Friction, Wear, and Auger Analysis of Iron Implanted With 1.5-MeV Nitrogen Ions

John Ferrante and William R. Jones, Jr.
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March 1982

Page 1, Left column, line 18: The concentration should be 6 at. %.

Page 4, right column, lines 22 and 23: The values of $n_{\text{max}}$ should be $5.0 \times 10^{21}$ atoms/cm$^3$ and 6 at. %.

Page 4, right column, line 25: The dose should be $2.8 \times 10^{17}$ atoms/cm$^2$.

Page 4, right column, line 29: The dose should be $5.0 \times 10^{17}$ atoms/cm$^2$.

Page 4, right column, lines 29 to 31: The sentence should read This agreement indicates that our calibration of depth and concentration was reasonable, and consequently...

Page 8, right column, line 24: The concentration should be $\sim 6$ at. %.

Page 9, left column, line 9: The word "product" should be changed to "quotient."

Page 9, left column: The equation should be

$$\frac{N}{Fe} = \frac{(N)}{(N/Fe)}_{2} = 0.0628$$

Page 9, left column, line 13: The values should be $N = 0.059$ and 5.9 at. %.

Page 9, left column, line 15: The concentration should be 6 at. %.

Page 9, right column, lines 7 and 8: The equation should be

$$n_{\text{max}} = (0.059)(8.49 \times 10^{22} \text{ atoms/cm}^3) = 5.0 \times 10^{21} \text{ atoms/cm}^3$$

Page 9, right column, lines 11 and 12: The equation should be

$$\text{dose} = \sqrt{\pi} \left(5.0 \times 10^{21} \text{ atoms/cm}^3 \right) \times (2.19 \times 10^{-5} \text{ cm}) = 2.8 \times 10^{17} \text{ atoms/cm}^2$$
Page 9, right column, line 13: The line should read

which is in agreement with the implanted dose (5.0 \times 10^{17} \text{ atoms/cm}^2) and

Page 10, right column: Equation (B5) should be

\[ r'^2 = R^2 - h'^2 - (R - h)^2 - 2h' (R - h) \]

Report documentation page, abstract, line 6: The concentration should be 6 at. %.
Friction, Wear, and Auger Analysis of Iron Implanted With 1.5-MeV Nitrogen Ions

John Ferrante and William R. Jones, Jr.
Lewis Research Center
Cleveland, Ohio
Summary

The effect of implantation of nitrogen ions (1.5 MeV) on the friction and wear characteristics of pure iron sliding against M-50 steel (unimplanted) was studied in a pin-on-disk sliding friction apparatus. Test conditions included room temperature (~25°C), a dry air atmosphere, a load of ½ kg (4.9 N), sliding velocities of 0.067 to 0.078 m/sec (~23 to 25 rpm), a pure hydrocarbon lubricant (n-hexadecane), and a nitrogen ion implantation dose of $5 \times 10^{17}$ ions/cm$^2$.

Small reductions in initial (~40 percent) and steady-state (~20 percent) wear rates were observed for nitrogen-implanted iron riders as compared with unimplanted controls. No differences in average friction coefficients were noted. Auger electron spectroscopy combined with argon ion bombardment revealed a subsurface Gaussian nitrogen distribution with a maximum concentration of 6 at.% at a depth of $8 \times 10^{-7}$ m (0.8 μm). Similar analysis within the wear scar (~2.0 x $10^{-5}$ m subsurface) of an implanted rider after 20 μm of wear yielded only background nitrogen concentration. No inward migration of nitrogen ions was observed.

Introduction

Ion implantation (ref. 1) is the process by which elements are injected into the surface region of a solid. This is accomplished by accelerating ions of the injected element in a vacuum chamber (~10$^{-5}$ torr) and allowing them to strike the solid substrate. Ion energies are usually in the range 10 to 500 keV.

