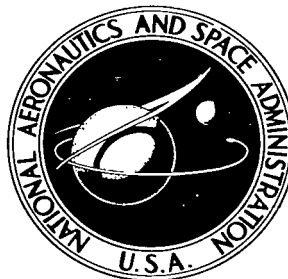


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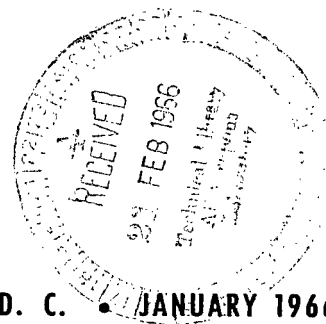
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INFLUENCE OF ORIENTATION OF GRAINS IN TUNGSTEN ON ITS FRICTION CHARACTERISTICS

by Donald H. Buckley
Lewis Research Center
Cleveland, Ohio



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SUMMARY

The influence of crystallographic orientation on the friction properties of tungsten was determined in both air and vacuum. Friction measurements were made with a 3/8-inch oriented sapphire ball sliding on a large grained tungsten disk, and the orientation of each grain (crystal) on the tungsten surface was determined. The two sapphire orientations examined were the (0001) plane, $[10\bar{1}0]$ direction, and the $(10\bar{1}0)$ plane, $[0001]$ direction, sliding on the tungsten surface.

The sapphire ball was loaded against the disk with a 500-gram load. The disk was then rotated to produce sliding velocities of 0.013 and 0.001 centimeter per second. The vacuum experiments were conducted with electron-cleaned surfaces at 10^{-10} millimeter of mercury.

The investigation indicates that tungsten exhibits anisotropic friction properties. Friction characteristics on different crystal planes in a particular direction did not differ markedly; considerable differences in friction coefficient, however, existed in changing crystallographic directions on particular planes. Further, marked differences in friction were observed with changes in crystallographic orientation of the sapphire. Friction decreased considerably when vacuum measurements were repeated in air.

INTRODUCTION

In the field of friction and wear, most materials have particular frictional characteristics which are based on their polycrystalline form. In recent years, however, measurements have been made on single crystals to determine if differences in friction and wear might exist for the various crystallographic planes exposed to the surface of a randomly oriented polycrystalline material. Studies have been conducted of the influence of single crystal orientation on the friction and wear behavior of sapphire in contact with various materials (refs. 1 to 5). Anisotropic frictional behavior has also been observed for metals. References 6 and 7 indicate the influence of crystallographic orientation on the friction characteristics of hexagonal metals, and references 8 and 9 indicate this influence for the

face-centered-cubic metal copper. These studies have been concerned with the orientation of one of the two materials in contact.

Although single crystal data can do much to elucidate the influence of crystal orientations on friction, they do not represent the polycrystalline material. The grain boundaries contribute markedly to friction behavior. While a grain may exhibit well-defined crystallographic slip behavior under deformation, the slip behavior at grain boundaries is not so well defined. Reference 10 has termed this a region for "noncrystallographic slip" and reference 11 refers to it as a "region of viscous flow, much like in a liquid."

The influence of crystallographic orientation on friction for body-centered cubic metals may not be as pronounced as observed for a face-centered cubic metal such as copper (ref. 8) possibly because of the increased number of slip systems. Hardness studies with the body-centered cubic metals indicate (refs. 12 and 13) anisotropic hardness behavior for silicon ferrite and columbium.

This investigation was conducted to determine with an oriented single crystal of sapphire sliding on a large polygrain (with each grain orientation determined) tungsten disk specimen (1) the effect of grain orientation and sliding direction on the friction behavior of tungsten, (2) the influence of changing the orientation of the sapphire and, (3) the effect of environment on such measurements. Experiments were conducted in both vacuum (10^{-10} mm Hg) and air (760 mm Hg) with a 3/8-inch-diameter sapphire ball sliding on a large-grain tungsten disk. The ball was loaded against the disk with a 500-gram load, and the disk was rotated to produce linear speeds of 0.013 and 0.001 centimeter per second.

APPARATUS

The basic elements of the apparatus used in this investigation (fig. 1) were the specimens (a 2-in. -diameter flat disk of tungsten and a 3/8-in. -diameter sapphire ball) mounted in a vacuum chamber. The disk specimen was driven through a magnetic drive coupling. The coupling had two 20-pole magnets 0.150 inch apart with a 0.030-inch diaphragm between magnet faces. The driver magnet, outside the vacuum system, was coupled to a hydraulic motor. The driver magnet was completely covered with a nickel-alloy housing (cutaway in fig. 1) and was mounted on one end of the shaft within the chamber. The end of the shaft opposite the magnet contained the disk specimen.

The ball and its holder specimen were supported in the specimen chamber by an arm mounted by gimbals and bellows to the chamber. A linkage at the end of the retaining arm opposite the ball specimen was connected to a strain-gage assembly. The assembly was used to measure frictional force. Load was applied through a deadweight loading system.

Attached to the lower end of the specimen chamber was a 400-liter-per-second ionization pump and a vac-sorption forepump. The pressure in the chamber was measured

adjacent to the specimen with a cold-cathode ionization gage. In the same plane as the specimens and the ionization gage was a diatron-type mass spectrometer (not shown in fig. 1) for determination of gases present in the vacuum system. A 20-foot, 3/16-inch-diameter stainless-steel coil was used for liquid-nitrogen and liquid-helium cryopumping of the vacuum system.

PROCEDURE

The large-grain tungsten used in this investigation was prepared by electron beam melting. Chemical analysis of the material used indicated 5 ppm carbon, 3 ppm oxygen, less than 3 ppm nitrogen, and small percentages (less than 2 ppm) of metallic elements calcium, chromium, cobalt, copper, iron, and nickel. The specimen was cut, finish-ground, and lapped prior to electropolishing. The specimen was then electropolished in a sodium hydroxide solution to remove the worked layer. After electropolishing, the specimen was mounted in a fixture and Laue patterns of the various grains were obtained. The orientations obtained are shown in figure 2 and their position on the unit triangle in figure 3. The actual specimen is shown in the photograph of figure 2; the diagrammatic sketch indicates the orientations.

In addition to crystallographic planes, directions are indicated in figure 2. (Planes are given in parentheses and directions in brackets.) A simple sketch in figure 3(a) defines these directions. The direction in the cubic system is normal to the plane.

