NASA TECHNICAL NOTE

NASA TN D-3593

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NASA TN D-3593

FRICTION CHARACTERISTICS OF SINGLE-CRYSTAL AND POLYCRYSTALLINE ALUMINUM OXIDE IN CONTACT WITH VARIOUS METALS IN VACUUM

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Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1966

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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 and polycrystalline form in sliding contact with various metals. Experiments were conducted in a vacuum to 10^{-10} torr with surfaces that were outgassed. The specimens were a hemispherical or a spherical surface sliding on a rotating flat. 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 show that, with metals sliding on sapphire, stresses result in fracture within the sapphire. These fracture stresses determine the measured friction. When metals were slid on polycrystalline aluminum oxide, adhesion of the metal to the aluminum oxide resulted in shear in the metals. Coefficients of friction for most hexagonal metals sliding on aluminum oxide were lower than for cubic metals sliding on aluminum oxide. Adhesion of metal to aluminum oxide occurs in air as well as with clean surfaces in vacuum.

INTRODUCTION

The fundamental concepts involved in the friction, adhesion, and wear behavior of metals in sliding contact with metals, in the absence of surface films, have been given considerable attention in the literature. In many engineering applications, however, there are other material combinations of equal interest. In dynamic seals, for example, ceramics and metals in contact with ceramics are of considerable interest. Such systems have not been explored so thoroughly as the metal-metal systems. A better understanding of the behavior of such materials in sliding contact is, therefore, needed.

Of the ceramic materials considered for use in mechanical devices, aluminum oxide has been given the most attention. A number of studies have been undertaken to examine its friction and/or wear behavior when in sliding contact with itself, particularly in its single-crystal form (refs. 1 to 5).

The marked differences in bonding and mechanical properties of metals and ceramics can be expected to influence the behavior observed at sliding interfaces. With metals in sliding contact with metals, interfacial bonding is metallic. With metals contacting ceramics such as aluminum oxide and with ceramics contacting ceramics, the bonding can be chemical. Many of the concepts related to properties that characterize metals and influence interfacial adhesion, shear, and friction may have to be modified in order to understand systems that involve ceramics. For example, plastic flow in metals is important to measured friction characteristics. Although plastic flow may occur at the sliding interface with a ceramic such as aluminum oxide in sliding contact with itself (refs. 1 and 6), it can hardly be expected to occur in aluminum oxide to any extent when aluminum oxide is in contact with soft metals. Friction experiments conducted in a vacuum can markedly simplify interfacial studies, since adsorbed films and metal surface oxides can be removed, and the influence of bulk properties on friction can be more confidently ascertained. Since aluminum oxide has been considered for use in mechanical systems and its friction characteristics when sliding on itself have been explored in reference 6, this investigation studied metals in sliding contact with aluminum oxide. Studies were conducted in vacuum with both single-crystal and polycrystalline metals and aluminum oxide, so that a better understanding of those factors that influence observed friction behavior could be gained.

MATERIALS

The single crystals of sapphire used in this investigation were all synthetic corundum (α-Al_2O_3 or white sapphire). Rider specimens consisted of 3/16-inch-diameter spheres. The disk specimens consisted of 1/8-inch-thick plates, $2\frac{1}{2}$ inches in diameter with a 1-inch hole in the center.

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

The single crystals of copper used were 99.999 percent pure as was the polycrystalline copper. All other metals were 99.99 percent pure.

SPECIMEN PREPARATION AND ORIENTATION

All the sapphire crystals (single-crystal aluminum oxide) were oriented after they had been received. The rider ball specimens were first oriented, by use of polarized light rotation, with respect to the crystal's optical axis. The basal plane of the crystal lies normal to the c or optical axis. The specimens were then X-rayed by the Laue back-reflection technique so that precise orientation and direction could be obtained. Another technique that was employed in locating the c-axis of the crystal is that described in reference 7. 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 three such leaves lying over the optical axis. The exact position was again located with Laue back reflection.

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

The polycrystalline aluminum oxide 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.

The metal specimens were electropolished before use to remove any worked layer and any contaminants that may have been introduced during mechanical finishing. The specimens were also rinsed in acetone and alcohol prior to insertion in the vacuum chamber.

After the vacuum system had been evacuated and baked out, the specimens were cleaned with electron bombardment, which resulted in specimen temperatures of 300° C. This type of heating is not analogous to more conventional heating techniques because in the former a large amount of energy can be put to the specimen surface by the impinging electrons. At somewhat higher levels than used herein, a frosted or thermally etched surface is obtained with sapphire.

