NBS TECHNICAL NOTE 862

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Application of Ion Beam Milling to the Characterization of Cracks in Metals

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The technique of ion beam milling coupled with subsequent optical and scanning electron microscopy has been applied as a means of detecting and characterizing small surface intersecting cracks. Two types of cracked specimens involving different metals were studied. Various orientations of the crack plane, crack direction, and ion beam were explored. The technique is capable of increasing the sensitivity for detection of small cracks and also removing distorted surface layers and revealing the crack more accurately.

Key words: Crack characterization; flaws; ion beam milling; metals; microscopy; surfaces.

Introduction

Ion beam milling has been investigated as a means of determining the size and shape of cracks in metals. This technique permits controlled removal of surface layers of material so that the detailed morphology of the exposed crack opening can then be examined using optical and electron microscopy. Application to very small cracks, cracks with opening dimensions of less than 1 μm, and the tip region of larger cracks was of primary interest. Direct viewing of the crack opening by means of optical microscopy or scanning electron microscopy (SEM) coupled with some form of serial surface removal appears to be the only means available for accurately assessing the crack opening geometry on a micrometer scale. Other conventional crack detection methods such as neutron and X-ray radiography do not permit resolution down to 1 μm. Acoustic and dye penetrant methods are useful in detecting cracks, however, they yield only limited information on crack geometry.

Small cracks or portions of cracks are particularly susceptible to being covered by oxide layers or obscured as a result of the deformation resulting from a mechanical sectioning operation. A method of preparing such surfaces without seriously disturbing the underlying crack geometry is needed. Mechanical means of surface removal even with careful metallographic polishing techniques produces metal flow which distorts or smears over the crack opening. This is especially true when ductile materials or very small openings are involved. Chemical methods of surface removal are unsatisfactory due to enhanced edge attack, differing disolution rates for different phases and problems associated with the penetration of corrosive chemical agents into the crack.

Ion beam milling as a surface preparation method appears to be less subject to the deficiencies noted above. With sufficiently low ion energies, radiation damage that occurs does not extend more than a hundred atomic layers below the surface. Any radiation damage effect could be minimized by maintaining the specimen at a low temperature. Care should be taken not to allow the specimen temperature to rise during ion bombardment. This paper describes the results of a study of ion beam milling at cracks under controlled conditions.

Ion Bombardment

It is well known that the removal of material by ion bombardment leads to the development of additional surface topography [1]. The selective nature of the attack at various crystal orientations, phases, grain boundaries, etc. is sometimes used as an alternative method for etching specimens. Other surface topography that develops has been attributed to specific ion bombardment conditions and is not related to any underlying structure [2]. Surface relief produced by these effects can be substantially reduced by milling at glancing angles to the surface while simultaneously rotating the specimen. This technique has been successfully employed to prepare transmission electron microscopy specimens where a surface smoothness on the order of 1 nm is required [3][4].

In recent years ion bombardment of solids has found a number of practical applications [5]. Among the best known are the doping of semiconductor materials by ion-implantation, the formation of thin films by sputter deposition, sputter etching in the fabrication of semiconductor devices and circuits and in the preparation of thin foils for transmission electron microscopy. The term sputtering refers to the ejection of surface atoms as a result of impact by incident ions.

Inert gases are used in sputtering or ion beam milling. More than one atom may be ejected for each incident ion. The yield, $S(\theta)$, is defined as the number of atoms ejected per incident ion and is a function of θ, the angle subtended by the incident ion direction and the target surface normal. $S(\theta)$ also depends on the incident ion and ejected atom species, the binding energy of the ejected atom, target surface temperature and incident ion energy. Ion energies employed are generally not greater than a few KeV. This, as well as other variables influencing sputtering yield, have been discussed in a number of works [2,3,5,6].

In connection with the general objective of demonstrating the usefulness of ion beam milling in examinations of crack opening geometry, a major part of this investigation was concerned with studying the effect of ion beam angle of incidence on the nature of the attack at crack edges. This is one of a class of problems concerned with predicting the surface topography that will develop from a given initial geometry under ion bombardment. If the yield $S(\theta)$ is known, then the rate at which a thickness, z, of material is removed from a plane surface having its normal at an angle θ to the beam direction is given by [7]

$$\frac{dz}{dt} = \frac{\Phi}{n} S(\theta) \cos \theta.$$

Here Φ is the number of ions incident per unit time on unit area normal to the beam direction and n is the number of atoms per unit volume of the bombarded surface. $S(\theta)$ [2,3,6] is generally found to vary in a way that is shown schematically in Fig. 1. Thus the rate of removal under constant ion flux is a minimum at normal incident ($\theta = 0$), rises to a maximum at $\theta = 60°$ to $70°$, then falls off rapidly to zero at $\theta = 90°$. Assuming a crack geometry shown schematically in cross section in Fig. 2, the crack opening dimensions should not be changed as long as the ion beam is parallel to the crack plane (the crack plane is perpendicular to the plane of the paper). On the other hand, if the beam direction does not lie in the crack plane, the leeward or down-beam crack wall will be subject to milling and the crack opening geometry changed. Even with the parallel orientation, however, the sharp crack edges may be subject to enhanced attack as was observed in this investigation. The simple geometry illustrated in Fig. 2 is not representative of real cracks in metals except in a few situations where cleavage or brittle fracture occurs. Theories have been developed that can deal with the more complicated topography actually realized [7-11]. However, they are based on a detailed knowledge of the yield coefficient $S(\theta)$. Although the relatively simple variation illustrated in Fig. 1 probably holds for amorphous materials, the situation for crystalline solids is far more complicated. $S(\theta)$ is not only a function of beam direction but also of crystal orientation and, as a consequence, faceting and other etch-like features develop. In addition, other important processes are not explicitly accounted for such as enhanced surface diffusion, redeposition and ion channelling affects, all of which may vary with topography. With a polycrystalline, multiphase material, the theoretical treatment of this problem may be for all practical purposes intractable.

