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EFFECT OF 3½ PERCENT SILICON ON ADHESION, FRICTION, AND WEAR OF IRON IN VARIOUS MEDIA

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16. Abstract

Sliding friction experiments were conducted with $3\frac{1}{2}$ percent silicon-iron single crystals, polycrystals, and rolled sheet. The various media of the experiment included vacuum, air, water, water with ferric chloride, hexadecane, hexadecane with oleic acid, and oleic acid. The results of these studies indicate that: (1) the friction is anisotropic with respect to single crystals and rolled sheet, (2) the brittle to ductile transition influences surface deformation but not friction coefficient, (3) the friction coefficient for silicon-iron is more sensitive to small concentrations of oxygen than pure iron, and (4) the presence of surface active species influenced the friction coefficient but had little effect on surface deformation.

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EFFECT OF $3\frac{1}{2}$ PERCENT SILICON ON ADHESION, FRICTION, AND WEAR OF IRON IN VARIOUS MEDIA

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SUMMARY

An investigation was conducted to determine the influence of silicon on certain properties of iron. These included adhesion, friction, deformation during sliding, and reactivity toward various environments. The experiments were sliding friction studies with a hemisphere sliding on a flat. The environments in which these experiments were conducted included vacuum, air, water, water with various concentrations of ferric chloride, hexadecane, hexadecane with various concentrations of oleic acid and oleic acid. Temperatures were from -195° to 500° C with loads on the hemispherical slider from 10 to 500 grams. The $3\frac{1}{2}$ percent silicon iron was examined in single crystal, polycrystalline, and rolled sheet form.

The results of this study indicate the brittle-to-ductile transition in silicon-iron influences surface deformation but has no measurable effect on friction. The friction properties of silicon iron were found to be anisotropic for both single crystals and rolled sheet. The presence of silicon in iron was found to increase the sensitivity of the friction to oxygen. With various surface active agents on silicon iron, friction coefficients were found to be sensitive to these species while surface deformation was not.

INTRODUCTION

The most commonly used alloys in lubrication systems are ferrous base materials. The majority of these alloys are multicomponent systems. It is known that the presence of certain constituents in such alloys is beneficial in reducing friction and wear. One such alloy constituent which has been known, to impart good wear resistance to metals is silicon (refs. 1 and 2). The influence of silicon on the adhesion, friction, and wear behavior of iron in the absence of other alloying elements has not been specifically determined.

The objective of this investigation was to examine the influence of silicon on the adhesion, friction, and wear of iron. A specific alloy $3\frac{1}{2}$ percent silicon iron was examined because it is a commonly used material in transformer applications and therefore its mechanical properties are well known. Experiments were conducted in various environments to determine the influence of that environment on adhesion, friction, and wear. Single, polycrystalline, and textured surfaces were examined.

Sliding friction experiments were conducted with a hemispherical surface in contact with a moving flat. The environments in which experiments were conducted included, air, water, water with ferric chloride surface active species, hexadecane, hexadecane containing surface active species, oleic acid and in vacuum. Sliding speeds were very low, 0.005 to 0.010 millimeter per second, and loads from 10 to 500 grams. Temperatures of experiments covered the range from -195° to 500° C.

MATERIALS

The iron silicon alloy was made from triple zone refined iron which was alloyed with high purity silicon. All specimens, after fabrication to shape and prior to experiments, were polished on abrasive papers and then electropolished in phosphoric acid. The single crystals were electric discharge machined to shape, polished on papers, and electropolished in phosphoric acid. The single crystals were oriented by use of LAUE X-ray techniques and the orientation specified are within $\pm 2^{\circ}$. The iron specimens used for comparative purposes were triple zone refined containing less than 8 ppm carbon. It was the same stock and lot from which the alloys were prepared.

The water used in these studies was triple distilled. The hexadecane was percolated through silica gel prior to use. The ferric chloride and oleic acid were reagent grade. The oxygen used in the vacuum studies was high purity reagent grade. Hydrogen gas used to clean surfaces in vacuum was high purity and was passed through a liquid nitrogen cooled molecular sieve prior to use.

APPARATUS

Two different experimental devices were used in these studies: one, for the fluid studies, which was a Bowden-Leben type apparatus (described in ref. 3); and second, for the vacuum studies, the apparatus is shown in figure 1.

