INFLUENCE OF SURFACE FILMS ON FRICTION AND DEFORMATION OF SINGLE-CRYSTAL AND POLYCRYSTALLINE GOLD

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ABSTRACT

Sliding friction experiments were conducted with single-crystal and polycrystalline gold with various media present. These included air, water, hexadecane and hexadecane with 0.02 weight percent stearic acid; experiments were also conducted with various gold halide films formed on the surface. The alloying of copper with gold was also employed to improve the adsorbability of various media to gold. Results of the investigation indicate that ordinary atmospheric contaminants influenced the friction and deformation of gold. Water will not wet clean gold and thus provides the same friction and deformation behavior as that observed in air. The halides of gold all reduced friction and deformation of gold; the most effective halides were the chlorides for friction and iodides for deformation. The addition of a small concentration of copper to gold improved the ability of an organic acid to lubricate gold.
Sliding friction experiments were conducted on single and polycrystalline gold with various media present. The media included air, water, hexadecane, hexadecane with 0.02 weight percent stearic acid carbon tetrachloride experiments were also conducted with various halides of gold formed on the surface. Friction experiments were conducted with a 1.6-millimeter-diameter sapphire ball sliding on a disk of gold at a sliding speed of 0.005 millimeter per second with the ball loaded against the disk at loads from 7 to 200 grams. The single-crystal orientation of gold examined were the (001) plane [100] direction and the (001) plane [110] direction.

The results of this investigation indicate that normal atmospheric contaminants, such as water vapor, influence the friction and surface deformation of gold. Water was found not to wet a clean gold surface, and it afforded no better surface protection than the normal atmospheric contaminants. The halides of gold all reduced friction and deformation. Of the halides, gold chlorides gave the greatest reduction in friction when compared to results obtained in the absence of surface films (under hexadecane). The addition of small concentrations of copper (0.25 to 5.0 wt. %) were found to improve, markedly, the ability of stearic acid to lubricate a gold surface. With single crystal experiments, the crystallographic direction influenced friction and deformation. On the (001) plane, friction and material removal were lower in the [100] direction than in the [110] direction.

INTRODUCTION

A metal of considerable interest in the field of lubrication, particularly in the area of electrical contacts, is gold. Gold is used in such applications because of its general resistance to the formation of undesirable surface films which increase interfacial contact resistance. Gold is also used in other specialized areas of lubrication such as the lubrication of bearings for a space environment. It is gold's resistance to the formation
of surface films, however, which has given rise to friction and wear problems. Surface films are frequently beneficial from the standpoint of friction and wear. Studies have been conducted on the friction and wear behavior of gold as well as on the ability of various lubricants to reduce its friction and wear (refs. 1 and 2).

Gold does not form a stable oxide at temperatures less than $900^\circ$ C in air or oxygen (refs. 3 and 4). Thus the effect of basic properties of the metal on friction and deformation in the absence of a surface oxide can readily be studied with this metal. Further, with simple vacuum degassing, a clean surface can be obtained; the influence of various relatively pure, surface active media on the friction and wear of gold can therefore be determined. While gold does not normally oxidize in air or oxygen, oxides (namely $\text{Au}_2\text{O}_3$) have been generated on a gold surface by electrochemical techniques (ref. 5).

The ability of hydrocarbons to adsorb on gold has been studied (refs. 6 and 7). It has been observed that gold surfaces will catalyze the formation of polymer films from hydrocarbon vapors (refs. 8 and 9). It has been shown that lubricants containing chloride provide the best surface protection for gold (ref. 2).

The objectives of this investigation were to determine (1) the friction and deformation behavior of single-crystal and polycrystalline gold, (2) the influence of various surface films on friction and deformation of gold, and (3) the effect of a surface active metal (copper) present in gold as an alloying agent on the ability to form protective surface films. Experiments were conducted with a sapphire sliding on either single-crystal or large grained polycrystalline gold surface. The single-crystal orientations examined were the (001) plane [100] direction and the (001) plane [110] direction. All experiments consisted of a single pass across the surface. The materials examined in contact with the gold surface included air, hexadecane, water, carbon tetrachloride, and the halides of gold formed by reaction on the surface.

