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EFFECT OF SURFACE ACTIVE MEDIA ON FRICTION, DEFORMATION, AND FRACTURE OF CALCIUM FLUORIDE

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16. Abstract

Sliding friction experiments were conducted on the (111) cleavage face of calcium fluoride single crystals in various environments including hexadecane, oleic acid, water, and dimethylsulfoxide. The results indicated that the friction and deformation behavior of calcium fluoride is extremely environment sensitive. The presence of the oleic acid and dimethylsulfoxide increased resistance to deformation of the calcium fluoride surfaces. The amount of deformation that occurred during sliding was strain-rate sensitive; the higher the sliding velocity the lower the amount of surface deformation. The mechanical behavior of the surface was sensitive to extremely small concentrations of surfactant.

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EFFECT OF SURFACE ACTIVE MEDIA ON FRICTION, DEFORMATION, AND FRACTURE OF CALCIUM FLUORIDE

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SUMMARY

An investigation was conducted to determine the influence of surface active fluids on the friction and deformation of calcium fluoride during sliding. The fluid environments examined were hexadecane, oleic acid, water, and dimethylsulfoxide. A 2.0-millimeter-diameter sapphire ball slid across the (111) cleavage face of calcium fluoride, submerged in the fluid, at speeds of 0.005 to 0.5 millimeter per second and loads of from 100 to 600 grams.

The results of the study indicated that the friction and deformation of calcium fluoride is very environment sensitive; the presence of oleic acid or dimethylsulfoxide on its surface increased the resistance of the surface to deformation. The amount of surface deformation that occurred with sliding was strain-rate sensitive; as the sliding velocity was increased, the extent of surface strain decreased. Extremely small concentrations of surface active agent were sufficient to influence friction and deformation. With dimethylsulfoxide, concentrations of 10^{-8} to 10^{-10} molar were observed to exert an influence.

INTRODUCTION

In the field of tribology, the presence of surface films as well as the mechanical behavior of surfaces are extremely important in understanding adhesion, friction, and wear of materials. Studies in material science have shown that the mechanical behavior of solid surfaces may be very markedly dependent on the film present on the surface (refs. 1 to 10). Thus, an understanding of this interrelation may help in the interpretation of adhesion, friction, and wear behavior.

The presence of surface films can influence the deformation behavior of surfaces by various mechanisms. These include (1) increase in strength by dissolution of the solid surface, Joffe effect (ref. 11); (2) surface hardening, Roscoe effect (refs. 12 and 13);

(3) surface softening, Rebinder effect (refs. 14 and 15); and (4) liquid metal embrittlement (ref. 16). Some of these mechanisms have been observed to affect inorganic materials and some metals, and other mechanisms, both types of surfaces.

Sliding friction experiments have shown the influence of surface active organic liquids on the deformation and fracture in lithium fluoride during sliding (ref. 17). Similar studies with aluminum (ref. 18) have shown an effect of surface active organics on the deformation of aluminum during sliding when the aluminum surface contained a 100-angstrom- (10^{-10}-m-) thick oxide. Aluminum also underwent surface embrittlement in the presence of liquid metals during sliding (ref. 18).

An inorganic solid of particular interest because of its usefulness as a high-temperature lubricant is calcium fluoride (refs. 19 and 20). Determination of the influence of surface active species on the deformation behavior of calcium fluoride could aid in the understanding of its lubrication behavior.

The objective of this investigation was to determine the influence of various fluid media on the friction and deformation of calcium fluoride crystals during sliding. The sliding experiments were conducted on the (111) cleavage face of calcium fluoride in hexadecane, oleic acid, water, dimethylsulfoxide (DMSO), and various concentrations of dimethylsulfoxide in water. The slider was a 2.0-millimeter-diameter sapphire ball. The loads employed ranged from 100 to 600 grams, and the sliding speeds ranged from 0.005 to 0.5 millimeter per second.

Studies of material property behavior can be made and more readily understood on single crystals than on polycrystals where many orientations and grain boundary effects must be considered. Observations made on the behavior of single crystal surfaces with respect to the influence of surface active species on deformation with sliding will certainly be applicable to the polycrystalline case.

APPARATUS

The apparatus used in this investigation is shown schematically in figure 1. The apparatus consisted basically of a MicroBierbaum hardness tester (ref. 21) 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. The rectangular (25 by 12 by 7 mm) calcium fluoride specimen was mounted in a dish of the fluid under study. The dish was mounted to a steel plate that was moved by the drive assembly.

The slider specimen, a 2.0-millimeter-diameter sapphire ball, was mounted in an arm above the calcium fluoride crystal surface. Loading was accomplished with the application of dead weights directly over the rider. The arm retaining the slider specimen had a strain-gage assembly for monitoring frictional force.