The specimens of the vacuum apparatus were a 2.54-centimeter-diameter flat disk and 0.475-centimeter hemispherically radiused rider specimen (shown in the insert of fig. 1). The disk specimen was mounted on a shaft which was magnetically driven by a

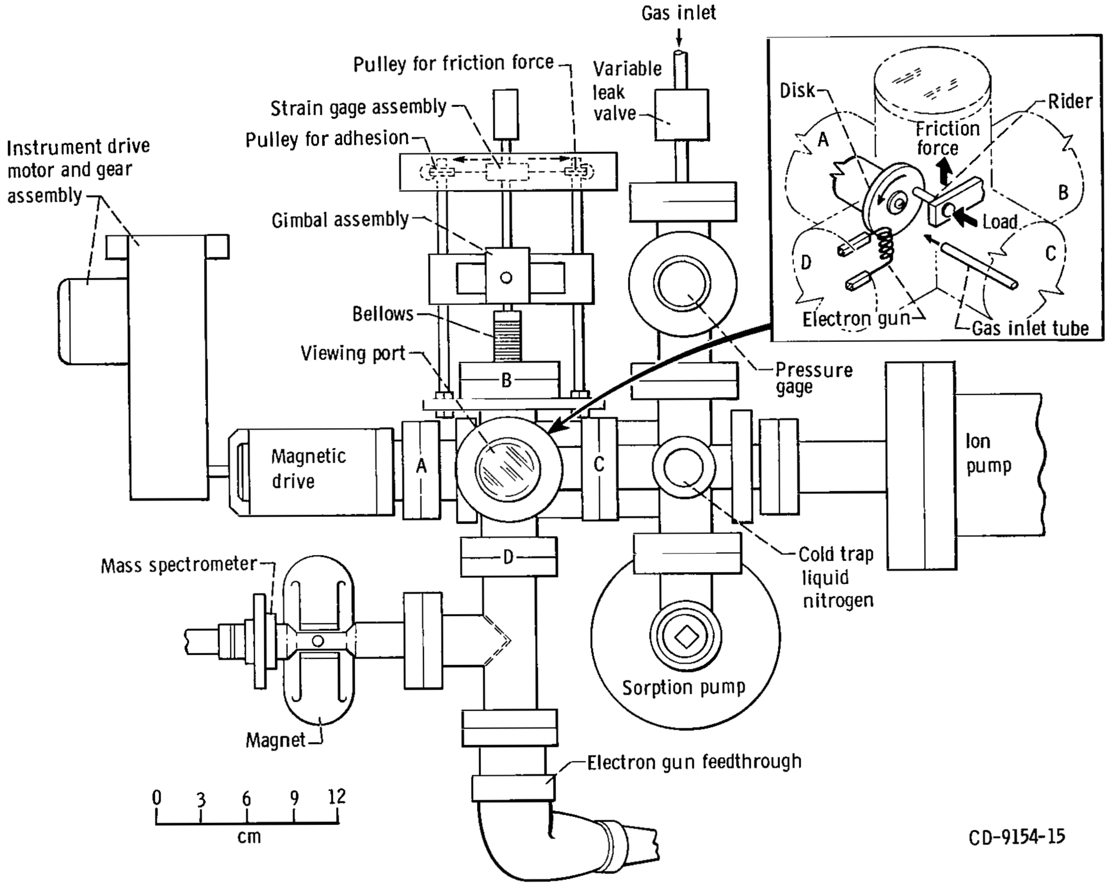


Figure 1. - Vacuum friction apparatus.

motor and gear assembly. The linear sliding velocity employed in these experiments was 0.001 centimeter per second.

The rider specimen was mounted in an arm which was gimbal mounted and bellows sealed to the vacuum-chamber wall. The rider specimen was loaded against the disk surface with dead-weight loading. Perpendicular to the loading force devices was a strain gage for monitoring friction force. Adhesive forces were measured by applying a force opposite the direction in which the load was applied. Breakaway forces were measured by filling a polyurethane bucket with liquid gallium. After the specimens separated, the gallium bucket was weighed and the breakaway load thus determined. Cleaning of specimen surfaces in the vacuum chamber was achieved by bombarding with electrons from an electron gun, and the gaseous species in the vacuum chamber were monitored with a mass spectrometer.

The vacuum system was rough pumped to 1 micron of mercury with a sorption pump, and pressures to 10^{-10} torr were achieved with an ion pump. A grid was provided in the pump throat to eliminate ionization in the specimen region. A liquid-nitrogen trap was used for cryopumping. Pressure was measured by a cold-cathode ionization gage, as well as by the pump current gage.

Gases were introduced into the vacuum chamber through a variable-leak valve. A separate vacuum sorption pump was used to evacuate the gas line connecting the cylinder of gas to the variable-leak valve. Both the gas line and the vacuum system were baked out with heating tapes and infrared lamps.

EXPERIMENTAL PROCEDURE

Fluid Experiments

In those experiments in which fluids were examined on iron-silicon surfaces, the specimens were thoroughly rinsed in alcohol after electropolishing and then placed in vacuum tubes. The specimens were heated to red heat in vacuum to remove surface adsorbates. After cooling the specimens to room temperature, the particular fluid in which the experiment was to be conducted was bled into the vacuum tube. From this point the specimens remained completely submerged under the fluid until the friction experiment was complete.

Vacuum Experiments

There are a number of techniques used to obtain clean surfaces in a vacuum. Two of the more commonly used are the ion-bombardment and the electron-beam bombardment techniques. Based on friction results obtained in an earlier study with tungsten which indicated more effective cleaning with the electron gun, the electron-beam cleaning of surfaces was selected for use in this investigation.

