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EFFECT OF SURFACE FILMS ON DEFORMATION OF ZINC SINGLE-CRYSTAL SURFACE DURING SLIDING

by Donald H. Buckley

Lewis Research Center

Cleveland, Ohio 44135

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16. Abstract

Sliding friction experiments were conducted on zinc single-crystal (0001) surfaces with sliding restricted to the $\langle 10\bar{1}0 \rangle$ directions. A ruby ball (9.0-mm diam) contacted the crystal surface under loads of from 50 to 300 g at sliding velocities from 0.02 to 14 mm/min. Surface films influenced plastic deformation. Oxides reduced plastic deformation, while organic films increased plasticity during sliding. Ion-implanted lead films also increased plasticity. Deformation mode was also influenced by the film present. With an oxide film, twins were observed, while with organic films, deformation was exclusively by slip. The higher the sliding velocity, the less the surface deformation. Track width increased with transition from the brittle to ductile state.

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SUMMARY

An investigation was conducted to determine the influence of surface films on the deformation of a zinc single crystal during sliding. A 9.0-millimeter diameter ruby ball slid on a zinc (0001) surface with sliding restricted to the $\langle 10\bar{1}0 \rangle$ directions. Loads of from 50 to 300 grams were used, and the sliding speed was varied from 0.02 to 14 millimeters per minute. Crystals were examined as cleaved, with an oxide-formed film, with the oxide film continuously removed, with hexadecane, with hexadecane containing stearic acid, and with ion-implanted lead.

The results of this study indicate that oxides on a zinc surface reduce deformation during sliding, while the presence of organic films increases plasticity or deformability. Lead films also increase plasticity. The mode of deformation was also influenced by the film. In dry sliding numerous twins formed, while with a lubricant present, deformation was exclusively by slip. The higher the sliding velocity, the less the amount of surface deformation. With increases in temperature the transition from the brittle to the ductile state resulted in an increase in surface deformation.

INTRODUCTION

Material studies have indicated that the presence of films on solid surfaces can markedly influence mechanical behavior (refs. 1 to 6). Mechanical deformation and understanding those conditions which influence it are important to adhesion, friction, and wear. The ability or inability of a material to deform plastically under certain conditions can influence contact area, plowing, fatigue, and fracture.

Metal oxides are known to increase the resistance of metals to mechanical deformation (refs. 4 and 5). The presence of organic films has a softening effect on metal surfaces in many instances (refs. 1 and 2) and yet a hardening or embrittling effect in others (ref. 6).

Sliding friction studies with inorganic crystals (lithium and calcium fluoride) indicate that the presence of organic films on these surfaces affects the nature of deformation during sliding (refs. 7 and 8). With aluminum metal a surface softening or increase in plasticity was noted with an organic film on the surface when the surface has been preoxidized (ref. 9).

Hexagonal metals with basal orientations (0001) parallel to the sliding interface exhibit lower friction and wear than cubic metals. The influence of surface films on the deformation of hexagonal metals is therefore of interest. Two hexagonal metals that have been examined and found to have mechanical behavior sensitive to surface films are zinc (ref. 5) and cadmium (ref. 4).

The objective of this investigation was to determine the influence of surface films on the deformation and friction of zinc. Sliding friction experiments were conducted on cleaved zinc (0001) surfaces with sliding in the $\langle 10\bar{1}0 \rangle$ direction. The effect of various films was examined. These films included oxides, organic fluids, organic friction films, and an ion-implanted metal.

MATERIALS

The zinc single crystal sliding surfaces used in these studies were obtained by cleaving a zinc single crystal (99.999 percent pure) cylinder into wafers 30 millimeters in diameter and 5 millimeters thick. The cleavage was done under liquid nitrogen along the natural cleavage plane of zinc, the (0001). The flat surfaces of the wafers were of the (0001) orientation. The cleavage process resulted in atomic flats of zinc. Other than for the presence of cleavage steps, this is a means of generating asperity-free metal surfaces.