The most important application of ion implantation has been in the semiconductor industry to introduce dopants into semiconductors (ref. 2). Other studies have shown that the implantation of certain elements (chromium, helium, and boron) can improve the corrosion resistance of steels and other alloys (refs. 3 to 5). Other applications have been in the areas of catalysis (ref. 6) and fatigue (ref. 7).

A number of investigators have reported substantial reductions in friction and wear of implanted surfaces. Molybdenum and sulfur implanted into the same steel surface reduced friction from 0.26 to 0.20 in unlubricated tests (ref. 8). Implantation of boron, nitrogen, and molybdenum ions reduced wear of nitriding steel by more than a factor of 10 (ref. 9). Similar results were obtained with nitrogen and carbon implanted in several different steels (ref. 10).

The present authors (ref. 11) reported the effect of low-dose ($5 \times 10^{15}$ ions/cm$^2$) nitrogen implantation on the friction and wear properties of pure iron in pin-on-disk experiments. No effects on either friction or wear were observed at this dose level.

More recently, it has become apparent that high nitrogen doses (>10$^{17}$ ions/cm$^2$) are required to affect the tribological properties of metals (ref. 12). However, the magnitude and direction (moving or stationary member) of these nitrogen implantation effects appear to be quite variable. Lu Russo, et al. (ref. 13) report nitrogen implantation effects on the unlubricated sliding wear of steel. A maximum reduction in the wear rate of 50 percent was observed at a dose of $2 \times 10^{17}$ ions/cm$^2$. A higher dose $8 \times 10^{17}$ ions/cm$^2$ yielded only a 10 percent wear reduction as compared with unimplanted steel. Hirvonen, et al. (ref. 14) indicate that nitrogen implantation not only reduces wear for stainless steels but also improves the load-carrying capacity, but only under boundary lubricating conditions. However, the life of a tool steel does not improve under milling conditions. Singer, et al. (ref. 15) report large effects (~six times) for titanium implantation in 52100 steel but no effect for nitrogen implantation in abrasive wear tests. The same authors (ref. 16) have reported detrimental nitrogen implantation effects on the abrasive wear resistance of 304 stainless steel.

The purpose of this investigation was to perform ion implantation studies under carefully controlled conditions. The pin-on-disk apparatus was selected since it is a classic technique for examining wear and is relatively easy to reproduce in any laboratory. The apparatus was placed in a plastic chamber so that the environment could be controlled. The sliding speed and load were selected to be low enough that elastohydrodynamic effects were minimized and the experiments were in the boundary lubrication regime. Since it is readily available and well characterized, n-hexadecane was chosen as the lubricant. Finally the rider (pin) material was standardized as pure iron to eliminate effects from varying materials. The riders (pins) were electropolished, annealed, and cleaned under fixed conditions in order to eliminate the effects of material preparation on the results. The disk material was selected to have a high hardness in order to make rider wear the controlling factor. Surface analysis along with ion bombardment were used to verify the presence of...
nitrogen as well as its distribution and quantity. In addition, to carefully control materials and experimental conditions, it was necessary to know the statistical level of confidence in the results. In the present study a sufficient number of tests were run on the same sample to establish repeatability and confidence level.

In this investigation the effects of high-dose ($5 \times 10^{17}$ ions/cm$^2$) nitrogen implantation (1.5 MeV) on the friction and wear properties of pure iron sliding against unimplanted M-50 steel in a pin-on-disk apparatus were studied. Other conditions included room temperature ($-25^\circ$ C), a dry air atmosphere, a load of $\frac{1}{2}$ kg (4.9 N), sliding velocities of 0.067 to 0.078 m/sec, and a pure hydrocarbon lubricant (n-hexadecane).

**Apparatus**

The pin-on-disk sliding friction apparatus is shown in figure 1. The test specimens were contained inside a plastic chamber so that the humidity of the test atmosphere could be controlled. A stationary 0.476-cm-radius, hemispherically tipped iron rider was placed in sliding contact with a rotating 6.3-cm-diameter (1.2-cm-thick) steel disk. A sliding speed in the range 0.067 to 0.078 m/sec was maintained. A normal load of $\frac{1}{2}$ kg (4.9 N) was applied with a deadweight.