APPARATUS

The apparatus used is shown in figure 1. The basic elements were the test specimens $\left(2\frac{1}{2}\text{-in.-diam flat disk and } \frac{3}{16}\text{-in.-rad. rider}\right)$, which were mounted in a vacuum chamber. The disk specimen was driven through a magnetic drive coupling, which consisted of two 20-pole magnets spaced axially 0.150 inch apart with a 0.030-inch diaphragm between the magnet faces. The drive magnet outside the vacuum system was coupled to a small elec-

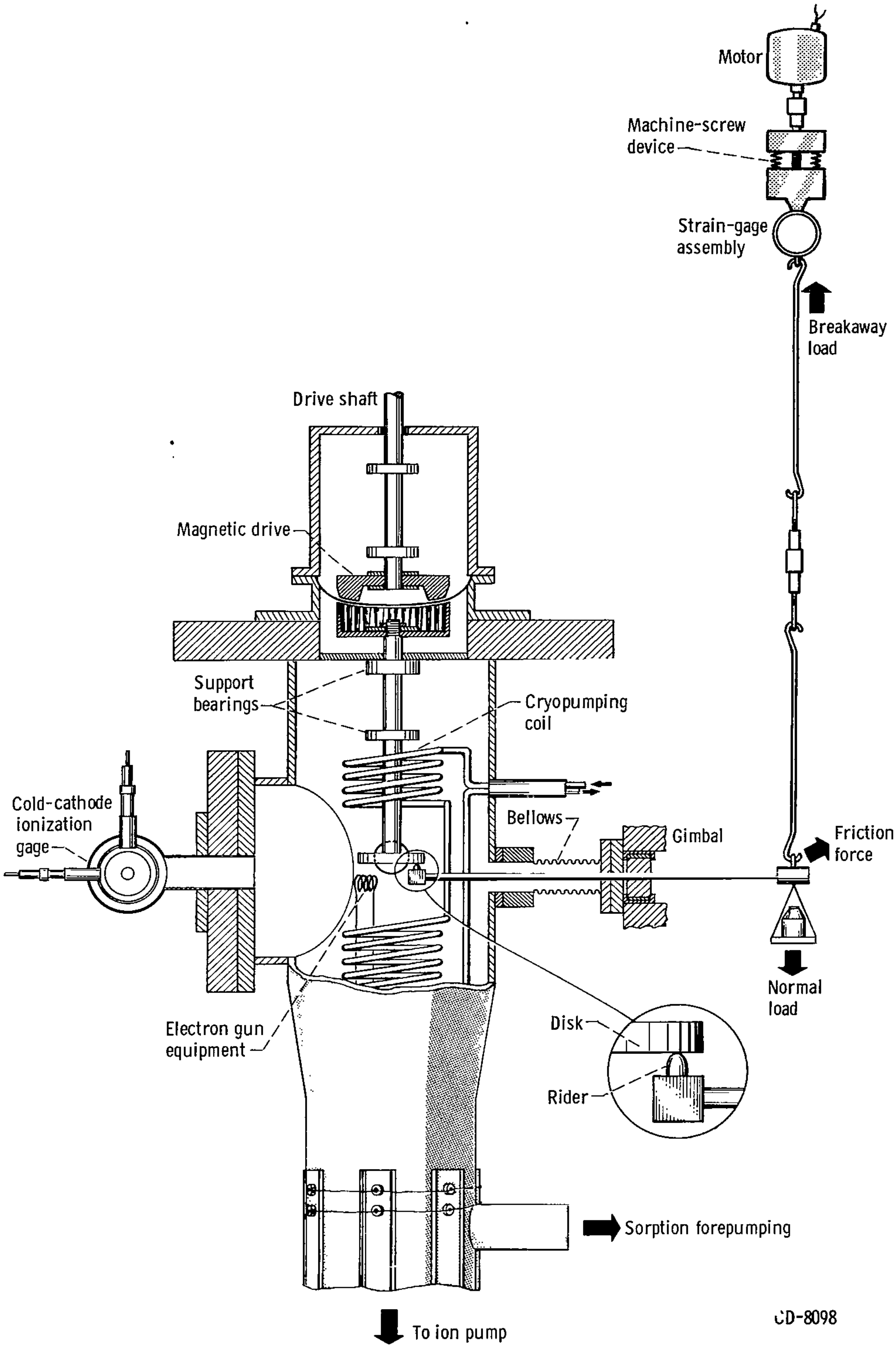


Figure 1. - Vacuum friction apparatus.

tric motor. The driven magnet was completely enclosed with a nickel-alloy housing (cutaway in fig. 1) 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 specimen chamber by an arm mounted by gimbals and sealed by a bellows to the chamber. A linkage at the end of the retaining arm away from the rider specimen was connected to a strain-gage assembly. The assembly was used to measure frictional force. Load was applied through a deadweight loading system. Directly opposite the load (at 180°) was a strain-gage assembly for measuring adhesion forces.

Attached to the lower end of the specimen chamber was a 500-liter-per-second ionization pump and a vac-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. 1) 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

The extremely hard nature of aluminum oxide when compared with pure soft metals indicates the possibility of marked plowing when aluminum oxide slides on metals. Two friction experiments were conducted with copper in sliding contact with sapphire to determine the influence of plowing on the measured friction coefficient for aluminum oxide in sliding contact with metals. In one experiment, a copper single crystal was the disk surface and in the other the rider specimen. The results obtained are presented in figure 2. With the sapphire rider sliding on the single-crystal copper disk, a high coefficient of friction was observed. Examination of the sapphire wear after the friction experiment indicated considerable adhesion of the copper to the sapphire; as a result, the sapphire was plucked out of the wear contact zone.