The slider used in these experiments was a 9.0-millimeter-diameter ruby ball. It had a very smooth fire-polished surface.

The hexadecane fluid was 99.999 percent pure and was olefin free. It was percolated through activated alumina just prior to use.

APPARATUS

The apparatus used in this investigation is shown schematically in figure 1. The apparatus consisted basically of a microscratch hardness tester to which a drive motor with a gear reduction head was attached to provide uniform motion, at various speeds, of the crystal surface under examination. When sliding experiments were conducted under a fluid media, the zinc crystal specimen was placed in a dish of the fluid as indicated in figure 1. The dish was mounted to a steel plate that was moved by the drive assembly.

The ruby ball was mounted in an arm above the zinc crystal surface. Loading was accomplished by the application of dead weights directly over the ruby ball. The arm retaining the ball specimen had a strain-gage assembly for monitoring frictional force.

The crystals were heated by placing a small brass block containing two cartridge heaters directly under the specimen. A Chromel-Alumel thermocouple was mounted directly on the top surface of the crystal for monitoring specimen temperature.

Cooling of the specimens to -195° C was accomplished with liquid nitrogen. The data obtained at -78° C were obtained by placing the crystal on a block of dry ice.

All experiments consisted of a single pass across the crystal surface. The total distance of travel was approximately 20 millimeters.

The entire apparatus was enclosed in a clear plastic box. The box was purged for 20 minutes prior to each experiment with argon. During the experiment a slight positive pressure was maintained in the box.

EXPERIMENTAL RESULTS

Temperature Effects

The effect of temperature on the deformation of a zinc (0001) surface when sliding in the $[10\bar{1}0]$ direction was measured over a range of temperatures. The track width produced on the surface as a result of sliding is presented in figure 2. At -195° and -78° C the deformation at a fixed load of 200 grams was less than 0.1 millimeter with a single pass of the ball across the surface. While the track width was essentially the same at both temperatures, fracture cracks were observed in the wear track at -195° C. The cracks were normal to the direction of sliding which indicated that they were associated with the $\{10\bar{1}0\}$ planes. Cleavage or fracture is normally not observed along this plane in pure zinc but is in zinc alloys containing as little as 0.13 percent cadmium (ref. 10). These cracks were not present at -78° C.

When the temperature was increased to 23° C, a very marked increase in track width occurred. The width continued to increase with further increases in temperature. The large increase in deformation of the surface when the temperature was increased from -78° to 23° C is associated with the brittle to ductile transition in zinc. At -78° and -195° C zinc, during the sliding process, behaves in a brittle manner. Plasticity and track width increase at 23° C and above because the zinc acts like all ductile metals above the brittle to ductile transition (ref. 11).

Twinning was observed to occur at 23° C in and along side the wear track. At the higher temperatures, 200° to 300° C, twins were not observed, which indicated that the critical resolved shear stress for slip must have been less than that for deformation twinning.

Effect of Surface Oxides

The effect of surface oxide (ZnO) on the deformation of a zinc crystal surface during sliding was determined. Three crystal wafers, all from the same cylinder, were examined. One surface was examined in dry sliding in the as-cleaved condition. The second crystal surface was oxidized in steam, and the third crystal was examined as cleaved under a 5-percent hydrochloric acid solution to dissolve the surface oxide continuously. The deformation track widths produced during these sliding experiments at various loads are presented in figure 3.

The data of figure 3 indicate that deformation during sliding was greatest with continual oxide removal under the 5-percent hydrochloric acid in water solution, intermediate with normal oxide on the as-cleaved crystal, and least with the preoxidized surface. Thus, the presence of the zinc oxide on its surface reduces deformation during sliding.

In addition to simple plastic deformation, some adhesion of zinc to the ruby surface occurred with the as-cleaved specimens. This could alter the nature of the deformation track. With both the preoxidized and the hydrochloric acid solution experiments, no evidence for adhesive transfer was noted. In fact, with the hydrochloric acid solution and its oxide removal, the friction coefficient was lower at all loads investigated than for the oxidized surface, as indicated by the data of figure 3.

