

NASA TECHNICAL NOTE



NASA TN D-3794

c.1

LOAN COPY: RETURN TO  
AFWL (WLIL-2)  
KIRTLAND AFB, N MEX

0130506



TECH LIBRARY KAFB, NM

NASA TN D-3794

# RECRYSTALLIZATION AND PREFERRED ORIENTATION IN SINGLE-CRYSTAL AND POLYCRYSTALLINE COPPER IN FRICTION STUDIES

*by Donald H. Buckley*  
*Lewis Research Center*  
*Cleveland, Ohio*





0130506

NASA TN D-3794

RECRYSTALLIZATION AND PREFERRED ORIENTATION IN SINGLE-  
CRYSTAL AND POLYCRYSTALLINE COPPER IN FRICTION STUDIES

By Donald H. Buckley

Lewis Research Center  
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

---

For sale by the Clearinghouse for Federal Scientific and Technical Information  
Springfield, Virginia 22151 - CFSTI price \$3.00

# RECRYSTALLIZATION AND PREFERRED ORIENTATION IN SINGLE-CRYSTAL AND POLYCRYSTALLINE COPPER IN FRICTION STUDIES

by Donald H. Buckley

Lewis Research Center

## SUMMARY

Friction experiments were conducted in vacuum ( $10^{-11}$  torr) with single-crystal and polycrystalline copper sliding on aluminum oxide. The studies were made with a hemispherical copper rider sliding on the flat surface of a rotating disk of aluminum oxide. The sliding speed was 0.001 centimeters per second, and the load varied from 50 to 1000 grams. The copper single crystal was oriented with the (111) plane parallel to the sliding interface and was oriented to slide in the preferred [110] slip direction.

This investigation indicated that at light loads the friction coefficient for the single-crystal copper is about 0.4, compared to 1.2 for the polycrystalline copper. At loads greater than 50 grams, the friction coefficients approach a common value because of the recrystallization and preferred orientation of both single-crystal and polycrystalline copper.

## INTRODUCTION

The influence of grain boundaries on the mechanical behavior of metals has been studied in considerable detail through the years (refs. 1 to 4). It is well established that the presence of grain boundaries in metals markedly influences the rate of strain hardening and hence the resolved shear stress. Since metal friction is a process of shear at the interface, grain boundaries should influence friction characteristics of materials in sliding contact.

Grain boundaries are transitional regions which link adjacent grains in a polycrystalline material. The atoms of each grain are oriented in a well-defined crystal lattice. The boundary serves to make the transition from the orientation of one grain to that of its neighbors. The width of a boundary, as well as the energy associated with it, depends upon the mismatch in the orientation of adjacent grains. Because the boundary

region represents atoms which are not in a balanced or equilibrium state, as in the grain itself, but rather in some distorted state, chemical reactions and diffusion will occur more rapidly there than in the bulk grains. These chemical reactions can influence the friction process.

With materials in sliding contact, high surface temperatures generated as a result of frictional heating may cause recrystallization (change in grain size) at the interface of contacting metals. The severe plastic deformation of material at the interface makes recrystallization more likely by lowering the temperature at which recrystallization will occur. Further, the influence of preferred orientation of crystallites (surface texturing) of metals in sliding contact is of interest. Since surface texturing occurs in rolling and drawing of metals, it would seem reasonable to assume that it also occurs in sliding. Even with recrystallization, texturing can take place with the newly formed crystallites.

This investigation was conducted to determine the friction characteristics and the nature of interfacial recrystallization of single-crystal and polycrystalline copper in sliding contact with aluminum oxide in vacuum. Friction experiments were conducted with a hemisphere of copper sliding on a flat-surface disk of polycrystalline aluminum oxide. The sliding speed was 0.001 centimeter per second, and the load varied from 50 to 1000 grams. The ambient pressure was  $10^{-11}$  torr and the temperature was  $20^{\circ}$  C.

These experiments were conducted in vacuum to reduce the influence of surface contaminants and oxides on the friction of copper. No experiments were conducted with copper sliding on copper because of complete seizure in vacuum. Aluminum oxide was selected as the mating surface because earlier work has shown that copper will adhere to aluminum oxide and shear will take place in the copper (ref. 5).