**Materials**

The riders were machined from polycrystalline iron rod (99.95 percent pure) and fully annealed before testing. Some unimplanted riders were tested in the machined state. Others were electropolished. All implanted riders had been electropolished before implantation. The disks were made of CVM M-50 steel with a Rockwell C hardness of 62 to 64. The lubricant was n-hexadecane with the properties listed in table I. The n-hexadecane was percolated through alumina and silica gel to remove impurities just before testing.

Specimens were implanted with nitrogen at the State University of New York at Albany. A dynamitron accelerator was used at a beam energy of 1.5 MeV. Beam currents to 20 $\mu$A were used. A total dose of $5 \times 10^{17}$ ions/cm$^2$ was attained.

**Test Procedure**

**Wear Tests**

Disk specimens were ground and lapped to a surface finish of $10 \times 10^{-8}$ m $R_a$. They were then scrubbed with a paste of levigated alumina and water. Riders were cleaned similarly but with a commercial (nonabrasive) detergent instead of alumina. All specimens were dried on clean filter paper.

Approximately 50 milliliters of lubricant was added to the lubricant cup. The chamber was purged with dry air (<50 ppm H$_2$O) for a minimum of 10 min. The disk was set in motion and the rider loaded against it. Frictional force was measured by a strain gage. Rider wear was determined periodically by stopping the test and measuring the wear scar diameter. Wear rates were then determined for each test and portions of each test by linear regression analysis.

**Auger Analysis**

To verify that the ion implantation had occurred as expected an Auger electron spectroscopy (AES) depth-profiling analysis (ref. 17) was performed on a region of the implanted disk. The depth profile consisted of AES elemental analysis simultaneously with argon ion bombardment. Consequently the distribution of implanted nitrogen with depth could be obtained with high spatial resolution.

The depth profiling was performed in an ultra-high-vacuum system with a base pressure of $1 \times 10^{-10}$ torr. The vacuum system was backfilled with argon to a pressure of $5 \times 10^{-5}$ torr. The surface was then bombarded with a beam of argon ions at a beam energy of 3000 eV and a current density of approximately

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity, N sec/m$^2$ (cP)</td>
<td>3.34</td>
</tr>
<tr>
<td>Density, g/milliliter</td>
<td>0.775</td>
</tr>
</tbody>
</table>

**TABLE I.** TYPICAL PROPERTIES OF LUBRICANT n-HEXADECANE AT 20°C
20 μA/cm² when focused. The beam was rastered, however, over an area of 0.5 cm by 0.5 cm to insure uniformity. In addition, the normal to the analyzed surface was at an angle of 60° to the ion beam direction. The electron beam for AES analysis was centered in the ion-bombarded region and had a beam energy of 2000 eV and beam currents ranging from 1.0 to 5.0 μA depending on the size of the nitrogen AES peak. The beam diameter was nominally 2.5 × 10⁻⁵ m (25 μm). The surface was then simultaneously sputtered and AES analyzed, with the peak-to-peak height of the nitrogen AES peak determined as a function of time. The sputtering rate was estimated by determining the time needed to sputter through a 1 × 10⁻⁷-m (1000-Å) tantalum oxide film. Since the surface was covered with an oxide and residual lubricant, the nascent surface was taken to be at the point where the low-energy (46 eV) iron peak (ref. 18), saturated (fig. 2).

The sputtering rate estimated from the tantalum oxide film was determined to be 1.14 nm/min. A profilometer trace at the sputtering crater gave good agreement with the calculated depth. This was used to calibrate the depth from the sputtering time. The nitrogen concentration was determined from the relative sensitivities of the 381-eV nitrogen and 703 eV iron peaks (ref. 18). Using this relative sensitivity and the experimental peak-to-peak height ratios (fig. 2) and assuming that the disk was pure iron enabled the atomic concentration of nitrogen with depth to be estimated.