The influence of a hydrocarbon lubricant on the deformation of the as-cleaved zinc crystals was examined. Both hexadecane and hexadecane containing 0.2 percent stearic acid were used. The results obtained together with the curves obtained for dry sliding are presented in figure 4.

The data of figure 4 indicate that the presence of surface active organics increases the plasticity and the resultant surface deformation during sliding. This is a manifestation of the Rebinder effect seen during a sliding friction experiment (refs. 1 and 2). At loads less than 200 grams the stearic acid reduced the deformation relative to the hexadecane alone. The higher the load, the less the difference, and at 200 grams and above, the presence of the stearic acid did not alter the observed behavior.

It is worthy of note in figure 4 that the curves for dry sliding and the lubricated condition converge to nearly the same value at a load of 300 grams. These results are as might be anticipated, that is, the higher the load, the greater the bulk deformation, and the less significant the surface influences.

The effect of steam preoxidation on the deformation in the presence and absence of hexadecane was also examined. The presence of the hexadecane also alters deformation behavior with the oxidized surface, as indicated by the surface profile traces of figure 5. The track is wider and not as deep with the hexadecane present.

When the zinc surface has been preoxidized, even the nature of the surface deformation in hexadecane is changed. This is evidenced in figure 6. In figure 6(a) with an unoxidized surface, plastic flow takes place with no evidence for twin formation. With the oxidized surface, however, a "ladder" of needlelike twins can be seen (fig. 6(b)). The thin lines running normal to the sliding direction are actually twins and not slipbands.

The presence of stearic acid in hexadecane would normally be expected to reduce the friction coefficient, and the data of figure 7 indicate that it does for zinc. At light loads the difference in friction in the presence and absence of stearic acid is greater than that at the heavier loads.

The total amount of plastic deformation is sensitive to strain rate just as observed in conventional mechanical testing of metals. Thus, it is not surprising to find in figure 8 that the track width generated on the zinc surface is dependent upon sliding velocity. Increasing the sliding velocity increases the strain rate, and this results in a decrease in the total amount of deformation observed.

Thin Metallic Films

In addition to natural oxides and organic films, thin metallic films have been observed to influence deformation. In most cases these films have been noted to increase the resistance to deformation (refs. 2 and 3). Most of the films applied to zinc have been capable of forming compounds or are somewhat soluble. A film of about 200×10^{-10} meter (200 Å) of lead was ion-plated to a zinc crystal surface (ion implantation). Lead and zinc are practically insoluble (ref. 12).

Sliding friction experimental results for a lead-coated zinc surface at various loads are presented in figure 9 along with a reference curve for the as-cleaved crystal surface. The results indicate that the presence of the lead film increases the deformability of the surface. Apparently, in the ion-implantation process the lead weakens the (0001) stable zinc lattice sufficiently to result in the increase in plasticity.

Figure 10 presents photographs of zinc (0001) surfaces after sliding under three conditions: (a) dry, (b) with the ion-plated lead, and (c) with hexadecane containing stearic acid. With dry sliding (fig. 10(a)) twins developed with sliding, and evidence of adhesion of zinc to the ruby ball is indicated by the darkened areas in the center of the photograph; these darkened areas represent pits or craters formed when the zinc transferred to the ruby. A cleavage step normal to the sliding track is shown in the upper portion of the photograph. Twins on a zinc surface with sliding have been observed by others (ref. 13).

In figure 10(b) the presence of lead in the zinc surficial layer has resulted in an inhibiting of twin formation. Close examination reveals the presence of twins within the wear track, but the number is considerably reduced over that in figure 10(a).

When a lubricant, hexadecane with 0.2 percent stearic acid, is used, there is a complete absence of twins (fig. 10(c)). Thus, the presence of various surface films not only quantitatively alters the surface deformation but also affects the mode. The absence of twins in figure 10(c) would indicate that deformation is entirely by slip.