The single crystals of sapphire used in this study consisted of 3/8-inch-diameter balls. The balls were initially oriented with polarized light to locate the optical axis, and then X-ray determinations were made for plane and direction. The balls were locked in a stainless-steel holder similar to that described in reference 5. The orientations were then rechecked.

After the specimens were mounted in the vacuum system, the system was evacuated, and the tungsten disk specimen was electron bombarded for 4 hours to remove adsorbed gases and surface oxides. The disk temperature at this time was approximately 1000° F. The specimens were cooled to room temperature before friction experiments.

RESULTS AND DISCUSSION

Elastic Behavior

The elastic behavior for various body-centered cubic metals and the degree of anisotropy in these metals has already been determined (ref. 14). Table I indicates the

degree of anisotropy in the elastic region for various body-centered cubic metals. A factor of unity is taken for the isotropic state. Examination of table I indicates that in the elastic region of all the metals listed tungsten is the only one that exhibits isotropic behavior.

In the region of plastic deformation, anisotropic behavior has been demonstrated for a silicon-iron alloy (ref. 12) and for niobium (ref. 13), both of which deviate considerably from 1.00 in table I. The behavior of tungsten in the plastic region with respect to anisotropic behavior is therefore of interest.

Deformation

In order to obtain some indication of anisotropic behavior for tungsten, preliminary experiments were conducted with a sapphire ball in a Bierbaum microhardness tester sliding across the flat surface of a tungsten single crystal. The crystal could be rotated for sliding in different crystallographic directions. The 1/16-inch sapphire ball was loaded against the tungsten surface under a load of 60 grams, and the tungsten crystal was moved to produce sliding speeds of 0.002 centimeter per second. The experiments were conducted in air at 75° F.

Examination of the photograph in figure 4(a) at a magnification of 140 shows the wear scar produced in the $[111]$ direction on the (211) plane and normal to the $[111]$ direction. The photomicrographs ($\times 700$) of the wear scars etch-pitted with Millner-Sass reagent (refs. 15 and 16) reveal anisotropic behavior on the (211) plane of tungsten (figs. 4(b) and (c)). Of particular interest is the high concentration of etch pits, and therefore dislocations, adjacent to the deformed region in the two sliding directions. There are a considerably greater number normal to the $[111]$ direction, (fig. 4(b)). These indicate a greater degree of deformation normal to the $[111]$ direction.

Friction Characteristics in Vacuum

From the results of the preliminary tests, it was decided to conduct friction experiments in vacuum with an oriented single crystal of sapphire sliding on a large-grained tungsten disk with known grain orientations. Data for sapphire in the literature have indicated a dependence of wear of various surfaces upon crystallographic orientation (refs. 2 to 5). With large grains in a polycrystalline matrix, the influence of crossing grain boundaries upon slip behavior can be determined. This situation does not exist in single crystals.

Experiments were conducted in a vacuum in order to reduce the influence of surface

oxides. In working with various crystallographic planes, marked differences in oxidation rates do occur. Reference 17 shows this for tungsten. With repeated passes over the same track, these differences in oxidation rate could influence friction properties. The (100) surface of tungsten oxidizes more rapidly than the (111) and (110) surfaces.

Friction data for the (0001) plane, $[10\bar{1}0]$ direction of sapphire sliding on the large grains of a tungsten disk specimen in a vacuum of 10^{-10} millimeter of mercury, a load of 500 grams, and speed of 0.013 centimeter per second are presented in figure 5(a). Light loads and low speeds were used to avoid high interface temperatures, which can cause surface recrystallization. The friction data are plotted as a function of arbitrary angular position. The crystallographic planes and the directions for each grain are indicated at the top of the figure and the position at which the grain boundaries in the tungsten disk surface were crossed by the sapphire slider are indicated with vertical lines. The experiment was repeated at a slower speed (0.001 cm/sec); and the results obtained are presented in figure 5(b).

The most obvious result of figure 5 is the marked change in friction properties with a change in slip systems in moving from one grain to another. Further, on any particular plane a change in direction results in a change in friction.

The effect of plane upon friction can be seen from an examination of friction on various planes in a particular crystallographic direction of figure 5. The friction coefficients from figure 5(b) for three planes in the $[110]$ direction are presented in table II. This table indicates a higher coefficient of friction in the $[110]$ direction on the (110) plane than on the (100) plane. Based on the relatively high coefficient of friction for the three planes and the relatively small differences in friction in general it could be said that, in tungsten, the plane, per se, does not exhibit a marked influence upon the friction coefficient. Changing direction on a particular plane does, however, appreciably influence friction; for example, on the (100) plane moving from the $[110]$ to the $[100]$ direction gave a coefficient of friction of 1.23, while moving in the $[100]$ direction decreased the coefficient to about 0.8. This difference is significant.

It is interesting to note that for the (100), (110), and (023) planes the maximum in friction is observed in the $[110]$ direction. On both (110) planes, the maximum in friction (1.3 to 1.35) was in the $[110]$ direction and a minimum (0.7 to 0.75) was about 45° to 50° from the $[110]$ direction (fig. 5(b)).

Hardness measurements were made on the (100) plane of tungsten in two crystallographic directions $[100]$ and the $[110]$. These hardness data are presented in figure 6. Each point represents the average of a number of individual hardness determinations. Hardness is lowest in the $[110]$ direction and maximum in the $[100]$ direction. When the friction data from the (100) plane of figure 5(b) are plotted below the hardness data in figure 6, the correlation between hardness and coefficient of friction is readily seen. Increase in hardness is accompanied by a decrease in friction coefficient.

In figure 5(a) for the (110) plane, friction was high in the [110] direction and near the minimum in the [100].

The same friction data obtained at a slower speed in figure 5(b) still indicate a decrease in friction coefficient in the [100] direction, but "saw tooth" type data are obtained. A minimum and maximum are reflected about every 20° .

Resolved shear-stress calculations and yield-strength data obtained from reference 18 are presented in figure 7 for the (110) plane in the body-centered cubic system. If the shear stress to yield pressure relation is used, a maximum in friction should be anticipated at about 22.5° from either the [100] or the [110] direction. This probability could explain, in part, the pulses in friction noted between the [110] and the [100] direction. Hardness data obtained on the (110) plane in the [100] and [110] directions indicate a marked increase in hardness in the [100] direction over hardness values obtained in the [110] direction (from unpublished data of Marvin Garfinkle of Lewis). These results are in agreement with friction data and further indicate the anisotropic behavior of tungsten.