Experimental

Ion beam milling was carried out in a bell jar vacuum system equipped with an oil diffusion pump and liquid nitrogen trap. A schematic diagram of the experimental arrangement is shown in Fig. 3. A vacuum of ∿20 mPa was maintained during milling operations. The ion gun was mounted on a translational stage to permit target area selection. A motor-driven turntable capable of rotating the specimen was also included in the system. The ion gun*† having approximate dimensions 2 x 6 x 8 cm was a hollow-anode, cold-cathode design. A potential of ∿5.6 kV was applied to the gun while argon was supplied as the ionizing gas. An ion beam was obtained through a circular 1 mm diameter gun (cathode) aperture. Both specimen current and gun beam current were monitored. The depth profiles of depressions produced in a chromium and nickel plated test specimen (described later) after milling for 1 hour at normal incidence with specimen-to-gun distances of 2, 4, and 9 mm are shown in Fig. 4.

*Microtron I, Commonwealth Scientific Corporation, Alexandria, Virginia.
†See footnote, Page 8.

Results and Discussion

Electroplated Penetrant Test Panel

Two different types of crack specimens have been investigated. The first was a penetrant test panel*† of the type used by the U.S. Air Force to assess the sensitivity of dye penetrant methods for detecting cracks. Three panel grades are available, termed coarse, medium and fine according to the crack opening dimensions. The fine grade of test panel was studied in this investigation. Crack openings were specified to be in the range 0.25 - 0.5 μm. The test panel consists of a brass plate 10 cm long, 6.7 cm wide and 0.32 mm thick. A layer of nickel 8.9 to 12.7 μm thick is electrodeposited onto one surface. This is followed by two layers of chromium, the first 0.64 to 0.76 μm and the second 2.0 to 2.4 μm thick. Cracks are introduced in the electroplated layers by bending the panel around a cylindrical arbor. A nickel-sensitive electrographic print providing a direct replica of the crack pattern is supplied with each panel. The fine, straight cracks provide a simple morphology for the investigation of ion milling techniques. A SEM micrograph of an unmilled crack is shown in Fig. 5. The crack opening in this case is actually less than 0.2 μm along much of its visible length.

In Fig. 6(a) a group of seven parallel cracks is shown after ion milling at normal incidence to the panel surface. Three zones of contrast are visible within the milled depression. The dark, outer zone is chromium, the lighter circular zone is the exposed nickel layer while the rough, etched area at the center of the depression is the relatively coarse grained brass substrate. A substantial increase in crack opening width is evident as cracks enter and pass through the milled depression. At the center of the depression the increase is more than 100 fold. On closer examination it appears that a groove has developed along the crack. This is contrary to the expected result illustrated in Fig. 2 and indicates the ease with which atoms at the crack edge are ejected. Also it is noted that a groove once formed is perpetuated by the milling process as seen in the etched brass area in Fig. 6(b). This occurs since material at the bottom of a crack will be milled as readily as surrounding surface area. Although normal incidence of the milling beam does distort the crack opening geometry, the enhanced attack can be exploited in order to reveal very small surface cracks that might otherwise escape detection.

In Fig. 7 a series of parallel cracks are shown after milling with the beam incident at $\theta = 60°$ to the surface normal and parallel to the plane of the cracks. The crack opening width appears to remain unchanged proceeding from outside the milled region until the crack disappears at the center of the milled depression where the brass

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*Available from Scientific Control Labs. Inc., Chicago, Illinois.

†See footnote page 8.

substrate has been exposed. Some etching attack has occurred at the crack edges as illustrated in Fig. 7(d). Some features that appear in the crack may result from irregularities that have been enhanced by milling or from debris trapped within the crack. Figure 8 shows a crack that has branched so that one segment is at an angle of approximately 45° to the beam direction. Considerable erosion in the vicinity of the leeward crack edge has occurred, since that wall was directly exposed to the ion beam. The amount of exposure is determined by the extent of shadowing produced by the opposite crack edge. The morphology exhibited in Fig. 8 indicates that the exposed portion of the leeward crack well has eroded at a relatively rapid rate in the direction of the beam, leaving a wide, shallow step along the crack.

In order to suppress the development of the etch-like morphology evident in both Fig. 7 and 8, a specimen was rotated about its surface normal while being milled at a 70° angle. Fig. 9 shows four cracks in that panel. Three cracks are approximately parallel to the axis about which the panel was bent. The fourth intersecting crack was steeply inclined to the bending axis. The surface has been milled to a depth of several micrometers in the nickel layer. Numerous conical features rising above the surface are present. In some cases the tips are truncated. Such features are often seen on ion etched surfaces and are associated with the presence of low yield inclusions [12]. In comparison, only a few cones were found in the panel shown in Fig. 7 and 8. A crack cannot be clearly discerned at the bottom of the steeply inclined groove in Fig. 9(a) and (b). Fig. 9(b) is the central region of Fig. 9(a) shown at a higher magnification. Either the crack has become so narrow that it is not resolved, or it did not extend to this depth. Note that the groove has developed in a relatively symmetrical manner as would be expected on the basis of the milling geometry. This geometry is not displayed, however, around the horizontal cracks. In fact, the crack visible in Fig. 9(b) appears to be along the lower side of the milled groove. The associated groove is also very irregular and ragged in appearance. An explanation for this morphology was found on examining another crack in a region that had been milled to the level of the nickel-chromium interface. The light region along that crack in Fig. 10 is exposed nickel while the darker surrounding area is chromium. The nickel along the crack contains numerous holes and appears to have been chemically attacked. The attack probably resulted from the electrochemical process customarily used to produce the nickel-sensitive electrographic print that displays the crack pattern. Cracks lying along the axis of bending appear to be most affected. This may be due to their larger opening and depth or possibly to the fact that some separation has occurred between the chromium and nickel layers as was suggested at some locations. Further study would be required to confirm the latter hypothesis.

Figure 11 is the crack shown in Fig. 10 at a location where the nickel layer has not been exposed. Here the crack opening geometry does not appear to have been substantially affected by the milling operation and the surrounding surface appears to be quite smooth, particularly when compared to that in Fig. 8.

