Adhesion and Friction of Iron-Base Binary Alloys in Contact With Silicon Carbide in Vacuum

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Summary

An investigation was conducted to determine the effect of alloying elements (Ti, Cr, Mn, Ni, Rh, and W) on the friction behavior of various iron-base, binary, solid-solution alloys in sliding contact with a single-crystal silicon carbide [0001] surface in vacuum. All single-pass sliding friction experiments were conducted with loads of 0.05 to 0.3 newton, at a sliding velocity of $3 \times 10^{-3}$ meter per minute, in a vacuum of $10^{-8}$ pascal, and at room temperature on the silicon carbide [0001] basal plane with sliding in the (1010) directions.

The results of the investigation indicate that the atomic size and concentration of the alloying elements are important factors in controlling the adhesion and friction of iron-base binary alloys. The coefficient of friction generally increases with an increase in solute concentration and with increases or decreases from unity in the solute-to-iron atomic radius ratio. The chemical activity of the alloying elements is also an important parameter in controlling the adhesion and friction of alloys: with increasing d bond character of the element, there is a decrease in adhesion and friction.

Introduction

Silicon carbide is widely used as an abrasive for industrial grinding. However, because the surface interactions involved are so complex, our knowledge of the process of grinding is limited. A better understanding of the fundamental behavior of silicon carbide during the process of grinding should allow improvements in its efficiency by more judicious selections of materials, lubricants, and operating parameters.

Fundamental studies on the surface interactions, that is, the friction and wear behavior of silicon carbide, should commence with clean surfaces of comparatively simple, that is, pure metals and binary or ternary alloys.

The present authors have conducted experimental studies to determine the tribophysical properties of single-crystal silicon carbide in contact with itself and with clean surfaces of pure metals in vacuum as well as of silicon carbide in contact with binary alloys.

It is well known that the presence of small amounts of elements in alloys can markedly alter their surface activity (refs. 1 to 3). The present authors found that small concentrations of chromium in iron resulted in an increase in the adhesion and friction of the alloys contacting silicon carbide (ref. 4). The exact role of alloying elements in the adhesion, friction, and wear of alloy-ceramic contacts was not clearly revealed in reference 4 because of the small selection of alloying elements.

The atomic size and concentration of the alloying elements are extremely important for the abrasive-wear and friction behavior of iron-base binary alloys in contact with silicon carbide. The coefficient of friction and abrasive wear volume generally decrease with increasing solute concentration (ref. 5). There is a correlation between the solute-to-iron atomic-radius ratio and the coefficient of friction. There is also a good relation between wear and the change in solute concentration. Friction and wear decrease as the solute-to-iron atomic-radius ratio either increases or decreases from unity. Of further interest are the effects of atomic size and concentration of alloying element on the adhesive properties of alloys in contact with silicon carbide.

This investigation was conducted to determine the effect of a wider range of alloying elements (such as Ti, Cr, Mn, Ni, Rh, and W) on the friction behavior of iron-base, binary, solid-solution alloys in sliding contact with a single-crystal silicon carbide (0001) surface. The single-pass, sliding-friction experiments were conducted with loads of 0.05 to 0.3 newton (5 to 30 g), at a sliding velocity of $3 \times 10^{-3}$ meter per minute, in a vacuum of $10^{-8}$ pascal, and at room temperature with sliding on
the silicon carbide (0001) basal plane in the (10\bar{1}0) directions. The alloys were all polycrystalline.

Materials

Table I presents the analyzed compositions in atomic percent of the iron-base alloys used in the present investigation. The iron-base binary alloys were prepared by Stephens and Witzke (ref. 7) who arc-melted the high-purity iron and high-purity alloying elements (Ti, Cr, Mn, Ni, Rh, and W). The

TABLE I. - CHEMICAL ANALYSIS* AND SOLUTE-TO-IRON ATOMIC-RADIUS RATIOS† FOR IRON-BASE BINARY ALLOYS

<table>
<thead>
<tr>
<th>Solute element</th>
<th>Analyzed solute content, at. %</th>
<th>Analyzed interstitial content, ppm by weight</th>
<th>Solute-to-iron atomic-radius ratios</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>1.02 2.08 3.66 8.12</td>
<td>56 69 94 7</td>
<td>1.1476</td>
</tr>
<tr>
<td>Cr</td>
<td>0.99 1.98 3.92 16.2</td>
<td>--- 50 85 9</td>
<td>1.0065</td>
</tr>
<tr>
<td>Mn</td>
<td>0.49 1.96 3.93 7.59</td>
<td>--- 39 85 9</td>
<td>0.9434</td>
</tr>
<tr>
<td>Ni</td>
<td>0.51 1.03 2.10 4.02 8.02 15.7</td>
<td>--- 90 9 5</td>
<td>0.9780</td>
</tr>
<tr>
<td>Rh</td>
<td>1.31 2.91 4.18 8.66</td>
<td>--- 175 22</td>
<td>1.0557</td>
</tr>
<tr>
<td>W</td>
<td>0.83 1.32 3.46 6.86</td>
<td>30 61 12 12</td>
<td>1.1052</td>
</tr>
</tbody>
</table>