## MATERIAL

The single-crystal and polycrystalline copper used in this investigation were all of 99.999-percent purity. The single crystals all had the (111) plane normal to the rod axis to within  $3^{\circ}$ . The polycrystalline aluminum oxide was of high purity (99.8 percent) and high density (99.9 percent) and had an average grain diameter of 0.023 millimeter.

After being finished to specimen shape, the single-crystal and polycrystalline metals were all electropolished with orthophosphoric acid to remove any worked or deformed surface layer. The orientations were then checked with the Laue back-reflection X-ray technique. The specimens were thoroughly rinsed with acetone and alcohol immediately prior to insertion into the vacuum chamber.

The reagent used for dislocation etch-pitting in this investigation is the same as that described in reference 6. It consisted of four parts of a saturated ferric chloride solu-

tion, four parts hydrochloric acid, one part acetic acid, and a trace of bromine (one drop per liter).

Polycrystalline aluminum oxide ( $\text{Al}_2\text{O}_3$ ) was selected as a mating surface because of the results obtained with copper sliding on it in an earlier investigation (ref. 5). Adhesion of copper to  $\text{Al}_2\text{O}_3$  occurs during sliding, with shear taking place in the copper. The shear property of the metal is, then, the factor determining friction. When the metals slide on themselves in vacuum, the large increase in true contact area with tangential displacement results in complete welding of the metal specimens.

## APPARATUS

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

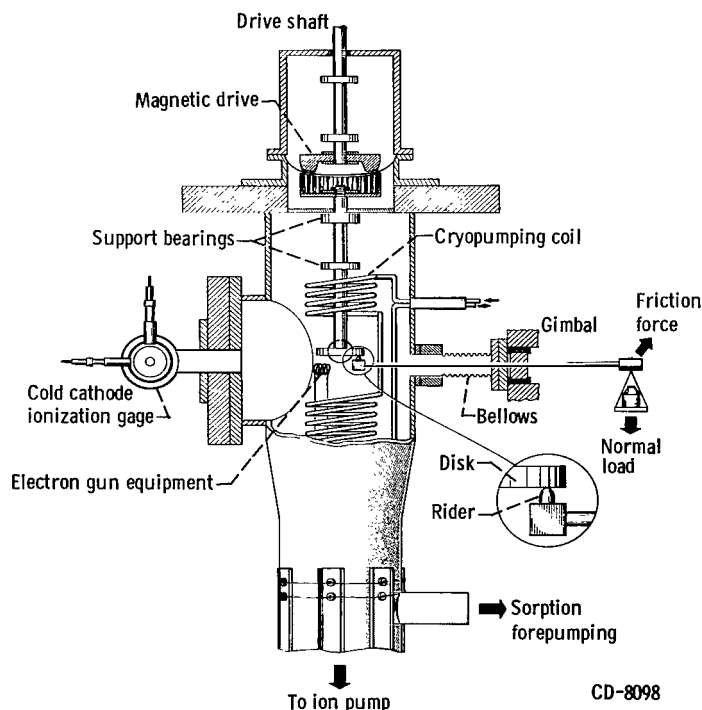


Figure 1. - Vacuum friction apparatus.

the shaft within the chamber. The disk specimen was at the lower end of the shaft (cutaway in fig. 1).

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 opposite end of the retaining arm from the rider specimen was connected to a strain-gage assembly. The assembly was used to measure frictional force. Load was applied through a dead-weight loading system. A 500-liter-per-second ionization pump and a vac-sorption forepump were attached to the lower end of the specimen chamber. The chamber pressure was measured with a cold-cathode ionization gage adjacent to the specimen. A diatron-type mass spectrometer (not shown in fig. 1) was used 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.

## PROCEDURE

The disk specimens of polycrystalline  $\text{Al}_2\text{O}_3$  were scrubbed with levigated alumina and rinsed with water and then with alcohol prior to insertion in the vacuum chamber. The metal specimens after electropolishing were rinsed with acetone and alcohol prior to being placed in the vacuum chamber. After pump down, the entire vacuum system was baked out overnight. The specimens were then electron-bombarded for 30 minutes to remove residual surface oxides and contaminants. During electron bombardment the specimen temperature was 400° C. Immediately after the specimens cooled to room temperature, the experiment was started.