With the copper single crystal sliding on sapphire, a coefficient of friction of 0.2 was obtained. The sapphire disk had areas in the wear track where shear or fracture of sapphire had occurred (fig. 2). In both experiments, adhesion of the copper to sapphire occurred. With the copper sliding on sapphire, the friction coefficient represents the forces necessary to cleave the sapphire once adhesion of copper to sapphire occurs. The friction coefficient was much lower than observed for sapphire sliding on sapphire (ref. 6) because chemical bonding of the metal to sapphire was believed to have occurred; this chemical bonding likely resulted in a marked increase in the lattice strain and the stresses within

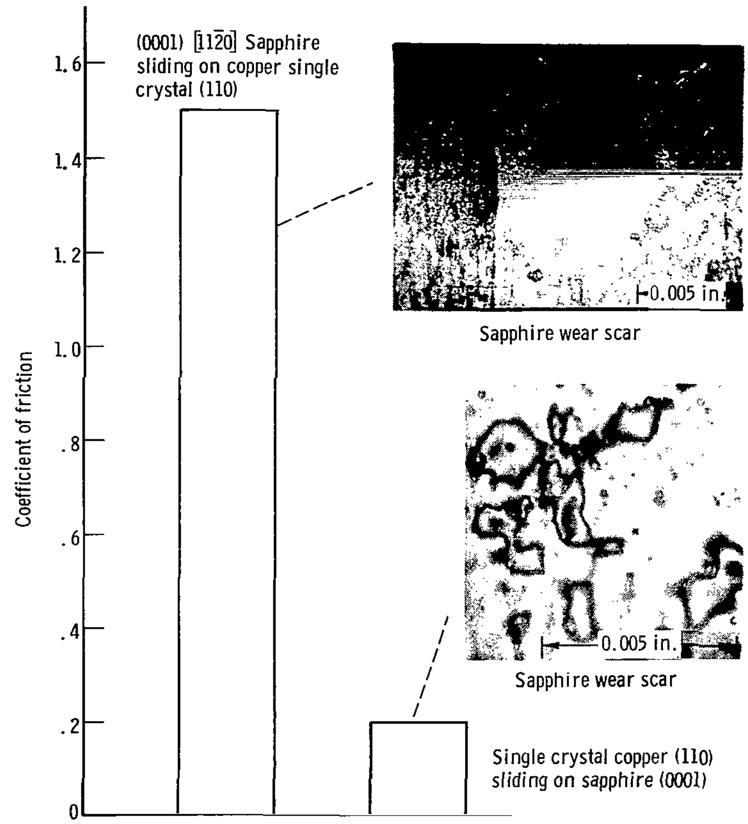


Figure 2. - Coefficient of friction for copper in sliding contact with sapphire in vacuum (10^{-10} torr). Load, 100 grams, sliding velocity, 0.013 centimeter per second.

the sapphire, which reduced appreciably the force to fracture. The chemical bonding of metals such as copper to sapphire is described in detail in references 8 to 13, and the reduction in sapphire shear strength with chemical bonding is described in references 13 to 16. With sapphire sliding on sapphire, the true contact area is small compared with that for soft metals such as copper in contact with sapphire. Plastic deformation dictates the observed friction for sapphire sliding on itself; in contrast, with metals sliding on sapphire, chemical bonding and fracture of sapphire occur.

The results obtained with sapphire sliding on the copper surface indicate that plowing, in this instance, contributed considerably to the friction force measured. Since the shear of sapphire took place in both experiments, the large differences in friction observed in figure 8 (p. 14) may be attributed to plowing effects.

Metals other than copper are of interest for lubrication systems. It would, therefore, be helpful to know the influence of adhesion of other metals to sapphire and its affect on friction. Further, in most lubrication systems, the aluminum oxide considered is in the polycrystalline form. Studies of metals in addition to copper sliding on polycrystalline