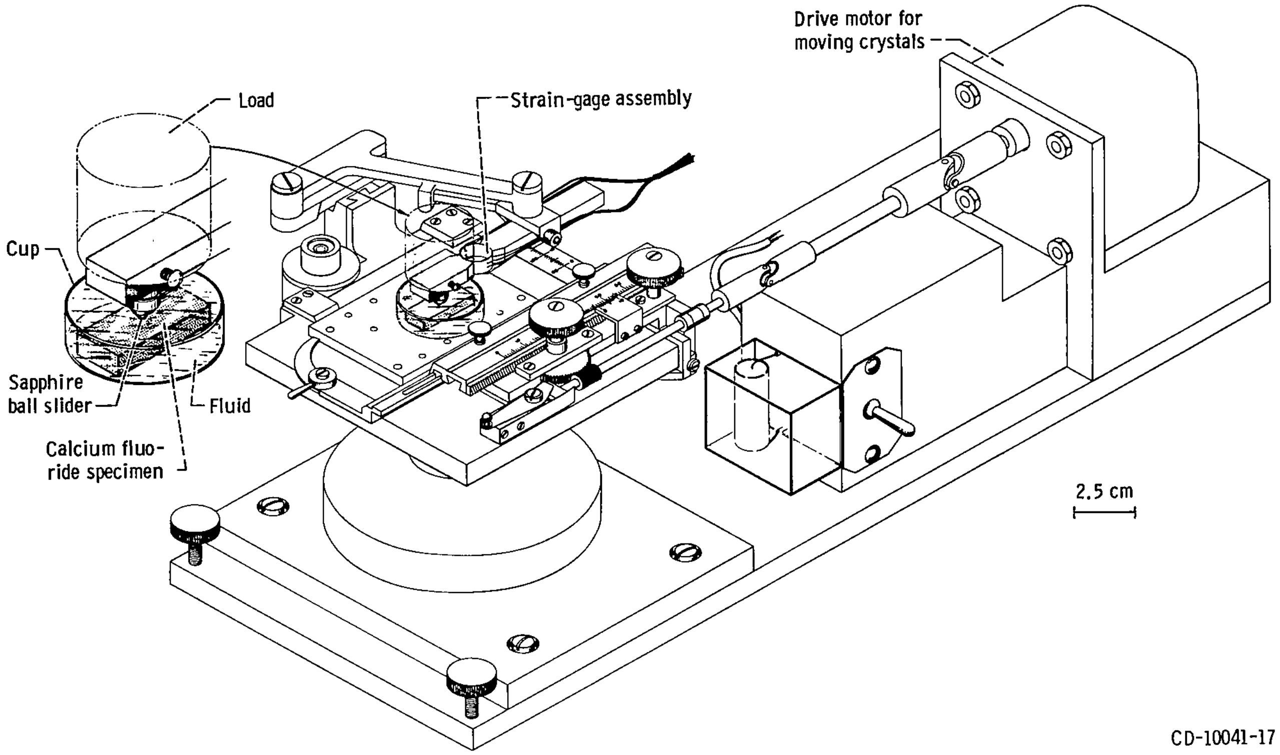


Figure 1. - Sliding friction apparatus.

The gear assembly attached to the motor allowed the sliding velocity to be varied from 0.005 to 0.5 millimeter per second. Loads on the 2.0-millimeter-diameter sapphire ball were varied from 100 to 600 grams. Motion was linear, which allowed for continuous sliding in the same crystallographic direction.

EXPERIMENTAL PROCEDURE

The calcium fluoride crystals used were cut to the 25- by 12- by 7-millimeter size with the (111) cleavage face as the flat surface on which sliding friction experiments were conducted (see fig. 2). The crystals were placed in a quartz vacuum tube and heated in a vacuum to 300° C and held at temperature for 12 hours. They were cooled to room temperature, the vacuum system was turned off, and the fluid media under study were admitted to the evacuated tube. The crystals were then placed into the dish of the friction apparatus and the sliding friction experiment was begun. The crystals were at all times submerged in the fluid media. The experiments were conducted in air at 30 percent relative humidity.

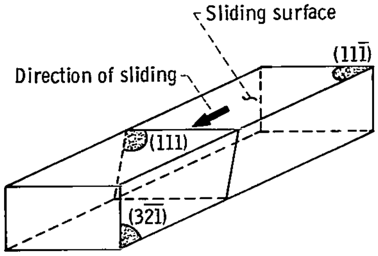


Figure 2. - Calcium fluoride crystal.

After completion of the friction experiment, the crystals were rinsed with solvents. The crystals were then etch pitted in concentrated sulfuric acid for 20 minutes to determine the extent of strain in the crystal. Then they were cleaved along the (111) planes (see fig. 2) and etch pitted to determine the extent of subsurface deformation and cleavage.

RESULTS AND DISCUSSION

When two surfaces slide over each other and one of the surfaces undergoes plastic deformation, a track is generated on the surface. This track is generally discernible with an optical microscope, and the profile of the deformed zone may be obtained with a surface profilometer. The track so observed does not, however, represent the true extent of deformation that has taken place in the material. The true extent of strain in the surface is a function of the extent of dislocation generation and motion along slip planes. These dislocations can frequently be revealed by etch pitting the surface and permit determining the extent of dislocation generation in the sliding contact area.

Some sliding friction experiments on the (111) face of calcium fluoride were conducted at various loads. The deformation track width was measured before and after etch pitting the surface. The data of figure 3 show the marked difference between the apparent extent of plastic deformation as revealed by optical examination of the track and that by etch pitting. Not only does a marked difference exist in the width of the track at all loads but also a notable difference in the slope of the curves as determined by the two techniques. As shown later in this section, examination of the real deformation can provide insight into the behavior of solids in sliding contact not revealed by studying only the apparent extent of surface deformation.

