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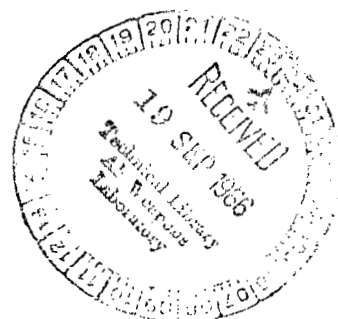
FRICTION CHARACTERISTICS OF SINGLE CRYSTAL AND POLYCRYSTALLINE ALUMINUM OXIDE IN CONTACT IN VACUUM

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SUMMARY

An investigation was conducted in vacuum to gain a better understanding of the friction and adhesion concepts of the ceramic material aluminum oxide in its single-crystal (sapphire) and polycrystalline forms in sliding contact with aluminum oxide of the same crystallinity. Experiments were conducted in a vacuum to 10^{-10} millimeter of mercury with surfaces which were outgassed. The specimens were a hemispherical or spherical surface sliding on a rotating flat surface. The materials were studied at rotating speeds of 0.013 centimeter per second, loads to 1500 grams, and ambient temperatures to 575° C.

The results of the investigation have shown that the friction characteristics for sapphire sliding on sapphire are highly anistropic and are determined by plastic deformation of the sapphire. With the polycrystalline material, friction coefficients obtained were intermediate between the values obtained for the prismatically and basally oriented single-crystal material.

INTRODUCTION

The relation of the adhesion and friction properties of metals in sliding contact in the absence of residual surface films is well established. For example, when clean metal surfaces come into intimate metallic contact, strong bonds of adhesion result (refs. 1 and 2). These bonds occur because of the interaction of the atomic bonding forces of the metals. The friction forces measured for metals under such conditions are then determined, primarily, by the bulk shear strength of the weaker of the two metals in contact. Physical properties which affect shear strength (e. g., mutual solubility, crystal structure, hardness, modulus of elasticity, ordering, and surface energy) can be expected to

influence the adhesion and friction forces.

The influence of surface films on the behavior characteristics of ceramic materials is much less clearly understood. Very little experimental work has been done with these materials to establish (1) the influence of adsorbates and surface films on their frictional properties, and (2) the type of surface interaction that will occur when surface films are absent.

With metals, the interfacial bonds are metallic; with ceramics the interfacial bonds are chemical. The differences in mechanical behavior of ceramics and metals (because of the atomic nature and bonding of these materials) is well known (ref. 3). For example, metals will deform plastically, whereas ceramics, while having extremely high strength, are normally extremely brittle and will fracture with little or no evidence of plastic flow. In recent years, however, it has been shown that many of these brittle materials will undergo varying degrees of plastic deformation at contacting interfaces under load and relative motion, much as metals do. Under such conditions plastic deformation has been observed in sodium chloride, magnesium oxide, and aluminum oxide (refs. 4 to 6).

The parameters which normally affect the mechanical behavior of metals (dislocations, vacancies, stacking faults, and crystal structure) may also affect the mechanical behavior of ceramics under such conditions. Dislocations, which normally have limited mobility in covalent crystals, become much more active with deformation. Some evidence for this conclusion is obtained from the work of reference 6. With the aid of dislocation concepts, a comparison is made of the slip behavior of aluminum oxide with that of hexagonal metals.

A ceramic material that has been the object of much study because of its low friction and wear characteristics in air is aluminum oxide (refs. 6 to 10). Of interest is the effect of removal of adsorbates on the bonding and friction character of aluminum oxide in both the single-crystal and polycrystalline forms. Further, with single crystals the knowledge of influence of orientation on friction could clarify data interpretation.

The objective of this investigation was to determine, in vacuum with outgassed surfaces, the friction characteristics of the ceramic material aluminum oxide in both single-crystal and polycrystalline forms in contact with the same forms in an attempt to clarify friction concepts.

BACKGROUND

Because of the desirable properties of ceramic materials (e.g., low friction and wear rates, good high temperature hardness characteristics, excellent corrosion resistance, etc.) these materials have been considered for lubrication in systems where more conventional bearing or seal materials cannot be used. Many investigators have looked

at the friction and/or wear properties of ceramics and particularly of aluminum oxide in its single-crystal form (sapphire).