Considerable data exist in the literature to indicate a dependence of friction and wear on crystallographic orientation for sapphire in contact with various metals (refs. 2, 3, and 5). References 2 and 5 indicate that the wear rate for sapphire is greatest in the direction of easiest shear on the (0001) plane. Further, reference 5 indicates that friction force increases as the wear rate increases.

In order to determine what influence orientation of the sapphire would have on the friction of the slip system in the tungsten disk, a sapphire ball was oriented with the (10 $\bar{1}$ 0) plane sliding parallel to the tungsten disk surface and in the [0001] direction. The results obtained in this experiment are presented in figure 8(a). Changing the crystallographic orientation of the sapphire resulted in a marked change in friction behavior on the slip planes of the tungsten disk. For example, the differences in friction coefficient on the (100) planes in the [110] and [100] directions was greater than was obtained with basal orientation of the sapphire. Further the friction coefficients in the (110) planes were generally greater than was observed with sapphire sliding in the [10 $\bar{1}$ 0] direction on the (0001) plane (fig. 5(a)).

Friction Characteristics in Air

In vacuum, the adhesion of tungsten to sapphire can markedly influence friction data obtained, since a thin transfer film of tungsten (to sapphire) could be sliding on itself. In air, however, an appreciable thickness of tungsten oxide is present and this oxide could inhibit metal transfer to sapphire and thereby reduce friction coefficients. Further, various crystal planes of tungsten exhibit different oxidation rates, which could also

influence observed friction results.

A friction experiment was conducted in air with sapphire ($10\bar{1}0$) plane sliding in the $[0001]$ direction on the large-grain tungsten disk specimen, and the results obtained are presented in figure 8(b). The most marked effect, as might be anticipated, is a general reduction in friction coefficient on all crystallographic planes of tungsten. It is interesting to note, for example, that, on the (100) plane, the difference in friction between the $[110]$ and $[100]$ directions represents a change in friction coefficient from 0.6 to 0.3. On the same plane and in the same directions in vacuum with surfaces cleaned by electron bombardment, the friction decreased from 0.96 to 0.24. The friction in these two directions differed by a factor of two in air and four in vacuum, which indicates that the oxide plays a role other than a simple equivalent reduction of adhesion of the tungsten to sapphire in different crystallographic directions. A similar effect is noted on the (110) plane.

Surface Deformation

The tungsten disk specimen and the sapphire rider specimen were carefully examined after each experiment by etch pitting and taking surface photomicrographs. Figure 9 shows wear scars on planes and in grain boundary regions between grains. Figure 9(a) shows wear tracks on the (110) to (123) planes across the grain boundary region. Note that just short of the grain boundary on the (110) plane a wear scar is not present. Just beyond the grain boundary in the (123) plane the wear scar is very heavy, which indicates possible difference in hardness on the two sides of the boundary. Similar results have been observed by others on copper surfaces (ref. 20).

Figure 9(b) indicates a stick-slip-type motion may have occurred with the sapphire oriented with the ($10\bar{1}0$) plane parallel to the tungsten surface and sliding in the $[0001]$ direction. The periodic areas of severe and much less severe deformation would seem to indicate a change in the type of motion with change in crystallographic direction.

Figures 9(c) and (d) are photomicrographs of a grain boundary between the (123) and the (023) planes. The sliding of sapphire took place from the (123) to the (023) plane. The boundary has been displaced under deformation by sliding into the (023) plane.

Figure 10 shows the surface of the wear area to the basal plane of sapphire after its sliding on tungsten in vacuum. Tungsten metal was transferred from the disk to the sapphire surface by adhesion of the metal to sapphire (fig. 10(a)). When this area was etched in orthophosphoric acid for 20 minutes at 320°C , the pattern of figure 10(b) was obtained. The etch pit reagent was that used in reference 20. Figure 10(d) is a photograph obtained at a higher magnification of these etch pits.

Reference 2 indicated that in sliding experiments sapphire underwent plastic deformation at room temperature. This plastic deformation was used (ref. 5) to explain the

increase in friction with increased wear rate in the direction of easiest shear for sapphire. If, however, the etch pit concentration is examined on the wear area of sapphire in figure 10(b), the amount of deformation does not appear to be severe. With metal under the same condition, the wear scar would be extremely densely populated with pits.

Figure 11 contains photomicrographs of the (100) plane of tungsten with a Knoop hardness indenter mark on the electropolished surface; figure 11(a) is unetched and figure 11(b) is etch-pitted with Millner-Sass reagent. Observation of the profile of etch pit concentration is interesting. The etch pit concentration is maximum in the direction of the indenter short axis and minimum in the long or longitudinal axis of the indenter. The angle of deformation to the surface is maximum in the direction of the short axis (approx. 22°) and minimum in the tips of the longitudinal axis (approx. 4°). The concentration of etch pits indicates then, as would be anticipated, the degree of deformation and dislocation buildup.

The friction characteristics observed in this investigation indicate a marked dependence of friction upon crystallographic direction and orientation. With the aluminum oxide rider specimen sliding on the (100) plane of tungsten, a minimum in friction was noted sliding in the [100] direction and a maximum in the [110] direction. Examination of the (100) plane of the body-centered cubic crystal indicates that the atomic density is greatest in the [100] and least in the [110] direction on the (100) plane. Hardness measurements also indicate this same dependence upon direction.

Friction and hardness measurements for magnesium oxide in reference 1 indicate a maximum hardness and a minimum in friction coefficient in the [110] direction and the reverse in the [100] direction on the (100) plane. These results are opposite of those obtained in this investigation. The difference occurred because magnesium oxide is a face-centered cubic rather than a body-centered cubic crystal. With face-centered cubic crystals, atomic density is greatest in the [110] and least in the [100] direction on the (100) plane. Consequently, the results are as might be anticipated from a consideration of atomic density.

Further evidence for possible dependence of friction coefficient upon atomic density is gained from an examination of the friction data on the (110) planes in the [110] and [100] directions. On the (110) plane the coefficient of friction is greatest in the [110] and least in the [100] direction or approaching the [100] direction. Again in the body-centered cubic system, atomic density would be least in the [110] direction and greatest in the [100] direction.