Sliding friction experiments were conducted in four media: hexadecane, oleic acid, water, and dimethylsulfoxide. Hexadecane was selected because it provides an inerting environment for the calcium fluoride crystal surface and therefore a point of reference. Oleic acid was selected because it is a surface active organic species used frequently by Rebinder in the establishment of the Rebinder effect (refs. 14 and 15). Water was used

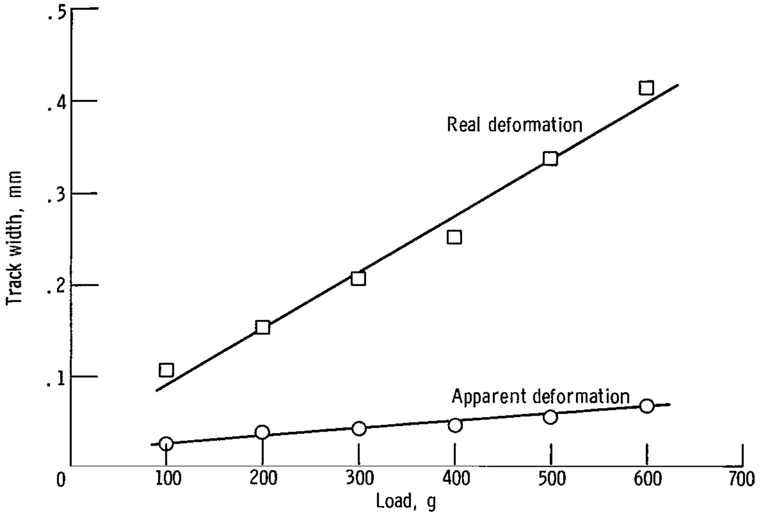


Figure 3. - Comparison of visible wear track generated on (111) calcium fluoride surface with real track width as indicated by dislocation etched pits. Sliding velocity, 0.005 millimeter per second; surface environment, dimethylsulfoxide; temperature, 20° C.

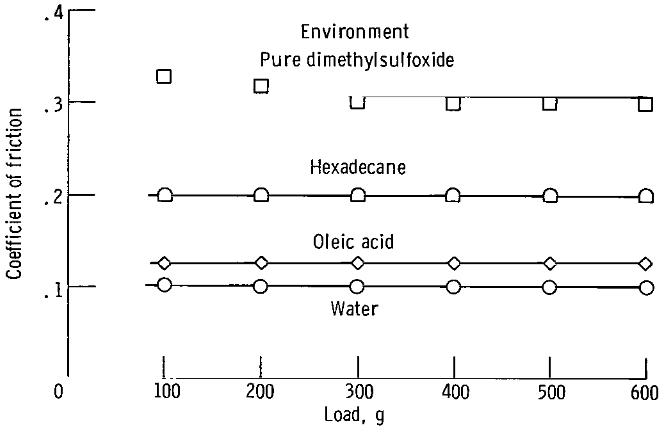


Figure 4. - Coefficient of friction for sapphire ball sliding on (111) cleavage face of calcium fluoride in various environments at various loads. Sliding velocity, 0.005 millimeter per second; temperature, 20° C; rider, sapphire ball.

because calcium fluoride is sometimes used as a solid film lubricant in moist air, and it would be of interest to know if water exerts any influence on the deformation and friction of calcium fluoride. Finally, dimethylsulfoxide was selected for examination because it has been observed to influence the deformation of calcium fluoride (ref. 1).

The friction coefficients measured for sliding on the (111) surface of calcium fluoride in all four media were measured at various loads, and the results obtained are presented in figure 4. The first observation made from figure 4 is that the friction coefficient obeys Amonton's law in that friction coefficient is independent of load. This relation has not been observed with other inorganic crystals (refs. 17 and 22).

The second observation made from figure 4 is that the friction coefficient for calcium fluoride is extremely environment sensitive. Whereas the surface active species, water and oleic acid, reduce the friction coefficient from that observed in hexadecane, DMSO on the surface results in an increase in the friction coefficient. It would appear that for inorganic crystals, unlike metals, not all surface films reduce friction of the clean surface. Depending on the nature of the surface active species, friction for calcium fluoride may increase or decrease from values measured in relatively inert hexadecane.

The friction results obtained in water were relatively low, which may be attributed to the formation of calcium hydroxide ($Ca(OH)_2$) on the crystal surface with sliding. Although the amount of $Ca(OH)_2$ that would form is small, as indicated by the thermodynamics of the reaction of water with calcium fluoride, very small concentrations of