Fatigue Crack in 6061 Aluminum Alloy Plate

In contrast to the relatively straight, planar cracks examined in the penetrant test panel, fatigue cracks in a relatively ductile aluminum alloy provided a more complicated geometry for the application of ion beam milling. Cracks were produced by partially fracturing 1.8 cm thick, flat plate, notched tensile specimens in low cycle fatigue. An optical photograph of a crack as it originally appeared on one surface of a plate is shown in Fig. 12(a). Figure 12(b) is an enlargement of a neutron radiograph of the crack taken parallel to the same surface. To obtain the radiograph, the crack was wedged open slightly and a penetrant containing gadolinium (large neutron scattering cross-section) was introduced. Aluminum itself is quite transparent to neutrons. The lower boundary of the projected crack shown in Fig. 12(b) coincides quite faithfully to the crack at the surface in 12(a) except for approximately the final 2 mm of length. The penetrant apparently did not fill the final tip region of the crack and hence it is not visible in the neutron radiograph.

An SEM micrograph of the crack tip region is shown at a higher magnification in Fig. 13. A relatively thick layer of scale and numerous surface scratches and flaws tend to obscure the crack. A surface layer approximately 0.25 mm in thickness was removed by hand grinding on SiC paper. The surface was then metallographically polished through a final 0.05 μm MgO abrasive following conventional procedures. An optical micrograph of the polished surface in the region of the crack tip is shown in Fig. 14(a). The crack is barely visible and has apparently been smeared over along portions of its length. This is demonstrated further in the SEM micrograph of Fig. 14(b).

The tip region of the crack was subjected to ion milling with the beam approximately parallel to the crack plane and 70° to the surface normal. Fig. 12(c) shows the entire length of the crack after milling at the tip region. The crack opening appears to decrease abruptly in the vicinity of the crack tip. This marks the end of the crack trace visible in the neutron radiograph (12(b)). The crack tip is apparently so narrow that adequate access of the gadolinium bearing penetrant was prevented. The milled tip region is shown at higher magnifications in the optical micrographs, Fig. 15(a) and (b). The orientation dependence of ion erosion along the crack edge here is similar to that demonstrated in the experiments performed on the penetrant test panel. There is little effect where the crack and beam are parallel; otherwise the leeward edge of the crack is eroded to leave a broad, shallow depression behind the crack. It is evident that at some locations the crack has followed grain boundaries. Note in particular the grain at A in Fig. 15(b). Upon encountering the grain, the crack has branched as is shown in the SEM micrograph of Fig. 16. The crack opening at some locations in Fig. 16 is less than 400 Å. The crack edges appear to be sharp and smooth without any evidence of local preferential etching effects. Apart from the obvious nonuniform milling associated with precipitates, inclusions and grain boundaries, the overall surface was smooth. Figure 17 shows the location of what appeared to be the crack tip with the approximate end marked by a rather large inclusion (shown in the upper left hand corner). There was considerable uncertainty concerning the actual path of the crack. Gaps existed where the crack could not be resolved. In order to reveal the crack in a more convincing manner, the surface was briefly milled at normal incidence. The result is shown in Fig. 18(a). The crack path is now quite visibly delineated by a groove which was shown to accompany ion milling at normal incidence. A portion of the crack length is shown at a higher magnification in Fig. 18(b). Although a dark line which is attributed to the crack is visible at most points, the crack opening is not actually resolved. This may be due to insufficient resolution or the crack may have closed when the applied tensile stress was released on termination of the fatigue test.

Summary

Experiments were performed to investigate the applicability of ion beam milling in revealing crack opening geometry and in detecting small surface cracks. Different beam orientations with respect to the specimen surface and crack plane were found to have a substantial effect on crack edge erosion. Penetrant sensitivity test panels containing planar cracks lying normal to the specimen surface were employed to evaluate the nature of these effects. With the ion beam normal to the surface and parallel to the crack plane, rapid erosion of the crack edges occurred producing a symmetrical groove along the crack, wide in relation to the crack opening width. When the ion beam was inclined at an angle of 60-70° to the surface normal but parallel to the crack plane, this preferential edge attack was nearly eliminated. However, at that inclination but with the beam no longer parallel to the crack plane, the leeward edge of the crack was rapidly eroded leading to a wide, shallow groove. Maintaining the same beam inclination while rotating the specimen about its surface normal resulted in a reduction in surface etching topography. The erosion of the crack edges also appeared to be minimized under these conditions.

The utility of the ion beam milling technique was demonstrated in revealing the nature of a fatigue crack in a mechanically polished 6061 aluminum alloy specimen. In addition, an unexpected subsurface corrosion attack was discovered along cracks in one of the penetrant sensitivity test panels.

Acknowledgments

The aluminum alloy plate was provided by Dr. D. Eitzen of the Mechanics Division. Neutron radiography of that plate was conducted by Dr. H. Berger of the Reactor Radiation Division. We are indebted to them for contributing to this study.

†Certain commercial products and instruments are identified in this paper in order to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards nor does it imply that the products or equipment identified are necessarily the best available for the purpose.

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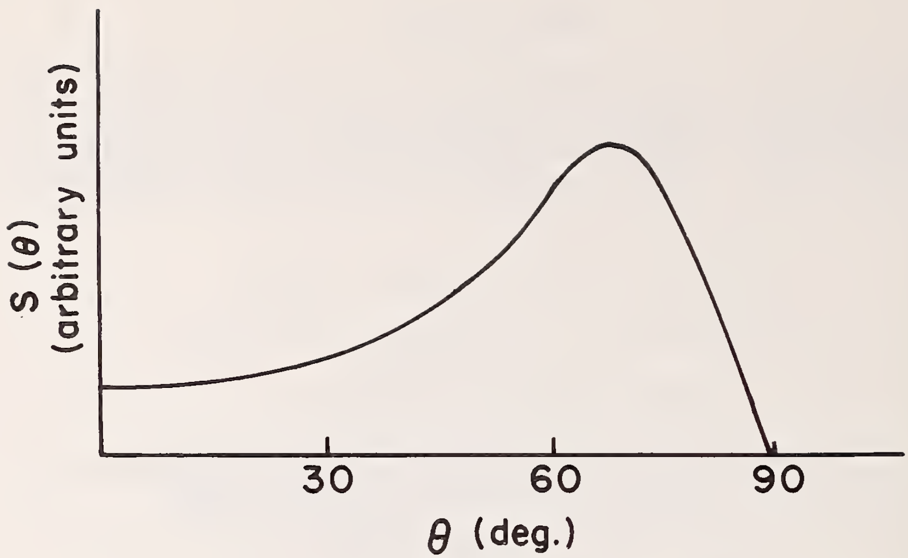

Figure 1 Schematic representation of the variation of yield with incident ion beam angle.