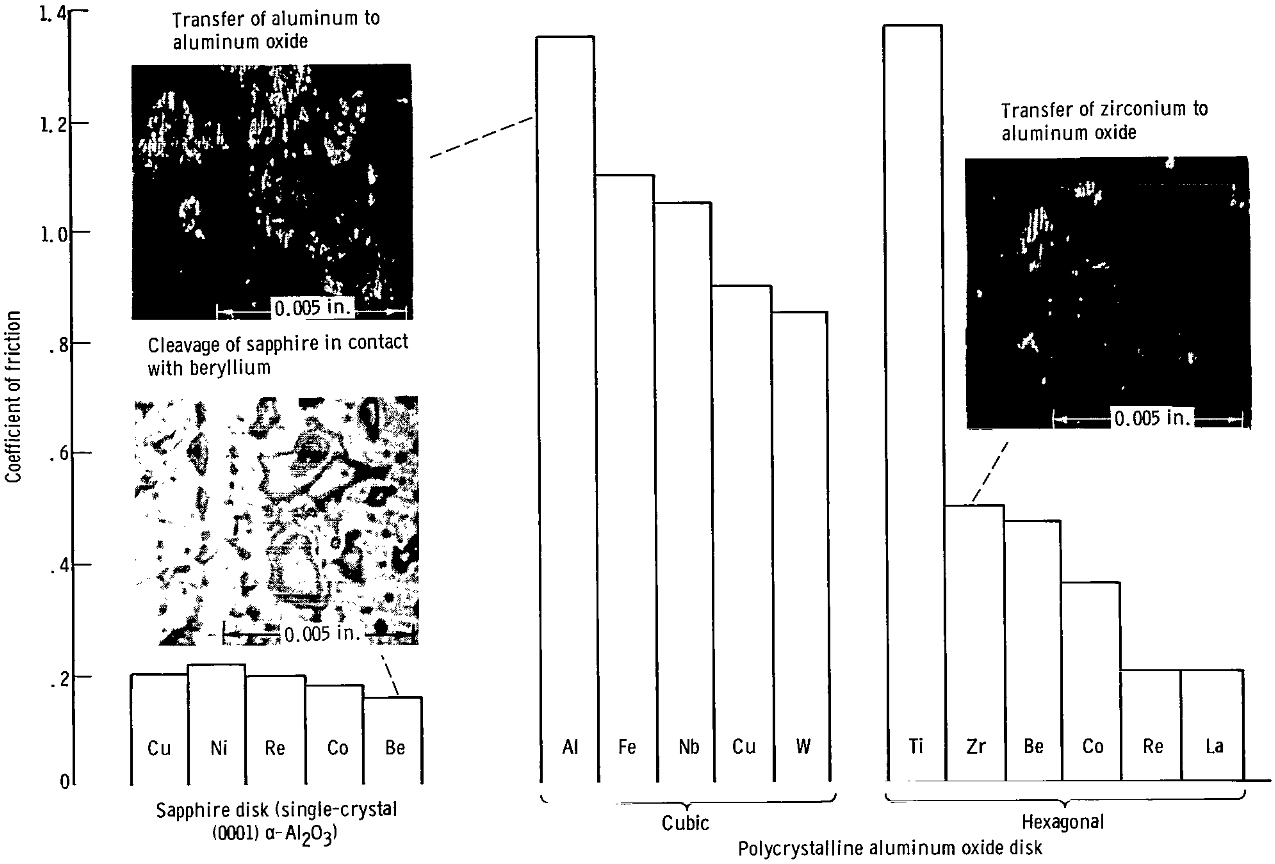


Figure 3. - Coefficient of friction for various metals sliding on aluminum oxide in vacuum (10^{-10} torr). Load, 1000 grams; sliding velocity, 0.013 centimeter per second; duration, 1 hour.

aluminum oxide would, therefore, be of interest. Friction experiments were conducted with various metals sliding on single-crystal and polycrystalline aluminum oxide, and the results obtained are presented in figure 3. With five different metals sliding on sapphire, the friction coefficient was approximately the same (fig. 3). With all the metals, fracture or cleavage occurred in the sapphire, which accounted for the similarity in friction. Adhesion of the metals to the sapphire resulted in stresses and fracture in the sapphire, as indicated for beryllium sliding on sapphire (fig. 3). In some instances, the pits in sapphire were 250 Å in depth.

With various metals sliding on polycrystalline aluminum oxide, much higher coefficients of friction were observed. The surfaces of all the polycrystalline aluminum oxide disks indicated transfer of metal to the disk surface. The transferred metal on aluminum oxide is shown in figure 3 for aluminum and zirconium. While fracture occurred in the sapphire with metals sliding on sapphire, shear of the metal was responsible for friction with metals sliding on polycrystalline aluminum oxide. For the cubic metals, the friction coefficients were those that might be predicted from the $f = S/H$ relation of reference 17 (where f is friction force, S is shear strength, and H is hardness). Since the metal was shearing and the disk surface was not undergoing notable plastic deformation (to increase the true contact area markedly as it does with metals in sliding contact with metals in vacuum), these values were as might be anticipated.

The friction data for the hexagonal metals were significantly less than for the cubic metals with one notable exception, namely, titanium. These differences in friction characteristics for cubic and hexagonal metals can be attributed to the differences in slip behavior in plastic deformation for hexagonal and cubic metals, as discussed in references 18 and 19.

The difference between friction behavior of titanium and other hexagonal metals that slip predominantly on basal planes, such as cobalt, beryllium, rhenium, and lanthanum, is that titanium slips primarily on prismatic planes during plastic deformation. Greater contact area and increased shear strength can then result for titanium with sliding, and the titanium will behave more like a cubic than a hexagonal metal (ref. 20).