Although much of the work with single crystals in the field of friction and wear, as well as in other areas, was to help elucidate the fundamental behavior of materials, single crystals are taking on significant practical importance. At one time single crystals could only be made in small sizes at large cost; very large crystals are available today. Furthermore, these materials are being employed in such mechanical devices as turbine blades for jet aircraft by a leading jet engine manufacturer to overcome the creep problems associated with polycrystalline materials at elevated temperatures. The concept of using single crystals as bearing or seal faces is conceivable, and if distinct advantages result, their use might become quite practical.

In order to assist those not familiar with the field of crystallography, figure 1 is supplied. Sapphire is a trigonal crystal consisting of a hexagonal closely packed lattice of oxygen atoms with the aluminum ions located at the octahedral interstices of the oxygen framework. In figure 1 sapphire is shown as hexagonal, since the trigonal system is a subsystem of the hexagonal. When a crystalline material is stressed under proper conditions, it will deform initially along well-defined prismatic and basal crystallographic planes, indicated in figure 1 as $(12\bar{1}0)$ and (0001) for sapphire. The parentheses ( ) indicate a plane and the braces { } indicate a complete set of equivalent planes. The displacement of the crystal under stress will occur in the specific directions indicated for sapphire in figure 1 as the $\langle 10\bar{1}0 \rangle$ directions for the $(12\bar{1}0)$ prismatic plane and the $\langle 11\bar{2}0 \rangle$ directions for the basal plane (0001). The brackets [ ] indicate a single direction and $\langle$ $\rangle$ indicate an equivalent set of directions. A slip plane and associated slip direction is then referred to as a slip system. To simplify the discussion the single plane will be designated by parentheses ( ) and direction by brackets [ ] throughout this report. Those familiar with crystallography know that the data will apply to equivalent planes and directions.

