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Ion implantation for improvement of wear properties of steels

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ION IMPLANTATION FOR IMPROVEMENT
OF WEAR PROPERTIES OF STEELS

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ABSTRACT

Previous research on ion implantation at U.M.R. has dealt with the development of a modified Falex Lubricant Tester as a valid wear test and its use in dose curve determination. Our recent work uses Auger surface analysis and scanning electron microscopy to gather support for a model that will tie together some of the many theories proposed as to why ion-implantation improves the surface related mechanical properties of steels.

ACKNOWLEDGEMENT

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Thanks are due to Dave Windes who performed all of the implantation work and Tim Sommerer who performed the Auger analysis. John Thompson is to be thanked for his assistance in performing the scanning electron microscopy. Mike Muehlemann, Bill Baker and Dane Crutcher are due the thanks that only the members of a team working for a common goal can appreciate.

Finally, thanks are due to the Kopper's Corporation for funding an interesting and worthwhile research program.

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I.INTRODUCTION

Ion implantation has progressed over the past decade from a method for doping silicon in semiconductor manufacturing to a method for improving the wear, fatigue and corrosion properties of metals and carbides. Research has been performed by groups around the world that supports the use of ion implantation as a surface modification technique in research laboratories as well as in industrial applications.

Ion implantation of various elements into steels and carbides has been shown to lower friction coefficients, improve wear properties, improve corrosion resistance and increase fatigue life. The shallow depth of penetration need not be a concern in many cases because it has been shown that the implanted species, or its influence, may last to much greater depths than the original implanted thickness.

Hartley (1) found in a survey of industrial contacts that over 80% were concerned most with problems related to wear. Other studies have been performed that cite corrosion and fracture, in addition to wear, as major contributors to industrial losses in terms of direct and indirect costs of maintaining and replacing facilities and equipment. Therefore, the idea of tailoring surface modification techniques to enhance resistance to wear, fatigue and corrosion becomes a very desirable goal.

Techniques for modifying surface properties of a metal, such as nitriding, chrome-plating, carburizing or ion implantation, have their own special effects which in turn create certain advantages and disadvantages. However, the basic objective is the same, namely to modify the surface in such a way that it possesses properties different from those of the bulk material. When comparing these processes, one of the first differences that becomes apparent is the depth of modification. In carburizing, case depths of 0.020" to 0.100" are not uncommon values. Nitriding yields typical case depths in the 0.008" to 0.025" range. Chrome plating thicknesses are in the range of 0.001" to 0.003" in decorative applications and up to a range of 0.005" to 0.050" for hard chrome plate needed for buildup and wear resistance.

As thin as these may sound thay are all much greater than the penetration depth for ion implantation. Penetration depths of thousands of angstroms are typical in ion implantation. A depth of 1000 angstroms or 4×10^{-7} inches for the range of implanted nitrogen seems insignificant compared to the other techniques, however very significant surface property modifications have been observed.

It was stated earlier that every process has its own peculiarities that create certain advantages and disadvantages. Dearnaley (2) has tabulated some of them for ion implantation. His views of the advantages and disadvantages are listed as Table I. It should be noticed that some of them are operator related, such as being an unfamiliar process, and some can be listed as advantages and disadvantages simultaneously, such as being a vacuum process. This list is not complete, as each operator or organization will have his own additions or changes, but it covers the major points.

Table I.