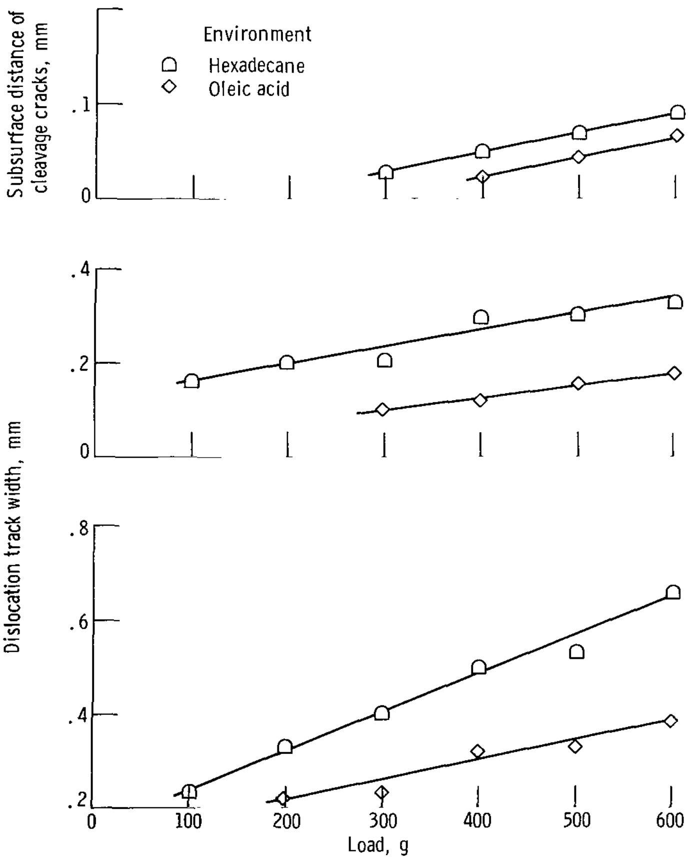


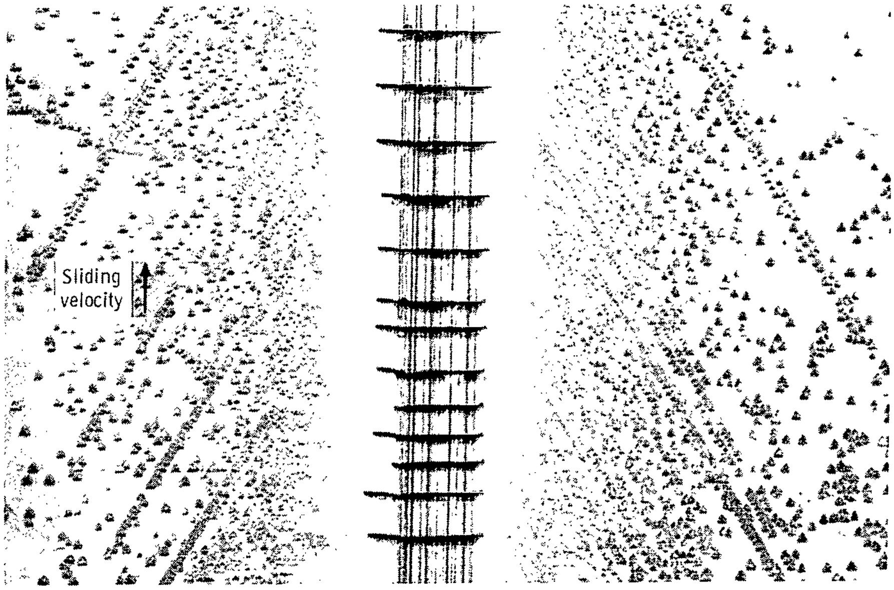
Figure 5. - Deformation and subsurface fracture as result of sliding on (111) crystal surface of calcium fluoride. Sliding velocity, 0.005 millimeter per second; temperature, 20° C; rider, sapphire ball. (No subsurface fracture cracks were observed in hexadecane at loads of 100 and 200 g.)

surface active species can influence friction, as shown later in this section. Calcium hydroxide has a layer lattice structure like that of cadmium iodine (CdI_2). Many layer lattice compounds have been shown to have good lubricating properties (ref. 23).

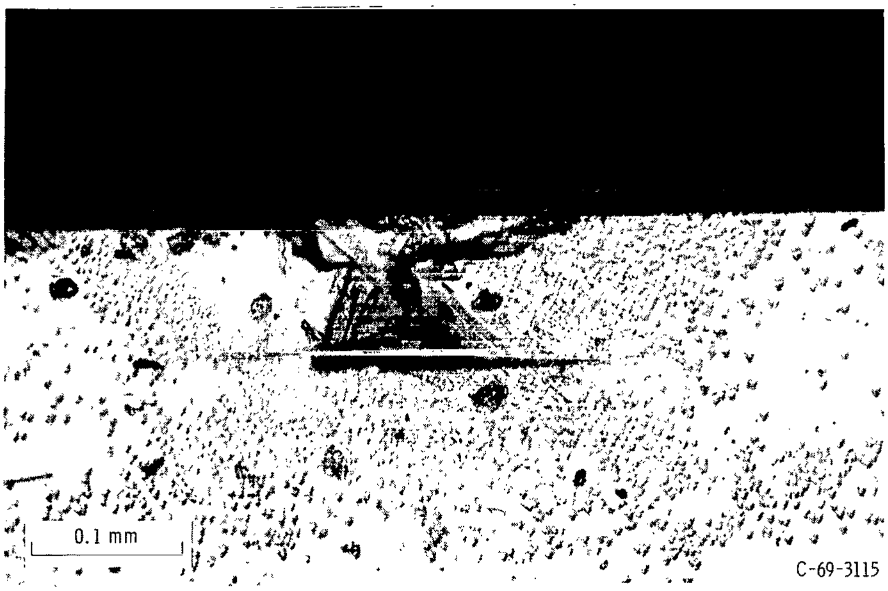
The amount of surface deformation that occurred with sliding at various loads in hexadecane and oleic acid is presented in figure 5. Both dislocation track width and depth of subsurface dislocations generated indicate, as might be anticipated, that deformation increases with increase in load. From results obtained in the relatively inert hexadecane at all loads, an unanticipated decrease was observed in the amount of surface deformation in the surface active medium, oleic acid. From the friction data of figure 4 and the deformation data of figure 5, less plastic deformation of the surface appears to occur in the presence of the surface active organic fluid (oleic acid). Accompanying less plastic deformation is less plowing and, consequently, lower friction.