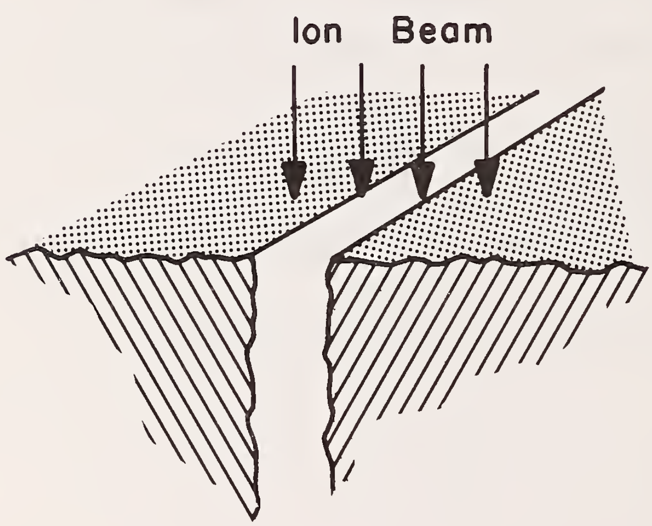

Figure 2 Idealized crack geometry intersecting a free surface.

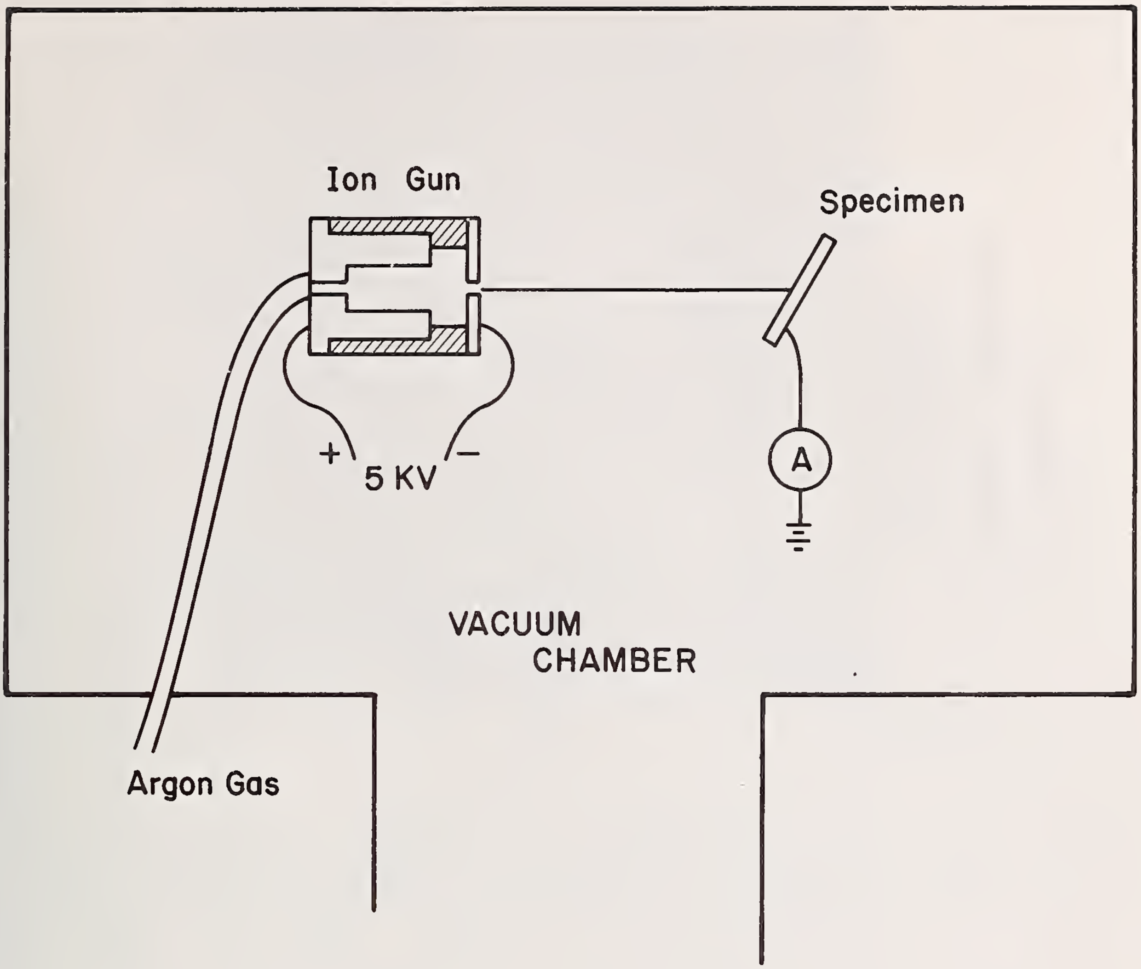

Figure 3 Schematic diagram of ion beam milling apparatus.