The iron surfaces used in this study were cleaned by electron bombardment in the vacuum chamber when the pressure reached 10^{-10} torr. The specimens were heated with the electron gun to a bulk temperature of 800° C (surface temperatures were considerably higher), and hydrogen gas was admitted to the chamber to reduce surface oxides. Once the surface oxides were reduced (as determined by the mass spectrometer) the temperature was increased to 1000° C, and the silicon-iron surface was electron bombarded for 3 hours to remove the adsorbed hydrogen.

Oxygen Adsorption

The specimens were cooled to room temperature after the electron bombardment, and oxygen was admitted to the specimen zone through a variable-leak valve and a tube. The gases were charged from their cylinders into a line outside the vacuum chamber. Prior to the admission of gas, the line was thoroughly evacuated with a sorption pump and was baked out. The evacuated supply line was then purged three times by alternately filling and evacuating it.

Mass spectrometer traces were obtained during gas admission to the specimen surface, during the friction experiment, and following the experiment. Background data for the mass spectrometer were obtained with a saturated specimen surface at room temperature, that is, when the specimen surface was effectively covered with a monolayer of oxygen.

EXPERIMENTAL RESULTS

Air and Fluid Media

Preliminary sliding friction experiments were conducted in air on the (001) plane of $3\frac{1}{2}$ percent silicon iron in two crystallographic directions, the $\langle 100 \rangle$ and the $\langle 110 \rangle$. The results obtained in these experiments are presented in table I. The coefficient of friction

TABLE I. - COEFFICIENT OF FRICTION AND TRACK WIDTH FOR $3\frac{1}{2}$-PERCENT SILICON-IRON SINGLE CRYSTALS IN VARIOUS MEDIA (001) PLANE

Media	Direction	Coefficient of friction[a]	Track width, mm
Air	100	0.15	0.055
	110	.20	.055
Hexadecane	100	0.20	0.050
Hexadecane and 0.2-percent oleic acid	100	0.08	0.055
Oleic acid	100	0.08	0.055

[a]Sliding velocity, 0.005 mm/sec; load, 500 g; ambient temperature, 20° C; rider specimen sapphire.

with sliding was lower on the (001) plane in the ⟨100⟩ than in the ⟨110⟩ directions. The track width generated in the sliding process was the same in both crystallographic directions. The results indicate that the most isotropic of the common metal crystal systems, namely, the body centered cubic, does exhibit anisotropic friction behavior. Similar results have been observed with another body centered cubic metal, tungsten, in earlier studies (refs. 4 and 5).

Experiments were conducted in hexadecane to exclude the influence of moisture on friction and deformation measurements and the results obtained are also presented in table I. With sliding the friction coefficient in hexadecane in the ⟨100⟩ direction was higher than was obtained in air. A slight decrease in track width was observed. If 0.2 percent oleic acid was added to the hexadecane a decrease in friction and a slight increase in track width occurred. If the same experiment was conducted in pure oleic acid, no further reduction in friction coefficient or change in track width was observed. These results then, indicate that a very small concentration of surface active agent (0.2 percent oleic acid) was as effective in reducing friction coefficient as 100 percent acid.

Bearing metals are usually polycrystalline. Experiments were therefore conducted with polycrystalline silicon iron in hexadecane, hexadecane containing oleic acid, and oleic acid. Data were also obtained with polycrystalline iron for comparative purposes, that is, to determine the influence of the silicon. The results obtained are presented in table II. A number of observations are apparent from an examination of table II. These are (1) the friction and track width for polycrystalline silicon iron in all three media were lower than those observed for the single crystals in table I, (2) friction and track width were lower for the silicon iron than for iron, (3) friction was sensitive to the presence of surface active species for both materials, deformation was not, and (4) the fric-

TABLE II. - EFFECT OF SILICON ON THE FRICTION AND WEAR OF POLYCRYSTALLINE IRON IN VARIOUS MEDIA

Media	Iron		$3\frac{1}{2}$-Percent silicon iron	
	Coefficient of friction (a)	Wear track width, mm	Coefficient of friction (a)	Track width, mm
Hexadecane	0.20	0.065	0.15	0.035
Hexadecane and 0.2-percent oleic acid	0.15	0.065	0.04	0.035
Oleic acid	0.092	0.065	0.03	0.035

[a]Rider sapphire ball; load, 500 g; sliding velocity, 0.005 mm/sec; ambient temperature 20° C.

tion of iron was sensitive to acid concentration. For silicon iron, less acid was required to reduce friction than for iron.

Earlier friction studies have shown that friction properties of metals are sensitive to texturing. Sliding friction experiments were therefore conducted on rolled silicon-iron sheet in two directions, normal to and parallel to the rolling direction. These experiments were conducted in a number of media and the results obtained are presented in table III. Both aqueous and hydrocarbon environments were examined. In an aqueous

TABLE III. - FRICTION AND WEAR OF $3\frac{1}{2}$-PERCENT SILICON IRON

ROLLED SHEET IN VARIOUS ENVIRONMENTS

Media	Parallel to rolling direction		Normal to rolling direction	
	Coefficient of friction	Track width, mm	Coefficient of friction	Track width, mm
Air	0.25	0.035	0.15	0.035
Water	0.075	0.035	0.05	0.035
Water and 10^{-4} M $FeCl_3$	0.075	0.035	0.05	0.040
Hexadecane	0.15	0.035	0.10	0.035
Hexadecane and 0.2-percent oleic acid	0.075	0.035	0.05	0.035

environment the presence of a surface active ion, namely, the chloride, known to influence both friction and deformation was examined.