*Manufacturer's analyses.
†Ref. 12.

solute concentrations ranged from approximately 0.5 atomic percent for those elements that have extremely limited solubility in iron up to approximately 16 atomic percent for those elements that form a series of solid solutions with iron.

The single-crystal silicon carbide used in these experiments was a 99.99-percent-pure compound of silicon and carbon and had a hexagonal-closed-packed crystal structure (table II).

Experimental Apparatus and Procedure

Apparatus

The apparatus used in the investigation was mounted in an ultrahigh-vacuum system. The apparatus measures adhesion, load, and friction. The vacuum system also contained tools for surface analysis, an Auger emission spectrometer (AES), and a low-energy-electron diffraction (LEED) system. The mechanism used for measuring adhesion, load, and friction is shown schematically in figure 1. A gimbal-mounted beam is projected into the vacuum system. The beam contains two flats machined normal to each other with two strain-gages mounted thereon. The 0.79-millimeter-radius alloy pin is mounted on the end of the beam. As a load is applied by moving the beam in the direction normal to the disk, it is measured by the strain gage. The vertical sliding motion of the pin along the disk surface is accomplished through a motorized gimbal assembly. Under an applied load the fric-
tation force is measured during vertical translation of
the strain gage mounted normal to that used to
measure load. This feature was used to examine the
coefficient of friction at various loads, as shown in
figure 1.

Specimen Preparation

The iron-base alloy pins and the silicon carbide
disks were polished with 3-micrometer-diameter
diamond powder and then 1-micrometer-diameter
aluminum oxide powder. Both pin and disk sur-
faces were rinsed with 200-proof ethyl alcohol be-
fore installation in the vacuum chamber.

The specimens were then placed in the vacuum
chamber, which was evacuated and baked out to a
pressure of 1.33 × 10⁻⁶ pascal (10⁻¹⁰ torr) where-
upon argon gas was bled back into the vacuum
chamber to a pressure of 1.3 pascals. A 1000-volt,
direct-current potential was applied, and the
specimens (both disk and pin) were argon-sputter
bombarded for 30 minutes. One hour after sputter-
ing had been completed, the vacuum chamber was
reevacuated, and AES (Auger emission spec-
troscopy) spectra of the disk surface were obtained
to determine the degree of surface cleanliness.
When the disks were clean, as evidenced by the
Auger spectra, friction experiments were con-
ducted.

Experimental Procedure

Loads of 0.05 to 0.3 newton were applied to the
pin-disk contact by deflecting the beam of figure 1.
Both load and friction force were continuously
monitored during the single-pass experiments. The
sliding velocity was 3 × 10⁻³ meter per minute. All
sliding was conducted with the system evacuated to
a pressure of 10⁻⁸ pascal.

Results and Discussion

Auger Analysis of Silicon Carbide Surfaces

An Auger emission spectroscopy spectrum of the
single-crystal silicon carbide [0001] surfaces obtain-
ed before sputter cleaning but after polishing and
bakeout is shown in figure 2(a). In addition to the
silicon and carbon peaks an oxygen peak is present.
The oxygen peak and the chemically shifted silicon
peaks at 78 and 89 electron volts (eV) indicate a
layer of SiO₂ on the silicon carbide surfaces as well
as a simple, adsorbed film of oxygen (refs. 8 and
9). The Auger spectrum taken after the silicon car-
bide surface had been sputter cleaned (fig. 2(b))
clearly reveals the silicon and carbon peaks at 92
and 272 eV, respectively; the oxygen peak is negli-
gible.
Figure 2. - Auger spectra of single-crystal silicon carbide (0001) surface.

(a) Before sputter cleaning.

(b) After sputter cleaning.
Tribological Behavior

Sliding-friction experiments were conducted with iron-base binary alloys in contact with single-crystal silicon carbide in vacuum. The binary-alloy systems were iron alloyed with various concentrations of titanium, chromium, manganese, nickel, rhodium, or tungsten. Figure 3 shows a typical friction-force trace resulting from such sliding. The friction-force traces obtained in this investigation are characterized by a sharp break in the friction force at point b in figure 3, that is, stick-slip behavior. The line a-b represents the region where both loading and tangential (shear) forces were being applied but where no gross sliding occurred. At point b the onset of slip occurs. Line b-c represents the region where the surfaces of alloy and silicon carbide are in rapid slip.