The results of figures 4 and 5 agree with the conclusions that Westwood et al. (ref. 1) reached from hardness measurements; namely, the presence of surface active species increases the hardness of calcium fluoride. This increase in deformation resistance may result from the chemisorption of the surface active species at dislocation sites, which inhibits or impedes dislocation motion with applied stress. The presence of oleic acid on the surface of calcium fluoride appears to produce a surface-hardening effect similar to that of oxides on metals, as was observed by Roscoe (ref. 12). Thus, although surface active organics increase the deformability of inorganic crystals (Rebinder effect), the presence of these materials on calcium fluoride causes a decrease in deformability. Examination of the deformation tracks showed that surface and subsurface cracks developed with sliding. The photographs of figure 6 show these cracks. The $(11\bar{1})$ cleavage cracks are shown on the surface in figure 6(a) together with etched pits revealing the extent of plastic deformation or strain occurring in the calcium fluoride. Figure 6(b) indicates one of the surface cracks, a (111) subsurface crack that is met by the $(11\bar{1})$ surface crack, as well as the extent of subsurface plastic deformation. These surface and subsurface cracks are discussed later in reference to experimental results obtained in water. It is of interest to note, however, that the surface environment plays an important role in the location of these cracks, as shown by the data of figure 5.

With the hexadecane at loads of 100 and 200 grams, no subsurface cracks were observed to form. With oleic acid, however, subsurface cracks were observed optically at all loads. Data for these cracks in oleic acid are not presented in figure 5 because, although the cracks were subsurface along (111) planes, they were close enough to the surface to make their exact location difficult to resolve. At the light loads (100 and 200 g), the stresses imposed on the crystal with sliding in hexadecane could be accommodated by strain. In oleic acid, however, the inhibiting environmental effect on strain resulted in the development of cracks.



(a) Plan view of track on (111) surface.



(b) Cross-sectional view of subsurface fracture cracks.

Figure 6. - Deformation and subsurface fracture of calcium flouride as result of sliding. Surface covered with oleic acid; sliding velocity, 0.005 millimeter per second; load, 600 grams; temperature, 20° C; rider, sapphire ball.

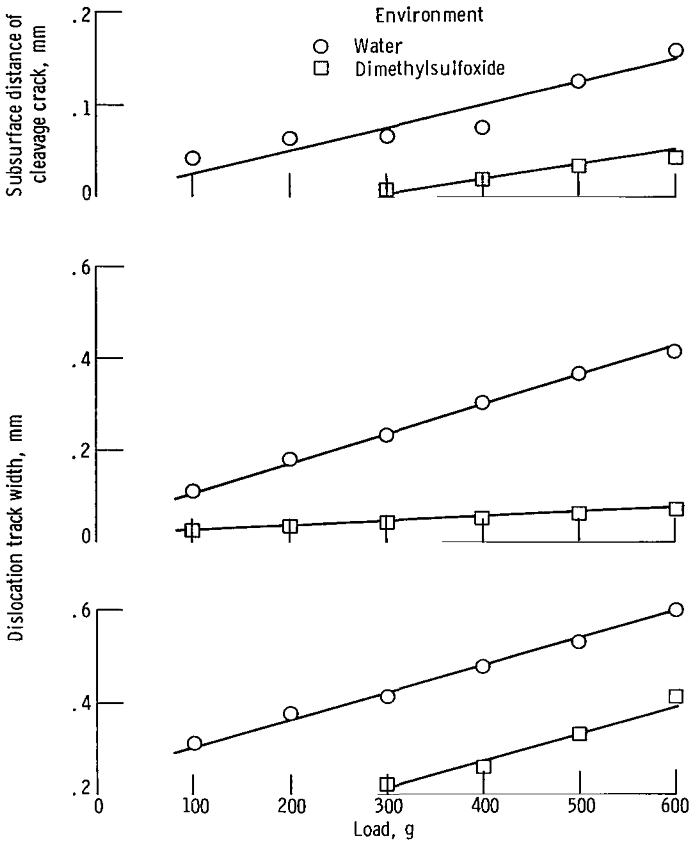


Figure 7. - Deformation and subsurface fracture as result of sliding on (111) crystal surface of calcium fluoride. Sliding velocity, 0.005 millimeter per second; temperature, 20° C; rider, sapphire ball; environments, distilled water and dimethylsulfoxide. (Subsurface cracks were observed in dimethylsulfoxide at loads of 100 and 200 g.)