**Results**

**Wear**

The results of unimplanted control tests for machined iron riders are presented in table II, and similar results but with the riders electropolished after machining are given in table III. Finally, the results for electropolished, nitrogen-implanted iron riders sliding against an unimplanted steel disk are presented in table IV. A summary of all the results appears in table V. Three different wear rates are given in these tables. The initial wear rate refers to measurements taken after the first 2 m of sliding, steady-state wear rate refers to measurements taken after approximately 15 to 150 m of sliding, and the overall wear rate takes all wear measurements into account. Initial wear rates were considered since implantation effects might be magnified in the early wear measurements because of the shallow implantation depth.

As expected, initial wear rates were much higher (by an order of magnitude) than steady-state rates, and the rate

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**TABLE II. – FRICTION AND WEAR RESULTS FOR UNIMPLANTED, MACHINED IRON RIDER**

[Unimplanted M-50 steel disk; 4.9-N load.]

<table>
<thead>
<tr>
<th>Test</th>
<th>Sliding velocity, m/sec</th>
<th>Steady-state friction coefficient</th>
<th>Initial</th>
<th>Steady state</th>
<th>Overall</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Wear rate, m³/N-m</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.072</td>
<td>0.14</td>
<td>58 × 10⁻¹⁵</td>
<td>5.7 × 10⁻¹⁵</td>
<td>7.6 × 10⁻¹⁵</td>
</tr>
<tr>
<td>2</td>
<td>0.067</td>
<td>0.15</td>
<td>79</td>
<td>6.2</td>
<td>8.0</td>
</tr>
<tr>
<td>3</td>
<td>0.077</td>
<td>0.14</td>
<td>57</td>
<td>9.3</td>
<td>10.0</td>
</tr>
<tr>
<td>4</td>
<td>0.070</td>
<td>0.15</td>
<td>36</td>
<td>5.7</td>
<td>6.2</td>
</tr>
<tr>
<td>5</td>
<td>0.069</td>
<td>0.15</td>
<td>45</td>
<td>3.6</td>
<td>4.4</td>
</tr>
<tr>
<td>6</td>
<td>0.075</td>
<td>0.15</td>
<td>30</td>
<td>4.4</td>
<td>5.0</td>
</tr>
<tr>
<td>7</td>
<td>0.072</td>
<td>0.14</td>
<td>60</td>
<td>7.7</td>
<td>9.9</td>
</tr>
<tr>
<td>8</td>
<td>0.071</td>
<td>0.15</td>
<td>71</td>
<td>10.0</td>
<td>11.0</td>
</tr>
</tbody>
</table>

**TABLE III. – FRICTION AND WEAR RESULTS FOR UNIMPLANTED, ELECTROPOLISHED IRON RIDER**

[Unimplanted M-50 steel disk; 4.9-N load.]

<table>
<thead>
<tr>
<th>Test</th>
<th>Sliding velocity, m/sec</th>
<th>Steady-state friction coefficient</th>
<th>Initial</th>
<th>Steady state</th>
<th>Overall</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Wear rate, m³/N-m</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>0.073</td>
<td>0.15</td>
<td>42 × 10⁻¹⁵</td>
<td>3.7 × 10⁻¹⁵</td>
<td>4.9 × 10⁻¹⁵</td>
</tr>
<tr>
<td>10</td>
<td>0.078</td>
<td>0.14</td>
<td>37</td>
<td>4.0</td>
<td>4.7</td>
</tr>
<tr>
<td>11</td>
<td>0.078</td>
<td>0.16</td>
<td>42</td>
<td>4.6</td>
<td>5.7</td>
</tr>
<tr>
<td>12</td>
<td>0.075</td>
<td>0.16</td>
<td>44</td>
<td>6.1</td>
<td>7.5</td>
</tr>
<tr>
<td>13</td>
<td>0.071</td>
<td>0.14</td>
<td>45</td>
<td>6.0</td>
<td>7.5</td>
</tr>
</tbody>
</table>
TABLE IV. - FRICTION AND WEAR RESULTS FOR NITROGEN-IMPLANTED, ELECTROPOLISHED IRON RIDER

[Unimplanted M-50 steel disk; 4.9-N load.]