It has been demonstrated in references 11 and 12 that sapphire will undergo plastic deformation on the basal plane (0001) in the $[11\bar{2}0]$ direction at temperatures above 900° C; sapphire also deforms prismatically on the prismatic plane $(12\bar{1}0)$ in the $[10\bar{1}0]$ direction at 1800° to 2000° C (ref. 13). Reference 11, with the aid of dislocation mechanisms,
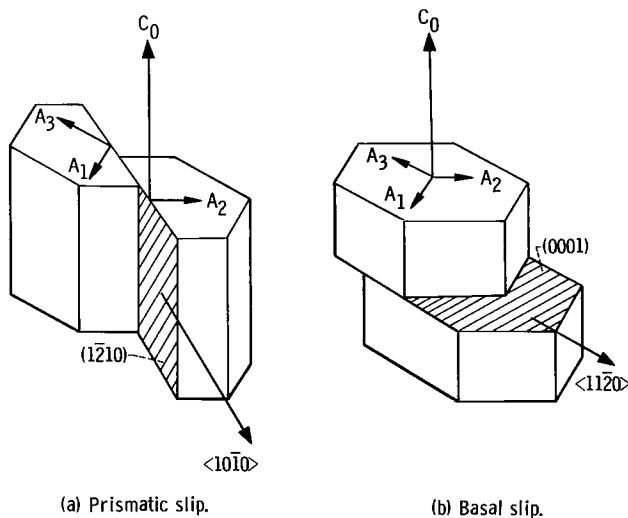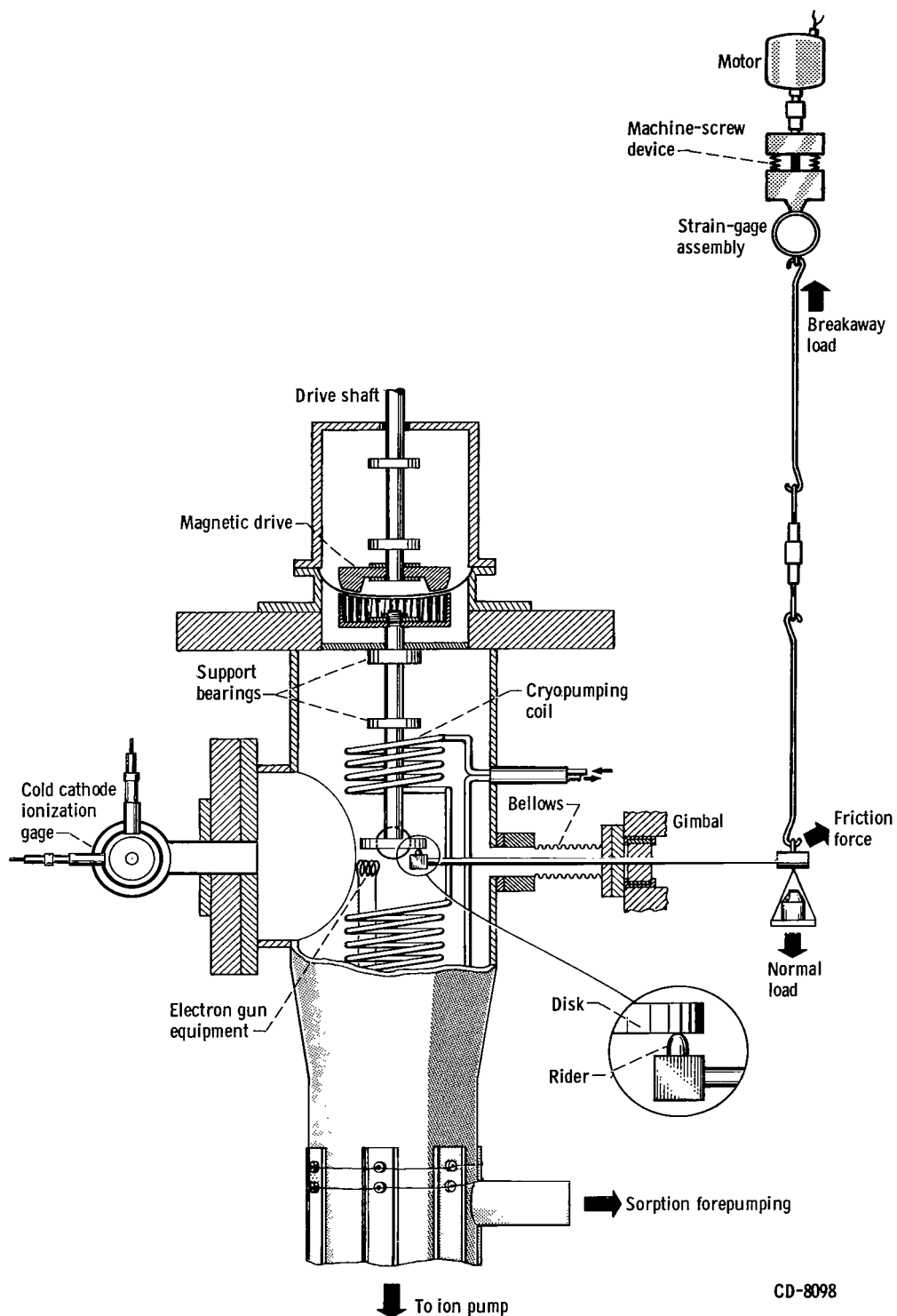

(a) Prismatic slip.   (b) Basal slip.

Figure 1. - Experimentally observed slip systems in sapphire (ref. 12). Directions A_1, A_2, A_3, and C_0 are indexing system for Miller-Bravais indices.

Figure 2. - Vacuum friction apparatus.

Motor

Machine-screw
device

Strain-gage
assembly

Breakaway
load

Drive shaft

Magnetic drive

Support
bearings

Cryopumping
coil

Cold cathode
ionization
gage

Bellows

Gimbal

Friction
force

Electron gun
equipment

Disk

Normal
load

Rider

Sorption forepumping

To ion pump

CD-8098

has further related the deformation behavior of sapphire to that of the structurally similar hexagonal metals. The work of reference 5 indicates that in wear experiments sapphire will undergo plastic deformation at room temperature with both basal and prismatic slip systems operating.

MATERIALS

The single crystals of sapphire used in this investigation were all synthetic corundum (α aluminum oxide or white sapphire). Rider specimens (see fig. 2) consisted of 3/16-inch-diameter spheres. The disk specimens of figure 2 consisted of 1/8-inch-thick plates, $2\frac{1}{2}$ inches in diameter, with a 1-inch hole in the center.

The polycrystalline aluminum oxide was of high density (99.9 percent) and high purity and had an average grain diameter of 0.023 millimeter. Rider specimens were prepared by radiusing the end of 3/8-inch-diameter rod. The disk specimens were $2\frac{1}{2}$ inches in diameter and 1/2 inch thick and had a hole through the center.