## EXPERIMENTAL RESULTS

The severe plastic deformation and high interface temperature which can be achieved in sliding-friction experiments create the probability of interfacial recrystallization. This is especially true when very high purity metals are used. The presence of alloying elements tends to increase the recrystallization temperature and thus decrease the probability of recrystallization. To determine the mechanical conditions at which surface recrystallization of copper occurs, friction experiments were conducted in vacuum with single-crystal and polycrystalline copper sliding on  $\text{Al}_2\text{O}_3$  at various loads. The sliding speed of these experiments was very low, 0.001 centimeter per second. The results of the experiments are presented in figure 2.

Examination of figure 2 indicates that, at a load of 50 grams, the friction coefficient

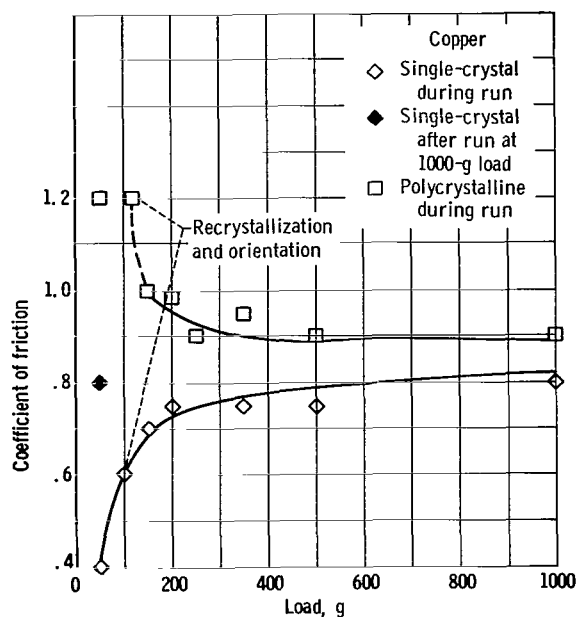


Figure 2. - Coefficient of friction for single-crystal and polycrystalline copper sliding on polycrystalline aluminum oxide in vacuum ( $10^{-11}$  torr). Copper oriented with (111) plane parallel to sliding interface; sliding velocity, 0.001 centimeter per second; no external specimen heating.

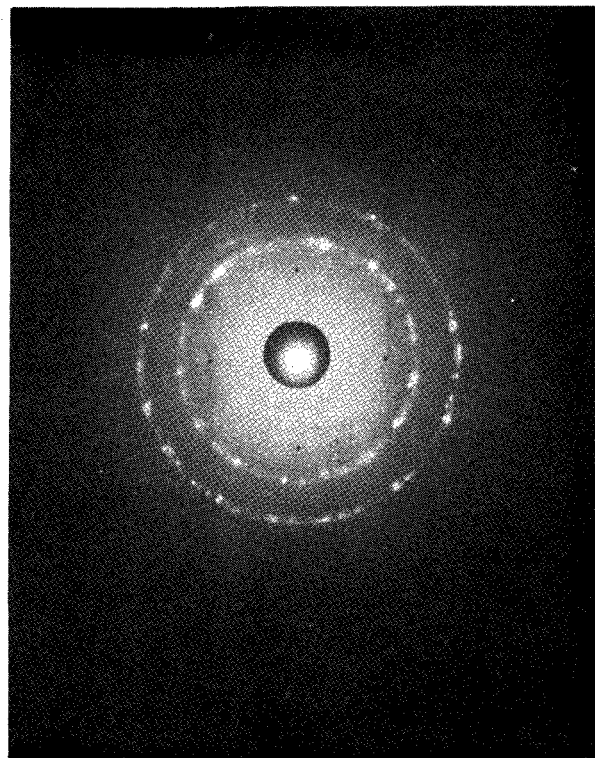
for randomly oriented polycrystalline copper (1.2) is three times greater than that for a copper single crystal with the (111) plane parallel to the sliding interface (0.4). Increasing the load results in an increase in the coefficient of friction for the copper single crystal and in a decrease in the coefficient of friction for the polycrystalline copper. If the loading were continued beyond 1000 grams, at some point the friction coefficients would become essentially the same.