ADVANTAGES AND DISADVANTAGES OF ION IMPLANTATION

ADVANTAGES	DISADVANTAGES
Versatility regarding ion species and substrate	High capital cost
Controllability	Shallow treatment
No buildup	Line-of-sight process
Clean vacuum process	Unfamiliar process
Applied to finished components	Requires in-vacuo manipulation
Monitored electrically	
Low power consumption	
No toxicity	

The use of acid baths or high temperatures have been a drawback to some of the other more conventional surface modification techniques. Ion implantation is performed under a vacuum and the temperature of the workpiece can be controlled by limiting the ion beam current and size relative to the workpiece (1). Hartley performed a case study of life improvement of tooling

TABLE II

CASE STUDY OF ION IMPLANTATION

APPLICATION	MATERIAL	ION TREATMENT	RESULT
Paper slitters	1C 1.6Cr Steel	$8\times10^{17}\ N/cm^2$	Cutting life x 2
Acetate punches	Cr-plate	$4\times10^{17}\ N/cm^2$	Improved product
Taps for drilling plastics	HSS	$8\times10^{17}\ N/cm^2$	Life x 5
Slitters for Synthetic rubber	WC-6% Co	$8\times10^{17}\ N/cm^2$	Life x 12
Tool inserts	4Ni 1Cr steel	$4\times10^{17}\ CO/cm^2$	Contamination x 1/3
Forming tools	12Cr 2C	$4\times10^{17}\ N/cm^2$	Much reduced adhesive wear
Dies for copper rod	WC-6% Co	$5\times10^{17}\ C/cm^2$	Throughput x 5
Drawing dies	WC-6% Co	$2\times10^{17}\ CO/cm^2$	Improved life
Dies for steel wire	WC-6% Co	$3\times10^{17}\ C/cm^2$	Wear rate x 1/3

through ion implantation and the rather exciting results that were obtained are depicted in Table II. Additional data (3) indicated that in some cases the improvement in tooling life persists after tools have been reground.

When examining this data, it is interesting to note the absence of reports of improved performance of chip cutting tools, such as drills or cutting tools. The tools that show improvement are ones that do not see continuous duty. This ties in well with experimental results reported by Hale et al. (4) and Hirvonen et al. (5) where improvement in wear was only observed on the member that did not see constant load conditions.

Although many properties can be changed through ion implantation, the Kopper's supported work at U.M.R. has been focused on wear property improvements. The aim is to determine the fundamental reasons why improvement occurs rather than to merely exploiting the final end result. This is a very lofty goal and can be summed up well in a quote from H. Herman (6).

> "Furthermore, some of the most significant implantation-induced effects are observed in commercial alloys. Here one is attempting to explain complex mechanical behavior, effected by difficult-to-characterize implantation processes, in a highly impure, structurally and chemically inhomogeneous alloys. (This situation, it is important to note, is consistent with the best tradition of industrial physical metallurgy.)"

Herman's quote summarizes very well the situation that exists in relating ion implantation and wear properties. It is well accepted that improvements by factors of ten to one hundred are possible. However, there have been differences of opinion as to why this occurs.

We have continued to use the cylinder-in-groove test geometry for our wear testing. Rather extensive tests have been performed to examine implanted and unimplanted pins at various time intervals. The research, that serves as the basis of this thesis, uses in-depth techniques of Auger surface analysis and electron microscopy to develop a model that relates wear and ion implantation in a manner that will be both consistant with previous research and yet provide a new model into the field.

II. RELEVANT TOPICS

The investigation of the use of ion implantation as a process to modify surface properties is relatively recent work. Nevertheless the results have been so impressive that commercial ion-implanters are now in the marketplace. Part of the reason for this impressive result is that ion implantation has been found to improve wear, corrosion and oxidation resistance, fatigue life and reduce the coefficient of friction in numerous alloy systems. It is not surprising to find that many facets of the results in these surface-related phenomenon are intertwined.

A.FRICTION

The coefficient of friction relates to wear because it correlates with the force parallel to the surface which is the same force that causes the most damage in wear. If the coefficient of friction can be reduced, the frictional forces and corresponding amount of wear can be reduced with the same applied normal force. Numerous researchers have investigated modification of the coefficient of friction with positive results. In a recent study sponsored by the Navy (7), it was found that implantation of Ti^+ into 52100 tool steel reduced the coefficient of friction from 0.8 to 0.3. Iwaki et al. (8) found in similar testing that implantation of Cr^+ into steel caused the coefficient of friction to decrease, but that the implantation of Cu^+ and Ni^+ caused the coefficient to increase. These results indicate that, not only can a dramatic decrease in the coefficient of friction be effected, but it can be modified up or down to suit your needs.