SUMMARY OF RESULTS

The results obtained in this investigation with single crystals, oriented sapphire, sliding on a large-grain tungsten disk specimen were as follows:

1. Differences in friction were observed with changes in crystallographic planes and directions for body-centered cubic tungsten. Greater differences in friction were observed with changes of crystallographic direction on a particular plane than with changes of planes in a specific direction. Hardness and deformation behavior furthermore indicated the anisotropic behavior of tungsten.

2. Marked changes were observed in friction characteristics as grain boundaries of tungsten were crossed. These changes are due to the changes in crystallographic slip systems in moving out of a grain, across the boundary, and into another grain.

3. On the (100) plane of tungsten in the polycrystalline matrix, friction and hardness observed in the $[100]$ direction were markedly less than in the $[110]$ direction.

4. Differences in friction were observed on large-grain tungsten with a change in the crystallographic orientation of sapphire. The differences in frictional behavior of tungsten were less notable with (0001) plane of sapphire sliding in the $[10\bar{1}0]$ direction on tungsten than with the $(10\bar{1}0)$ plane parallel to the tungsten surface and sliding in the $[0001]$ direction.

5. Grain boundary displacement was observed under deformation between two tungsten grains.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 19, 1965.

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TABLE I. - ANISOTROPIC ELASTIC FACTOR FOR
VARIOUS BODY-CENTERED CUBIC ELEMENTS^a

Elements	Anisotropy, A (b)
Lithium	9.39
Sodium	8.14
Potassium	6.47
Iron	2.41
Tantalum	1.56
Tungsten	1.00
Vanadium	.779
Chromium	.714
Molybdenum	.757
Niobium	.513

^aRef. 14.

^b $A = 2C_{44}/C_{11} - C_{12}$ where C is an elastic constant; 1.00 for isotropic materials.

TABLE II. - INFLUENCE OF CRYSTALLOGRAPHIC PLANES
ON FRICTION IN LARGE-GRAINED TUNGSTEN

[Slider, sapphire; (0001) plane; $[10\bar{1}0]$ direction; load, 500 g; sliding velocity, 0.001 cm/sec; 10^{-10} mm Hg].

Crystal plane	Crystal direction	Coefficient of friction
(100)	$[110]$	1.23
(110)	$[110]$	1.35
(110)	$[110]$	1.30
(023)	$[110]$	1.11

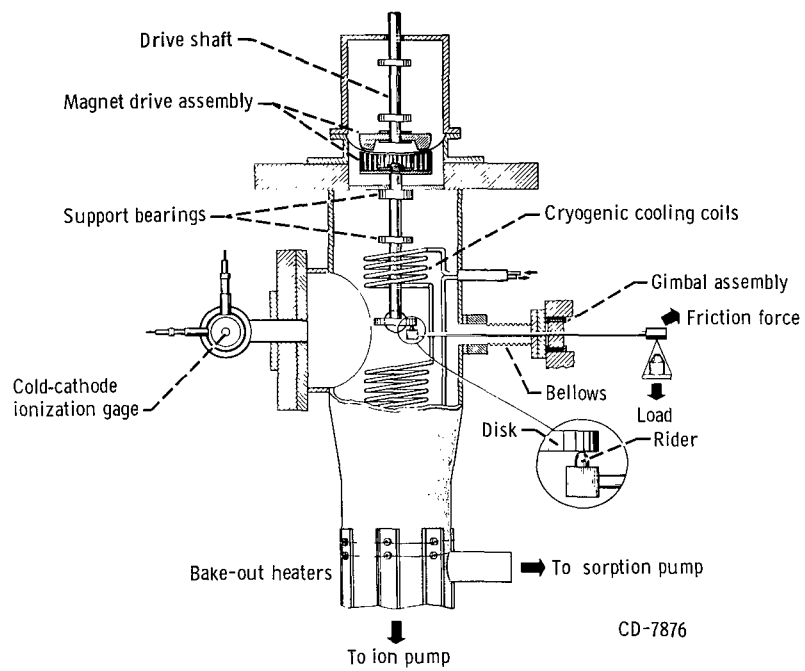
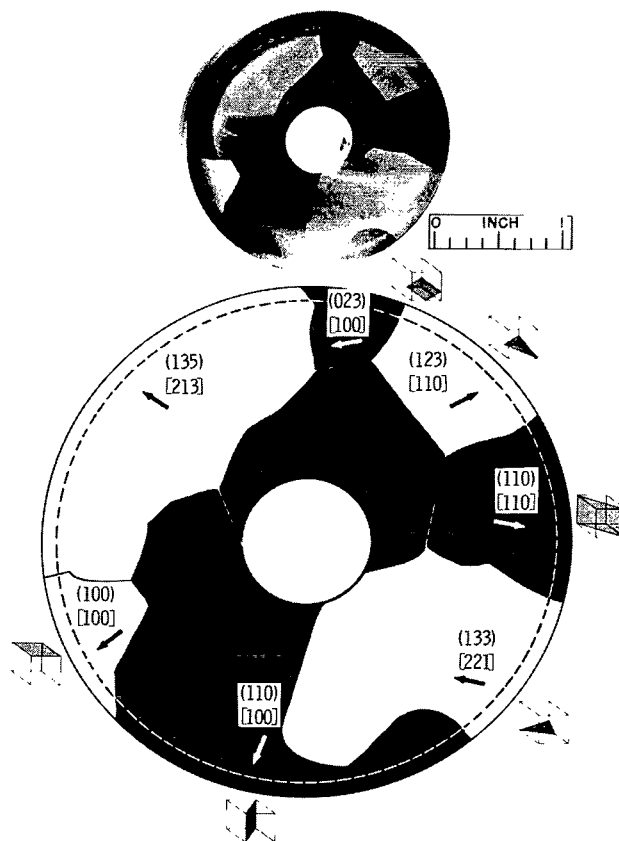


Figure 1. - High-vacuum friction and wear apparatus.



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Figure 2. - Large-grain polycrystalline tungsten disk specimen with crystallographic orientations and directions.