SPECIMEN PREPARATION AND ORIENTATION

All specimens of sapphire (single-crystal aluminum oxide which shall be referred to herein as sapphire) were oriented at Lewis. The rider ball specimens were first oriented with respect to the optical axis of the crystal by using polarized light rotation. The basal plane of the crystal lies normal to the optical or c-axis. The specimens were then X-rayed, by the Laue back-reflection technique, to obtain precise orientation and direction. Another technique which was employed for locating the c-axis of the crystal was that described in reference 13. In applying this method, the as-received crystal was etched with orthophosphoric acid at 320° C. A cloverleaf pattern became etched on the surface of the sapphire with the center of the three leaves lying over the optical axis. The exact location was then again located with the Laue back-reflection method.

In order to obtain other orientations the crystals were rotated with a single-crystal goniometer-orienter. The ball was mounted in a stainless steel tube fitting, the body of which was bored in a taper to accommodate a 3/16-inch-diameter ball. The orientations were then rechecked with the Laue back-reflection technique. The specimen holder used was similar to that used in reference 8.

The polycrystalline aluminum oxide used in these studies was obtained in the form of disk and rider specimens. All specimens were thoroughly cleaned with aqua regia before use. All sapphire and polycrystalline aluminum oxide specimens were thoroughly rinsed with acetone and alcohol before insertion into the vacuum chamber. After the vacuum

system had been evacuated and baked, the specimens were cleaned by heating to 300° C.

APPARATUS

The apparatus used in this investigation is shown in figure 2. The basic elements of the apparatus were the test specimens (a $2\frac{1}{2}$-in.-diam flat disk and a 3/16-in.-rad. rider) mounted in a vacuum chamber. The disk specimen was driven by means of a magnetic drive coupling. The coupling consisted of two 20-pole magnets axially spaced 0.150 inch apart with a 0.030-inch diaphragm between the magnet faces. The driver magnet, outside the vacuum system, was coupled to a low-speed electric motor (a speed of 0.013 cm/sec was used). The driven magnet was completely enclosed with a nickel-alloy housing (cutaway in fig. 2) and was mounted at the upper end of the shaft within the chamber. The disk specimen was at the lower end of the shaft.

The rider specimen was supported in the chamber by an arm that was mounted by gimbals and sealed to the chamber by a bellows (fig. 2). A linkage at the end of the retaining arm away from the rider specimen was connected to a strain-gage assembly to measure frictional force. Load was applied through a deadweight loading system as shown. Directly opposite the load (at 180°) was a strain-gage assembly for measuring adhesion forces (fig. 2).

Attached to the lower end of the specimen chamber was a 500-liter-per-second ionization pump and a vacuum-sorption forepump. The pressure in the chamber adjacent to the specimen was measured with a cold-cathode ionization gage. Also present in the apparatus was a diatron-type mass spectrometer (not shown in fig. 2) for determination of gases present in the vacuum system. A 20-foot, 5/16-inch-diameter stainless-steel coil was used for liquid-nitrogen and liquid-helium cryopumping of the vacuum system. The vacuum chamber and specimens were baked out at 200° C for 16 hours prior to each experiment.