The marked change in friction with increasing load for the two forms of copper may be related to surface recrystallization. X-ray analysis of the sliding surface after sliding with a load of 1000 grams indicated the presence of a recrystallized layer on the wear surface of both the single-crystal and polycrystalline materials (fig. 3). Although the X-ray technique did not detect recrystallized layers at the lighter loads, electron diffraction revealed a recrystallized surface layer at a load of 100 grams for the single crystal (see fig. 4). At 50 grams recrystallized film was not noted; however, subsurface to the wear area a high concentration of Kikuchi lines and lattice distortion was observed. This wear area itself gave a pattern for severely deformed material.

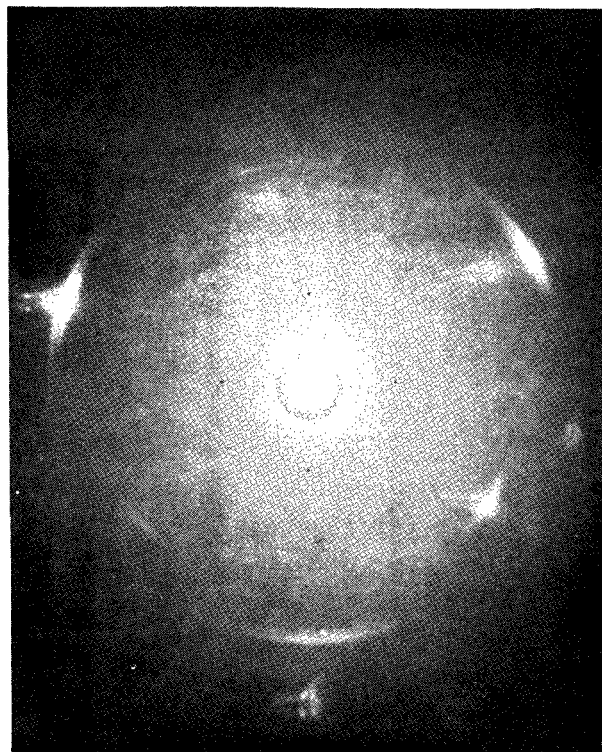
The difference in friction coefficient for the single-crystal and polycrystalline copper at a load of 50 grams was anticipated from shear and deformation behavior of single-crystal polycrystalline materials. With the single crystal oriented for slip on the preferred slip plane (111) in the preferred slip direction [110], a minimum of shear stress



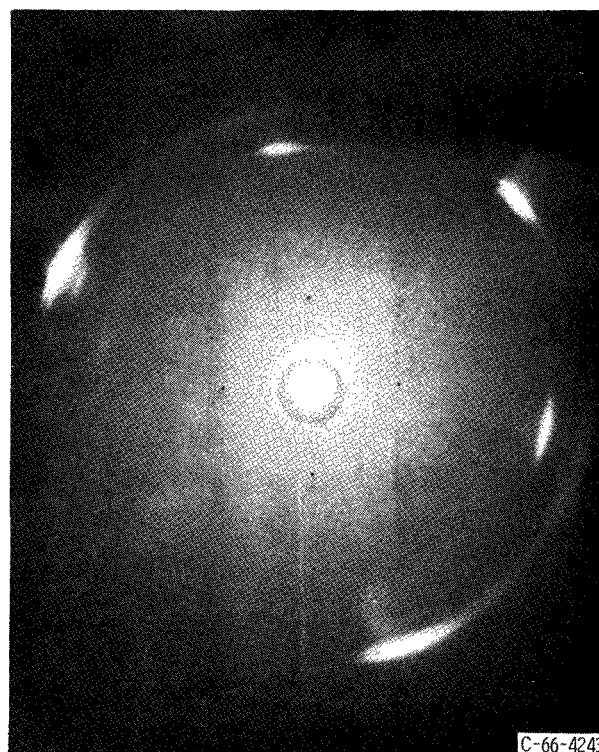
(a) Single-crystal copper before experiment.



(b) Polycrystalline copper before experiment.



(c) Single-crystal copper after experiment.



(d) Polycrystalline copper after experiment.

Figure 3. - X-ray Laue patterns for wear areas in single-crystal and polycrystalline copper after sliding on aluminum oxide in vacuum ( $10^{-11}$  torr). Sliding velocity, 0.001 centimeter per second; load, 1000 grams; no external specimen heating.