**MATERIALS**

The gold used in these studies was 99.999 percent pure. The single crystals were solid gold disks, 1.27 centimeters in diameter by 1.27 centimeters thick. The polycrystalline specimens were solid gold disks, 2.54 centimeters in diameter by 1.27 centimeters thick. The hexadecane was reagent grade, olefin free. The water was distilled and freshly boiled. The carbon tetrachloride was reagent grade, as were all of the materials used in the formation of the halides of gold.

**APPARATUS**

The apparatus used in this investigation is shown schematically in figure 1. The apparatus consisted basically of a Microbierbaum hardness tester (ref. 10) to which a drive
Figure 1. - Sliding friction apparatus.
motor was attached in order to provide uniform motion of the crystal specimens under examination. The drive motor, through a gear assembly, moved the lower specimen from right to left. The speed at which the crystal moved across the stage was 0.005 millimeter per second.

The rider specimen was a sapphire ball 1.6 millimeters in diameter which was locked in a holder. The arm containing the rider had a strain gage assembly for measuring frictional force. The output was recorded on a strip chart recorder. The sapphire ball was loaded against the gold surface by the application of dead weights directly over the rider. The load range was 7 to 200 grams.

EXPERIMENTAL PROCEDURE

The gold used was fully annealed prior to the start of each experiment and the surface was electropolished. For those experiments in which a film was present on the surface, the gold was heated in vacuum to 500°C; after cooling the gold to room temperature, the material to be adsorbed to the surface was admitted to the vacuum chamber, and the system was bled to atmospheric pressure in the media.

Water would not wet the clean gold surface but formed a drop (see fig. 2(a)), and this experiment was conducted by forming a water "cap," that is, a hemisphere of water which made contact with the gold disk surface at the circumferential edge (see fig. 2(b)). The gold specimens were removed from the vacuum system and mounted on a glass slide with rubber cement.

The single-crystal orientations of the gold were determined by Laue X-ray techniques. The orientations specified are within 2°.

Because of the extremely soft nature of annealed gold, it is very susceptible to the embeddibility of hard particles. For this reason, the gold surfaces used were polished with diamond paste (conventional metallurgical) rather than aluminum oxide prior to electropolishing. Reference 11 has shown that even electropolishing will not remove embedded aluminum oxide from gold. After diamond polishing and electropolishing, the

![Water drop and cap](image-url)  
(a) Water drop.  
(b) Water cap.  
Figure 2. Technique used to maintain water layer over gold specimen surface.
surfaces used herein were heated to 900°C in air to burn out the residual diamond. This was done prior to the vacuum adsorption treatment already described.

Two approaches were used to develop halide films on the surface of gold, (1) fused salt reactions as described in reference 12 and (2) a reaction of the gold surface at red heat in vacuum with acid vapors. Both were found equally effective. The later technique was adapted because it did not roughen the surface as did the fused salt reaction.

RESULTS AND DISCUSSION

Effect of Atmospheric Contaminants on Friction and Deformation

The use of gold in sliding electrical contact applications, as well as other specialized areas of lubrication, warrants an understanding of the influence of conventional atmospheric constituents on friction and deformation behavior. In order to gain such an understanding, sliding friction experiments were conducted (1) in air, (2) under hexadecane (to exclude these contaminants), and (3) with water, one of the chief constituents of the atmosphere known to have an effect on the friction and deformation behavior of many materials. The results obtained in some of these experiments are presented in figures 3 to 6.
Figure 4. Photomicrographs of wear surfaces to polycrystalline gold after sliding in air at 100-gram load. Sliding velocity, 0.005 millimeter per second; temperature, 20° C.
The results of sliding friction experiments with single-crystal and polycrystalline gold in moist air (50% humidity) are presented in figure 3. The coefficient of the sliding wear track, as developed from a single pass across the surface, are plotted as a function of load. At light loads, the friction coefficient for both single-crystal and polycrystalline gold deviate from Amonton's law. At loads greater than 25 grams this deviation was not observed. As will be shown later, the deviation at light loads may be attributed to the presence of physically adsorbed films.