DISCUSSION

The normal cleavage plane for zinc is the basal (0001) plane (ref. 10). In sliding friction studies in this investigation at -195° C, cleavage or fracture was observed along $\{10\bar{1}0\}$ planes. An examination of the literature indicates that while zinc cleaves along only the (0001) planes, zinc alloys containing 0.03 percent cadmium will also cleave along the $\{10\bar{1}0\}$ planes (ref. 14). The zinc employed in these studies was 99.999 percent pure, and the observed fracture cannot therefore be related to the presence of alloying elements but must be attributed to the forces operating on the $\{10\bar{1}0\}$ planes as a result of the sliding process. Tangential motion associated with the sliding process will exert a tensile force on these planes.

The brittle to ductile transition of the zinc surface results in an increase in plasticity in the sliding friction experiment. Studies with other metals have indicated an influence of this transition on friction coefficient (ref. 15) and surface deformation during sliding (ref. 16). With a body-centered cubic iron-silicon alloy in reference 16, no evidence for brittle fracture or cleavage was observed even at -195° C, while in this study with the hexagonal metal zinc fracture was observed. This may be related to the larger number of slip systems in the body-centered cubic system allowing for deformation to occur.

In figure 3 the presence of oxides increased the resistance of the zinc surface to plastic deformation. The influence of oxides on increasing the resistance of hexagonal metals to plastic flow in mechanical testing has already been observed (refs. 4 and 5). It is interesting to note the marked effect it produces on plastic flow during sliding in figure 3 and that the resistance is a function of film thickness. With continuous oxide removal under the hydrochloric acid, deformation is greatest. With the deliberately preoxidized surface, deformation is least. It cannot be argued that adhesion is playing a role in the observed results because in figure 4 the friction force for the oxide-covered surface is greater than that for the surface run under the hydrochloric acid solution. This difference indicates that the amount of metallic adhesion is greater in dry sliding over the oxide than it is in sliding under the hydrochloric acid solution. Plowing is greater in the latter case and on that basis alone the friction force would be expected to be higher. The fact that it is not indicates that the adhesive force must be low. Thus, the presence of oxides on metal surfaces increases the resistance of the surface to plastic deformation.

Corrosion is not believed to have influenced the results obtained in figure 3 under hydrochloric acid. Experiments were conducted with a 10-percent hydrochloric acid solution, and the track widths measured were within the experimental error of values presented in figure 3 for a 5-percent acid solution. If corrosion played a part, track widths should have been larger in the 10-percent solution.

In contrast to oxides on a zinc (0001) surface, which decrease surface deformation, the presence of hexadecane or hexadecane with 0.2 percent stearic acid actually causes an increase in the surface plasticity. This observation is commonly referred to as the Rebinder effect and has been observed with a number of materials in the presence of organic fluids (refs. 1 and 2).

The sensitivity of the zinc surface to strain rate as reflected by a change in the sliding velocity in figure 8 indicates that there are conventional mechanical property measurements such as strain-rate measurements of materials which may provide an insight into the effect of sliding speed. This is particularly true since a similar observation was made with an inorganic crystal in reference 8. This may, in part, explain why at slow sliding speeds friction force is usually higher than at high sliding speeds. At the low speeds, plowing is greater than at high speeds. The significance of this effect will be influenced by the creep characteristics of the materials involved.

The experimental results of figure 9 indicated that ion-implanted lead increases the ductility of the zinc (0001) surface. In reference 2 both gold- and copper-plated zinc crystals had slightly higher tensile strengths than the same crystal without plating. Both gold and copper have appreciable solubilities with zinc, and gold is capable of forming compounds (ref. 12). Lead is nearly insoluble in zinc and does not form compounds (ref. 12). While the two former metals, namely, gold and copper, could conceivably result in surface strengthening due to solid solution or compound formation, this cannot occur with lead.

The (0001) plane of zinc is the atomically most dense and electronically the most stable, having the lowest surface energy. Ion implantation of foreign atoms can only introduce defects and disrupt zinc cohesive bonds. This must be sufficient to weaken the resistance of surficial layers to deformation. The penetration of foreign atoms into the lattice will also reduce the stacking fault energy.