The surface and subsurface deformation, as well as the formation of subsurface fracture cracks with sliding in water and DMSO are presented in figure 7. Although both water and DMSO are surface active with respect to calcium fluoride, marked differences in the ability of the calcium fluoride to deform with sliding is evident from the data of figure 7. Plastic deformation in DMSO is markedly less than in water, which indicates, as with hexadecane and oleic acid, that deformation of calcium fluoride during sliding is environment sensitive.

It is of interest to note that while the calcium fluoride surface was more resistant to deformation in DMSO than in water, the friction coefficient in water was lower. This result would indicate that with DMSO a lower plowing component of friction exists; the ability of the calcium hydroxide surface film to reduce adhesion and provide a low shear strength film may, however, mask the deformation effects. The calcium hydroxide (referred to earlier in this section) that can form in water may provide a lower shear strength surface film (possibly because of its layer lattice structure) than DMSO. The friction force is a function of the area in contact and the shear strength of the interfacial film. While, in DMSO, the area of contact may be reduced because of the increased surface hardness, the shear strength of chemisorbed DMSO may be considerably higher than that of calcium hydroxide.

Subsurface microcracks were observed to form in both water and DMSO at all loads examined (fig. 7). The presence of one such crack in calcium fluoride under a 300-gram load in water is shown in figure 8.

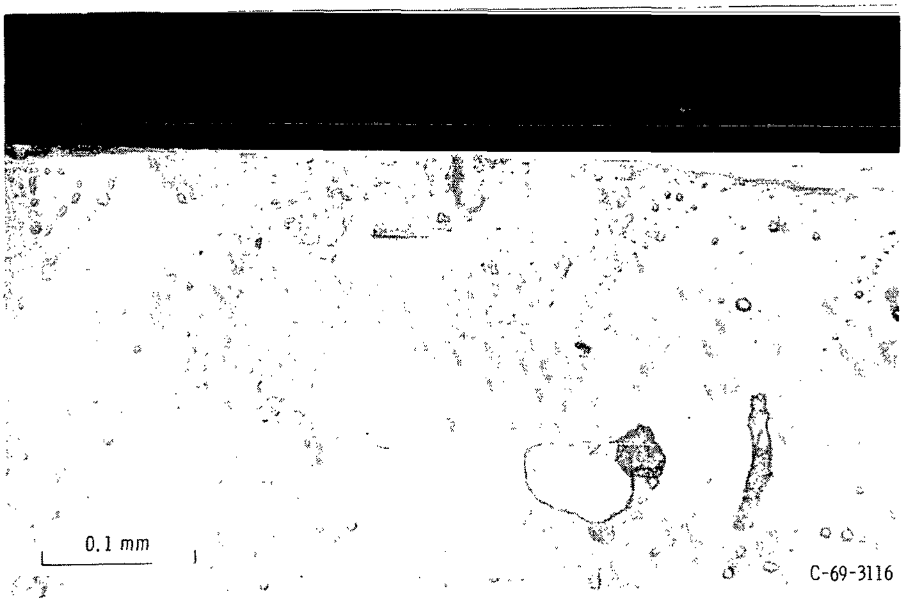
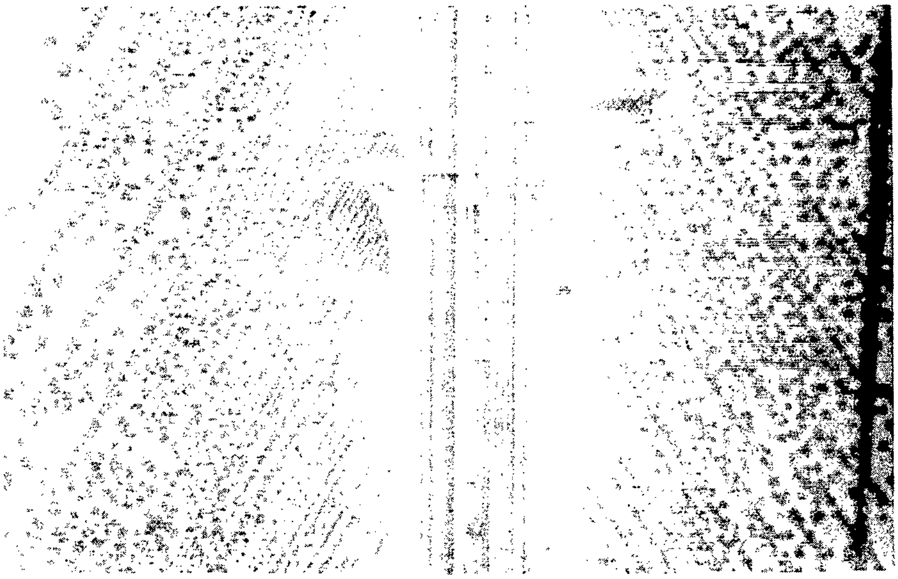
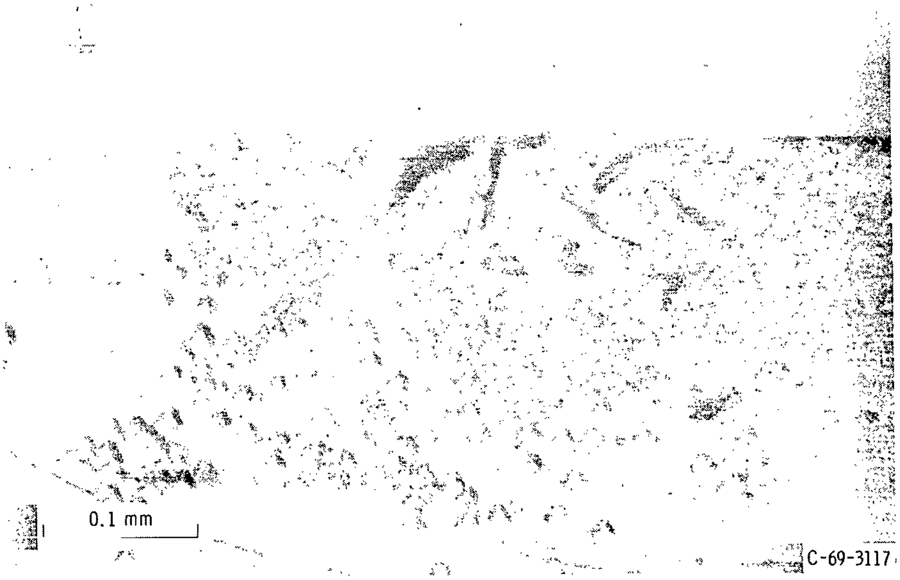