At the contacting interface, chemical bonding is believed a major contribution for the adhesion of metal to aluminum oxide. With sapphire, the stresses produced resulted in a weakening and fracture of sapphire, while with the polycrystalline aluminum oxide, it was shear of the metal. If chemical bonding is responsible for adhesion, experiments with metals that do not form stable oxides should reveal no evidence for fracture of sapphire; also, friction coefficients less than those in figure 3 should be obtained. Friction experiments were conducted with gold and silver sliding on sapphire in vacuum. Gold does not have a stable oxide, and the oxide of silver dissociates at moderate vacuum. The results obtained in these experiments are presented in figure 4. When sliding on sapphire, both silver and gold exhibited friction coefficients that were approximately half of those obtained for the metals forming metal oxides in figure 3. Further, photomicrographs of the

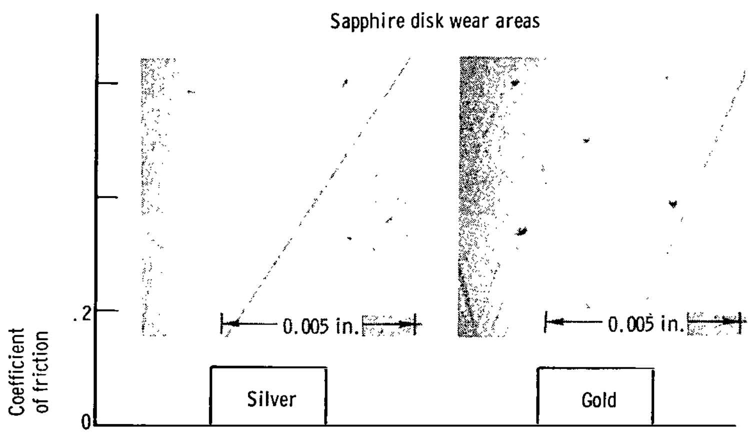


Figure 4. - Coefficient of friction for gold and silver riders sliding on sapphire in vacuum (10^{-10} torr). Sliding velocity, 0.013 centimeter per second; ambient temperature, 25° C (77° F); duration, 1 hour.

TABLE I. - RELATIVE SHEAR STRENGTHS MEASURED FOR VARIOUS METALS AND METAL OXIDES IN COMPRESSION TWISTING (REFS. 21 AND 22)

Metal	Shear strength, S_s, kg/mm^2	Metal oxide	Shear strength, S_s, kg/mm^2
Aluminum	31.0	Al_2O_3	94.0
Chromium	122.0	Cr_2O_3	134.0
Cobalt	63.0	Co_2O_3	117.0
Copper	49.0	Cu_2O	103.0
Iron	100.0	Fe_2O_3	167.0
		PbO	24.0
Lead	6.80	PbO_2	99.0
		Pb_3O_4	81.0
Molybdenum	121.0	MoO_3	111.0
Nickel	87.0	NiO	119.0
Silver	47.0	Ag_2O	35.0
Titanium	130.0	TiO_2	145.0
Tungsten	128.0	WO_3	140.0
Zinc	18.4	ZnO	126.0
Zirconium	45.0	ZrO_2	121.0

wear areas indicated no evidence for fracture in the sapphire. Even if an oxide of silver should form at the sliding interface, it has a lower shear strength than the metal (see table I and refs. 21 and 22), and shearing in the silver oxide for the experiments with polycrystalline aluminum oxide would be expected. With sapphire, a weakening in the sapphire might still be anticipated. Similar experiments with silver and gold sliding on polycrystalline aluminum oxide revealed no evidence of metal transfer to the aluminum oxide, but friction coefficients were considerably higher than those presented in figure 4.

The results of figures 3 and 4 indicate that chemical bonding is important in the friction resulting from adhesion of metals to aluminum oxide. Possible sites for chemical bonding and van der Waals interaction can be seen with the aid of figure 5 for the (0001) plane of sapphire. The bonding of metals to this surface is discussed in reference 14. In references 11 and 14, the adhesion of metals to single-crystal and polycrystalline aluminum oxide (ref. 14) is related to the free energy of formation of the metal oxides. Experimental evidence for such reactions is presented in reference 23 in contact-angle studies of liquid metals on sapphire.

The schematic diagram of the rider and disk in figure 5 illustrates that three types of bonds are of concern with metals sliding on aluminum oxide: metal to metal, interfacial metal to oxygen where stable oxides will form, and bonding in the aluminum oxide. If