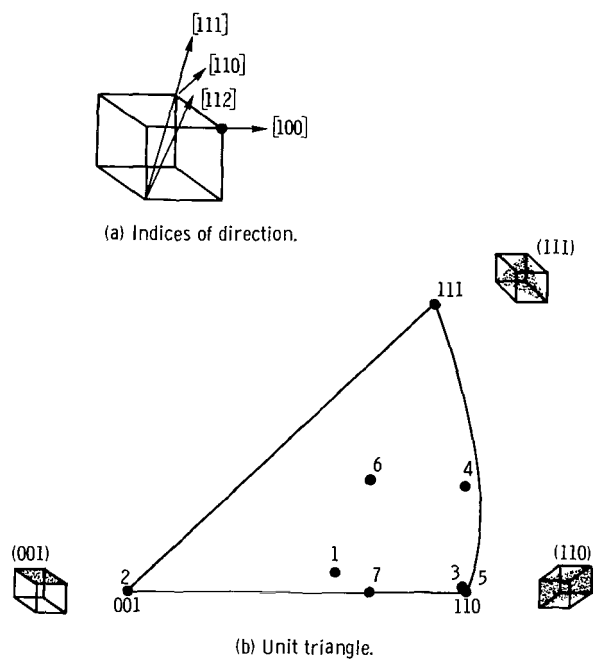
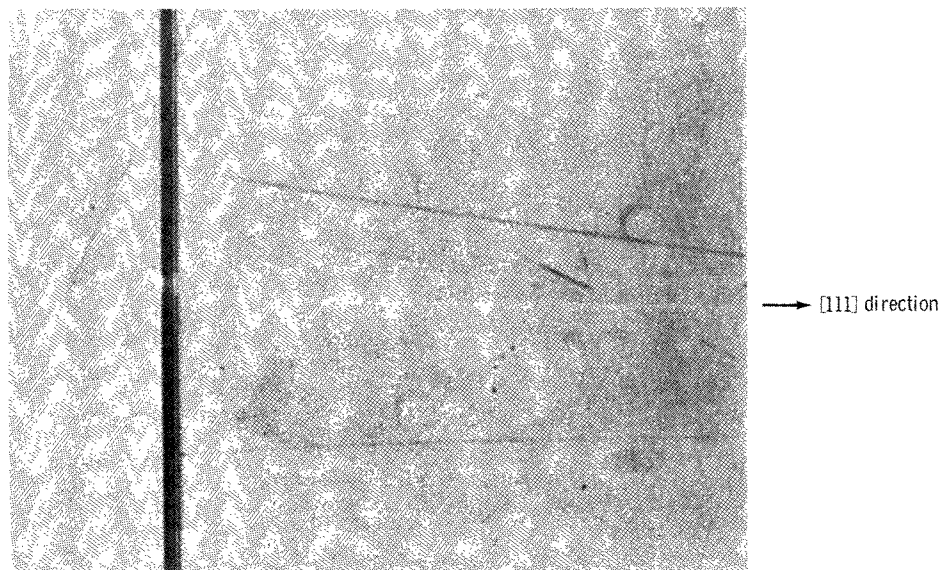
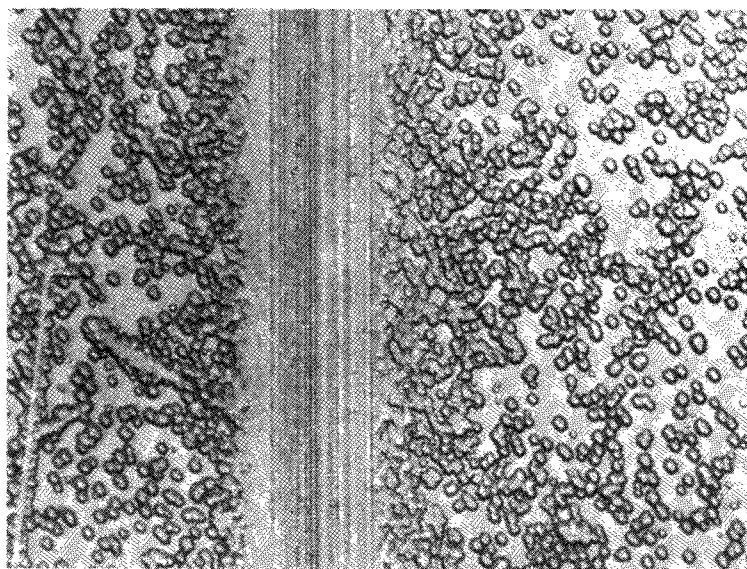


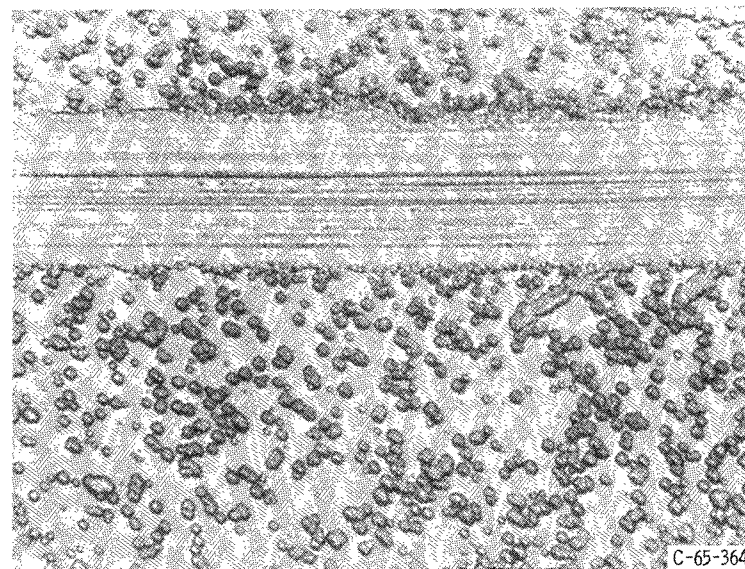
Figure 3. - Crystallographic direction and orientation on unit triangle for grains in tungsten disk specimen.



(a) Normal to and in $[111]$ direction.



(b) Normal to $[111]$ direction. Etch-pitted with Millner-Sass reagent. X700.



(c) In $[111]$ direction. Etch-pitted with Millner-Sass reagent. X700.

Figure 4. - Wear scars showing influence of direction of sliding of sapphire ball on (211) plane of tungsten single crystal in air. Ball diameter, $1/16$ inch; load, 60 grams; sliding velocity, 0.002 centimeter per second; temperature, 75° F. (Reduced 20 percent in printing.)