RESULTS AND DISCUSSION

Sapphire on Sapphire

The friction coefficient for sapphire, outgassed in a vacuum, was measured for the randomly oriented material in reference 7. With outgassing temperatures to 1000° C the coefficient of friction measured was 0.9 at the higher temperatures. The work of references 5 and 8 has indicated that the wear characteristics of sapphire are anistropic. Experiments were conducted with two orientations of a sapphire ball to determine if
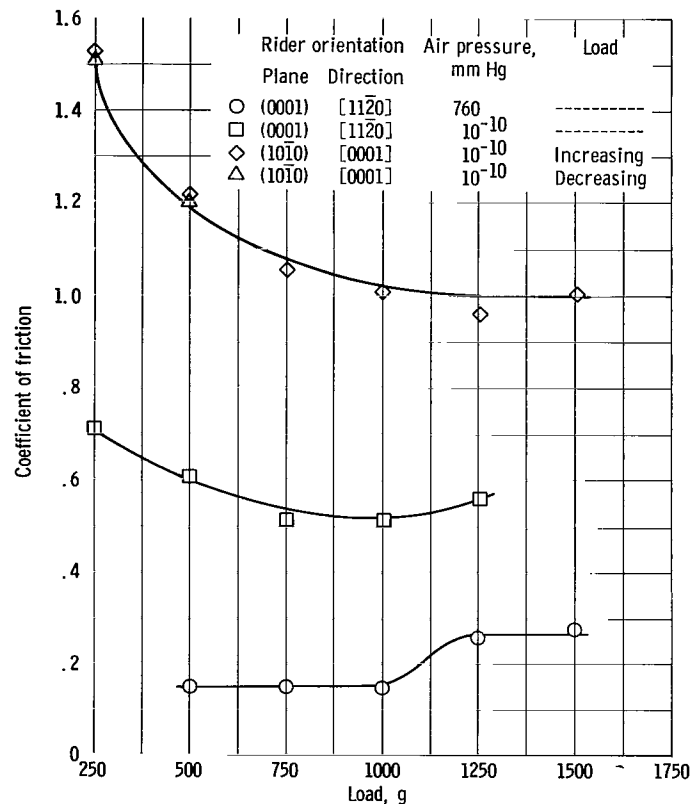
Figure 3. - Coefficient of friction as function of load for sapphire sliding on sapphire in vacuum of 10^{-10} millimeter of mercury and in air at 760 millimeters of mercury. Sliding velocity 0.013 centimeter per second; temperature 25° C; specimen outgassed by electron gun at 300° C; disk specimen had (0001) plane parallel to sliding interface.

friction characteristics were also anisotropic. The first orientation involved the (0001) plane and the [11$\bar{2}$0] direction, and the second the (10$\bar{1}$0) plane and the [0001] direction; the rider was sliding on a disk of sapphire having the basal plane essentially parallel (within 4°) to the sliding interface. The results obtained in these experiments are presented in figure 3 together with data for orientation in the (0001) plane and the [11$\bar{2}$0] direction examined in air to show the influence of adsorbed films.

The data of figure 3 would indicate that the friction characteristics of sapphire are highly anisotropic. At a moderate load of 250 grams the friction coefficient for the basal orientation was less than one-half that obtained for the prismatic orientation. As load was increased to 1000 grams, both orientations showed a decrease in friction coefficient. The marked differences in friction for the two orientations are maintained, however.

With metals, experiments conducted in vacuum serve to reduce the presence of adsorbed films and surface oxides. Sapphire, however, has a layer of oxygen atoms as an inherent part of its surface structure and therefore it is primarily the adsorbed films
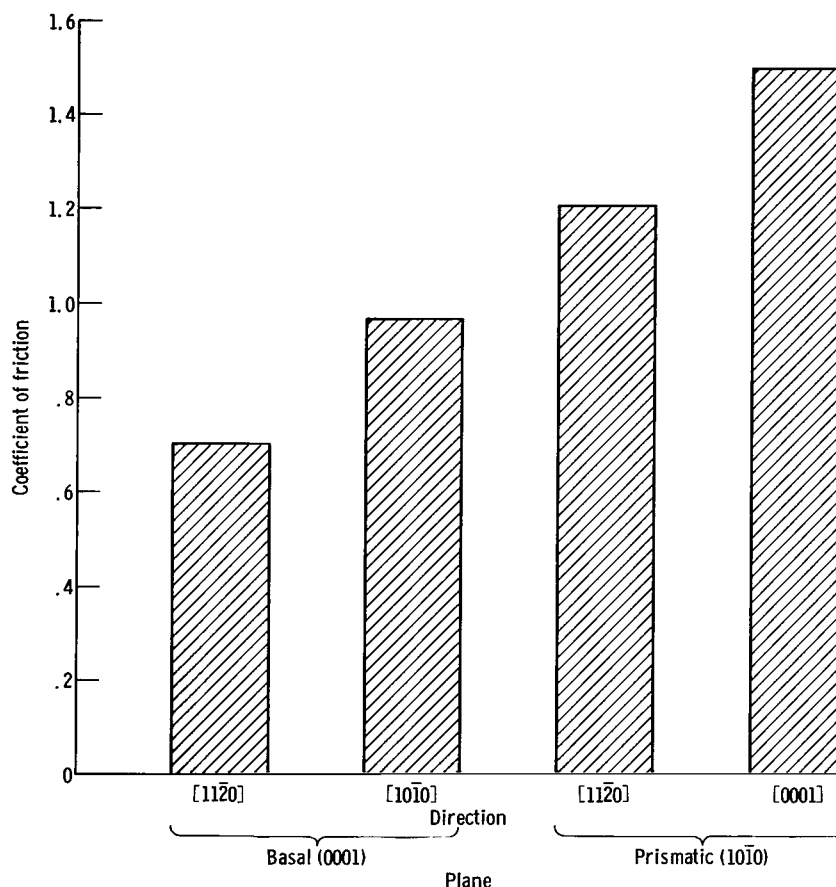
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Figure 4. - Influence of crystallographic direction on coefficient of friction for sapphire sliding on sapphire in vacuum of 10^{-10} millimeter of mercury. Load, 250 grams; sliding velocity, 0.013 centimeter per second; disk oriented with basal plane parallel to sliding interface.