CONCLUSIONS

Based upon the sliding friction experiments conducted in this investigation with a zinc single crystal (0001) surface sliding $\langle 10\bar{1}0 \rangle$ in the directions with various surface films present, the following conclusions are drawn:

1. The deformation of zinc single crystal surfaces is environment sensitive. The presence of oxide increases the resistance of the surface to plastic deformation during sliding, while the presence of an organic film increased surface plasticity. Ion-implanted lead also was observed to increase plastic flow.

2. Sliding velocity affects the amount of plastic deformation observed. The higher the sliding speed, the lower the amount of surface deformation.

3. The brittle to ductile transition influences the deformation. Deformation is markedly less in the brittle zone. Further, fracture cracks were observed at -195° C along $\{10\bar{1}0\}$ planes. Fracture along these planes had not been previously observed. The formation of fracture cracks had not been observed with a body-centered cubic alloy at the same temperature.

4. The mode of deformation with sliding was sensitive to the surface film present. With oxide deformation twins formed readily. With a lead film the formation of twins was reduced and with an organic lubricant, deformation twins were completely absent from the deformation track.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 17, 1971,
129-03.

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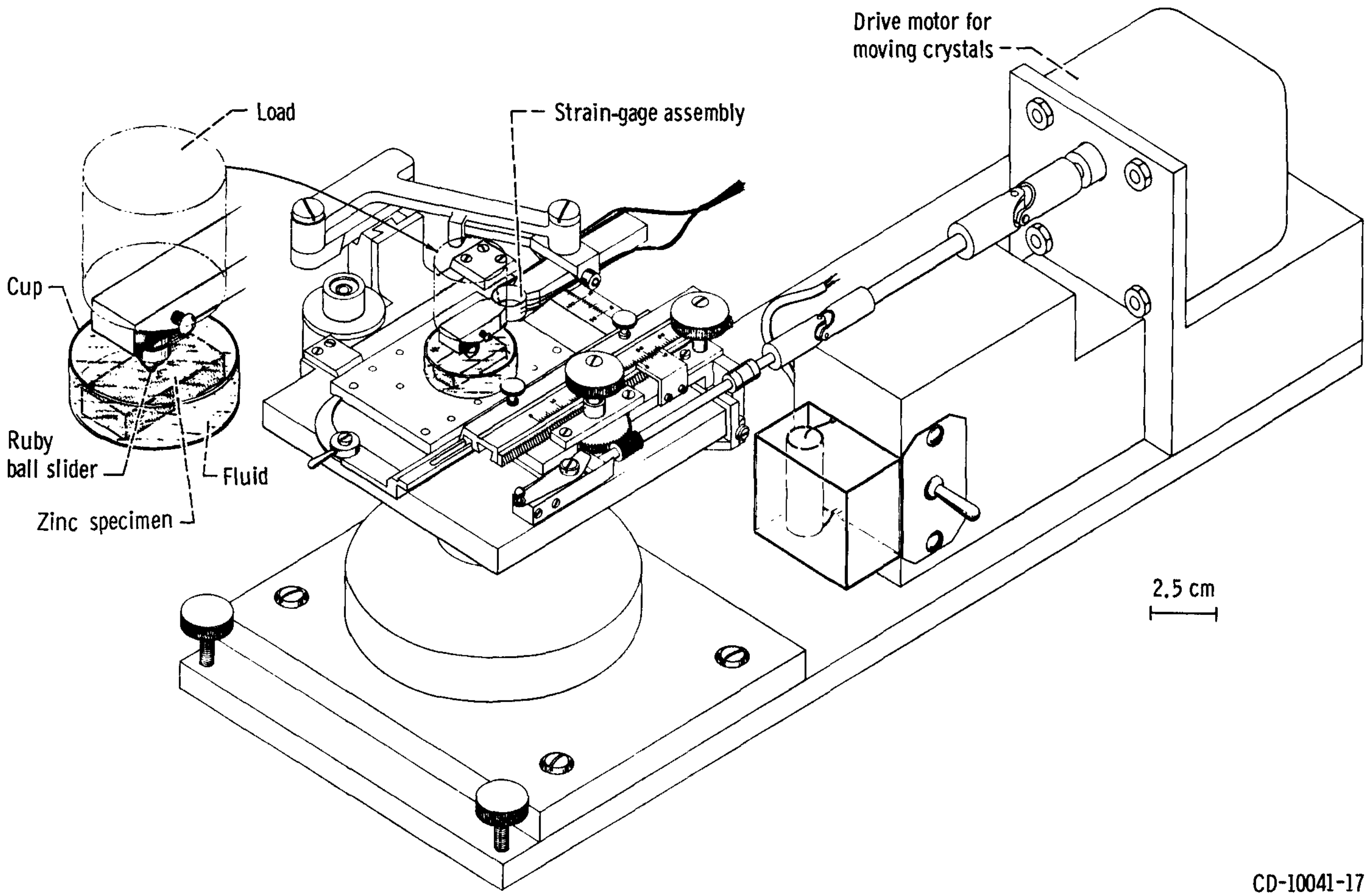