<table>
<thead>
<tr>
<th>Test</th>
<th>Sliding velocity, m/sec</th>
<th>Steady-state friction coefficient</th>
<th>Initial Wear rate, m³/N·m</th>
<th>Steady state Overall Average friction coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>0.078</td>
<td>0.15</td>
<td>18 × 10⁻¹⁵ 2.1 × 10⁻¹⁵ 2.8 × 10⁻¹⁵</td>
<td>0.15 ± 0.005a</td>
</tr>
<tr>
<td>15</td>
<td>0.075</td>
<td>0.15</td>
<td>20 2.8 3.7</td>
<td>0.15 ± 0.004a</td>
</tr>
<tr>
<td>16</td>
<td>0.073</td>
<td>0.15</td>
<td>37 3.2 4.0</td>
<td>0.15 ± 0.004a</td>
</tr>
<tr>
<td>17</td>
<td>0.078</td>
<td>0.16</td>
<td>33 4.4 5.4</td>
<td>0.15 ± 0.004a</td>
</tr>
<tr>
<td>18</td>
<td>0.074</td>
<td>0.15</td>
<td>34 5.0 6.6</td>
<td>0.15 ± 0.004a</td>
</tr>
<tr>
<td>19</td>
<td>0.071</td>
<td>0.15</td>
<td>30 4.2 5.3</td>
<td>0.15 ± 0.004a</td>
</tr>
<tr>
<td>20</td>
<td>0.070</td>
<td>0.15</td>
<td>37 5.5 6.6</td>
<td>0.15 ± 0.004a</td>
</tr>
</tbody>
</table>

variations were greater. Wear (both initial and steady state) appeared to be lower for the electropolished unimplanted rider than for the machined control rider. However, statistical analysis using small-sample theory (ref. 19) indicated no significant difference. Similar analyses comparing the initial wear rates for an unimplanted, electropolished rider and a nitrogen-implanted, electropolished rider yielded a difference, significant at a 99-percent confidence level. The same analysis of the steady-state rates had a 95-percent confidence level. So the wear rate reductions for nitrogen implantation for initial (~40 percent) and steady state (~20 percent) are statistically valid.

Rider wear volume as a function of sliding distance is plotted in figure 3 for machined unimplanted, electropolished unimplanted, and electropolished nitrogen-implanted riders, respectively. These data have been replotted in a different format in figure 4. Here incremental wear rates between each wear measurement are plotted as a function of sliding distance.

Friction

A typical trace of friction as a function of time is presented in figure 5. All tests, both implanted, and unimplanted, yielded similar traces. All were characterized by an initial friction spike at startup followed by a lower constant value (usually ±0.005) for the remainder of the test. Values taken from the steady-state portion of the trace are given for each test in tables II to IV. The steady-state friction coefficients are averaged in table V. Essentially, the same value was obtained for all three test series.

The results of the AES depth profile were curve fit to a Gaussian (ref. 20) by linear regression. The equation for the Gaussian is

\[ n(x) = n_{\text{max}} e^{-\frac{(x-x_0)^2}{2\sigma^2}} \]  

where \( n \) is the atomic concentration, \( x \) is the depth, \( x_0 \) is the range, and \( \sigma \) is the width. The dose can be determined from

\[ n_{\text{max}} = \frac{\text{dose}}{\sqrt{2\pi}\sigma} \]  

A typical AES spectrum from the implanted disk is shown in figure 2. The nitrogen AES peak at increased sensitivity is shown in figure 6. The variation in nitrogen concentration with depth is shown in figure 7. The values for the fitting parameter from equation (1) were found to be \( x_0 = 8.8 \times 10^{-7} \) m (0.88 \( \mu \)m) and \( \sigma = 2.2 \times 10^{-7} \) m (0.22 \( \mu \)m). The value of \( n_{\text{max}} \) was calculated to be \( 5.6 \times 10^{23} \) atoms/cm\(^3\) or approximately 8 at.\%.