The friction coefficients for single-crystal and polycrystalline gold were essentially the same. In contrast, however, the deformation of the gold surface with changes in load was greater for the single-crystal (001) [110] orientation than with the randomly oriented grains of the polycrystal. This difference might be anticipated since the single crystal will deform plastically more readily than the polycrystal.

Sections of the wear surface on polycrystalline gold are shown in figure 4. In figure 4(a), slip bands are visible about the wear track. They are seen to extend some distance in front of the terminal point of the track. The photograph was taken in one large grain of the polycrystalline disk. The bands are then associated with the orientation of the particular grain identified in the photo. The presence of slip bands in front of the terminal point of the rider indicates a region of deformed material which is already work hardened to some degree as will be shown later. This region of material which deformed plastically ahead of the rider has been referred to as a frontal bulge, and the degree of deformation and extent of work hardening in this area has been very carefully studied for copper, another face-centered cubic metal (ref. 13). Figure 4(b) indicates the generation of wear debris in the process of sliding across a grain boundary. A buildup of material causes a segment on the surface of the solid disk to lose contact with the sapphire rider; this interrupted contact is visible on the surface as an undeformed region just beyond the grain boundary and the debris.

In order to exclude the normal atmospheric contaminants from directly influencing friction and deformation behavior, some experiments were conducted with polycrystalline gold under hexadecane. The gold specimens were heated to red heat in vacuum, and then, after cooling to room temperature, high purity hexadecane was bled into the vacuum chambers containing the gold. The sample was removed, and a liquid layer of hexadecane (formed by condensation) was maintained on the surface as shown in figure 2(b). Friction and deformation data as a function of load in sliding friction experiments are presented in figure 5. The coefficient of friction at light loads was higher than in air. At the heavier loads, the friction coefficient was essentially the same. The width of the wear track was greater at all loads, indicating greater deformation of the gold in sliding friction experiments in the absence of atmospheric contaminants. The friction and deformation data indicate that the normal physically adsorbed atmospheric contaminants do provide some degree of surface protection for gold.
Figure 5. - Coefficient of friction and width of wear track for single pass for sapphire slider on polycrystalline gold run under hexadecane. Sliding velocity, 0.005 millimeter per second; temperature, 20°C.

Figure 6. - Coefficient of friction and width of wear track for single pass of sapphire slider on polycrystalline gold under water. Sliding velocity, 0.005 millimeter per second; temperature, 20°C.
The atmospheric contaminant which is known to influence lubrication behavior markedly is water vapor. Friction and deformation experiments, therefore, conducted with water on the surface of gold. The gold was again heated to red heat in vacuum as described earlier and, after cooling to room temperature, freshly boiled triple distilled water vapor was admitted to the system. Water would not wet the clean gold surface. The water formed spheres which would not remain on the surface. Water was maintained on the surface by the technique shown in figure 2(b) for the sliding experiments. While gold has been shown to be hydrophobic, an oxidized gold surface is hydrophilic (ref. 14).

The results obtained in sliding friction experiments with water are presented in figure 6. The results of figure 6 indicate that the data are essentially the same as those of figure 3 for experiments conducted in air. Since gold does not form stable oxides at room temperature in air, water vapor may be and is most likely, the principle surface contaminant in the atmosphere accounting for the differences in the values of figures 3 and 5. Water vapor physically adsorbed to gold appears then to reduce friction at light loads and reduce deformation at all the loads examined in these studies.