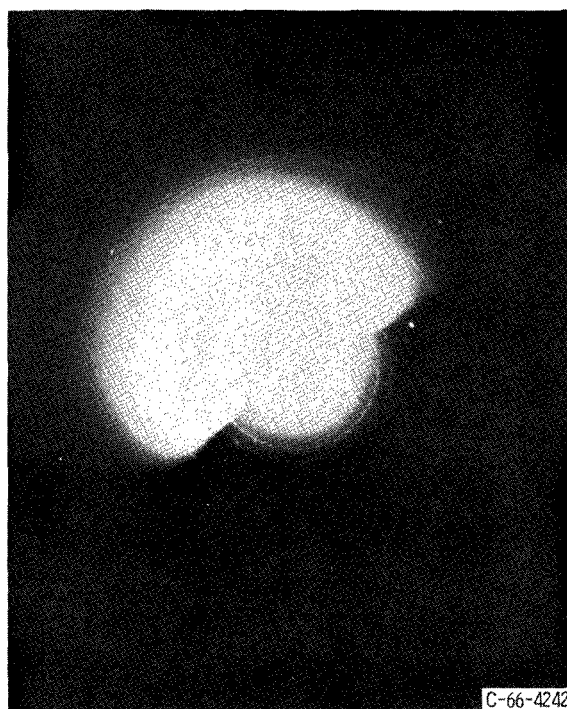


Figure 4. - Electron diffraction pattern for single crystal of copper after sliding on aluminum oxide in vacuum ( $10^{-11}$  torr). Sliding velocity, 0.001 centimeter per second; load, 100 grams; no external specimen heating.

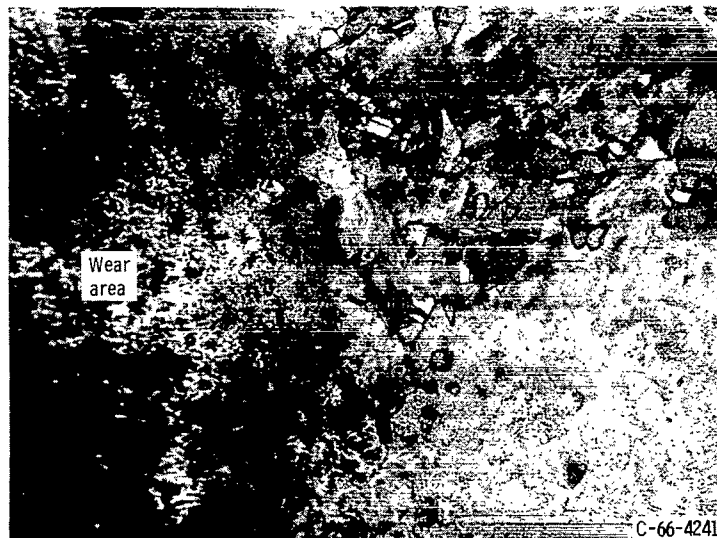
might be anticipated in sliding. For copper, the (111) plane is the most dense atomically and the spacing between planes is the greatest. The resistance to shear is therefore at a minimum on this plane. However, the polycrystalline material contains grain boundaries and individual crystallites of varied crystallographic orientations. Therefore, any crystallite orientation other than the preferred could be expected to exhibit a higher shear stress. Furthermore, the grain boundaries act as barriers to the motion of slip planes and slip plane dislocations.

Although a recrystallized surface layer was observed with electron diffraction for the 100-gram-load condition, the friction coefficient was about one-half that obtained for the polycrystalline material. These results indicate that the subsurface material still exerts an influence on friction. As mentioned earlier, with the 1000-gram load the recrystallized layer was thick enough to be detected by X-rays. As the recrystallized interface layer thickens (with increasing load), its effect on friction becomes significant and friction coefficients for the single-crystal and polycrystalline materials approach one another.

The wear-scar regions for the single-crystal and polycrystalline copper are shown after etch pitting (fig. 5). After loading with 1000 grams, the grains adjacent to the wear scar for the polycrystalline material exhibited marked twinning. The twins, however,



(a) Polycrystalline copper. X250.



(b) Recrystallized area on single-crystal copper. X150.