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TABLE V. - SUMMARY OF FRICTION AND WEAR TESTS

<table>
<thead>
<tr>
<th>Rider preparation</th>
<th>Initial</th>
<th>Steady state</th>
<th>Overall</th>
<th>Average friction coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unimplanted, machined rider</td>
<td>55 × 10⁻¹⁵ ± 17a</td>
<td>6.5 × 10⁻¹⁵ ± 2.3a</td>
<td>6.9 × 10⁻¹⁵ ± 2.2a</td>
<td>0.15 ± 0.005a</td>
</tr>
<tr>
<td>Unimplanted, electropolished rider</td>
<td>43 ± 5.1a</td>
<td>4.9 ± 1.1a</td>
<td>5.4 ± 1.3a</td>
<td>0.15 ± 0.01a</td>
</tr>
<tr>
<td>Nitrogen-implanted, electropolished rider</td>
<td>27 ± 8.4a</td>
<td>3.9 ± 1.2a</td>
<td>4.4 ± 1.3a</td>
<td>0.15 ± 0.004a</td>
</tr>
</tbody>
</table>

*Standard deviation.
Figure 3. Rider wear volume as a function of sliding distance. Load, 4.9 N; sliding velocity ~0.07 m/sec; lubricant, n-hexadecane; disk, unimplanted M-50 steel.
Figure 4. - Incremental wear rate as a function of sliding distance. Load, 4.9 N; sliding velocity, 0.07 m/sec; lubricant, n-hexadecane; disk, unimplanted M-50 steel.

(a) Unimplanted iron rider.

(b) Unimplanted, electropolished iron rider.

(c) Nitrogen-implanted, electropolished iron rider.

Figure 5. - Typical friction trace.

Figure 6. - Nitrogen Auger peak.
A further analysis is called for in the present study. Since the riders were hemispherical, the radial distribution of nitrogen across the wear scar was not uniform. This nonuniformity of distribution is analyzed in appendix B. The results of this analysis are presented in figure 8. In that figure the fractional area of the wear scar within a specified depth is shown as a function of the maximum depth at the center of the wear scar. The depths selected were the depth at which the maximum nitrogen distribution occurred to a depth slightly more than this value plus a Gaussian half-width \( \sigma \). As figure 8 shows, the percentage of the area varies rapidly at first and then varies much more slowly. At a center depth of 10 \( \mu \text{m} \), well beyond the implanted range, approximately 10 percent of the area is still in a region with high nitrogen concentration.

**Hardness Measurements**

To determine the effects of implantation on hardness, Vickers microhardness measurements were performed on an M-50 disk in implanted and unimplanted regions. A load was used with an average penetration depth of approximately 2 to 3 \( \mu \text{m} \). This samples the implanted region, which has a maximum dose at \( \sim 1 \mu \text{m} \). The results were averagehardnesses of 996 \( \pm 35 \) kg/mm\(^2\) in the implanted region and 821 \( \pm 38 \) kg/mm\(^2\) in the unimplanted region, an indication of increased hardness due to implantation.