The data of table III indicate that, in all environments examined, friction coefficients were lower normal than parallel to the direction of rolling. The width of the track generated during sliding, however, seemed to be relatively insensitive to sliding direction and environment.

While a decrease in friction coefficient occurred with iron in changing acid concentration from 0.2 percent to 100 percent oleic acid no significant change in friction was observed with silicon iron (table II). In order to determine if the silicon iron was sensitive to oleic acid concentration a series of experiments were conducted with various percents of oleic acid in hexadecane. The results obtained in these experiments are presented in figure 2.

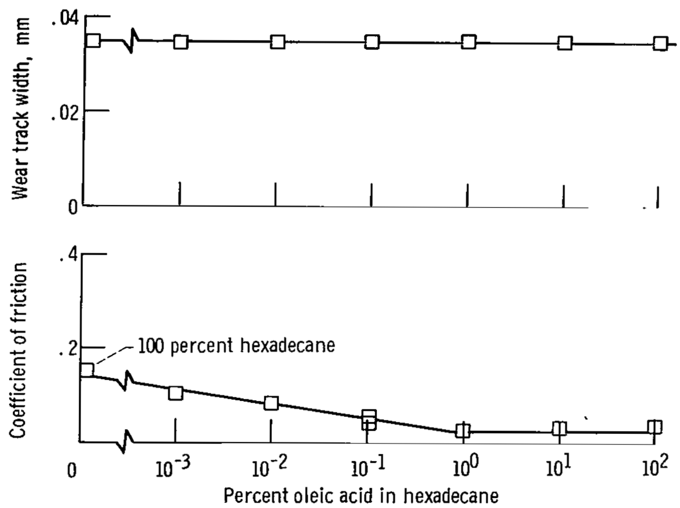


Figure 2. - Influence of pleic acid concentration in hexadecane on friction and wear of $3\frac{1}{2}$ percent silicon-iron. Rider specimen, sapphire; sliding velocity, 0.005 millimeter per second; load, 500 grams; ambient temperature, 20° C.

The coefficient of friction for silicon iron is influenced by oleic acid concentrations up to about 1 percent. Beyond 1 percent, increases in acid concentration seem to exert very little influence on friction coefficient. It is interesting to note that, over the entire range of oleic acid concentrations examined, the surface deformation with sliding, as determined by track width, was essentially the same (fig. 2).

The oleic acid concentration in hexadecane selected for use in tables I to III was the same as used by P. A. Rebinder in experiments where increase in plasticity for iron was observed in the presence of surface active oleic acid (ref. 6). The track widths reported herein for iron in table II and for silicon iron in tables I to III and figure 2 indicate an insensitivity of plasticity to the presence of surface active oleic acid.

Experiments were also conducted with silicon iron covered with an aqueous solution containing various concentrations of a surface active ion. The results obtained in sliding friction experiments over a range of ferric chloride concentrations are presented in figure 3. In figure 3 the friction coefficient and track width developed during sliding were found to be essentially insensitive to chloride ion concentration. The data of figure 3 are for randomly oriented polycrystalline iron. When the surface is textured by rolling, a lower friction coefficient is observed (see table III).

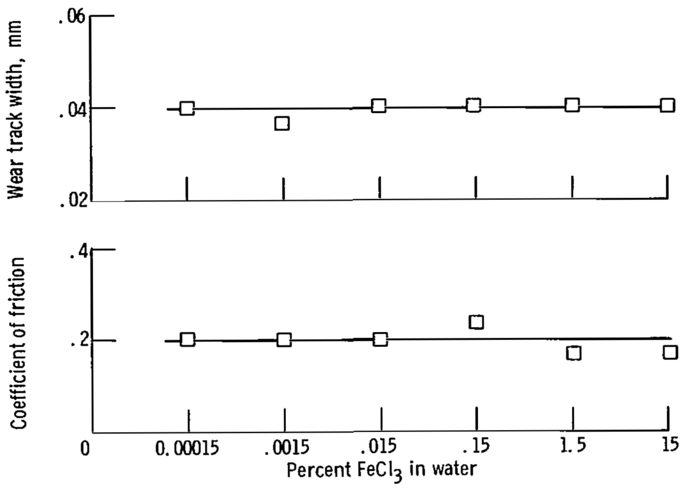


Figure 3. - Influence of ferric chloride concentration in water on friction and wear of $3\frac{1}{2}$-percent silicon-iron. Rider specimen, sapphire; sliding velocity, 0.005 millimeter per second; load, 500 grams; ambient temperature, 20° C.