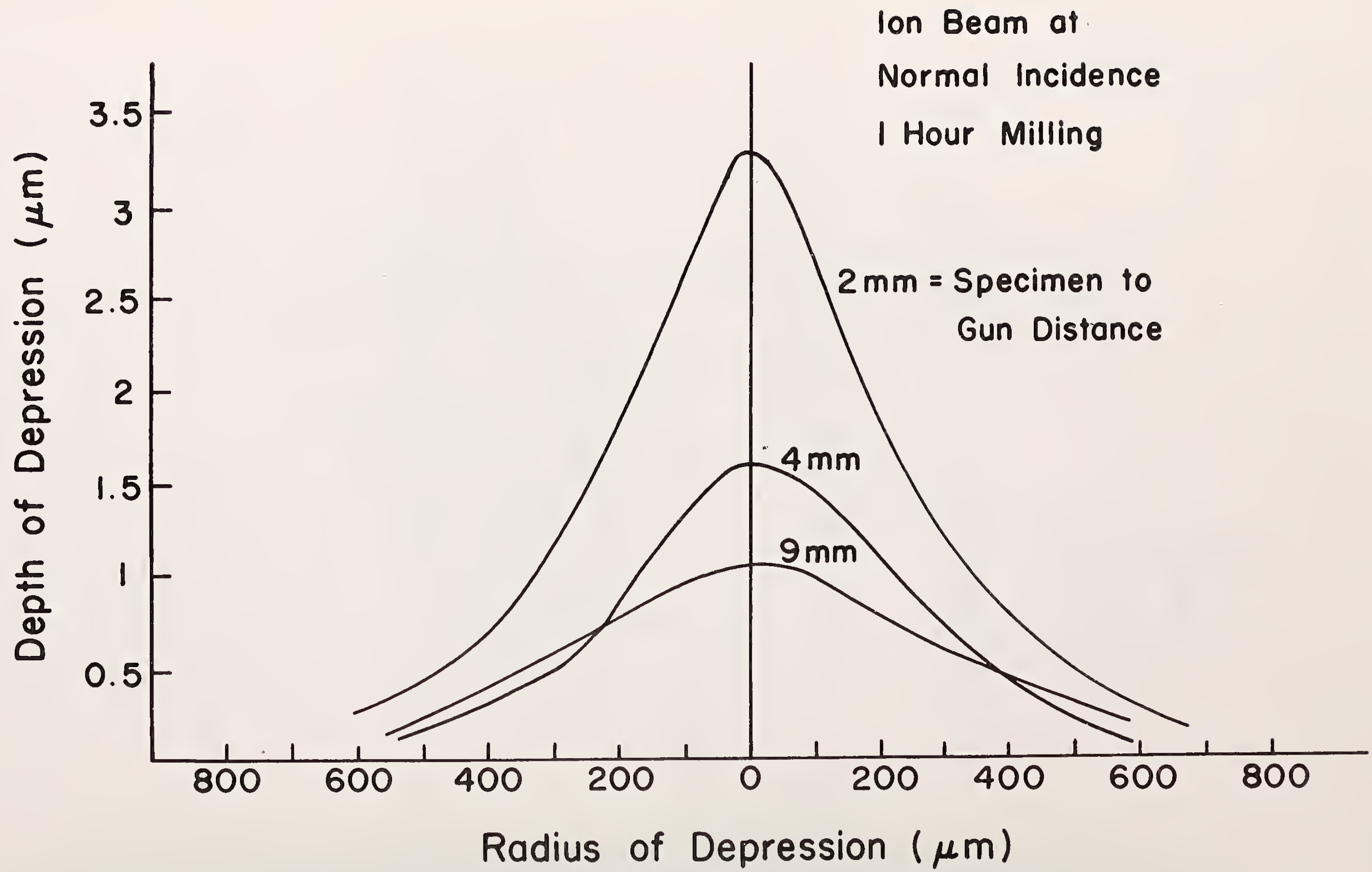

Figure 4 Depth and radius of ion milled depression for various gun-specimen distances.

Figure 5 Scanning electron micrograph of a crack in a panel.

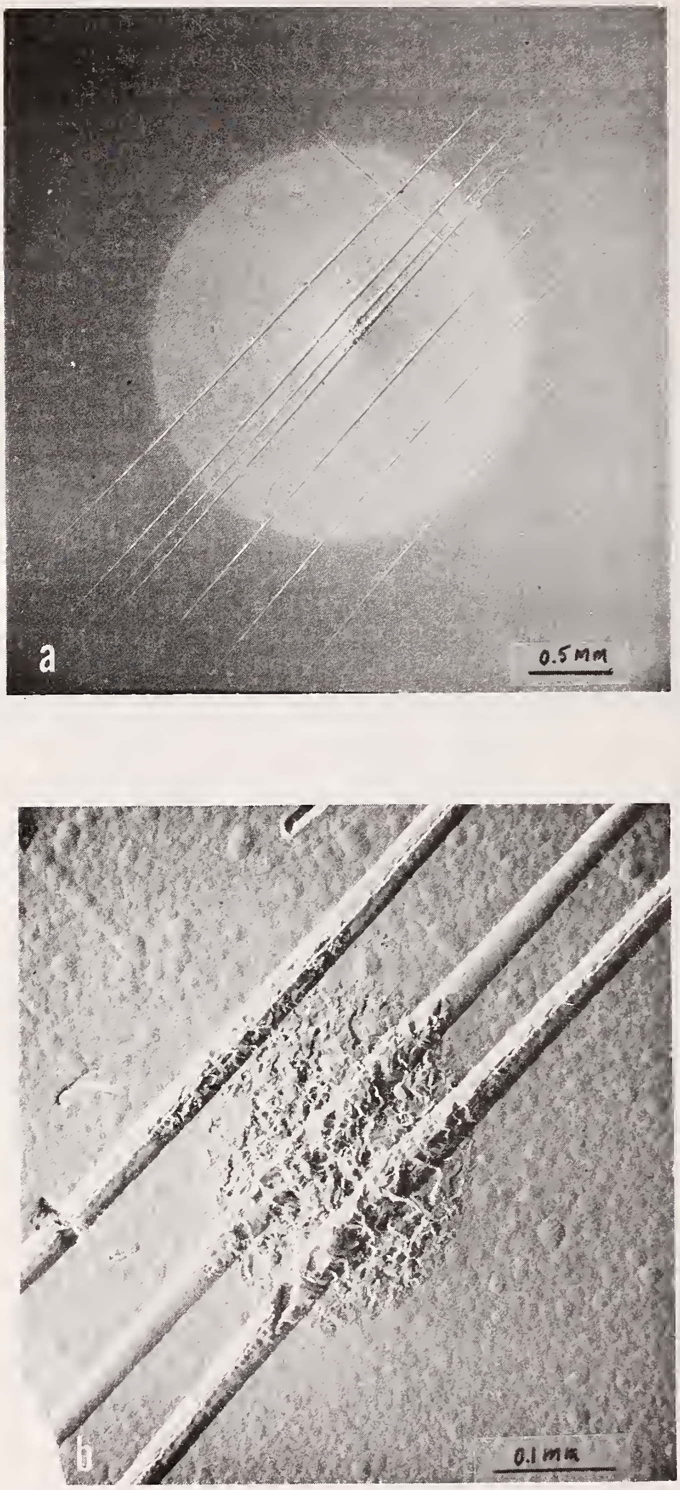

Figure 6 Scanning electron micrographs of cracks in an ion milled area.