**Discussion**

Using pin wear to determine the effects of ion implantation is quite a severe test. In the initial stages of the test (e.g., \( \sim 2 \) m of sliding distance) the depth worn at the center of the wear scar (\( \sim 5 \mu \text{m} \)) was well beyond the implanted range. Although this depth varied with radial position, a large fraction of the contact region was beyond the range of the implantation. Consequently in this study a relatively high implantation energy (1.5 MeV) was selected to give a large range (\( \sim 1 \mu \text{m} \)). In other current studies, much lower energies are being used (\( \sim 50 \) keV with ranges of 0.1 to 0.2 \( \mu \text{m} \)). In all these studies, effects well beyond the implantation range have been observed. Generally, inward diffusion of the implanted species during the wear process is offered as an explanation for the exceptional depths of effectiveness (ref. 12). Our observations indicated decreased wear well beyond the implantation range. Effects are still seen in the mild wear range, where final wear depth on the pin is about 20 \( \mu \text{m} \). However, in light of the analysis presented in appendix B and figure 8 these results can be understood simply in terms of the geometry of the contact. A fraction of the area comparable to wear reduction still is within the implantation range. Thus there is no strong evidence for effects beyond the implantation range. Auger analysis of these final wear scars revealed no significant nitrogen concentration near the center of the scars, that is, no higher than was observed by Jones and Ferrante (ref. 11), where a null result was obtained. The extended range of effectiveness...
in the other studies may be due to strain fields resulting from the implantation but well beyond the range of the implanted ions. However, implantation with argon (ref. 22) showed no improvement.

Bolster and Singer (ref. 16) observed a reduction in abrasive wear in a carbon steel and an increase in wear in 304 stainless steel. Bolster and Singer performed one of the few studies where repeatability was checked. However, even in their elegant studies the scatter was such that at times no effect was observed at approximately the same conditions. Hartley (ref. 12) attributes the improved wear resistance of ferrous materials to a formation of martensitic structures with the nitrogen. Most studies to date have been performed on alloys, which are complicated combinations of materials. The fact that a positive result is found in pure iron should help sort out mechanisms concerning minor components in alloys. For example, the nitrogen could be interacting with the iron to produce martensitic hardening (ref. 12) without worry about what other interactions may be taking place with other alloy components. In iron chromium alloys, chemical interactions of nitrogen with chromium rather than with iron would be far more likely because chromium nitrides have a higher free energy of formation than iron nitrides.

A final point of interest is which component shows an effect in a sliding or rolling contact. Hirvonen, et al. (ref. 22) observed no effect on wear in the stationary component in a crossed-cylinders configuration. This would not necessarily be surprising, since in this case the stationary component undergoes constant wear. With low implantation energies the range of implantation is only of the order of 0.1 μm and could be expected to wear away quickly even with an enhanced depth of effectiveness. Surprisingly, Hirvonen found that implantation of the rotating component decreases wear in the stationary component. In the present study a decrease in wear of the stationary component (the rider, which is undergoing continuous wear) was observed. This was probably due to our greater depth of implantation. Consequently a wear reduction on the member undergoing continuous wear in a sliding configuration was observed.

As indicated, a number of interesting experiments have been done concerning the effects of ion implantation on wear. To date, however, there is no clear-cut understanding of the physical mechanisms involved. Therefore effort now should be directed toward designing experiments that would clarify these mechanisms.

**Summary of Results**

A sliding pin-on-disk apparatus was used to determine the effect of nitrogen ion implantation on the friction and wear characteristics of pure iron sliding against M-50 steel. The major results were as follows:

1. Small reductions were effected in initial (~40 percent) and steady-state (~20 percent) wear rates of nitrogen-implanted iron riders.
2. Average friction coefficients were not affected by nitrogen ion implantation.
3. A Gaussian nitrogen distribution with a maximum concentration of ~ at.% at a depth of $8 \times 10^{-7}$ m (0.8 μm) was observed in an unworn, implanted disk by Auger electron spectroscopy (AES).
4. AES revealed only background nitrogen concentration within the wear scar of an implanted rider. No inward migration of nitrogen ions was observed.
5. The present results can be understood simply in terms of a geometrical analysis of the pin-on-disk configuration and not in terms of any effect beyond the range of implantation.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, August 26, 1981
Appendix A