VACUUM STUDIES

When iron surfaces are carefully cleaned in a vacuum environment, touch contact under negligible load is sufficient to produce strong bonding between the surfaces. The forces are strong enough to prevent tangential motion between the surfaces and under such conditions friction coefficient loses its meaning (ref. 7). The presence of certain alloying elements has been found to exert a marked influence on reducing such adhesive forces. In reference 8, it was demonstrated that adhesive forces between surfaces could be appreciably reduced if the carbon content in iron were sufficiently high.

Experiments were conducted with $3\frac{1}{2}$ percent silicon iron in sliding contact with itself in vacuum after surface cleaning. The specimens were single crystals with (001) planes in contact. The first observation made was that, when the $3\frac{1}{2}$ percent silicon iron was cleaned in vacuum in the same manner as the iron of reference 7, sliding occurred and friction forces could be measured. The friction coefficient under such conditions was high as indicated in figure 4 (4.0 at all loads). Further, when the load was removed, the specimens remained adhered to one another and considerable force was required to separate the surfaces. For example, after a load of 200 grams had been applied, the tensile force required to separate the surfaces was 1050 grams.

After sliding at various loads, microindentation hardness measurements were made in the wear track. The values obtained using a 20-gram load on the hardness indentor are presented in figure 4. Microhardness increased with increase in load on the slider to loads of 200 grams. At 300 grams the microhardness was essentially the same as that

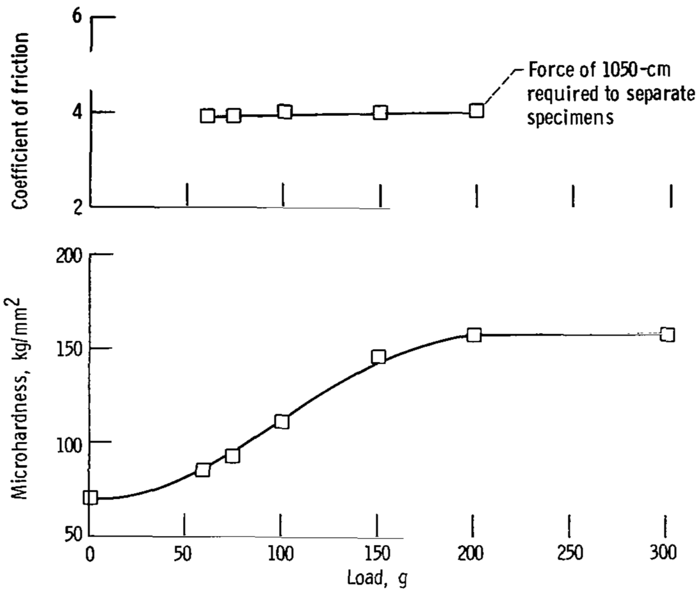


Figure 4. - Coefficient of friction and surface hardness after single-pass sliding-friction experiment: (001) rider sliding on (001) disk of iron - $3\frac{1}{2}$-percent silicon. Sliding velocity, 0.001 centimeter per second; ambient temperature, 20° C; ambient pressure, 10^{-10} torr. Note: adhesion of specimens occurred at all loads, and force was required to separate surfaces.

obtained at 200 grams. This indicated that the silicon iron may be fully work hardened in the wear track at a 200-gram load.

The nature of the friction traces obtained with (001) silicon iron sliding on itself is shown in figure 5. A relatively high friction force was recorded during sliding with periodic slips developed in the trace when the welded junctions broke at the low speeds

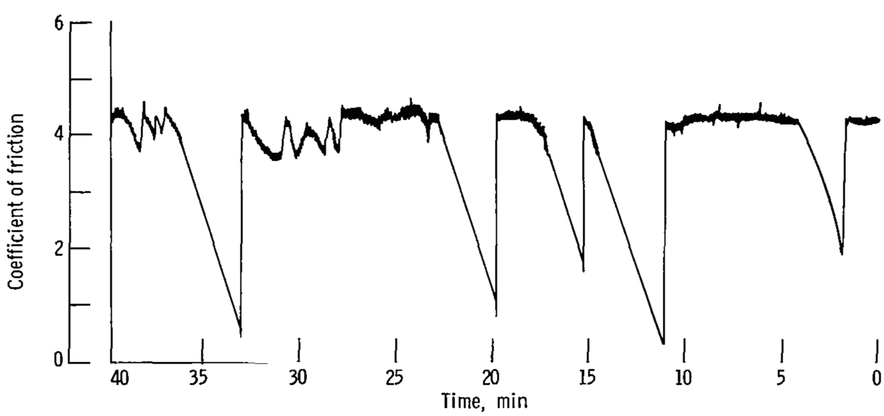
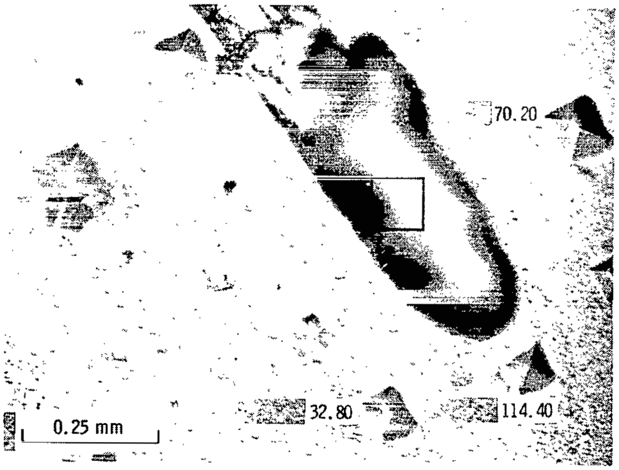