Influence of Halide Lubricating Films on the Friction and Deformation of Gold

While it is useful to know that water vapor reduces the friction and deformation behavior of gold, the material is still not effectively lubricated in this environment. The friction coefficients of figures 3 and 6 are much higher than that normally associated with boundary lubrication. The problem then exists as to what will provide an effective lubricating film. Oxides for most metals are very effective in reducing friction. Gold does not readily form oxides, but it does form halides. The halides of gold were, therefore, examined to determine their ability to reduce the friction and deformation of gold. If such films are effective in reducing friction and deformation, they could be preformed on surfaces to be lubricated, such as electrical contacts or operated in an environment of the media (liquid) for such applications as bearings.

With polycrystalline materials there are a variety of orientations present on a surface. Some of the grain orientations may be more chemically active than others. The reason for this is more readily apparent with the aid of a discussion of figure 7. The photomicrograph of figure 7 is of a polycrystalline gold surface reacted with hydrogen iodide. Examination of various grains revealed a marked variation in the color and, therefore, film thickness of the gold iodide on each grain. The variation is due simply to the differences in the rates of reactivity of various grain (orientations) of gold with hydrogen iodide. The variations in reactivity of various orientations of different metals is well known (ref. 15).
Figure 7. - Photomicrograph of polycrystalline gold after reaction to form gold iodide.

TABLE I. - COEFFICIENT OF FRICTION AND DEFORMATION OF GOLD SURFACE WITH VARIOUS FILMS PRESENT

[Sliding velocity, 0.005 mm/sec; load, 100 g; temperature, 20° C; slider sapphire ball; air environment.]

<table>
<thead>
<tr>
<th>Surface film</th>
<th>Single-crystal (001) surface</th>
<th>Crystallographic direction</th>
<th>Coefficient of friction</th>
<th>Track width, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>None</td>
<td></td>
<td>[110]</td>
<td>0.40</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[100]</td>
<td>0.34</td>
<td>0.18</td>
</tr>
<tr>
<td>AuF</td>
<td></td>
<td>[110]</td>
<td>0.29</td>
<td>0.11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[100]</td>
<td>0.20</td>
<td>0.16</td>
</tr>
<tr>
<td>aAuCl</td>
<td></td>
<td>[110]</td>
<td>0.15</td>
<td>0.11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[100]</td>
<td>0.09</td>
<td>0.16</td>
</tr>
<tr>
<td>aAuBr</td>
<td></td>
<td>[110]</td>
<td>0.28</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[100]</td>
<td>0.23</td>
<td>0.13</td>
</tr>
<tr>
<td>aAuI</td>
<td></td>
<td>[110]</td>
<td>0.25</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[100]</td>
<td>0.22</td>
<td>0.12</td>
</tr>
</tbody>
</table>

aIdentified by X-ray diffraction (from generation of thicker films than used in experiments).
In order to avoid averaging of friction and deformation on various gold surfaces, experiments were conducted with single crystals of gold. The crystals were heated to red heat in vacuum and then reacted while hot with hydrogen fluoride, hydrogen chloride, hydrogen bromide, or hydrogen iodide. The vacuum system was bled to atmospheric pressure in the various halogen media. Thick films were prepared for X-ray analysis. For sliding friction experiments, the films were extremely thin and undetectable to the naked eye (50 - 100 Å).

Friction and deformation results obtained on the (001) plane of gold in two crystallographic directions, the [110] and [100] with various halide films present, are given in table I. Examination of table I indicates that the friction coefficient and deformation were less in a particular direction with any of the films present than in their absence. The most effective film in reducing friction coefficient was the chloride film. With respect to deformation, however, the iodide was more effective.

It is of interest to note in table I that friction coefficient was, in each case, less in the [100] than in the [110] direction. Further, the track width was always greater in the [100] than in the [110] direction. These differences, as will be discussed later, may be related to the nature of surface plowing.