Another result of the Navy sponsored study is important (7). Ti^+ ions that were implanted at 50 keV reduced the coefficient to 0.3 with a fluence of 2 x 10^{17} Ti^+ / cm^2, however it took 5 x 10^{17} Ti^+ / cm^2 to get an equivalent reduction when implanted at 190 keV. This result indicates that the closer the implanted species is to the surface the more effective that it is. This is almost an expected result when you consider that friction is a surface related property.

Shepard and Suh (9) show the importance of the coefficient of friction in their work. Using a computer model and assuming a thin, hard surface layer and no reduction of the friction coefficient, they found that there was no noticeable reduction in subsurface stresses which could contribute to deformation and consequently surface wear. However, when a reduction of frictional forces is considered, there is a substantial reduction of subsurface deformation and stresses, which in turn would reduce wear. Iwaki et al. (8) have indicated that the reduction in friction is caused by the oxygenation of the implanted species. Now we begin to see the complex interaction of the surface related properties.

B. OXIDATION

It has been indicated that there exists possible ties between oxidation and friction. It may also be anticipated that oxidation and corrosion, which are surface sensitive processes, respond to ion implantation (10). Dearnaley (11) listed a few guiding principles for corrosion resistance that tie oxidation firmly into the picture. If you can perform any of the following, then you may be able to reduce the rate of corrosion.

1) Form a coherent oxide layer
2) Block short-circuit diffusion paths
3) Induce catalytic effects
4) Induce oxide plasticity effects
5) Modify oxide defect population
6) Modify oxide conductivity

Since ion implantation is performed under a vacuum, it would seem that the low partial pressure of oxygen would preclude the formation of any oxides during implantation. However, electron diffraction patterns have shown that impurity oxygen and carbon atoms can become incorporated into the implanted layer during implantation and combine with the metallic species. Iron and chromium have been identified in a spinel typified by $FeCr_2O_4$ (1). Under the effects of ion bombardment the enhanced migration of impurity oxygen atoms inward and of iron atoms outward leads to the formation of an iron oxide zone on the outermost layers of the sample (12). The formation of these surface oxides is also substantiated by the changes in surface color. These have been documented by Hartley (12) as well as by the research at U.M.R.

It is suggested by Baumvol (13) that the reduction in the oxidation rate, due to ion implantation, is due to the inhibition of the outward diffusion of iron cations through the scale. This inhibition can be caused by several mechanisms cited earlier by Dearnaley, such as coherent oxide formation or blocking of short-circuit diffusion paths (11). The modification of diffusion kinetics is also accompanied by an improvment of the adherence of the oxide layer (13).

The oxidation rate of the surface is tied into the wear rate of the material in work done by Kerridge (13), Hartley (14), Goode et al. (15) and Rowson and Quinn (16). In these works the oxidative theory of wear is discussed. The oxidative theory of wear states that as surfaces come into contact the asperities meet and deform. This deformation causes the surfaces to heat and therefore oxidize. The subsequent oxide layer is the material that now controls the rate of wear.

C. WEAR

It has been shown through the works of various researchers that friction, oxidation and corrosion affect or contribute to wear. In order to explore wear in more detail wear must be defined. Wear can be classified into four main groups: 1) abrasive wear, 2) adhesive wear, 3) surface fatigue wear, and 4) corrosive (including oxidative) wear (17).

Abrasive wear may be defined as damage to a surface by a harder material. This hard material may be introduced between two rubbing surfaces from outside; it may be formed in-situ by oxidation and other chemical processes; or it may be the material forming the second surface.