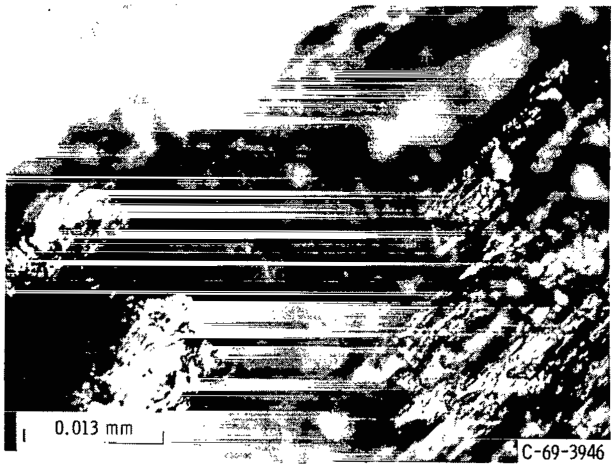
Figure 5. - Friction trace for (001) plane of iron - $3\frac{1}{2}$-percent silicon single-crystal in sliding contact with itself in vacuum. Sliding velocity 0.001 centimeter per second; load, 60 grams; ambient temperature, 20° C; ambient pressure, 10^{-10} torr.

employed of 0.001 centimeter per second. Note the extended period of the stick portion of the stick-slip curve.

Photomicrographs of the wear scars generated on the iron-silicon surface are presented in figure 6. The wear track was not a continuous path on the surface but rather



(a) Wear track.



(b) Enlarged wear track (see area of box in (a)).

Figure 6. - Photomicrographs showing adhesion of (001) iron - 3.5-percent silicon to itself during sliding-friction experiments. Sliding velocity, 0.001 centimeter per second; load, 60 grams; ambient temperature, 20° C; ambient pressure, 10^{-10} torr.

consisted of localized welded areas developing in streaks over the surface. At the head of such streaks was a large buildup of metal due to adhesion and transfer. One such area is shown in figure 6(a). Microindentation hardness measurements made in and about this region indicated marked differences in hardness. Maximum hardness was found in the adhered or built-up metal (150 kg/mm^2), with the area in front of the buildup softer (114 kg/mm^2) but not as soft as the metal outside the sliding zone (70 kg/mm^2). The increase in hardness of the region in front of the buildup of adhered material indicates that silicon iron is being deformed plastically for some distance ahead of the slider. Figure 6(b) is an enlarged view of the built-up region indicating the extent of surface damage.

Experiments were conducted to determine the influence of oxygen on the adhesion and friction of the silicon-iron crystals; the friction coefficient as a function of oxygen exposure in torr-seconds is presented in figure 7. With increases in oxygen exposure to 10^{-2} torr-second, friction coefficient continued to decrease. Beyond this oxygen exposure, no change in friction was observed. It is interesting to note the marked influence a small quantity of oxygen has on the coefficient of friction. These results indicate the extreme sensitivity of friction to surface conditions.

For purposes of comparison, data taken from reference 7 for iron as a function of oxy-

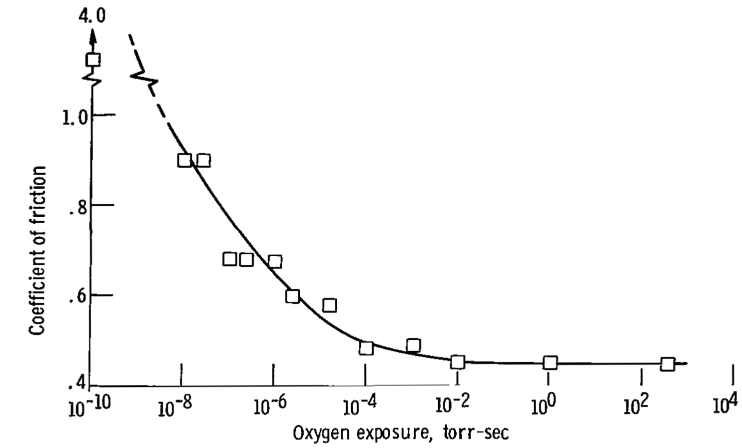


Figure 7. - Coefficient of friction for iron - $3\frac{1}{2}$-percent silicon single crystal (001) sliding on iron - $3\frac{1}{2}$-percent silicon single crystal (001) in vacuum as function of oxygen exposure. Sliding velocity, 0.001 centimeter per second; load, 60 grams; ambient temperature, 20° C; ambient pressure, 10^{-10} torr.