The chloride film on the gold (001) surface was examined at various loads, and the results obtained are presented in figure 8. The curves of figure 3 are also presented for Chloride Film Curve from Figure 3

![Graph](image)

Figure 8. - Coefficient of friction and wear track for single-crystal gold (001) surface with chloride film present. Sliding direction on crystal surface [110]; sliding velocity, 0.005 millimeter per second.
Virgin or undeformed surface
In wear track
Load, 100 g
Load, 200 g

<table>
<thead>
<tr>
<th>Location of microhardness measurement on surface</th>
<th>Microhardness without AuCl film present, kg/mm$^2$</th>
<th>Microhardness with AuCl film on Au surface, kg/mm$^2$</th>
<th>Change in hardness with film, kg/mm$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin or undeformed surface</td>
<td>10.72</td>
<td>14.84</td>
<td>4.12</td>
</tr>
<tr>
<td>In wear track</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Load, 100 g</td>
<td>16.80</td>
<td>21.02</td>
<td>4.22</td>
</tr>
<tr>
<td>Load, 200 g</td>
<td>16.80</td>
<td>21.02</td>
<td>4.22</td>
</tr>
</tbody>
</table>

$^a$DPH measurements with 20-g load.

The observed differences in hardness in the presence and absence of gold chloride film, are attributable to the so-called "Roscoe effect" (ref. 16). Roscoe in 1936
observed that cadmium oxide on the surface of cadmium increased its resistance to plastic deformation. Later, the work of reference 17 confirmed Roscoe's observations with zinc crystals. More recently, the observed Roscoe effect has been explained for aluminum in terms of modern dislocation theory (ref. 18). The egress of dislocations from the crystals surface with plastic deformation is impeded by the presence of the oxide film. Thus, if oxides can influence the ability of surfaces to deform plastically, chlorides could have a similar effect.

Another interesting observation of table II is that hardness measurements in the wear track indicate that 100-gram loads in each instance appear to represent complete hardening since no further increase in microhardness was observed at 200 grams.

Figure 9 shows microhardness indentations in the gold surface. As shown in table II and figure 9, the hardness is greater (from the size of the indentations) in the wear track. Of equal interest is that the indentation just ahead of the terminal point of the wear track is smaller in size than the indentation to either side of it. This indicates that the region ahead of the rider has undergone work hardening due to slip and the resulting deformation. The region in this area was elevated (equivalent to the frontal bulge of ref. 13).

![Figure 9. Microhardness indents on single-crystal gold surface (001). Sliding direction [110]; load, 200 grams; sliding velocity, 0.005 millimeter per second; temperature, 20°C.](image)
Figure 10. - Photomicrographs of cracks developed in single-crystal gold surface with chloride film present. Sliding direction [110]; load, 200 grams; sliding velocity, 0.005 millimeter per second; temperature, 20° C. Lower photograph is a magnification of circled area in upper photograph.
At a load of 200 grams in the presence of the chloride film, severe ductile fracture of the gold in the wear track itself was observed. This effect is indicated in the two photomicrographs of figure 10. A complete series of cracks, normal to the sliding direction, were observed the length of the wear track on single crystal gold. These cracks resulted from a single pass of the slider (sapphire) across the surface. These same surface cracks were observed in some, but not all grains of polycrystalline gold under identical experimental conditions. This fracture was not observed in the absence of a chloride film.

The method employed for the development of the halide films on the gold was relatively severe. Such films can possibly be formed by more conventional techniques employed in lubrication system such as the use of a chlorine containing fluid. Sliding friction experiments were, therefore, conducted with a liquid containing chemically bound chlorine, namely, carbon tetrachloride, acting as the lubricant. The results of these experiments, together with the results from experiments in air and with a chloride film formed by surface reaction prior to the experiment, are presented in figure 11. The data of figure 11 indicate that, while the carbon tetrachloride offers a reduction in friction and deformation compared to the results in air, the fluid was not as effective as the
preformed films. These results might be anticipated on the basis of the difficulty of forming surface films on gold.

The marked difference in deformation resulting from changing crystallographic direction on a single-crystal surface can be seen with the surface profile traces of figure 12. As already discussed, sliding in the [110] direction on the (001) surface resulted in a bulge of material in front of the rider specimen. Surface traces across the wear track reveal no evidence of plastic flow of gold to the sides of the track. In contrast, however, sliding in the [100] direction resulted in the development of considerable plastic flow to the sides of the track. It is also of interest to note that the amount of material removed in the [100] direction is about half that observed in the [110] direction.