which are removed during vacuum degassing. The differences in friction coefficients for the basal orientation of sapphire in air at 10^{-10} millimeter of mercury in figure 3 may be related to the influence of these films on the adhesion characteristics of sapphire.

The influence of crystallographic direction for both prismatic and basal orientations was determined, and the results obtained are presented in figure 4. With the basal orientation, a lower coefficient of friction was observed in the preferred slip direction [11$\bar{2}$0]. These data are quite similar to those observed for the hexagonal-metal beryllium in reference 13. With beryllium the friction coefficient on the basal plane was lowest in the preferred [11$\bar{2}$0] slip direction, which also corresponds to closest atomic packing. An interesting point is that, although sapphire slips in the [11$\bar{2}$0] direction, it has its closest oxygen atomic packing in the [10$\bar{1}$0] direction. Reference 6 explains this anomalous behavior of sapphire with the aid of dislocation partials.
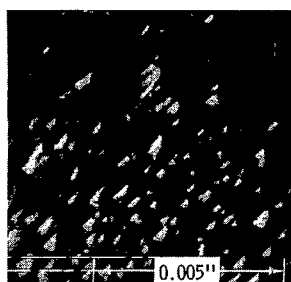
The friction coefficient for the prismatic orientation of the sapphire rider was determined at various temperatures in vacuum, and the results obtained are presented in
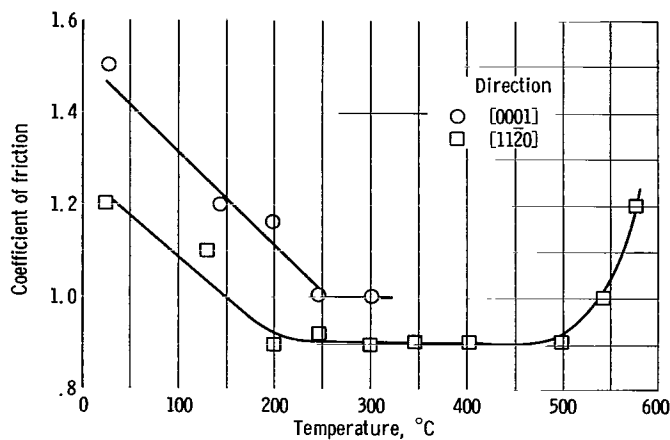
(a) Friction coefficient as function of temperature.
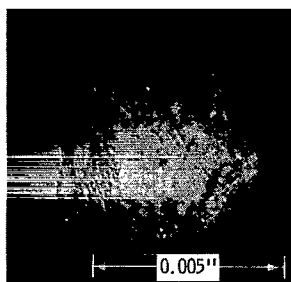
(b) Photomicrograph of wear scar after experiment.

(c) Photomicrograph of dislocation etch pits on (0001) plane after experiment; etched with orthophosphoric acid at 320° C.

Figure 5. - Influence of temperature and direction on friction for prismatic orientation of sapphire (10$\bar{1}$0) plane parallel to sliding interface. Load, 250 grams; sliding velocity, 0.013 centimeter per second; ambient pressure, 10^{-8} to 10^{-10} millimeter of mercury; disk oriented with basal plane parallel to interface.

figure 5. Data were obtained for two crystallographic directions, [0001] and [11$\bar{2}$0]. The data of figure 5 indicate that, for both directions, the friction coefficient decreases to a temperature of 200° to 250° C, then remains essentially constant to about 500° C. At temperatures above 500° C the friction coefficient for the preferred slip direction begins to increase markedly. Photomicrographs of the wear scar for the prismatic surface are shown in figure 5 together with the etch pits in the basal plane, which are the result of new dislocations on the prismatic planes. These dislocations are nonexistent in the virgin crystal, but are generated as a result of plastic deformation on the prismatic orientation in the sliding process. A convenient method for determining if plastic deformation in crystals has occurred involves etch-pitting to observe dislocation behavior. Reference 12 has described this technique and the correlation of etch pits with dislocations based on the Nye formula in detail. This technique was used to examine plastic deformation in the crystals mentioned in figures 3 to 5. In order to distinguish between dislocations present in the as-received crystal and those resulting from plastic deformation, a double-etching technique was used. The wear scar shown in figure 5 is on the prismatic plane, but etch-pitting for prismatic dislocations must be done on the basal plane.

