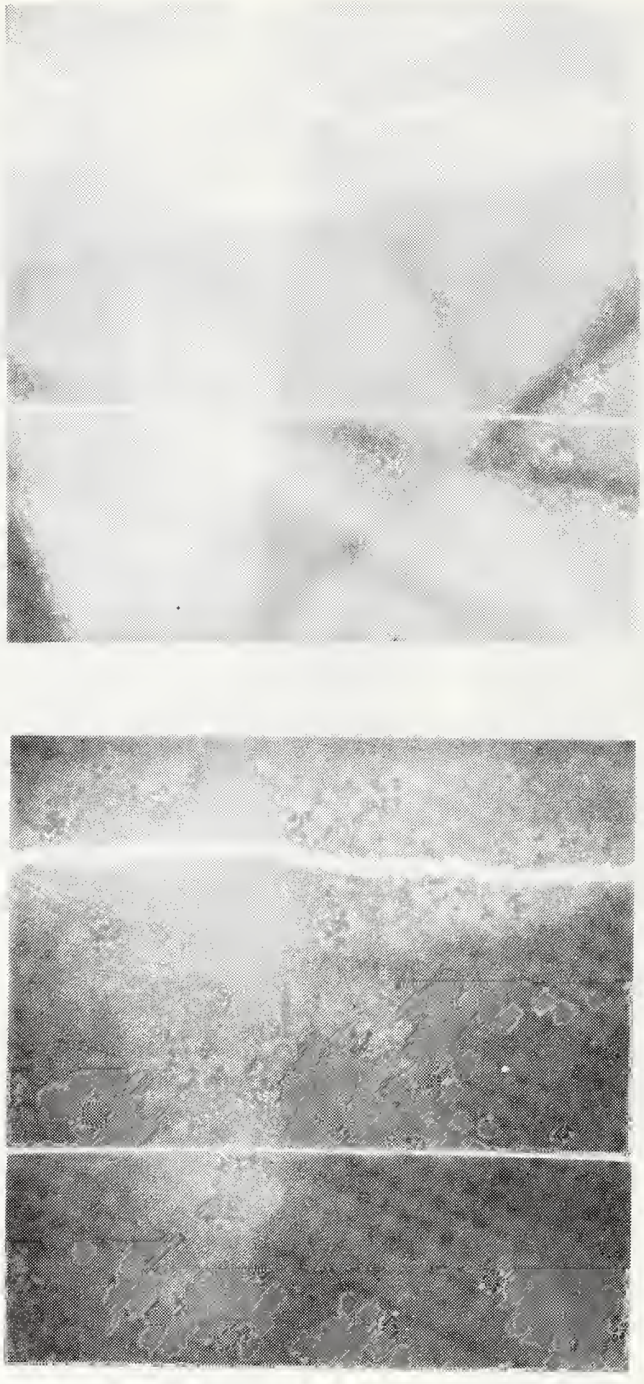

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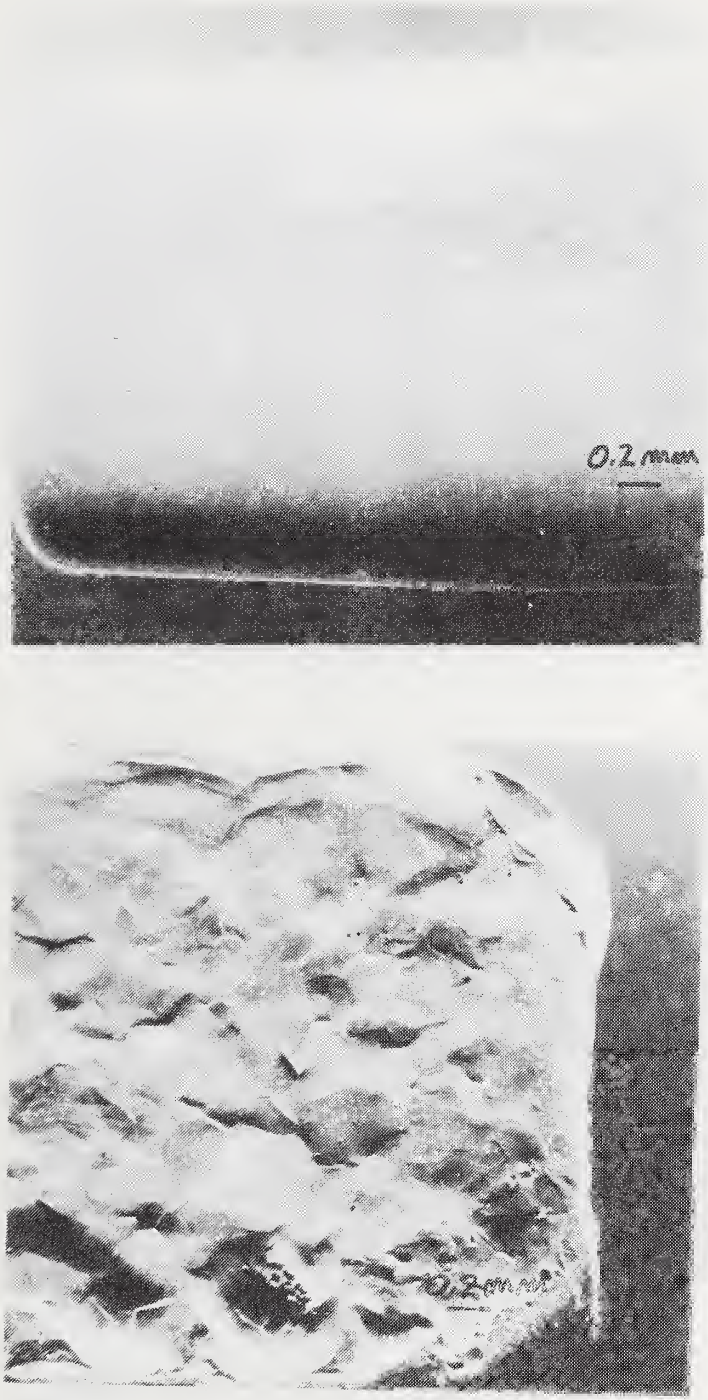