Figure 8. - Development of subsurface fracture crack in calcium fluoride as result of sliding. Surface covered with water. Sliding velocity, 0.005 millimeter per second; load, 300 grams; temperature, 20° C, rider, sapphire ball.

Friction is a function of both the plowing term associated with sliding on the crystal surface and adhesion at the interface between the sapphire ball and the calcium fluoride. Both these terms are influenced by the adsorbed film present on the crystal surface. Further, friction can influence the zone of maximum shear stress and therefore deformation. Thus, an interrelation exists among adhesion, deformation, adsorbed films, and friction. In DMSO, surface cracks were observed at all loads, but in water, surface cracks appeared only at 500- and 600-gram loads. The presence of both surface and subsurface microcracks in water at a 500-gram load is shown in figure 9. In water at a load of 500 grams, the surface cracks first became visible, and their occurrence was sparse (contrast fig. 9(a) to fig. 6(a)). In DMSO the cracks appeared at more frequent intervals, similar to that shown for oleic acid in figure 6(a).



(a) Track on (111) surface.



(b) Subsurface cracks.

Figure 9. - Deformation and subsurface fracture of calcium fluoride as result of sliding. Surface covered with water. Sliding velocity, 0.005 millimeter per second; load, 500 grams; temperature, 20° C; rider, sapphire ball.

In both oleic acid and DMSO (surface-hardening media), the subsurface cracks occurred nearer the surface than in hexadecane and water (see figs. 5 and 7). The stress field analysis for a circular contact (ref. 24) indicates that the maximum stress changes from a point below the surface for low friction coefficients to a position on the surface at a friction coefficient of about 0.27. The friction coefficient in water was 0.10 at all loads, and surface cracks began to form when the load exceeded 400 grams.

Note that the subsurface distance for the formation of (111) cleavage cracks increased with the increased plasticity of the crystal surface and that it occurred within the zone of strain. These observations suggest that, when the applied stress could no longer be accommodated by slip along $\{100\}$ slip planes, subsurface fracture cracks occurred in the zone of maximum subsurface stress along (111) cleavage planes. As in the case of water with oleic acid and DMSO, when the stresses became sufficiently high (at loads of 500 and 600 g), not only did slip along $\{100\}$ planes occur and cracks develop along (111) planes but also cracks were observed along $(1\bar{1}1)$ planes.

The presence of extremely small concentrations of DMSO in water influenced the friction coefficient and surface deformation. The data of figure 10 indicate that molar concentrations of 10^{-8} to 10^{-10} DMSO in water were sufficient to exert an influence on

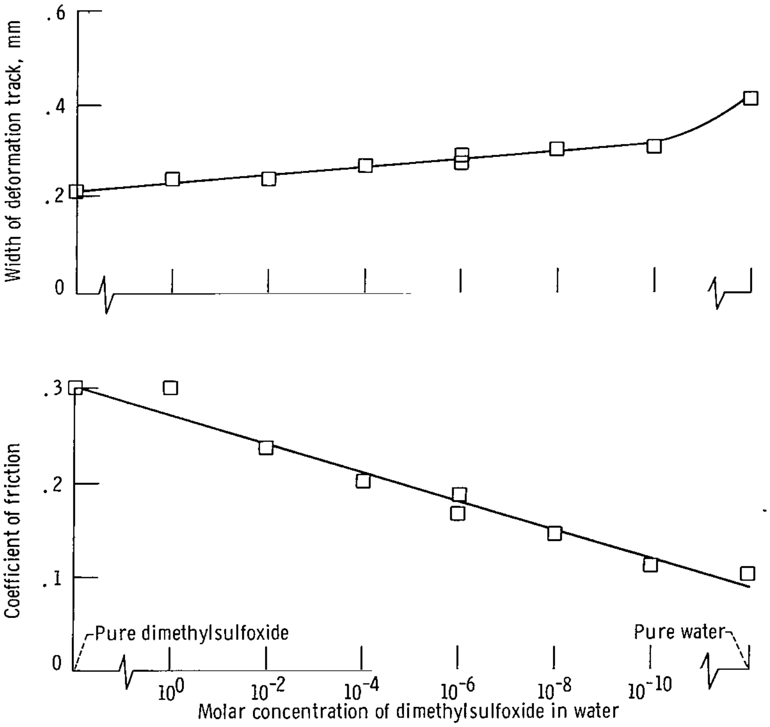


Figure 10. - Coefficient of friction and dislocation track width for sapphire ball sliding on (111) cleavage surface of calcium fluoride in various concentrations of dimethylsulfoxide in water. Sliding velocity, 0.005 millimeter per second; load, 300 grams; temperature, 20° C.