The results shown in figures 3 to 5 indicate that the friction characteristics of sapphire are highly anisotropic. Etch-pitting of sapphire crystals after friction experiments was further marked evidence of plastic deformation of the crystals. The friction behavior of sapphire in these figures is very analogous to that reported for hexagonal metals in references 13 and 14. In sliding-friction experiments with hexagonal metals the friction coefficient was always less on preferred slip planes in preferred slip direc-

tions than for other slip systems. Similar results were obtained in this investigation. The preferred slip plane for sapphire is the basal plane under deformation. Furthermore, the preferred slip direction is the [11$\bar{2}$0] direction when the crystal is deformed plastically. With plastic deformation occurring at the sliding interface, the prismatic orientation of sapphire would be expected to exhibit a higher coefficient of friction than the basal orientation. While there are a number of prismatic planes that can slip in the deformation process, only a single set of planes is involved with the basal plane oriented parallel to the sliding interface.

When plastic deformation occurs at the sliding interface and more slip planes are involved, applied stresses, in the form of the load, may be distributed over a number of equivalent planes. With the basal plane parallel to the sliding interface, any applied normal load can only tend to compress basal planes. Even with metals such as beryllium, this orientation will support tremendous loading, to the point where the metal literally explodes, with no evidence of slip occurring on other slip planes. A larger true contact area can then occur in the sliding process with prismatic than with basal orientation. Further, when a number of prismatic planes undergo slip, interaction of such slip planes can occur to produce locks similar to the Lomer-Cottrell locks observed in the face-centered cubic metals. Such locks can produce marked increases in shear strength.

The lower coefficient of friction measured for the basal orientation of sapphire sliding on sapphire, shown in figure 3, is what might be predicted from observed slip behavior. In the plastic deformation experiments of reference 12 very little resolved shear stress was necessary to initiate slip on basal planes while the shearing stress for prismatic slip was at a maximum.

In figure 5, adhesion at the sliding interface with the prismatic orientations resulted in prismatic slip. With hexagonal metals such as cobalt, orientation of single crystals for prismatic slip results in slip plane and slip plane dislocation interaction, in strain hardening, and consequently, in an increase in measured friction. Cross slip mechanisms which can account for strain hardening in metals must be modified for sapphire if only two slip systems operate. Cross slip on the same system may not be possible, but prismatic plane dislocations can interact with stacking faults of the basal planes to produce strain-hardening. Reference 15 refers to strain-hardening from prismatic slip in sapphire. This interaction of basal stacking faults cannot occur for the basal slip mechanism because both the basal dislocations and stacking faults lie in the same plane.

The shear strength for sapphire calculated from friction measurements of this investigation, together with bulk shear strength data obtained from references 4, 16 and 17, is presented in figure 6. The surface shear strength is from 20 to 30 times that of the bulk shear strength. Similar results have been reported for a number of inorganic crystals in reference 3. A possible explanation for this increase may be that indicated in reference 16, which states that microscopic plastic deformation occurring at the
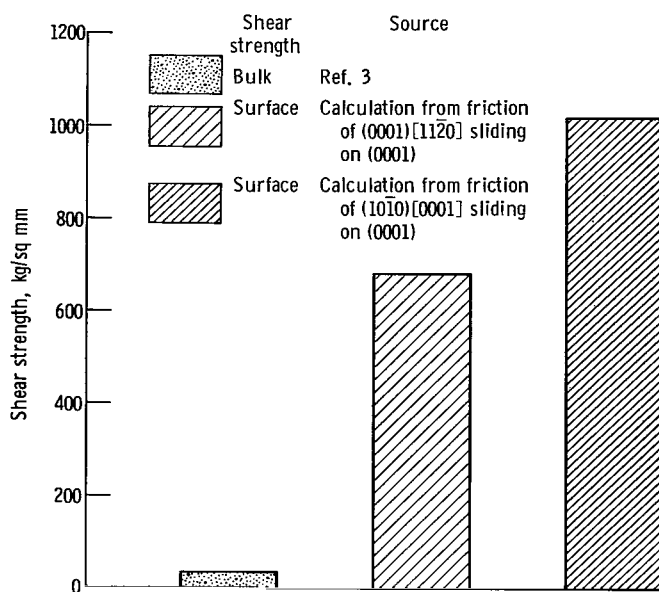
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Figure 6. – Bulk shear strength and surface shear strength data for sapphire calculated from vacuum friction experiments.