Adhesive wear is characterized by the interaction of asperities, causing metal to be transferred from one surface to another. A particularly severe form of adhesive wear is known as scuffing.

Surface fatigue is the predominant mode of failure when a surface, such as a bearing, undergoes repeated high contact stresses. The distribution of Hertzian stresses is such that the maximum stresses occur below the surface. When a defect is created and then propagates under a cyclic or fatigue load, it propagates towards the surface. Once the defect reaches the surface a piece of metal detaches leaving a pit.

Corrosive and oxidative wear take place when sliding occurs in a corrosive or oxidative environment. Normally a film forms which may act as a passive layer slowing or arresting further reaction. However, in corrosive wear, sliding interrupts the film, causing a combination of further corrosive attack coupled with another wear mechanism.

Now that we have an idea of what wear is, the question becomes, "Is there a standard test for measuring wear?". The answer is a resounding, no! Since most people involved with wear have their own special circumstances, they have invariably developed their own test that most closely models their situations. The bulk of wear research involving ion implantation has used three basic test geometries and set-ups. They are 1) Pin-on-disc, 2) Crossed cylinders, and 3) Cylinder-in-groove.

A loaded pin wears against a rotating disc in a geometry similar to a phonograph needle on a record in the pin-on-disc test (18). The pin and disc are normally submerged in a bath or sprayed with a lubricant. The wear rate of the wear couple is usually assessed by the loss of material from the pin.

In the crossed cylinder test two cylinders are arranged at 90° to each other. The lower cylinder rotates in a lubricant bath while load is applied through the upper, stationary cylinder. The wear rate is determined by optical measurment of the wear scar on the stationary member (19).

A modified Falex Lubricant Tester is used with the cylinder-in-groove test. A rotating pin is loaded between two V-blocks submerged in a lubricant bath (20). Wear measurements are taken directly from the mass loss of the pin, which is somewhat different from the previous tests where wear measurements are made on the stationary member.

Even though different ions have been used, most generally nitrogen ions are used as the implanted species in wear tests of implanted steel. Nitrogen is used due to its ease of ionizing and the large beam currents that are possible. Implantation of nitrogen into steels causes the wear rate to drop roughly 10 to 100 times if the dose exceeds 2×10^{17} N^{+}/cm^{2} (5),(14),(21). This now gives us a common link to use in examining the results of the reported wear tests.

One difference between the tests is that in the cylinder-in-groove and crossed cylinder tests, a reduction in wear rate of the stationary member was found only if the rotating member was implanted (4),(5). In the pin-on-disc test they generally measure the wear rate of the couple by the mass loss of the pin and a reduction in wear is seen. This may be due to higher contact stresses and thus higher wear rates than the pin-on-disc tests, but it is uncertain at this time.

Early work on improvement of wear through ion implantation seemed to center on showing that ion implantation created a hard surface layer, similar to nitriding or carburizing, which decreased the adhesive component of wear by hardening the surface (22),(23). Contributing factors to this line of thought were discoveries that 20% to 40% of the implanted species remained after wear tracks were much deeper than the original implanted depths (1),(2), (18),(24). The premise was that the implanted nitrogen forms coherent intermetallic compounds of Fe_4N and Fe_2N at the surface, which either harden the surface or cause surface asperities to flow under loading, causing lubrication to proceed more efficiently (14),(25). The nitrogen is proposed to diffuse ahead of the wear front through a network of subsurface dislocations so as to maintain a hard surface of just the thickness to most effectively reduce wear (18),(26).

Work done by Pollock et al. (27) shows that the nitrogen does diffuse into the steel, but the diffusion is minor. Their tests found no evidence of nitrogen remaining after the surface had worn to a depth of twice the implanted depth.

Work done on the improvement of wear properties of steels with the implantation of aluminum agrees with the Pollock work. Predicted diffusion rates of aluminum through iron is only on the order of several atomic diameters for the recorded test time (9). Therefore it would be very unlikely that diffusion of the implanted species ahead of the wear front would be possible.