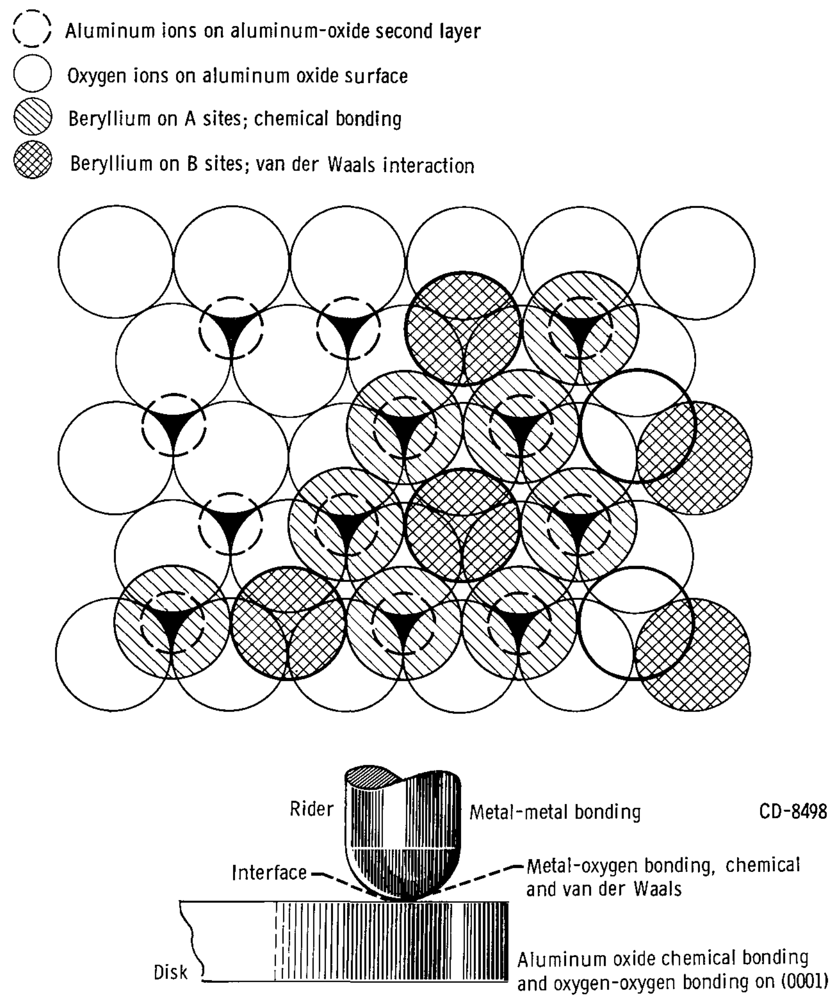
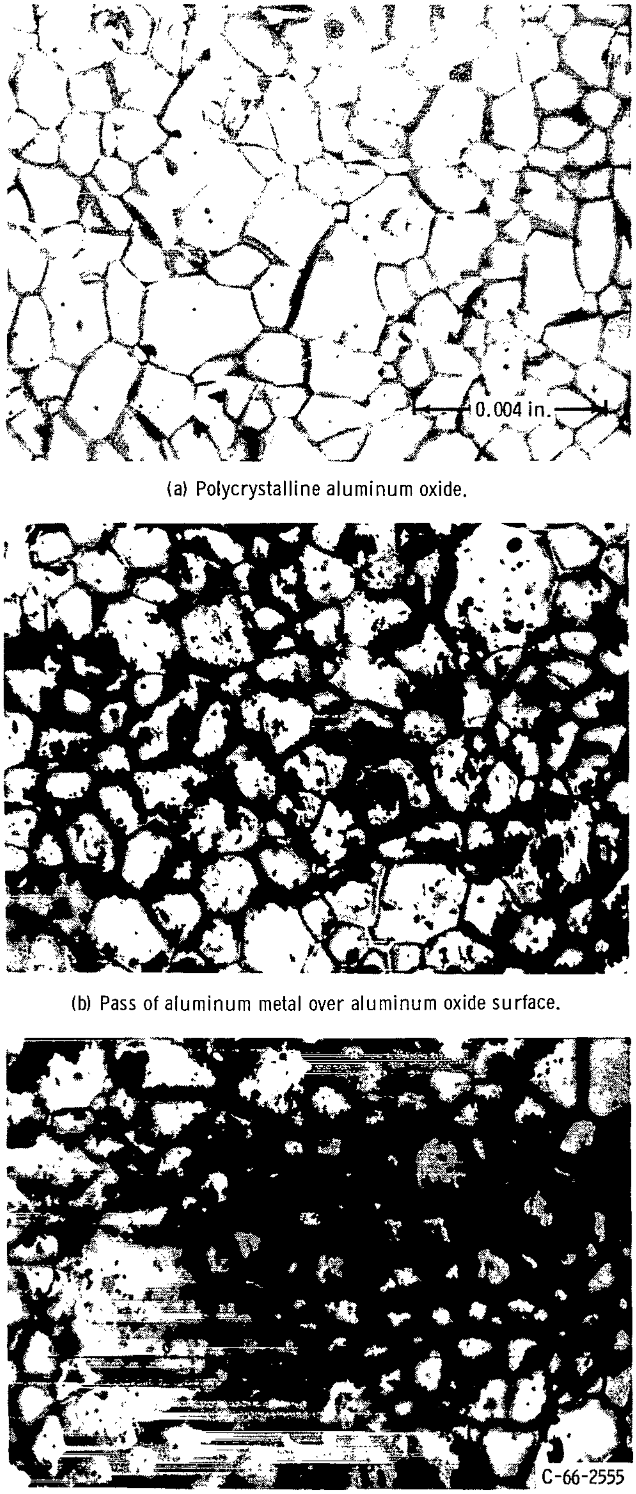


Figure 5. - Nature of surface interaction and bonding of metal to sapphire (0001) plane direction.

metal oxides do form, shear must take place in either the sapphire, the metal oxide, or the metal. With sapphire, the chemical bonding produced stresses and fracture of sapphire. For polycrystalline aluminum oxide, shear of the metal takes place. Table I (refs. 21 and 22) indicates that for most of the metals listed, the stable oxides, if they were to form, would exhibit higher shear strengths than the metal, and shear would be expected to occur in the metal. There are a few exceptions in table I, for example, silver. Figure 6 explains the reason for metals transferring to polycrystalline aluminum oxide and for shear taking place in the metal while fracture occurs in single-crystal aluminum oxide. Examination of the surface of polycrystalline aluminum oxide indicates a preferred adherence of metal to the grain boundary region of the aluminum oxide. Most of the metal transferred to the aluminum oxide surface is in the grain boundary region. The bulk grains represent a satisfied or equilibrium state for the oxygen-aluminum lattice.