Figure 5. - Photomicrographs of wear areas in single-crystal and polycrystalline copper after sliding on aluminum oxide in vacuum ( $10^{-11}$  torr). Sliding velocity, 0.001 centimeter per second; load, 1000 grams; no external specimen heating.

look more like annealing than deformation twins. Because face-centered cubic metals are not noted for deformation twinning (only body-centered cubic and closely packed hexagonal are so noted), and because high interface temperatures are achieved, these twins can be considered to be annealing twins. With the single crystal, recrystallization occurred not only in the wear area but also on the surface of the crystal adjacent to the wear area, as shown in the photomicrograph (fig. 5(b)). These "surface crystallites" are extremely interesting. A very light electropolish readily removed them, and they did not reappear on subsequent etch pitting. These results indicate that the thickness of the recrystallized layer was, indeed, very small.

## DISCUSSION

The threefold difference in friction coefficient for the single-crystal and polycrystalline copper of figure 2 is extremely interesting. The marked difference in the stress-strain curve for copper is shown in figure 6, which was taken from reference 3. Polycrystalline copper shows shear stress, for a 30 percent strain, approximately three times greater than that of single-crystal copper.

If the coefficient of friction is, as predicted by the classic theory of friction, the shear force divided by the yield strength, little difference in friction should be observed

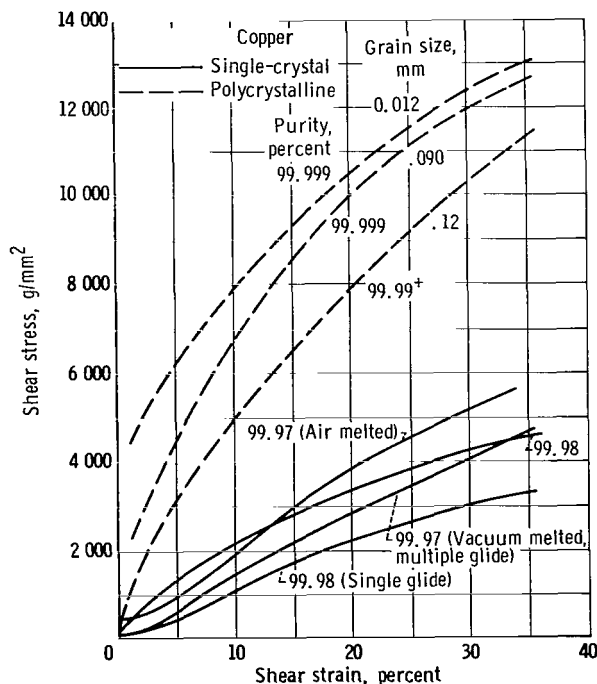


Figure 6. - Curves of shear stress as function of shear strain for copper (ref. 3).

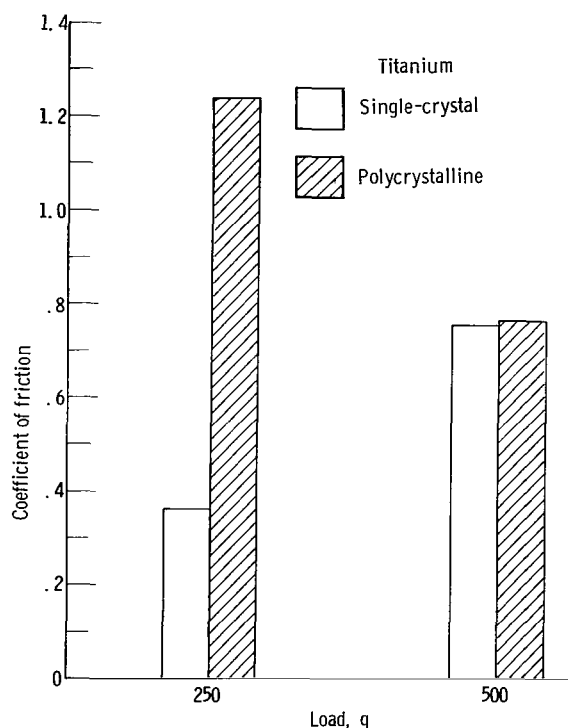


Figure 7. - Coefficient of friction for single-crystal and polycrystalline titanium sliding on polycrystalline titanium in vacuum ( $10^{-9}$  torr). Titanium oriented with prismatic plane parallel to sliding interface (deviation,  $11^\circ$ ); sliding velocity, 2.28 centimeters per second; no external specimen heating. (Ref. 7.)

for the two crystalline forms of copper. The shear strength should change with the yield strength. One fairly evident explanation appears to prevail. It is related to the adhesion of the copper to the aluminum oxide with shear taking place in the metal. Examination of the contact area after the experiments revealed that large pieces of copper adhered to the aluminum oxide with very little difference in amount for the two forms of copper.