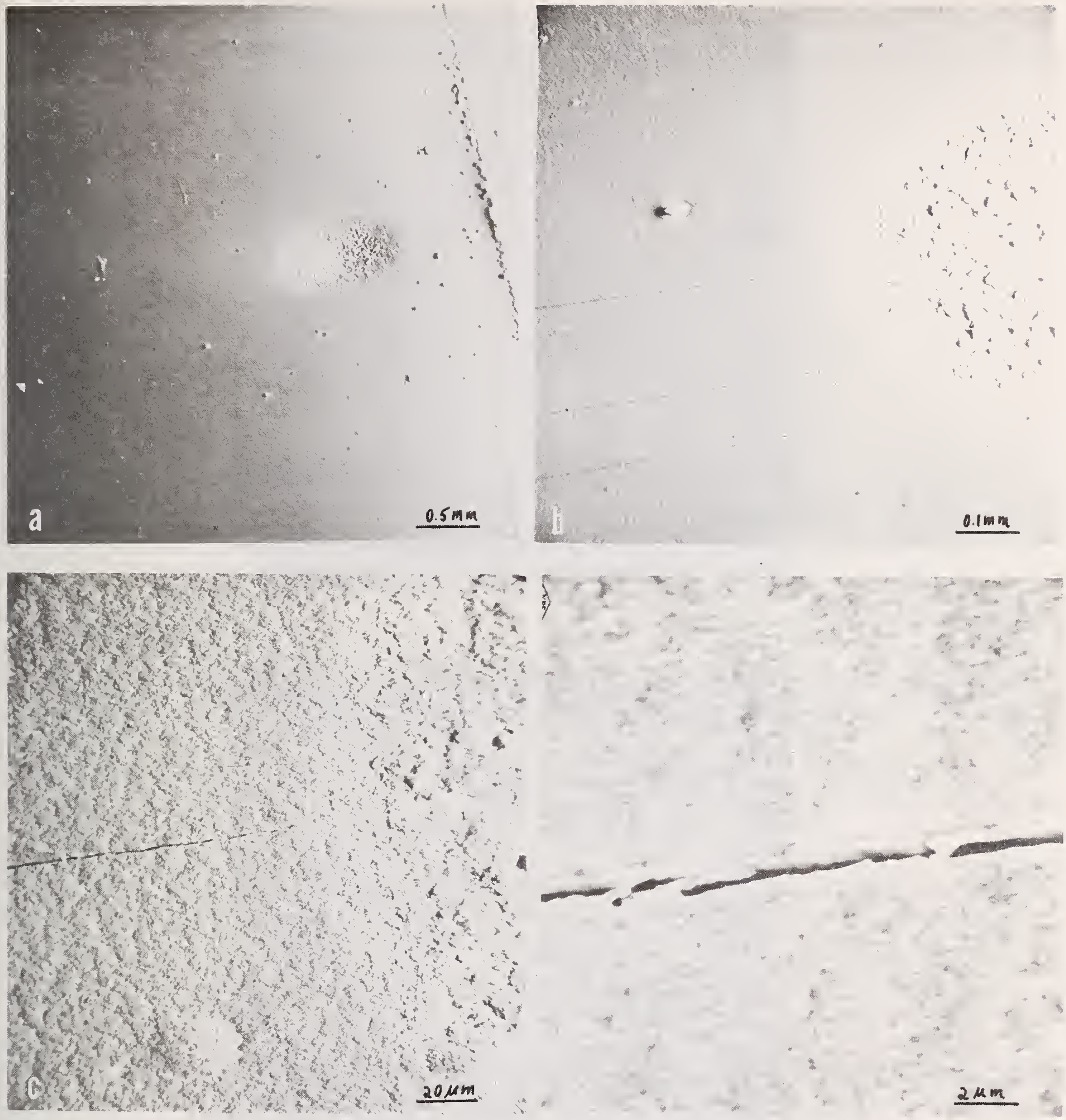

Figure 7 Details of crack in a panel after milling at 60° orientation (see text).

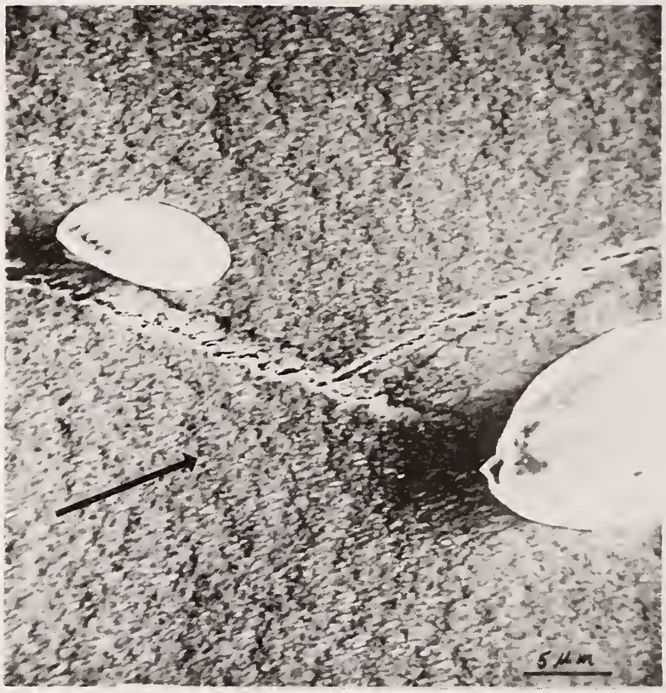

Figure 8 Effect of enhanced milling at one edge of crack in a panel. Arrow indicates beam direction.