Figure 1. - Sliding friction apparatus.

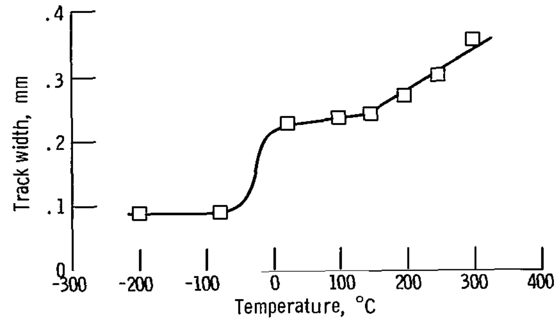


Figure 2. - Width of deformation track produced by surface in sliding ruby ball on zinc single crystal (0001) surface in [10$\bar{1}$0] direction at various temperatures. Sliding velocity, 1.4 millimeters per minute; load, 200 grams; dry argon atmosphere.

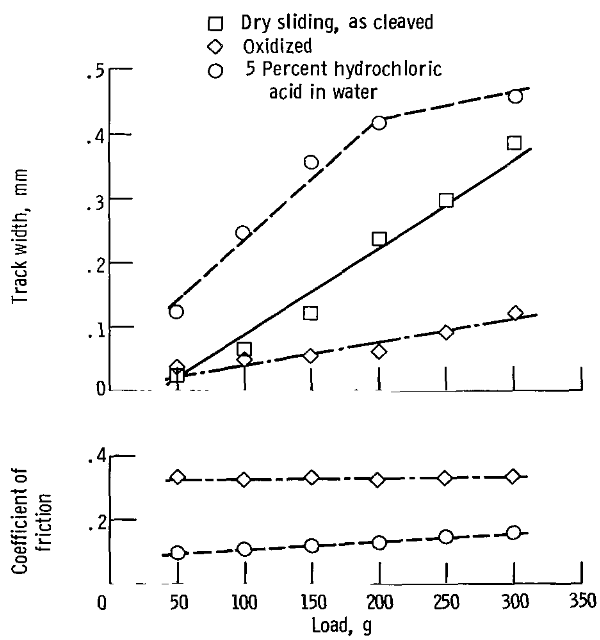


Figure 3. - Width of wear track and coefficient of friction produced with ruby ball sliding on zinc single crystal (0001) surface in [10$\bar{1}$0] direction. Sliding velocity, 1.4 millimeters per minute; temperature, 23° C; dry argon atmosphere.