The differences in friction behavior (fig. 2) rest with the shear behavior of the copper. For the single crystal with a 50-gram load, shear occurs along the (111) plane, and the effect of strain hardening with sliding increases the shear stress at a rate only one-third of that observed for the polycrystalline material (fig. 6). As the load is increased, however, recrystallization and orientation occur at the interface. Because the polycrystalline film has a higher shear strength than the single crystal, an increase

in friction is observed. The grain boundaries act as barriers to the motion of slip-plane dislocations in the individual crystallites and therefore increase shear stress. For the polycrystalline material at a load of 50 grams, the strain-hardening rate is very high because of the random nature of crystallite orientations at the interface (fig. 6). As the load is increased, recrystallization and orientation again take place at the interface. Preferred orientation of the crystallites at the interface reduce shear strength, and a reduction in friction is observed. Since the nature of the surface film is the same at high loads, the friction coefficients become essentially the same.

Results similar to those obtained in this investigation with copper sliding on aluminum oxide were observed in reference 7 with single-crystal and polycrystalline titanium. The data obtained are shown in figure 7. With titanium the friction coefficient for the single crystal on the preferred slip system was appreciably less than that for the polycrystalline form. Load changes resulted in a decrease in friction for the polycrystal and an increase for the single crystal. With titanium, recrystallization occurred at a higher load (500 grams) than it did in this study (100 g). This fivefold difference in load to initiate interfacial surface recrystallization is not surprising since a marked difference in recrystallization temperature exists for these two metals (higher for titanium).

## SUMMARY OF RESULTS

From the results of an investigation with single-crystal and polycrystalline copper sliding on aluminum in vacuum ( $10^{-11}$  torr) the following summation is made:

Although differences in friction coefficients exist for single-crystal and polycrystalline copper at light loads, a nearly common friction coefficient is obtained for the two forms of copper at higher loads. This effect is due to surface recrystallization and preferred orientation of copper at the sliding interface. X-ray data indicate the surface recrystallized layers to be the same at a 1000-gram load.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, October 13, 1966,  
129-03-13-02-22.

## REFERENCES

1. Dieter, George E.: Mechanical Metallurgy. McGraw-Hill Book Co., Inc., 1961.
2. Barrett, Charles S.: Structure of Metals, Crystallographic Methods, Principles, and Data. McGraw-Hill Book Co., Inc., 1943.
3. McLean, Donald: Grain Boundaries in Metals. Clarendon Press, Oxford, 1957.
4. Jaswon, M. A.; and Richman, M. H.: Some Mechanical Effects of Grain Boundaries. Tech. Rep. No. 6, Brown University, June 1964.
5. Buckley, Donald H.: Friction Characteristics of Single-Crystal and Polycrystalline Aluminum Oxide in Contact With Various Metals in Vacuum. NASA TN D-3593, 1966.
6. Young, F. W., Jr.: Etch Pits at Dislocations in Copper. Jour. Appl. Phys. vol. 32, no. 2, Feb. 1961, pp. 192-201.
7. Buckley, Donald H.: Influence of Crystal Orientation on Friction Characteristics of Titanium Single Crystals in Vacuum. NASA TN D-2988, 1965.

*"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."*

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

## NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

**TECHNICAL REPORTS:** Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

**TECHNICAL NOTES:** Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

**TECHNICAL MEMORANDUMS:** Information receiving limited distribution because of preliminary data, security classification, or other reasons.

**CONTRACTOR REPORTS:** Technical information generated in connection with a NASA contract or grant and released under NASA auspices.

**TECHNICAL TRANSLATIONS:** Information published in a foreign language considered to merit NASA distribution in English.

**TECHNICAL REPRINTS:** Information derived from NASA activities and initially published in the form of journal articles.

**SPECIAL PUBLICATIONS:** Information derived from or of value to NASA activities but not necessarily reporting the results of individual NASA-programmed scientific efforts. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

*Details on the availability of these publications may be obtained from:*

SCIENTIFIC AND TECHNICAL INFORMATION DIVISION  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
Washington, D.C. 20546