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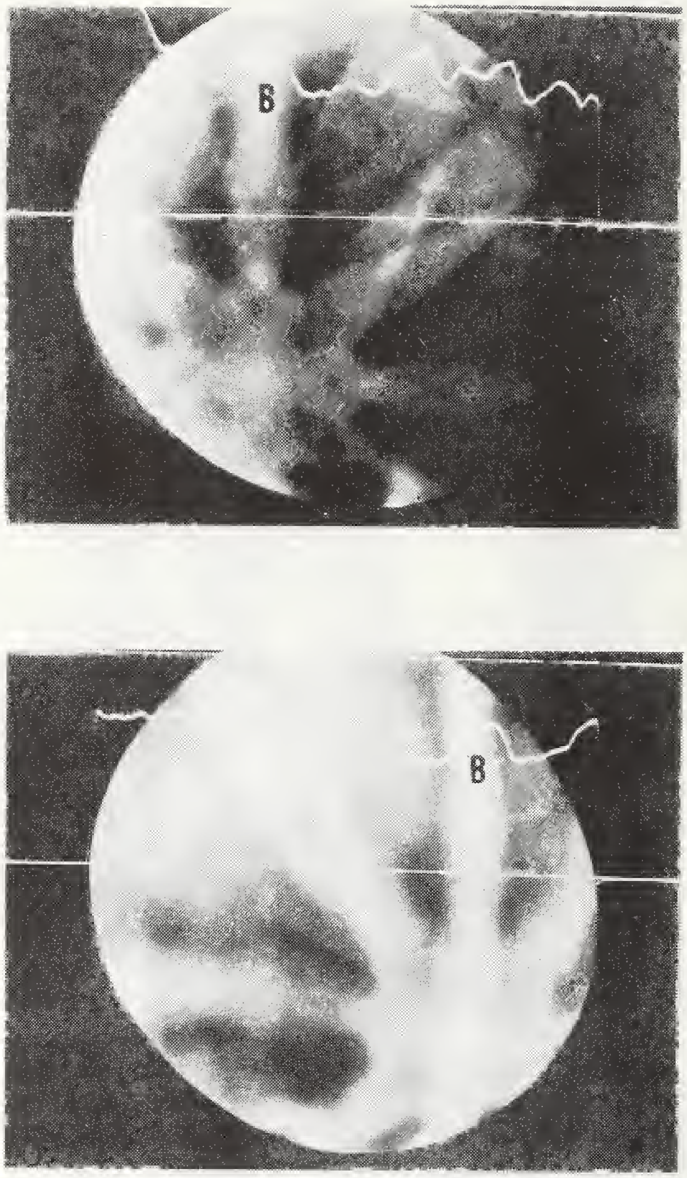