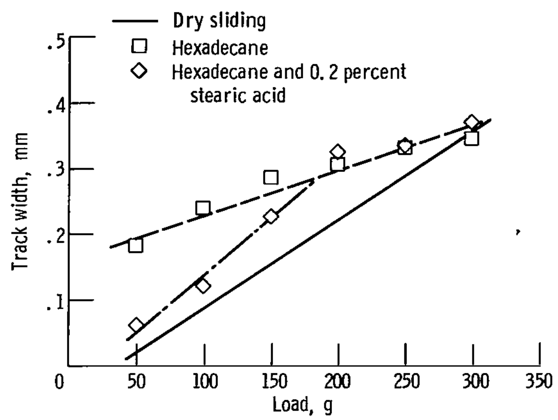
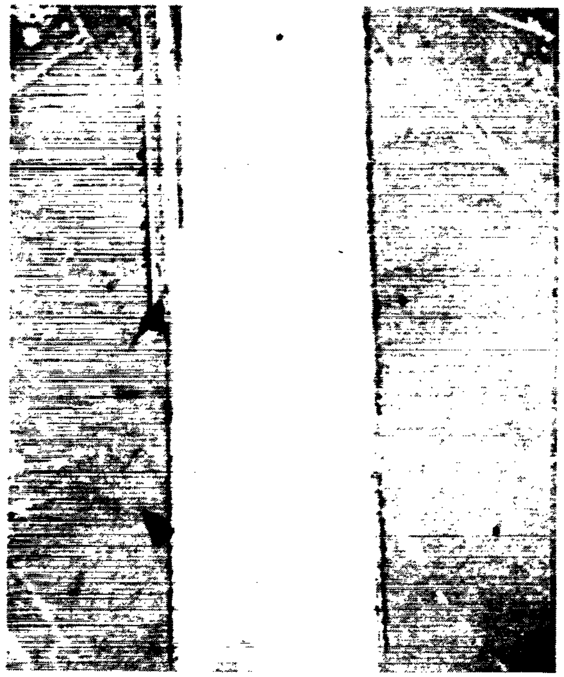


Figure 4. - Width of deformation track produced by sliding ruby ball on zinc single crystal (0001) surface in [10$\bar{1}$0] direction. Sliding velocity, 1.4 millimeters per minute; temperature, 23° C; dry argon atmosphere.

(a) Preoxidized dry sliding.

(b) Preoxidized hexadecane.

Figure 5. - Surface profile of deformation tracks formed in sliding ruby ball on zinc (0001) surface in [10$\bar{1}$0] direction.



(a) Unoxidized surface.

(b) Oxidized surface.

Figure 6. - Deformation tracks developed on zinc (0001) surface in sliding contact with ruby ball under 200-gram load and in hexadecane.

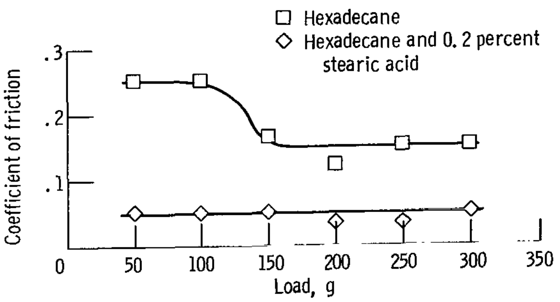


Figure 7. - Coefficient of friction with ruby ball sliding on zinc single crystal (0001) surface in [10$\bar{1}$0] direction. Sliding velocity, 1.4 millimeters per minute; temperature, 23° C; dry argon atmosphere.

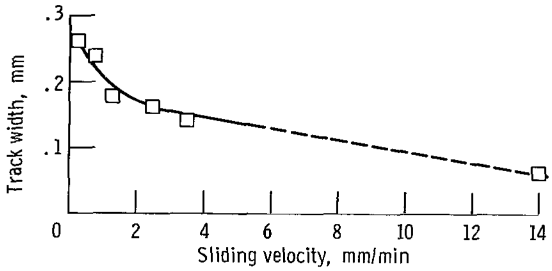


Figure 8. - Width of deformation track produced by sliding ruby ball on zinc single crystal (0001) surface in [10$\bar{1}$0] direction at various sliding velocities in hexadecane. Sliding velocity, 1.4 millimeters per minute; temperature, 23° C; dry argon atmosphere.