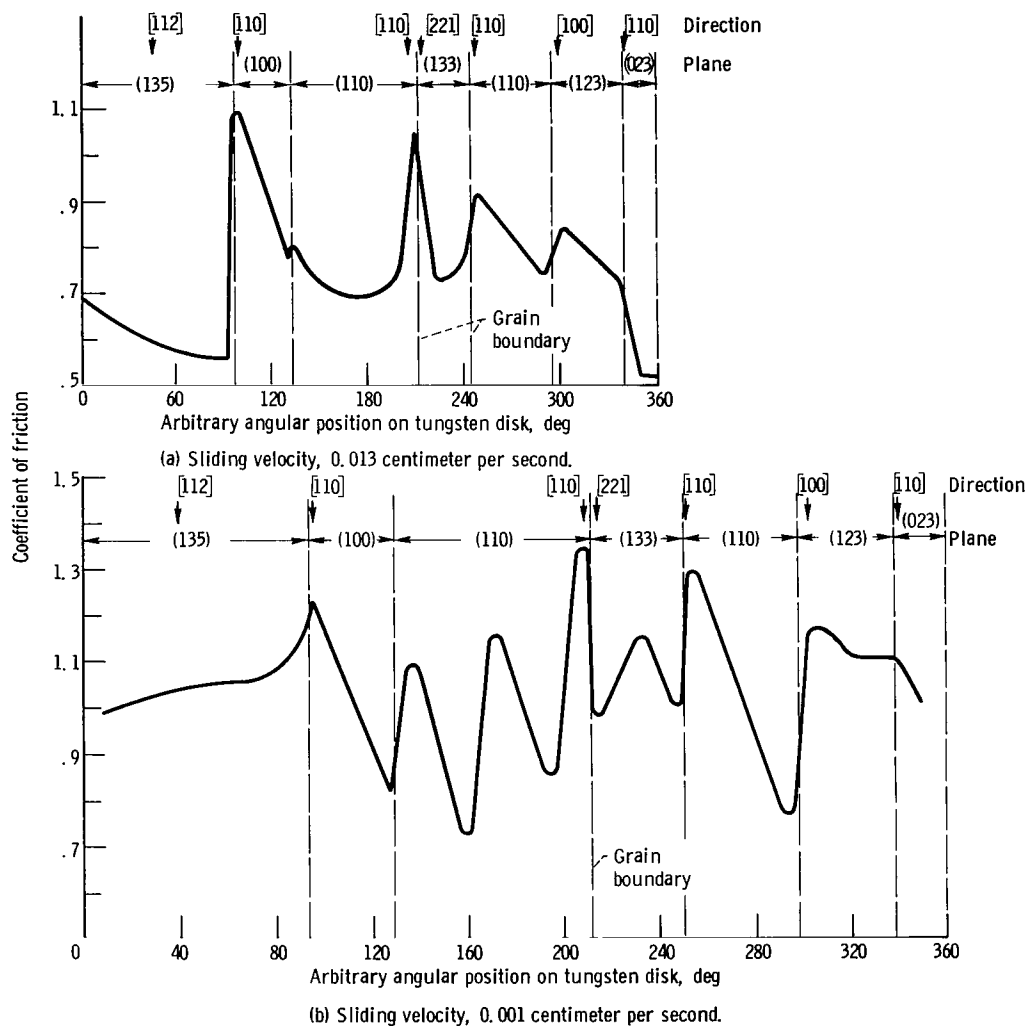


Figure 5. - Coefficient of friction of sapphire (0001) plane sliding in $[10\bar{1}0]$ direction on large-grain tungsten in vacuum (10^{-10} mm Hg). Load, 500 grams.

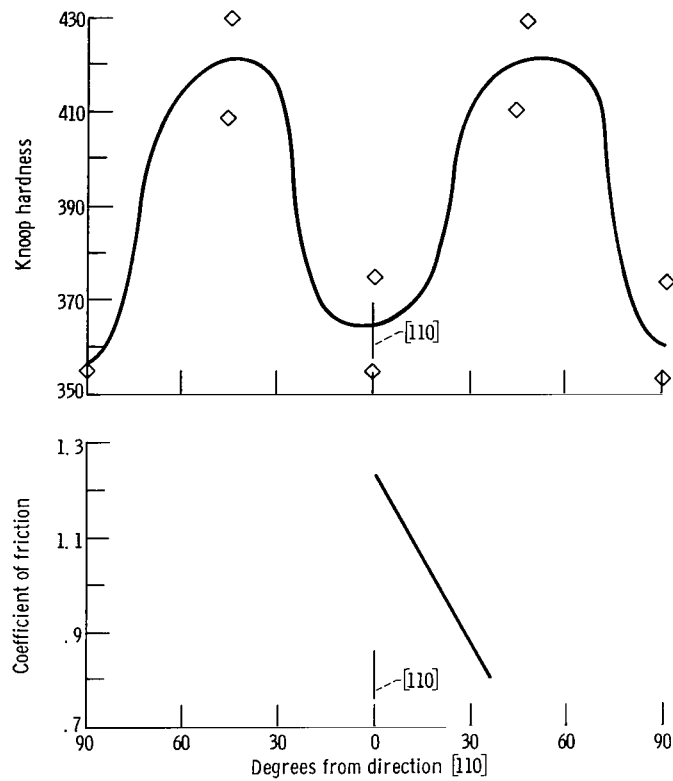


Figure 6. - Variation of hardness and friction coefficient in (100) plane of tungsten in two crystallographic directions. Friction experiments with (0001) sapphire rider sliding in $[10\bar{1}0]$ direction in vacuum (10^{-10} mm Hg). Load, 500 grams; sliding velocity, 0.01 centimeter per second.

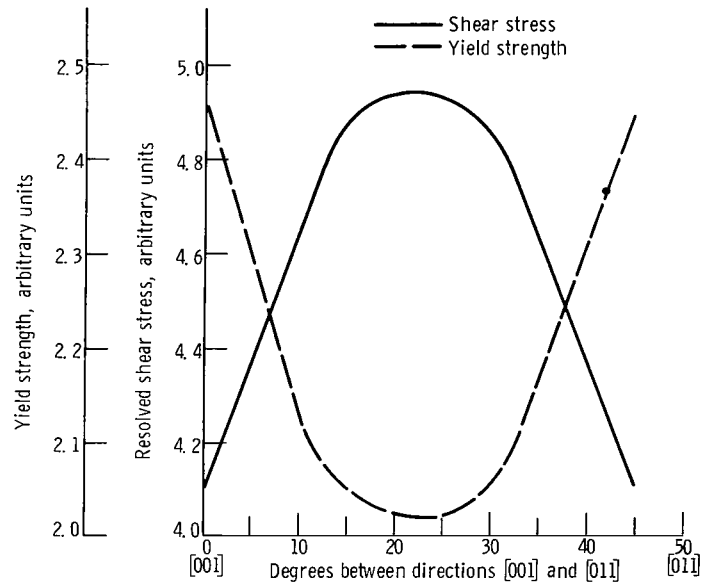


Figure 7. - Yield strength and shear stress in (110) plane of body-centered cubic crystals in various crystallographic directions (ref. 18).