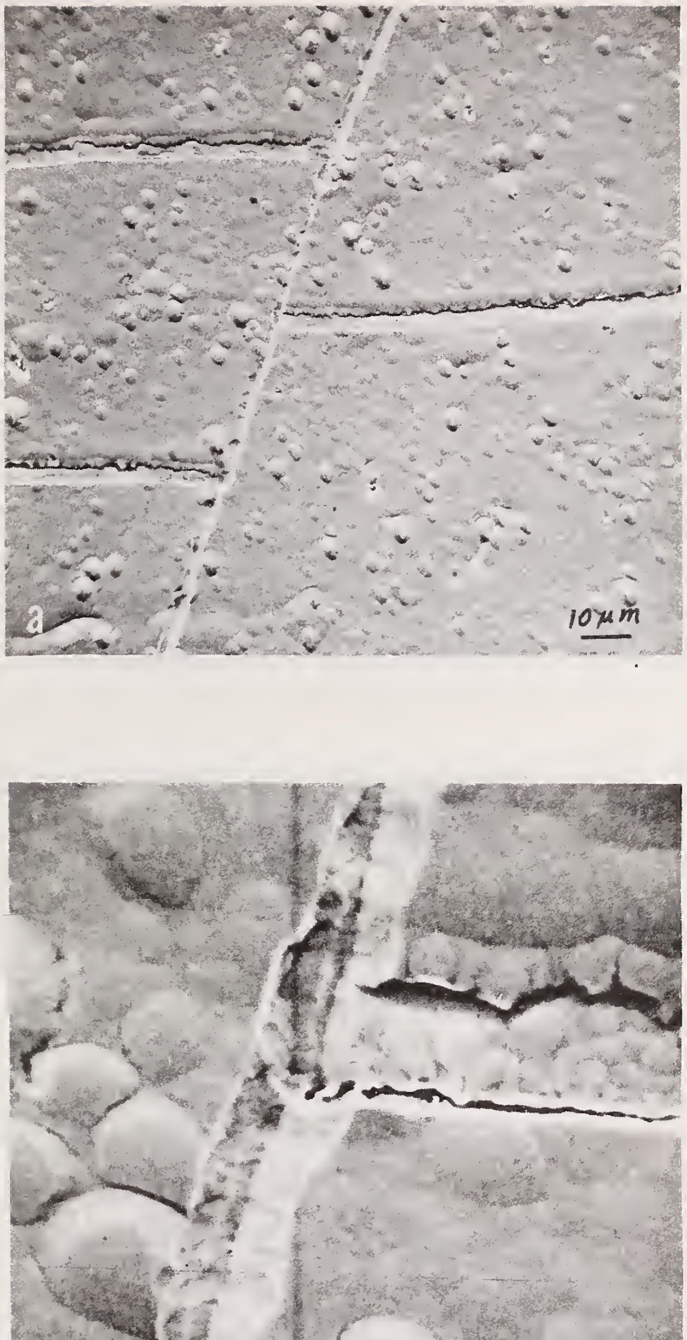

Figure 9 Specimen rotated during ion milling at 70° orientation. Note in (b) crack and unusual groove associated with it.

Figure 10 Evidence for chemical attack in nickel region associated with crack in test panel.

Figure 11 Crack in panel at location of upper chromium layer where subsurface attack is not seen.

Figure 12 Fatigue crack in 6061 aluminum plate. (a) Optical photograph of original surface, (b) neutron radiograph, (c) optical photograph after polishing surface and ion milling.

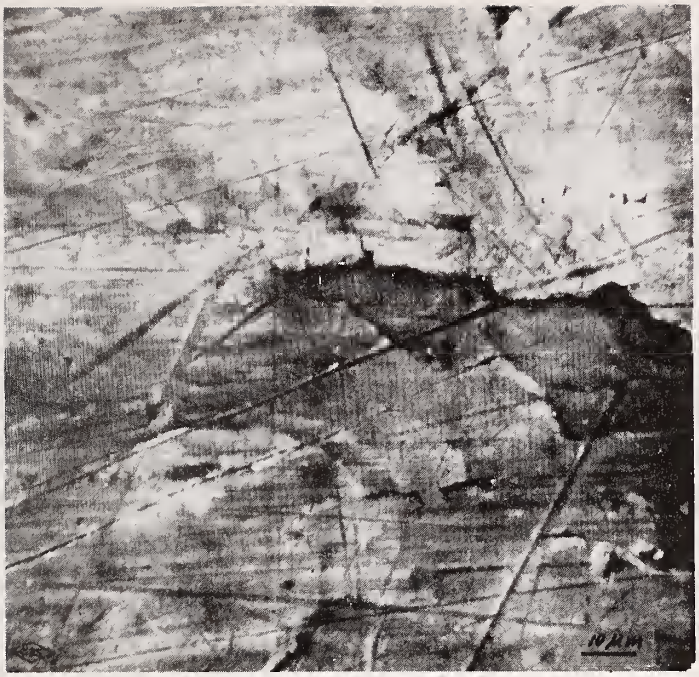

Figure 13 Scanning electron micrograph near the crack tip on original surface.

Figure 14 Crack observed after metallographic polishing of surface (a) optical, (b) SEM photograph.

Figure 15 Effect of ion milling the polished surface near the crack tip (see text). Location at A in (a) is shown at higher magnification in (b).

Figure 16 Branching of crack near tip revealed by ion milling. SEM micrograph.

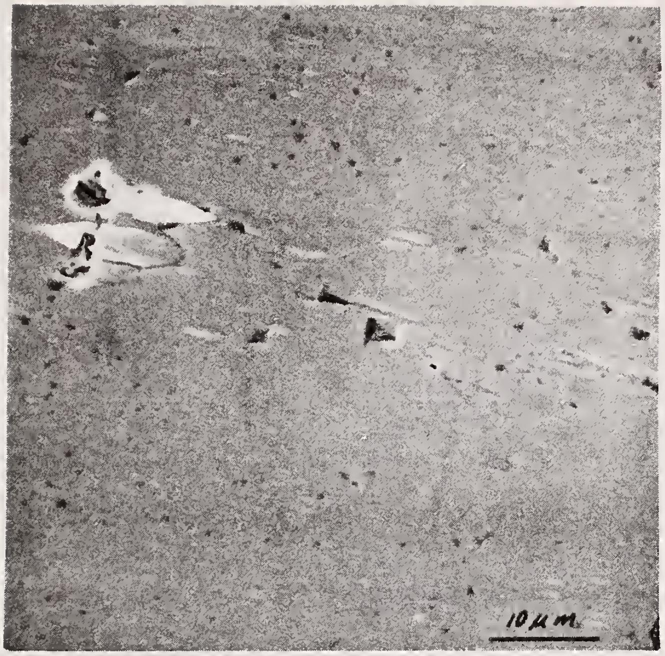

Figure 17 Region of crack tip after ion milling the surface at 70° orientation. SEM micrograph.