Figure 7. – Coefficient of friction as function of load for polycrystalline aluminum oxide compared with that for single-crystal aluminum oxide. Sliding velocity, 0.013 centimeter per second; temperature, 25° C; ambient pressure, 10^{-10} millimeter of mercury.

sliding interface permits relief of stresses in the sapphire, resulting in an increase in shear strength.

The friction data of this investigation indicate, as did the wear data of reference 5, that plastic deformation dictates the sliding behavior of sapphire. The friction behavior of sapphire is much like that observed with hexagonal metals in that it depends upon slip systems which operate during plastic deformation.

Polycrystalline Aluminum Oxide

In polycrystalline materials, a number of individual crystallites of differing or random orientation are held together by a transition region or grain boundary. The grain boundaries in aluminum oxide are the weak members, and fracture nearly always occurs intergranularly. The bulk shear strengths for single and polycrystalline aluminum oxide are nearly the same. Reference 4 cites a value of 34 kilograms per square millimeter for the shear strength of sapphire, and reference 18 cites a value of 28 kilograms per square millimeter for polycrystalline aluminum oxide. Based upon the similarity of other mechanical properties of aluminum oxide, it might be anticipated that the single-crystal and polycrystalline forms would exhibit similar friction characteristics. Friction measurements were made on polycrystalline aluminum oxide sliding
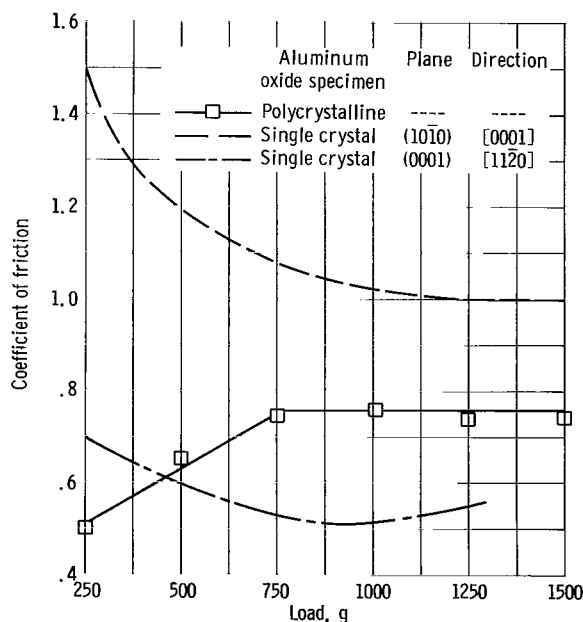
on itself in vacuum, and the results obtained are presented in figure 7. At loads in excess of 750 grams a friction coefficient of 0.75 was obtained, a value intermediate between those of the extreme single-crystal orientations of figure 3. The single-crystal curves are included in figure 7 for comparative purposes.

SUMMARY OF RESULTS

Based on the experimental results obtained in an investigation of aluminum oxide (single crystal and polycrystalline) sliding on aluminum oxide of the same crystallinity in vacuum, the following summary remarks are made:

1. The friction characteristics for sapphire (single-crystal aluminum oxide) sliding on sapphire are highly anisotropic. Lower friction coefficients were observed with basal than with prismatic orientations at the sliding interface. The results obtained indicate that plastic deformation of sapphire may dictate its friction behavior.

2. Polycrystalline aluminum oxide exhibits friction coefficients intermediate between those observed with the two single-crystal orientations (basal and prismatic) at loads in excess of 500 grams.

3. The friction coefficient for single-crystal aluminum oxide (sapphire) is markedly less in air than in vacuum, which indicates the influence of adsorbed films.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 10, 1966,
123-03-13-02-22.

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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