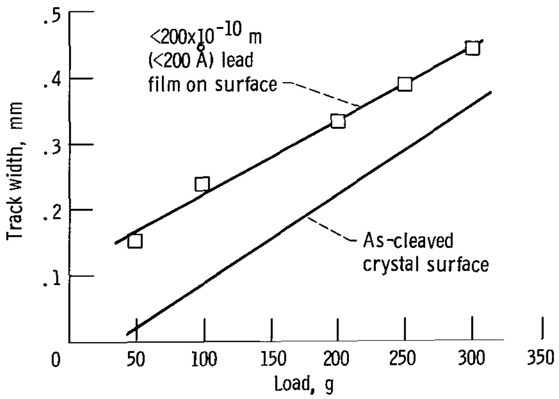
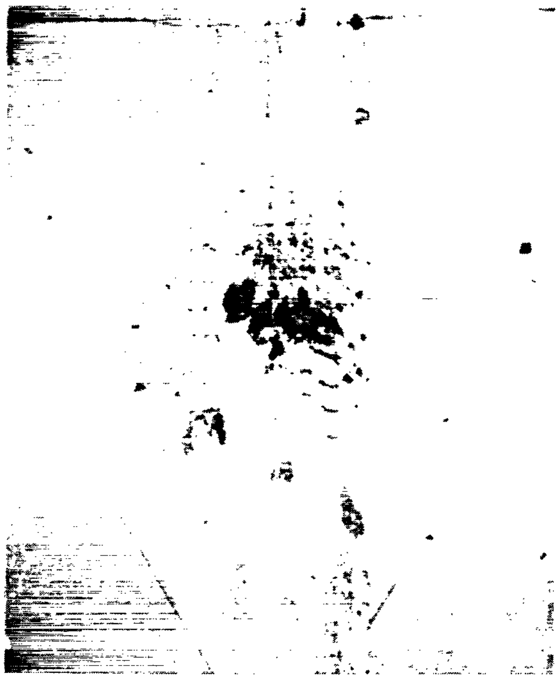
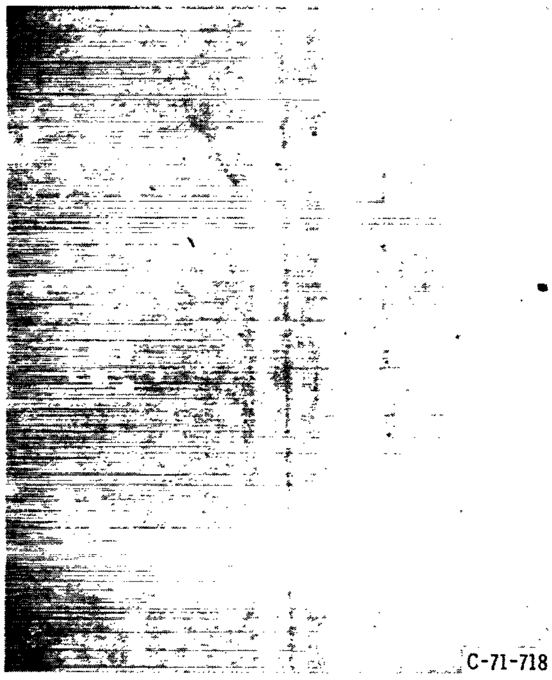


Figure 9. - Width of deformation track produced by sliding ruby ball on zinc single crystal (0001) surface in [10$\bar{1}$0] direction with ion-plated lead film. Sliding velocity, 1.4 millimeters per minute; temperature, 23° C; dry argon atmosphere.



(a) Dry sliding.

(b) Ion-plated lead film (appox. 200×10^{-10} m; 200 A).



(c) Hexadecane and 0.2 percent stearic acid.

Figure 10. - Deformation tracks formed on zinc (0001) surface in [10$\bar{1}$0] direction with ruby ball slider. Load, 200 grams.

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