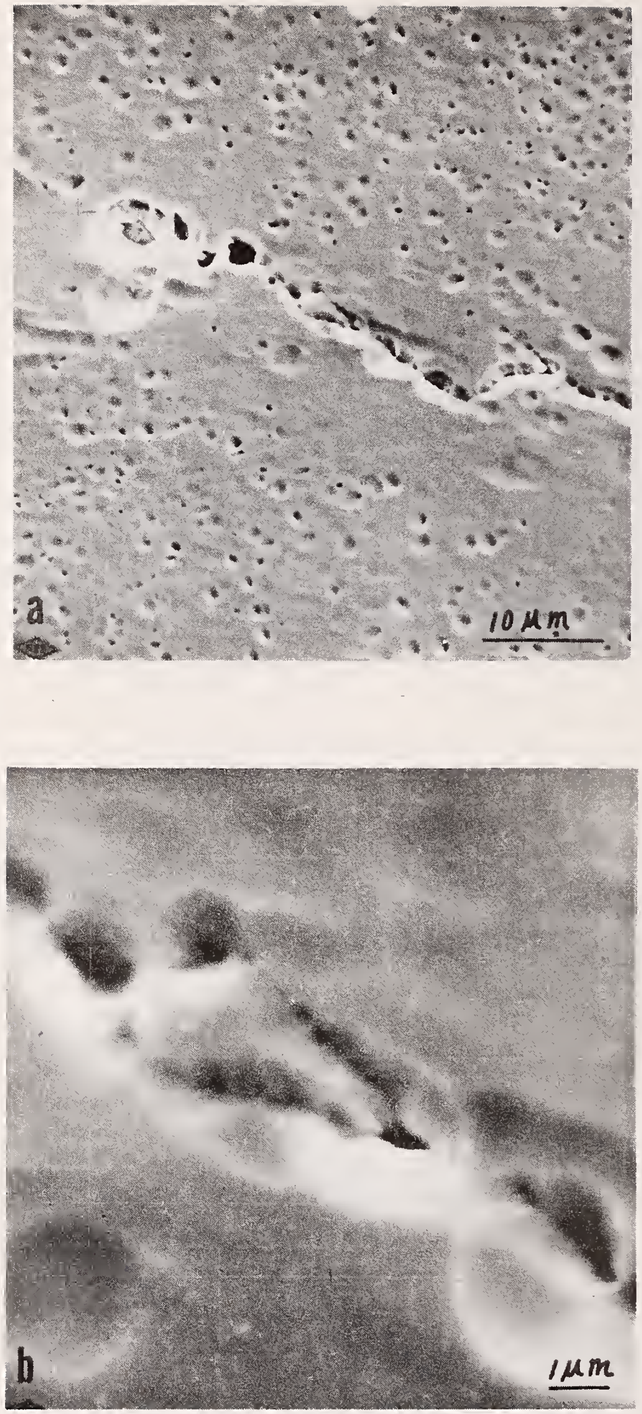

Figure 18 (a) Region of crack tip after additional ion milling at normal incidence to further delineate crack location. (b) Region near center of (a) at higher magnification.

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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.*)

The technique of ion beam milling coupled with subsequent optical and scanning electron microscopy has been applied as a means of detecting and characterizing small surface intersecting cracks. Two types of cracked specimens involving different metals were studied. Various orientations of the crack plane, crack direction, and ion beam were explored. The technique is capable of increasing the sensitivity for detection of small cracks and also removing distorted surface layers and revealing the crack more accurately.

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*)

Cracks; flaws; ion beam milling; metals; microscopy; surfaces

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Studies and compilations designed mainly for the mathematician and theoretical physicist. Topics in mathematical statistics, theory of experiment design, numerical analysis, theoretical physics and chemistry, logical design and programming of computers and computer systems. Short numerical tables. Issued quarterly. Annual subscription: Domestic, $9.00; Foreign, $11.25.

DIMENSIONS/NBS (formerly Technical News Bulletin)—This monthly magazine is published to inform scientists, engineers, businessmen, industry, teachers, students, and consumers of the latest advances in science and technology, with primary emphasis on the work at NBS. The magazine highlights and reviews such issues as energy research, fire protection, building technology, metric conversion, pollution abatement, health and safety, and consumer product performance. In addition, it reports the results of Bureau programs in measurement standards and techniques, properties of matter and materials, engineering standards and services, instrumentation, and automatic data processing.

Annual subscription: Domestic, $9.45; Foreign, $11.85.

NONPERIODICALS

Monographs—Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NBS, NBS annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

Applied Mathematics Series—Mathematical tables, manuals, and studies of special interest to physicists, engineers, chemists, biologists, mathematicians, computer programmers, and others engaged in scientific and technical work.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a world-wide program coordinated by NBS. Program under authority of National Standard Data Act (Public Law 90-396).

NOTE: At present the principal publication outlet for these data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St. N. W., Wash. D. C. 20056.

Building Science Series—Disseminates technical information developed at the Bureau on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NBS under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The purpose of the standards is to establish nationally recognized requirements for products, and to provide all concerned interests with a basis for common understanding of the characteristics of the products. NBS administers this program as a supplement to the activities of the private sector standardizing organizations.

Federal Information Processing Standards Publications (FIPS PUBS)—Publications in this series collectively constitute the Federal Information Processing Standards Register. Register serves as the official source of information in the Federal Government regarding standards issued by NBS pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

Consumer Information Series—Practical information, based on NBS research and experience. covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

NBS Interagency Reports (NBSIR)—A special series of interim or final reports on work performed by NBS for outside sponsors (both government and non-government). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Service (Springfield, Va. 22161) in paper copy or microfiche form.

Order NBS publications (except NBSIR's and Bibliographic Subscription Services) from: Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.

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Cryogenic Data Center Current Awareness Service

A literature survey issued biweekly. Annual subscription: Domestic, $20.00; foreign, $25.00.

Liquefied Natural Gas. A literature survey issued quarterly. Annual subscription: $20.00.

Superconducting Devices and Materials. A literature survey issued quarterly. Annual subscription: $20.00. Send subscription orders and remittances for the preceding bibliographic services to National Technical Information Service, Springfield, Va. 22161.

Electromagnetic Metrology Current Awareness Service Issued monthly. Annual subscription: $100.00 (Special rates for multi-subscriptions). Send subscription order and remittance to Electromagnetics Division, National Bureau of Standards, Boulder, Colo. 80302.