The coefficient of static friction \( \mu \) is defined as \( \mu = F_{\text{max}}/W \), where \( F_{\text{max}} \) is the friction force at which the first sharp break is observed in the friction force trace (see point b in fig. 3) and \( W \) is the normal load.

Figure 4 presents a scanning electron micrograph and an X-ray map of a wear track on the silicon carbide generated by a single pass of the 8.12-at% titanium-iron alloy rider. In the X-ray energy dispersive map (fig. 4(b)), the concentrations of white spots correspond to those locations in the micrograph (fig. 4(a)) where copious amounts of alloy have transferred. It is obvious from those photographs that a large amount of alloy transfers to the silicon carbide surface. In figure 4(a) the light area, where a lot of alloy transfer is evident, was the contact area before sliding of the rider. It is the area where the surfaces of alloy and silicon carbide where sticking to each other and where strong interfacial adhesion occurred. This area, where both the loading and tangential (shear) forces were applied to the specimen, corresponds with the region a-b of figure 3.

Figures 5(a) and (b) are scanning electron micrographs (SEM) of the typical rider wear scar on an iron-base binary alloy (in this case 8.12 at% Ti-Fe alloy) after it has slid on a silicon carbide surface. The size of wear scar is comparable with that of the area of alloy transfer shown in figure 4. The wear scars reveal a large number of small grooves and microcracks, formed primarily by shearing of the interfaces and in the bulk of the alloy. Close examination of figure 5(b) indicates that the cracks are very small, are in the wear scar, and propagate nearly perpendicular to the sliding direction. All friction-force traces are characterized by the sharp break of friction force; and the shearings at the interface and in the bulk are the main factors responsible for the observed friction behavior. All iron-base binary alloys transfer to silicon carbide. The transfer characteristics will be discussed in detail in the next section.

The coefficients of friction for a number of iron-base binary alloys (alloying elements Ti, Cr, Mn, Ni, Rh, and W) in contact with silicon carbide were examined as functions of load. Figure 6 presents typical plots of the coefficient of friction, calculated from the first maximum peak height in friction-force traces, as a function of load for the various titanium-iron alloys. The plots indicate no significant change in the coefficient of friction with load over the load range (0.05 to 0.3 N).

Alloying Element Effect on Friction

The averaging coefficients of friction over the load range for iron-base binary alloys are presented in figure 7 as functions of solute concentration. The data of these figures indicate that the coefficient of friction generally increases markedly with the presence of alloying element in the pure iron and that it increases with further increases in the concentration of alloying element, but not so markedly as the initial increase. The increasing rate in the coefficient of friction strongly depends on the alloying element.

The average coefficient of friction for pure iron in sliding contact with single-crystal carbide was about 0.5 (ref. 6), obtained under the same experimental conditions as those of this investigation. The coefficient of friction was about 0.6 for pure titanium, 0.5 for pure nickel and tungsten, and 0.4 for pure rhodium. The coefficients of friction for
Figure 4. - Titanium-iron binary alloy (8.12 at% Ti) transfer to single-crystal silicon carbide (0001) surface at start of sliding. Silicon carbide (0001) surface; sliding direction $\langle 10\bar{1}0 \rangle$; sliding velocity, 3 mm/min; load, 0.2 N; room temperature; vacuum pressure, $10^{-8}$ Pa.
Figure 5. - Wear scar on titanium-iron binary alloy (8.12 at % Ti) rider showing grooves and cracks. Single pass on SiC (0001) surface; sliding direction \( \langle 10\bar{1}0 \rangle \); sliding velocity, 3 mm/min; load, 0.2 N; room temperature; vacuum pressure, \( 10^{-8} \) Pa.
Figure 6. - Coefficient of friction, calculated from first maximum-peak height in friction-force trace, as function of load for various titanium-iron alloys sliding on single-crystal silicon carbide (0001) surface. Single pass of alloy rider; sliding direction, (1010); sliding velocity, 3 mm/min; room temperature; vacuum pressure, 10^-8 Pa.

(a) 8.12 at% Ti.  
(b) 3.86 at% Ti.  
(c) 2.08 at% Ti.  
(d) 1.02 at% Ti.

Figure 7. - Coefficient of friction for various iron-base binary alloys as function of solute concentration. Single-pass sliding on single-crystal silicon carbide (0001) surface; sliding direction, (1010); sliding velocity, 3 mm/min; load, 0.2 N; room temperature; vacuum pressure, 10^-8 Pa.