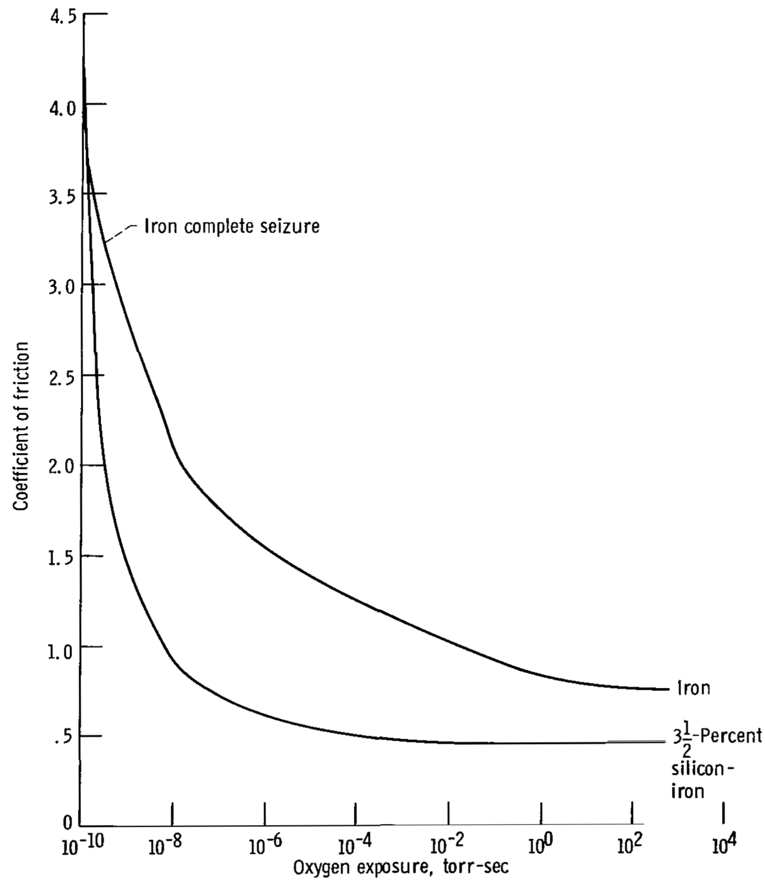


Figure 8. - Coefficient of friction for iron and $3\frac{1}{2}$-percent silicon-iron as function of oxygen exposure. Sliding velocity, 0.001 centimeter per second; ambient temperature, 20° C; ambient pressure, 10^{-10} torr.

gen exposure, are presented with the plot of figure 7 in figure 8. The curves of figure 8 indicate that the addition of $3\frac{1}{2}$ percent silicon to iron reduces appreciably the friction coefficient at equivalent oxygen exposures. These results are not surprising in light of the affinity of silicon for oxygen (the silicon acting as an oxygen getter).

Most metals, with the exception of the face-centered cubic, exhibit some type of temperature dependent brittleness. Brittle fracture in steels, for example, has been a matter of concern for many years. Brittle fracture or cleavage cracks can develop at local highly stressed regions of a material. The nature of surface behavior of a metal such as iron or alloy such as silicon-iron in this brittle region during sliding has not been examined.

Experiments were conducted with $3\frac{1}{2}$ percent silicon-iron crystals, (001) orientation, to determine the influence of the ductile-brittle transition on friction and surface deformation during sliding. In these experiments the disk specimen was cooled to the experimental temperature. In order to minimize the heat input to the surface during the sliding process, an aluminum oxide slider was used. The results obtained with silicon-iron surfaces in vacuum at temperatures from -195° to 500° C are presented in figures 9 and 10.

The friction coefficient on the (001) surface of silicon iron was measured in two crystallographic directions, the $\langle 100 \rangle$ and the $\langle 110 \rangle$, and the data obtained and presented in figure 9 indicate anisotropic friction behavior. Friction was lower in the $\langle 100 \rangle$ directions. These results are similar to those obtained in table I where friction was lower in the $\langle 100 \rangle$ directions in the presence of surface oxides. It is interesting to note that, with sliding in the $\langle 110 \rangle$ directions, friction was high (1.0) at the low temperatures. Above room temperature, however, the friction coefficient began to decrease. This result is somewhat surprising since, at the high temperatures, the material is most ductile. It must be indicated here that examination of the sliding surface after experiments at each temperature revealed no evidence of the formation of brittle cracks.