Calibration of Depth of Implantation and Concentration of Implanted Nitrogen

The first step in the calibrations was to obtain the relative AES sensitivities for the 703-eV iron peak and the nitrogen peak from the PHI handbook (ref. 18), giving \( \frac{N}{Fe} = 1.64 \). The equivalent ratio of AES peak heights was then obtained by ratioing the nitrogen to iron AES peaks at the maximum in the Gaussian distribution curve, giving \( \frac{N}{Fe} = 0.103 \). The \( \frac{N}{Fe} \) is the actual atomic ratio of nitrogen to iron.

\[
\frac{N}{Fe} = \left( \frac{N}{Fe} \right)_1 \left( \frac{N}{Fe} \right)_2 = 0.169
\]

Assuming the total adds to unity, \( N + Fe = 1 \). Solving these equations simultaneously gives \( N = 0.145 \), or 14.5 at.%. Therefore nitrogen has a concentration of approximately 15 at.% at the maximum.

Next we can estimate the implanted dose from our curve fit to a Gaussian (ref. 18)

\[
dose = \frac{\text{dose}}{\sqrt{2\pi} \sigma} \]

As an approximation we assume that the atomic density of the implanted region is the same as that of body-centered cubic iron \((8.49 \times 10^{22} \text{ atoms/cm}^3)\). Therefore

\[
\frac{N}{Fe} = (0.145) \times (8.49 \times 10^{22} \text{ atoms/cm}^3) = 1.22 \times 10^{22} \text{ atoms/cm}^3
\]

We obtain \( \sigma \) from the curve fit to the Gaussian, where \( \sigma = 2.19 \times 10^5 \text{ cm} \); and thus from equation (1)

\[
dose = \sqrt{2\pi} (1.22 \times 10^{22} \text{ atoms/cm}^3) \times (2.19 \times 10^{-5} \text{ cm}) = 6.7 \times 10^{17} \text{ atoms/cm}^2
\]

which is in good agreement with the implanted dose and which independently indicates that the sputtering depth calibration is reasonable.
Appendix B
Height Removed as a Function of Radial Position on the Wear Scar

where
\( A \) area of wear scar
\( \Delta A \) implanted area
\( h \) maximum height removed at center of wear scar
\( h' \) height removed at \( r' \)
\( R \) radius of rider
\( r \) radius of wear scar
\( r' \) radial position on wear scar

By applying some simple geometry and trigonometry we can derive the desired relation. From the law of Cosines, on triangle BCD

\[
R^2 = h'^2 + X^2 - 2h'X \cos \theta \tag{B1}
\]

(\( \theta \) is defined by the angle BCD), where

\[
\theta = 90^\circ + \phi
\]

\[
X = \sqrt{(R - h)^2 + r'^2} \tag{B2}
\]

\[
\sin \phi = \frac{R - h}{X} \tag{B3}
\]

\[
h = R - \sqrt{R^2 - r^2} \tag{B4}
\]

Substituting into equation (B1) and rearranging give

\[
r'^2 = R^2 - h'^2 - 2(R - h)^2 - 2h'(R - h) \tag{B5}
\]

Finally

\[
\frac{\Delta A}{A} = \frac{r^2 - r'^2}{r^2} = 1 - \left( \frac{r'}{r} \right)^2 \tag{B6}
\]

Therefore, from equations (B4) and (B5) we can calculate \( \Delta A/A \) as a function of \( h \), with \( h' \) as a parameter (fig. 8).
References

The effect of implantation of 1.5-MeV nitrogen ions on the friction and wear characteristics of pure iron sliding against steel was studied in a pin-on-disk apparatus. An implantation dose of $5 \times 10^{17}$ ions/cm² was used. Small reductions in initial (~40 percent) and steady-state (~20 percent) wear rates were observed for nitrogen-implanted iron riders compared with unimplanted controls. Auger electron spectroscopy revealed a subsurface Gaussian nitrogen distribution with a maximum concentration of 15 at.% at a depth of $8 \times 10^{-7}$ m. A similar analysis within the wear scar of an implanted rider after 20 μm of wear yielded only background nitrogen concentration, thus giving no evidence for diffusion of nitrogen beyond the implanted range.