(a) Manganese-iron alloy.  
(b) Nickel-iron alloy.  
(c) Chromium-iron alloy.  
(d) Rhodium-iron alloy.  
(e) Tungsten-iron alloy.  
(f) Titanium-iron alloy.
the alloys are generally much higher, as much as twice those for the pure metals.

Figure 8 presents the average coefficients of friction for the various alloys of figure 7 as functions of solute-to-iron atomic-radius ratio. In figure 8(a) the solute concentrations are all about 4 atomic percent. In figure 8(b) the coefficients of friction for the maximum solute concentration (see table 1) of each alloy are plotted. The maximum solute concentrations are up to approximately 16 atomic percent.

There appears to be good agreement between the adhesion and friction and the solute-to-iron atomic-radius ratio. The correlation of the coefficient of friction and solute-to-iron atomic-radius ratio is separated into two cases: first, the case for alloying with manganese and nickel, which have smaller atomic radii than iron; and, second, the case for alloying with chromium, rhodium, tungsten, and titanium, which have larger atomic radii than iron. The coefficients of friction increase generally as the solute-to-iron atomic-radius ratio increases or de-

![Figure 8. Coefficients of friction for iron-based binary alloys as function of solute-to-iron atomic radius ratio. Single-pass sliding on single-crystal silicon carbide (0001) surface; sliding direction, ⟨1010⟩; sliding velocity, 3 mm/min; load, 0.2 N; room temperature; vacuum pressure, 10⁻⁸ Pa.](image)

creases from unity. The increasing rate of the coefficients of friction for alloying elements that have smaller atomic radii than iron are much greater than that for alloying elements that have larger atomic radii than iron. The atomic-size ratio values reported here are from reference 7. The correlations indicate that the atomic size of the solute is an important factor in controlling the adhesion and friction in iron-base binary alloys as well as the abrasive wear and friction reported by the present authors (ref. 5) and the alloy hardening reported by Stephens and Witzkie (ref. 7).

A more detailed examination of figures 8(a) and (b) indicates that the correlations for manganese and nickel are better than those for titanium, tungsten, rhodium, and chromium. The coefficient of friction for rhodium is relatively low, and that for titanium is relatively high. The relative chemical activity of the transition metals (metals with partially filled d shell) as a group can be ascertained from their percent d bond character, after Pauling (ref. 10). The authors already determined that the coefficient of friction for silicon carbide in contact with various transition metals was related to the d bond character, that is, chemical activity of the metal (ref. 6). The more active the metal, the higher the coefficient of friction. Figure 9 shows the reciprocal d bond character of metals calculated from the data of reference 10. The greater the reciprocal percent d bond character, the more active the metal and the higher the coefficient of friction (ref. 6).

Rhodium-iron alloys in contact with silicon carbide showed relatively low friction. On the other
hand, titanium-iron alloys showed relatively high friction. The results seem to be related to the chemical activity of alloying elements, that is, the rhodium is less active, and titanium is more active, as indicated in figure 9. The good correlation for manganese, nickel, and chromium in figure 8 is due to the reciprocal percent d bond character for those being the almost same value for each.

Conclusions

As a result of sliding friction experiments conducted with various iron-base binary alloys in contact with single-crystal silicon carbide in vacuum, the following conclusions are drawn:

1. The atomic size and the concentration of the alloying element are important factors in controlling the adhesion and friction of iron-base, binary alloys.

2. The coefficient of friction generally increases markedly with the presence of any concentration of alloying element in the pure metal and more gradually with increasing the concentration of alloying element.

3. The coefficients of friction generally increase as the solute-to-iron atomic-radius ratio increases or decreases from unity.

4. The chemical activity of the alloying elements is also an important parameter in the controlling adhesion and friction of alloys. With increasing d bond character, there is a decrease in adhesion and friction.

5. All iron-base binary alloys transfer to silicon carbide.

References


ADHESION AND FRICTION OF IRON-BASE BINARY ALLOYS IN CONTACT WITH SILICON CARBIDE IN VACUUM

Kazuhisa Miyoshi and Donald H. Buckley

Single-pass sliding friction experiments were conducted with various iron-base binary alloys (alloying elements were Ti, Cr, Mn, Ni, Rh, and W) in contact with a single-crystal silicon carbide \{0001\} surface in vacuum. Results indicate that atomic size and concentration of alloying elements play an important role in controlling adhesion and friction properties of iron-base binary alloys. The coefficient of friction generally increases with an increase in solute concentration. The coefficient of friction increases linearly as the solute-to-iron atomic-radius ratio increases or decreases from unity. The chemical activity of the alloying elements is also an important parameter in controlling adhesion and friction of alloys, as these latter properties are highly dependent upon the d bond character of the elements.
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