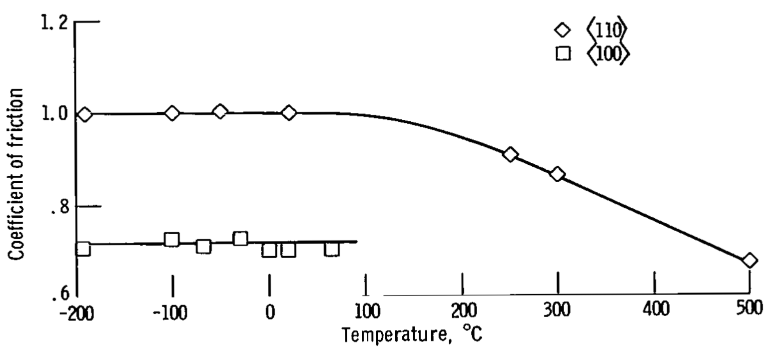


Figure 9. - Coefficient of friction of single-crystal (001) surface of iron - $3\frac{1}{2}$-percent silicon as function of crystallographic direction in vacuum. Rider specimen, sapphire; sliding velocity, 0.001 centimeter per second; load, 60 grams; ambient pressure, 10^{-10} torr.

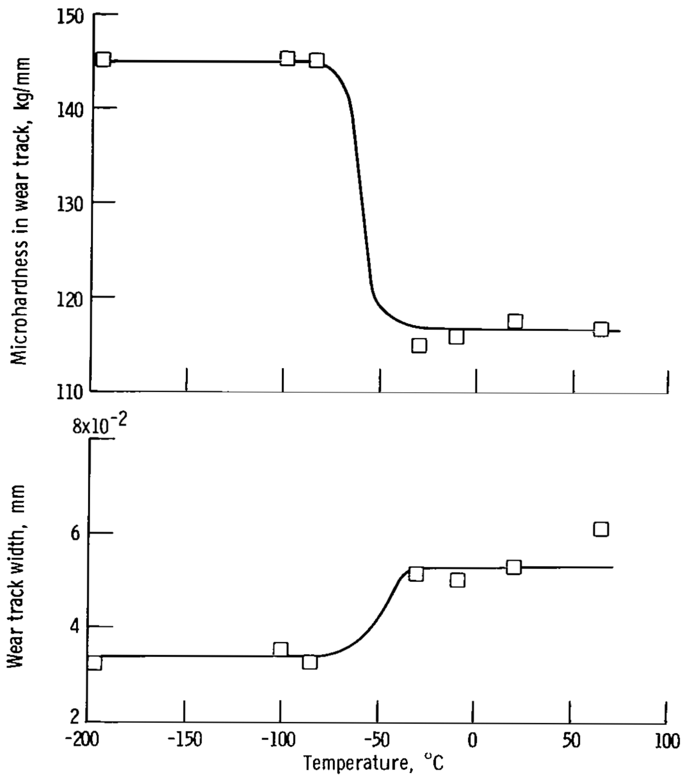


Figure 10. - Deformation and work hardening measurements in sliding sliding-friction experiments with (001) iron - $3\frac{1}{2}$-percent silicon in vacuum. Rider specimen, sapphire; sliding velocity, 0.001 centimeter per second; load, 60 grams; ambient pressure, 10^{-10} torr.

The normal brittle to ductile transition of silicon iron is in the temperature range of -80° to -100° C. This transition temperature, however, can be radically shifted by changes in strain rate and the composition or microstructure of a particular material. (ref. 9). The friction data of figure 9 indicate that the coefficient of friction for silicon iron is insensitive to the brittle-to-ductile transition.

In contrast to friction, the surface deformation during sliding and microhardness in the wear track were markedly influenced by the brittle-to-ductile transition as indicated by the data of figure 10. The wear track width increased markedly and the microhardness decreased in the transition region. These results are in agreement with what might be anticipated. The wear track width has increased because of increased plasticity and the microhardness has decreased because the energy put into work hardening is spread to a larger mass of metal in the contact zone, the result being less strain hardening.

It would seem that a plausible reason for the lack of response of friction to the brittle-to-ductile transition in figure 9 may be that, while the true area of contact increases with the transition, the shear strength decreases; the net effect is no change in friction. In examination of wear tracks upon completion of experiments with etch pitting, plastic flow at all temperatures was seen subsurface.

SUMMARY OF RESULTS

From the data obtained in this investigation with $3\frac{1}{2}$ percent silicon iron in various media during sliding friction experiments, the following summary remarks are made:

1. As might be anticipated, the brittle-to-ductile transition on the (001) surface has a marked influence on surface deformation and work hardening in the wear track. No effect of the transition on friction coefficient, however, was observed.

2. The friction coefficient is anisotropic. In sliding on the (001) plane, friction was found to be lower in the $\langle 100 \rangle$ than in the $\langle 110 \rangle$ directions.

3. Extremely small quantities of oxygen are sufficient to markedly reduce the friction coefficient for silicon iron. Oxygen exposures of 10^{-8} torr-second was sufficient to reduce the friction coefficient from 4.0 to 0.9. Oxygen was more effective in reducing friction of iron silicon than it was in reducing the friction of iron.

4. The friction coefficient for silicon-iron rolled sheet was anisotropic. Friction was lower normal to than in the rolling direction in various media.

5. The presence of surface active species were found to influence the coefficient of friction for silicon iron but had little or no effect on surface deformation.

Lewis Research Center,
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