surface behavior during sliding. Figure 10 indicates that even impurities in lubricating fluids may be present in sufficient quantities to exert an influence on friction and deformation if the surface mechanical behavior is environment sensitive.

The nature of bonding of the DMSO to the crystal surface is of interest because it exerts such a strong influence on friction and deformation. A friction experiment was therefore conducted in which DMSO was admitted to the evacuated quartz tube after the crystal had been heated for 12 hours at 300° C. After the crystal surface had been completely covered with DMSO, it was removed from the fluid and rinsed in distilled water; then a sliding experiment was conducted under distilled water. The coefficient of friction measured was 0.25. Although this value is below the 0.3 measured in pure DMSO, it does indicate (since the value is markedly higher than that observed in water) that the water did not displace the adsorbed DMSO. Since the data of figure 10 were obtained with a decrease in concentration of DMSO in water, the results may simply represent a continuous decrease in the amount of DMSO surface coverage.

In most situations, an increase in sliding velocity results in a decrease in friction

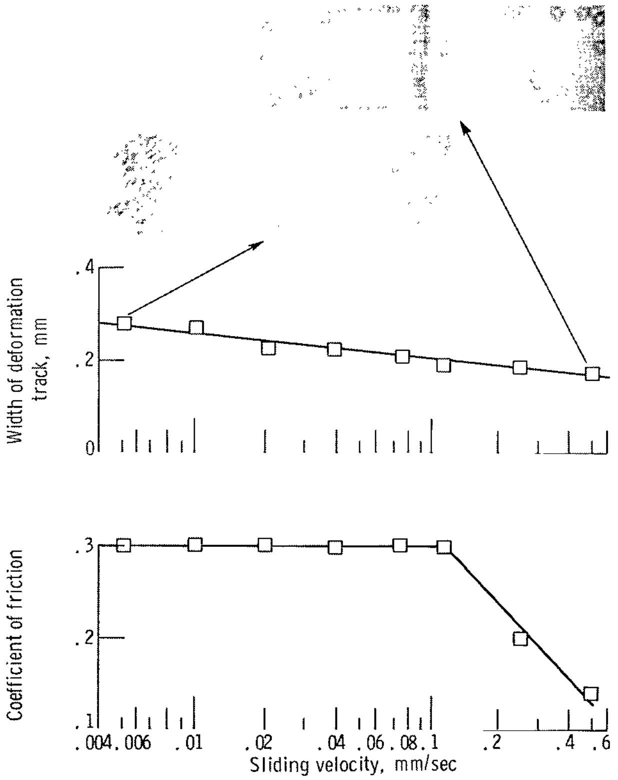


Figure 11. - Coefficient of friction and dislocation track width for sapphire ball sliding on (111) cleavage face of calcium fluoride as function of sliding velocity in 1 molar concentration of dimethylsulfoxide. Load, 300 grams; temperature, 20° C; rider, sapphire ball.

coefficient. In most mechanical testing, the strain observed is related to the rate of strain. Thus, observations of superplasticity in metals are intimately related to strain rate. Sliding friction experiments were conducted in a 1-molar DMSO solution at various sliding velocities, and the measured friction coefficient as well as the width of the deformation track are presented in figure 11. With increase in sliding speed, the width of the deformation track continued to decrease, which indicated that the deformation of calcium fluoride during sliding is strain rate sensitive. The faster the sapphire ball moved across the surface, the less the observed strain. At the higher sliding velocities, the friction began to decrease. At these higher sliding speeds, the decrease in plastic deformation may have become significant enough to influence the plowing term in the determination of friction. The photographic inserts show the difference in the extent of deformation.

SUMMARY OF RESULTS

Sliding friction experiments were conducted on the (111) cleavage face of calcium fluoride single crystals in four environments: hexadecane, oleic acid, water, and dimethylsulfoxide. The following results were obtained:

1. The friction and deformation behavior of calcium fluoride is extremely environment sensitive. The presence of surface active species such as oleic acid and dimethylsulfoxide increased the surface resistance to deformation with sliding.
2. The amount of strain that occurs during the sliding process is sensitive to the rate of strain or the rate at which sliding takes place.
3. Extremely small concentrations of surface active species influenced friction and deformation. With dimethylsulfoxide in water, concentrations of 10^{-8} to 10^{-10} molar exerted an effect.
4. In addition to plastic deformation of calcium fluoride with sliding, cleavage cracks formed on both the surface and subsurface. The number and depth of these cleavage cracks were sensitive to both load and the environmental surface species present.
5. Amonton's law (friction coefficient is independent of load) held for calcium fluoride. This law has not held true for other inorganic crystals.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 12, 1969,
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>
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