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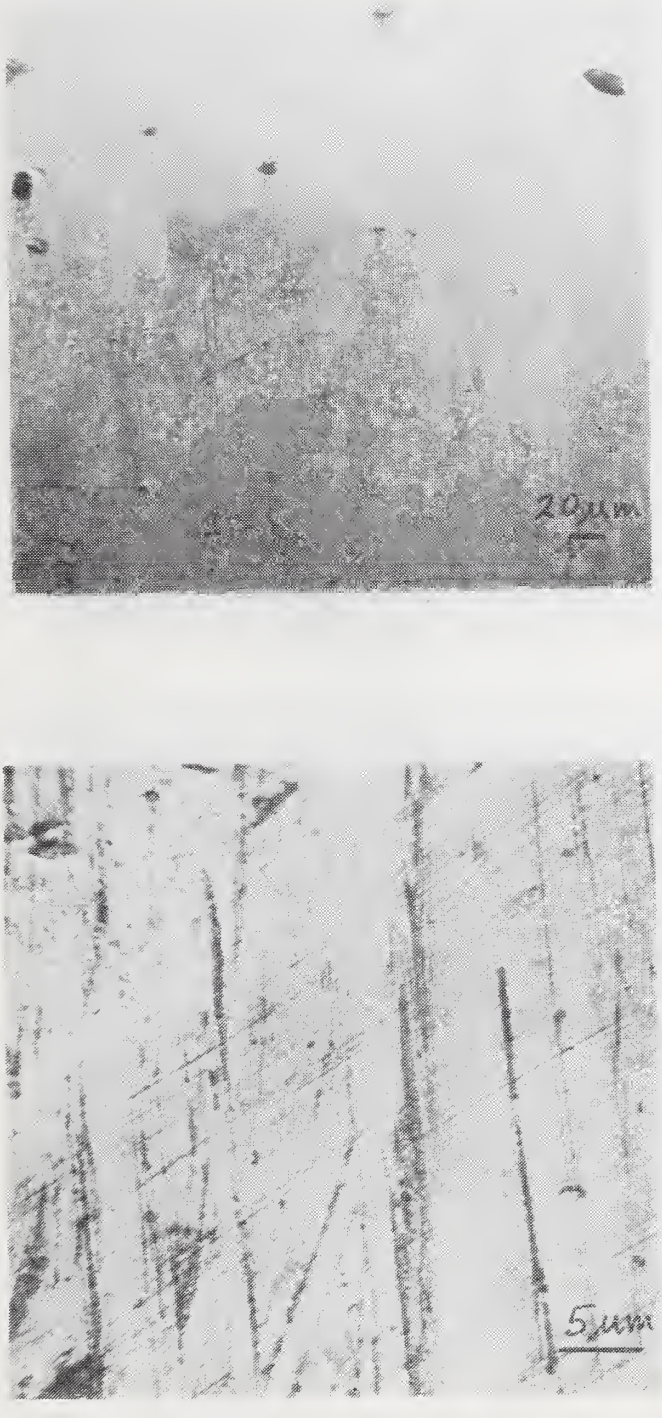