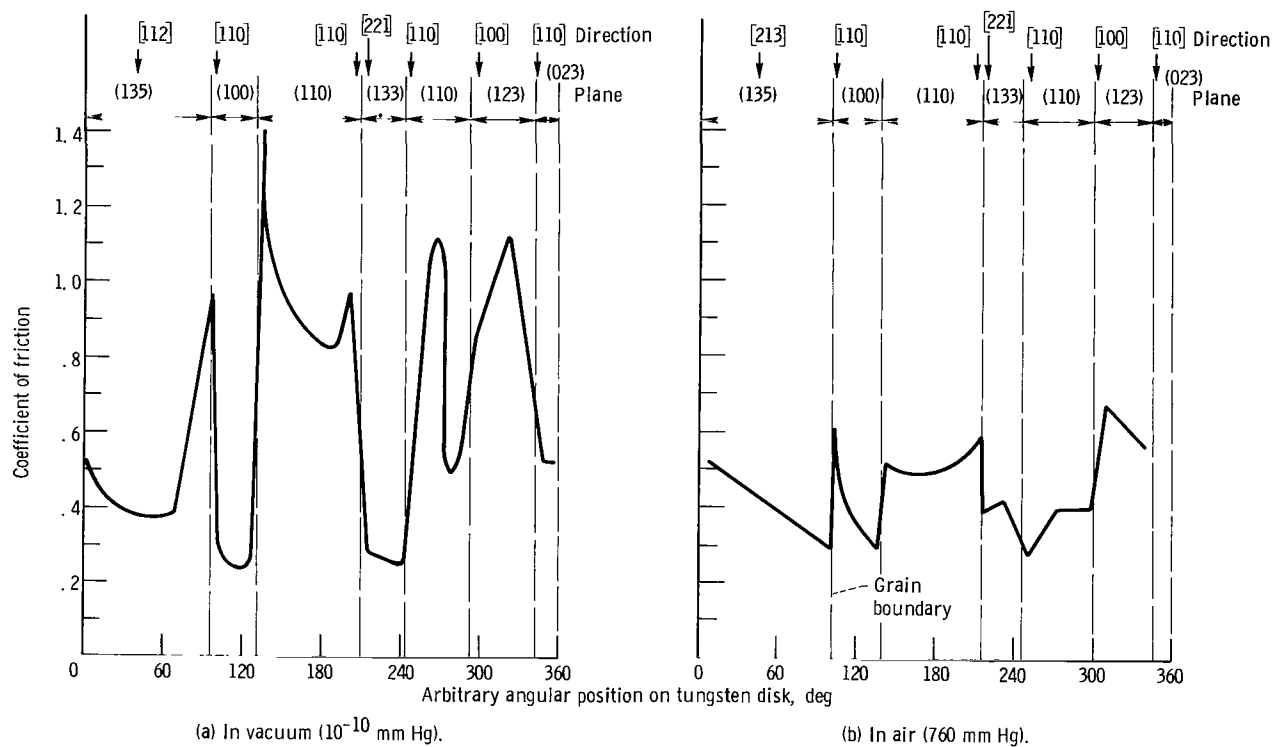
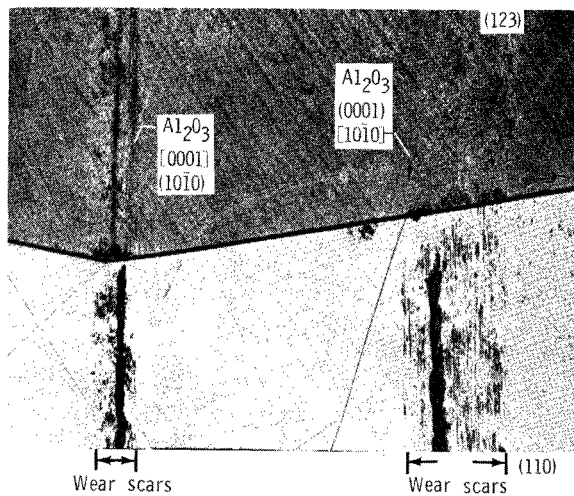
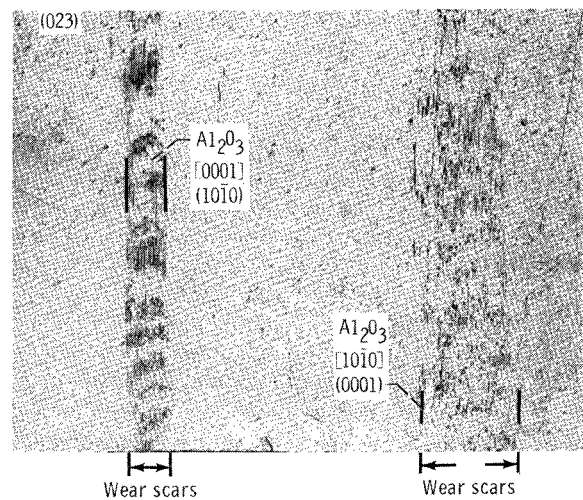


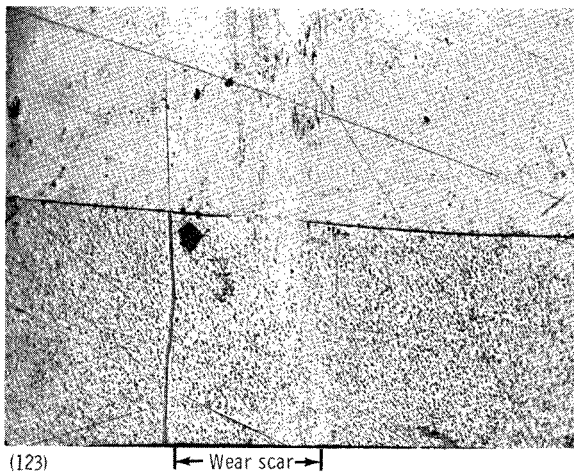
Figure 8. - Coefficient of friction of sapphire ($10\bar{1}0$) plane sliding in $[0001]$ direction on polycrystalline tungsten. Load, 500 grams; sliding velocity, 0.013 centimeter per second.