Effect of Alloying on Improving the Lubrication of Gold Surfaces

The most frequently used materials to lubricate surface are hydrocarbons. It would, therefore, be ideal for some lubrication applications where gold might be involved (e.g.,
bearings), to be able to use hydrocarbons for the lubrication of gold. The difficulty is that hydrocarbon will not normally chemisorb to gold (refs. 6 and 7). Can the problem be overcome such that hydrocarbon will effectively lubricate gold? Thus far, only altering the surface film has been considered. Chemisorption might be achieved with alloying by the addition of a small concentration of an active metal to gold. Small concentrations of copper were, therefore, added to gold in an attempt to improve the ability to absorb films. The results of such an effort on hardness, friction, and deformation are presented in figure 13.

Friction and deformation experiments were conducted (fig. 13) as a function of copper content using, as the lubricant, 0.02 percent stearic acid in the carrier hexadecane. Increasing the copper content resulted in a decrease in both friction and the width of the wear track. The addition of just 2.5 weight percent copper to gold resulted in a friction coefficient one-third that of pure gold and a 38 percent reduction in the width of the wear track.

The friction and deformation results are not conclusive, because the point could be raised that hardening is occurring, due to the alloying; and this is responsible for the observed reduction in friction and deformation. Microhardness results presented in fig-
ure 13 indicate that the hardness of the gold is not appreciably altered with additions of copper to 2.5 weight percent. At 5.0 weight percent, a significant increase in hardness was observed. The hardness measurements indicate that, at all compositions, the work hardening of the solid solution is not affected by changes in composition. The most important conclusion to be drawn from these data is that, in the region where there was little or no change in hardness, the greatest reduction in friction and deformation occurred, indicating that alloying can be effectively utilized to improve the adsorption nature of gold surface and thereby improve friction and the resistance to deformation. Better lubricating performance has been observed in the lubrication of copper by the addition of alloying elements, such as aluminum and silicon (ref. 19). The observed effects of reference 19 are very similar to those seen in this investigation with the addition of copper to gold.

CONCLUSIONS

Based upon the results obtained in sliding friction experiments in this investigation with single and polycrystalline gold, the following summary remarks are made:

1. While gold is normally insensitive to the presence of ordinary film forming materials, its friction and deformation are found to be influenced by the presence of some atmospheric contaminants such as water vapor. At light loads, both friction and deformation of polycrystalline gold were greater under hexadecane (a blanketeting media) than in air.

2. The addition of small concentrations of copper to gold (0.25 to 5.0 percent) in a solid solution were found to improve the ability of the gold to be lubricated by an organic acid (stearic).

3. Gold halides were found to reduce friction and deformation of gold; of the halides, gold chloride was the most effective for reducing friction while the iodide was most effective in reducing deformation.

4. The presence of the gold halides on the surface were found to produce a hardening effect on the gold (Roscoe effect). With gold chloride present, the undeformed and the deformed surfaces were harder than the same gold surfaces without the chloride film. This was true for both single-crystal and polycrystalline gold.

5. In the presence of water vapor, the friction or deformation of a gold surface was the same as in air. This result might be expected since water does not wet clean gold.

6. The presence of a liquid containing chlorine (carbon tetrachloride) did not provide as effective surface protection as did a reacted solid surface film.

7. With the loads of 100 and 200 grams employed in this study, the surface was found to be fully work hardened in a single pass of the rider over disk surface.
8. With a single pass of the rider over the surface, fracture cracks developed in the wear track with a chloride film present. These cracks were not observed in the absence of the halide film.

9. On a single-crystal (001) surface, friction was lower and plowing less marked in the [100] crystallographic direction than in the [110] direction in all media.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 11, 1968
129-03-13-02-22.

REFERENCES


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