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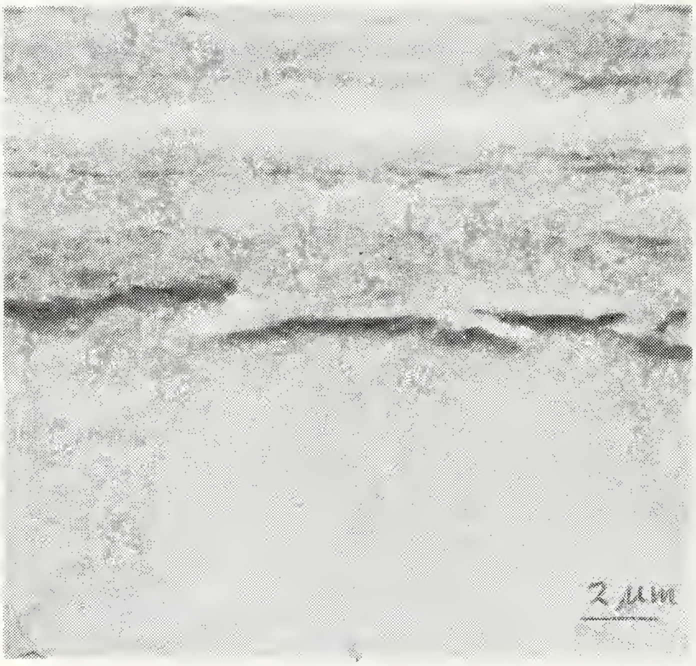
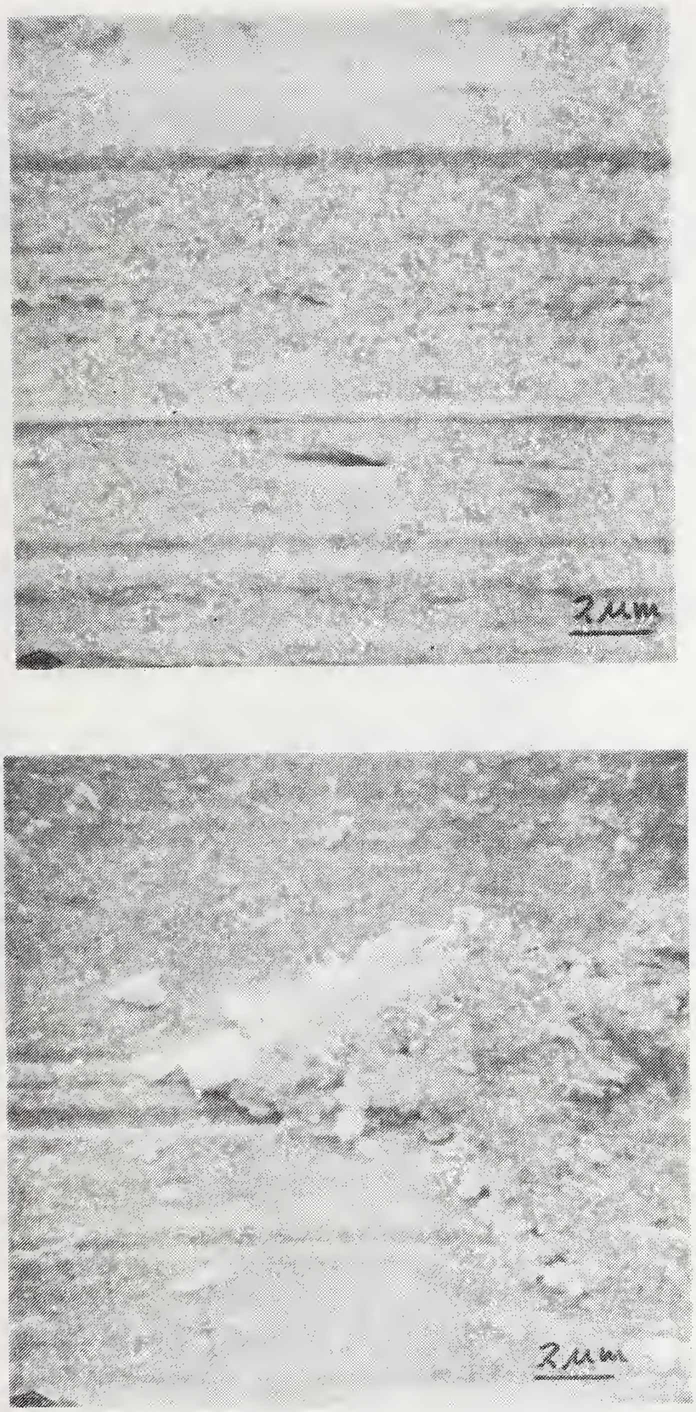


Fig. 10. (a) Wear track on high purity electropolished iron sheet specimen after 400 cycles under oil.
(b) Details at track edge.



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15. SUPPLEMENTARY NOTES

16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

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17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Abrasives; debris; electron channeling; iron; mechanical strain; metals; wear.

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