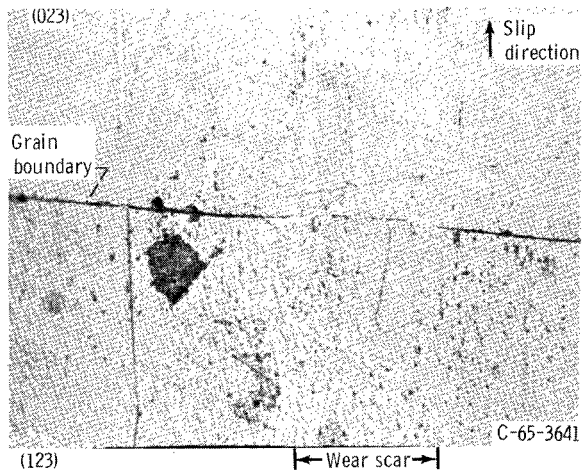
(a) Grain boundary of (110) to (123) planes. X250.



(b) Plane (110). X250.

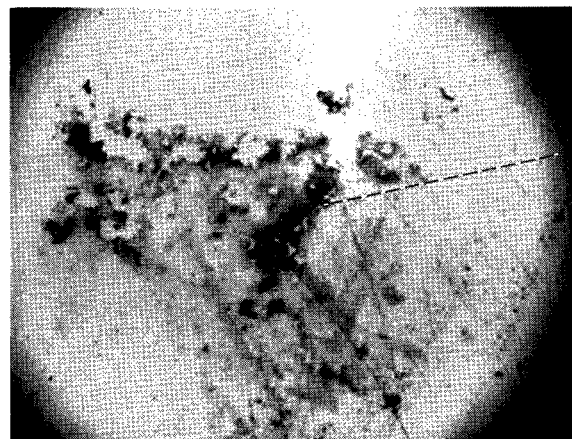


(c) Grain boundary of (123) to (023) planes. X250.

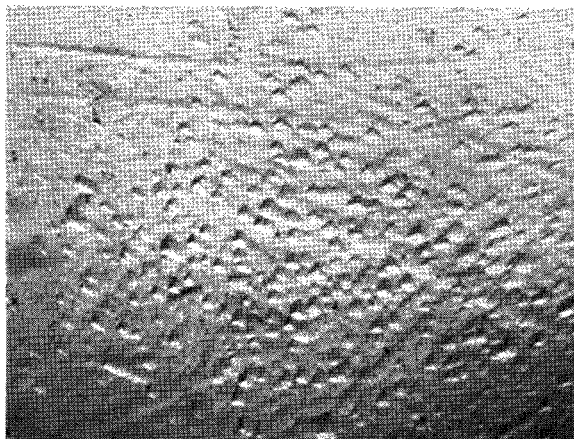


(d) Grain boundary of (123) to (023) planes. X875.

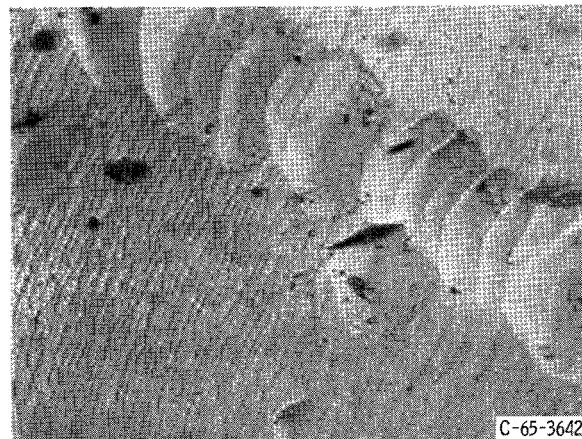
Figure 9. - Photomicrographs of grain and grain boundary regions of tungsten disk surface after wear experiments. (Reduced 20 percent in printing.)



(a) Unetched wear area. X140.

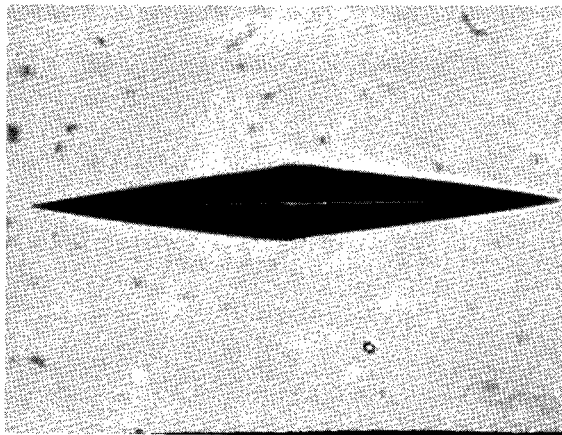


(b) Etch pitted wear area. X140.

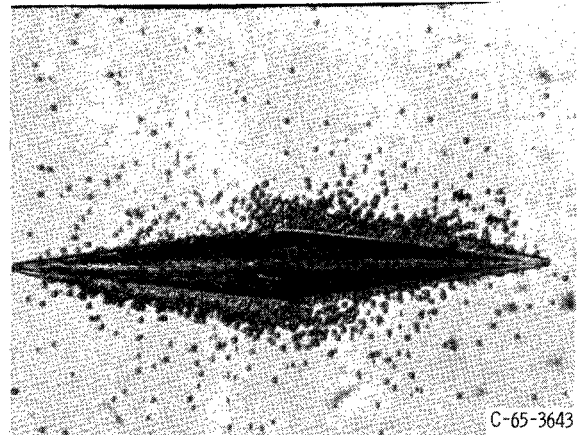


(c) Etch pits. X750.

Figure 10. - Sapphire single crystal wear scar in region of (0001) plane. Etch pits reveal dislocation concentrations. Ball diameter, 3/8 inch. (Reduced 20 percent in printing.)



(a) Unetched.



(b) Etched.

Figure 11. - Knoop indenter mark on (100) plane of tungsten both electropolished and unetched and electropolished and etched with Millner-Sass reagent for etch pits. X500. (Reduced 20 percent in printing.)

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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