(a) Polycrystalline aluminum oxide.

(b) Pass of aluminum metal over aluminum oxide surface.

(c) Repeated passes of metal over aluminum oxide surface.

Figure 6. - Polycrystalline aluminum oxide surface with and without transferred aluminum.

The grain boundary, in contrast, is a stressed or distorted arrangement of atoms that serves as a connecting link between the orientations of two adjacent grains or individual crystallites. The highly stressed condition is demonstrated for aluminum oxide in that it will almost always fracture intergranularly (ref. 15).

If chemical reaction of metals with the oxygen of the aluminum oxide accounts for a portion of the observed adhesion, a greater tendency for reaction to occur at the grain boundaries might be anticipated. With metals, evidence for greater reactivity in grain boundaries is demonstrated by preferred chemical etching of grain boundaries and preferred diffusion into grain boundaries.

With aluminum oxide, it may be anticipated that, in the bulk grains of the material, a stoichiometric distribution of aluminum and oxygen atoms exists in a regular array. Stoichiometric conditions cannot exist in the grain boundaries, for, if they did, there would be no grain boundary; that is, if it is accepted that the boundary is a distorted lattice connecting two undistorted orientations, then at any point in the boundary where distortion exists, nonstoichiometric conditions must exist of necessity. Such a condition increases the tendency for chemical reaction to occur at these sites on the surface when a metal is in sliding contact.

The single-crystal aluminum oxide has no grain boundaries to accommodate preferred sites for chemical reactivity. Here, the metal must compete with aluminum for oxygen. If chemical reaction occurs, lattice strain must occur in the sapphire because of differences in lattice parameters and structures of the two oxides. Such a strain can produce marked stresses in sapphire. It is easy to produce such stresses in sapphire, and reference 16 indicates that simply dropping silicon carbide particles onto a sapphire surface from a few inches is sufficient to produce marked stresses in the sapphire. Some investigators have observed such stresses in other inorganic crystals produced by such objects as dust particles. These stresses, then, must account for the ease of sapphire fracture. Chemical bonding of the metal to sapphire must occur, or only cracks in the sapphire might be anticipated. The removal of discrete particles of sapphire, as shown in figures 2 and 3, indicates marked adhesion of the sapphire to the metal rider.

Since shear takes place with metals sliding on polycrystalline sapphire, the properties of the metals should influence friction. Changes in the mechanical properties of metals with temperature are well known. Further, in many applications where aluminum is oxide in sliding contact with metals, elevated temperatures may be anticipated. A metal that is well characterized with respect to changes in mechanical properties with temperature is copper.

Friction experiments were also conducted with copper sliding on polycrystalline aluminum oxide at various temperatures in vacuum. The results of this experiment are shown in figure 7. In figure 7, increasing temperature produced some decrease in friction coefficient. At temperatures in excess of 370° C (698° F), the friction coefficient

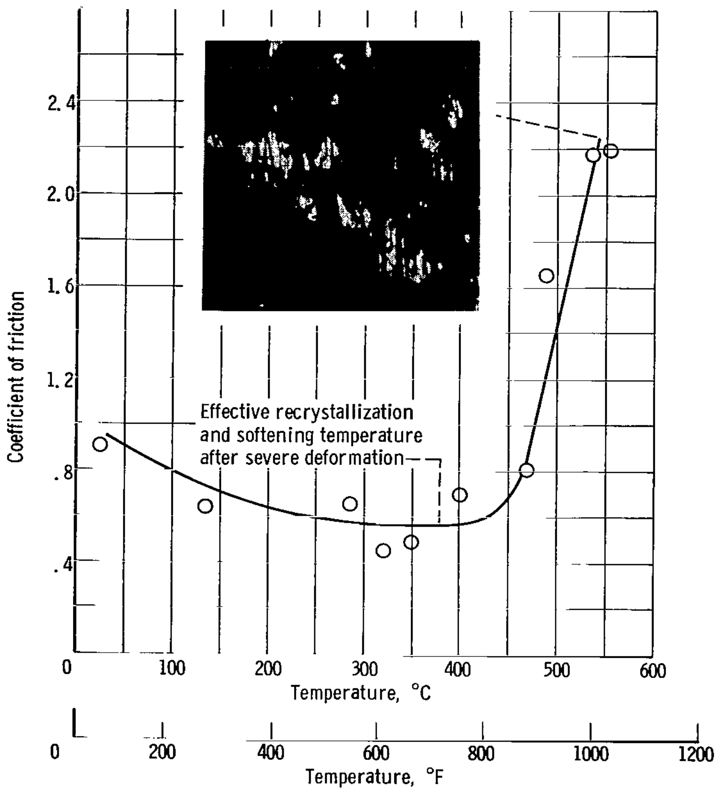


Figure 7. - Coefficient of friction for copper sliding on polycrystalline aluminum oxide in vacuum (10^{-10} torr) at various temperatures. Load, 1000 grams.

rose rapidly, and at 500° C (932° F), it was in excess of 2.0. The recrystallization and marked softening of copper will occur at temperatures below approximately 350° C (662° F) for deformed copper. Copper metal is extremely prone to work hardening and, with increased sliding at the lower temperatures, the decrease in friction may be due to an increase in the hardness of copper. In reference 23, a hardness increase of 150 percent was observed for copper wear debris in sliding-friction experiments. Once recrystallization has occurred, marked softening and an increase in contact area occurs as a result of yielding in the copper; increased shear area accounts for the marked increase in friction observed in spite of reduced shear strength that accompanies softening.

Although adhesion of metals to polycrystalline aluminum oxide occurs in vacuum for most applications, aluminum oxide will not be in a vacuum environment, and surface adsorbates will be present. The presence of adsorbates on aluminum oxide could influence markedly the adhesion and, therefore, friction properties of metals in sliding contact with aluminum oxide.

Experiments were therefore conducted in both vacuum and air with nickel sliding on polycrystalline aluminum oxide to determine the influence of adsorbed films and metal

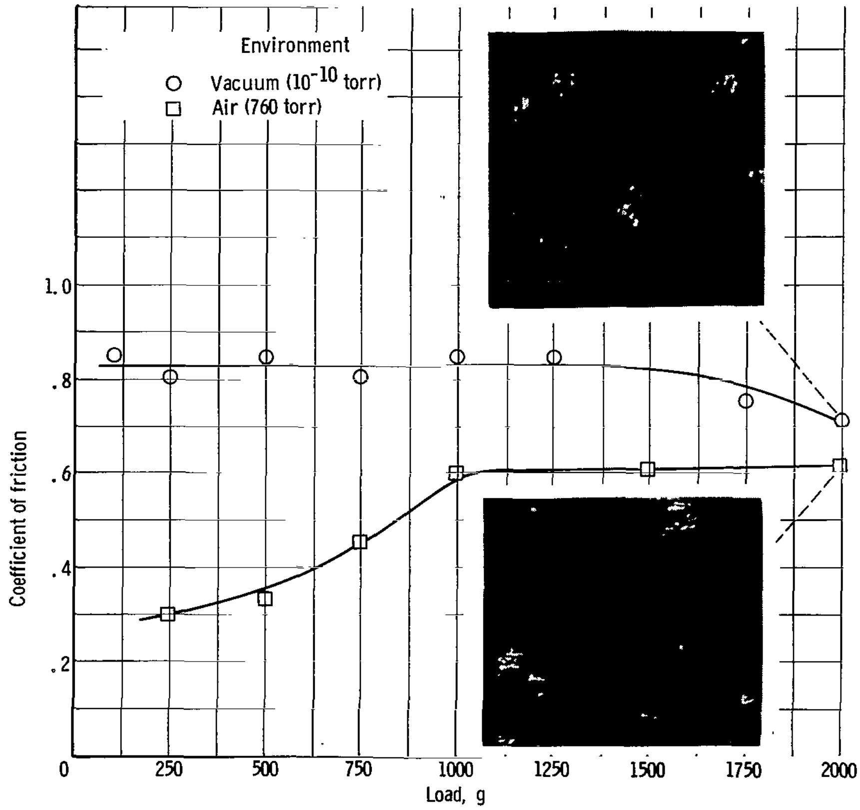


Figure 8. - Coefficient of friction for nickel sliding on polycrystalline aluminum oxide in vacuum and in air. Load, 1000 grams; sliding velocity, 0.013 centimeter per second; specimens outgassed with electron bombardment.

oxides on the friction characteristics of a metal - aluminum oxide combination. The results obtained are presented in figure 8. At a load of 250 grams, a marked difference in friction coefficients exists. As the load on the nickel rider specimen is increased, however, the friction coefficient for the air experiment increases rapidly. Photomicrographs taken after each experiment reveal the presence of transferred metal to aluminum oxide. The bonding, then, of nickel to aluminum oxide occurred with surface films present.

SUMMARY OF RESULTS

The following experimental results were obtained in an investigation of aluminum oxide (single-crystal and polycrystalline) sliding in contact with various metals in vacuum:

1. With metals in sliding contact with single-crystal aluminum oxide, the adhesion of metal to aluminum oxide resulted in shear or fracture of the sapphire; with metals in sliding contact with polycrystalline aluminum oxide, shear occurred in the metal.

2. The friction coefficients for cubic metals sliding on polycrystalline aluminum

oxide correlated with values calculated from bulk shear strength of the metals. The friction coefficients for hexagonal metals sliding on the aluminum oxide were markedly less. These differences in friction for cubic and hexagonal metals are related to differences in crystallographic slip behavior.

3. At relatively moderate conditions (load and low speeds), adhesion of metals to aluminum oxide occurred in air as well as in vacuum.

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