Once again we are confronted with the fact that wear is an intensely complex mechanism and introducing ion implantation causes more interactions to be considered.

D. FATIGUE

One possibility as to why the wear improvements have been seen primarily on the rotating member in the crossed cylinder and cylinder-in-groove tests is that ion implantation improves fatigue life (4). It was suggested by Hirvonen et al. (5) that nitrogen implantation inhibits either the crack initiation or crack growth rate associated with wear.

It was suggested by Chakrabortty et al. (17) that the changes in cyclic behavior of metals is due to three possible factors: 1) surface alloying and subsequent lowering of stacking fault energy, 2) surface stresses caused by implanted ions and 3) implantation induced damage and substructure.

Fatigue cracks generally originate at the surface, unless pre-existing defects or complex stress states cause subsurface origins (28). This surface relationship is where ion inplantation has its greatest contribution. Jata and Starke (29) found that ion implantation can cause homogenization of surface slip, but that the surface defect structure, caused by the implantation alone, was not sufficient to cause that effect. They implanted copper with Cu^+ ions and found no improvement of fatigue properties, although dislocations and defect substructures were indeed present.

Herman (6) proposes that the products of ion implantation, i.e. $Fe_{16}N_2$ caused by nitrogen implantation, act to strengthen the matrix and make dislocation motion and consequently surface emerging slip, more homogeneous. Major slip inhomogenieties are thus reduced and an increase in fatigue life is the result.

Researchers at Georgia Tech (17) have used transmission electron microscopy and x-ray analysis to determine surface residual stresses and defect substructure as a result of ion implantation. They have shown that when Al^+ and Cr^+ are implanted into polycrystalline copper that an improvement in fatigue life is accompanied by residual surface compression. Implantation of B^+ on the other hand decreases the fatigue life and is accompanied by residual tension at the surface (30).

There has been a great deal of research performed on fatigue where ion implantation has been shown to improve fatigue properties of steels (31), titanium alloys (5) and copper (32). These results add to the versatility of ion implantation over other surface modification methods.

An aging phenomenon was observed by Hu et al. (33) on the fatigue properties of nitrogen implanted steels. Implantation of nitrogen into AISI 1018 steel caused a lifetime improvement of approximately 2.5 in fatigue. Samples that were implanted and aged, however, showed much more significant results. Samples that were aged for four months at room temperature showed an improvement of 100 in fatigue life. Another series of specimens were implanted and aged for six hours at 100°C and saw a factor of ten improvement over the unimplanted baseline. Herman (6) proposes that this effect may be due to segregation of nitrogen at dislocations, forming nitrides and thereby having a major effect on fatigue by slip homogenization.

III. EXPERIMENTAL PROCEDURE

A Falex Lubricant Testing Machine, shown in Figure 1, was used to evaluate the wear performance of implanted and unimplanted pins under lubricated conditions. It is documented in a thesis written by Meng at U.M.R. (34) that the setup shown with a spring load gage was not adequate for our tests. The mechanical gage was replaced with a loadcell connected to a 15.00 volt D.C. power supply and an AIM 65 microcomputer as shown in Figure 2. This setup has proved to be very reliable.

Each test uses standard Falex #10 pins made of AISI 3135 steel and two V-blocks made of AISI 1137 steel. The pins are polished on a drill press using sandpaper in graduated order of 240, 320, 400, and 600 grit. This is to get consistency from test to test. Each set of V-blocks is polished using 400 grit sandpaper.

The pin is weighed prior to testing and is loaded in a configuration shown in Figures 3 and 4 while submerged in a lubricant. As the pin wears, the pin/block dimension decreases, thus the decreasing the load. Our tests are run with a 200 pound applied load and are stopped when there has been a 10% drop in load or a time limit of 240 minutes has been met.

The U.M.R. accelerator, affectionately known as

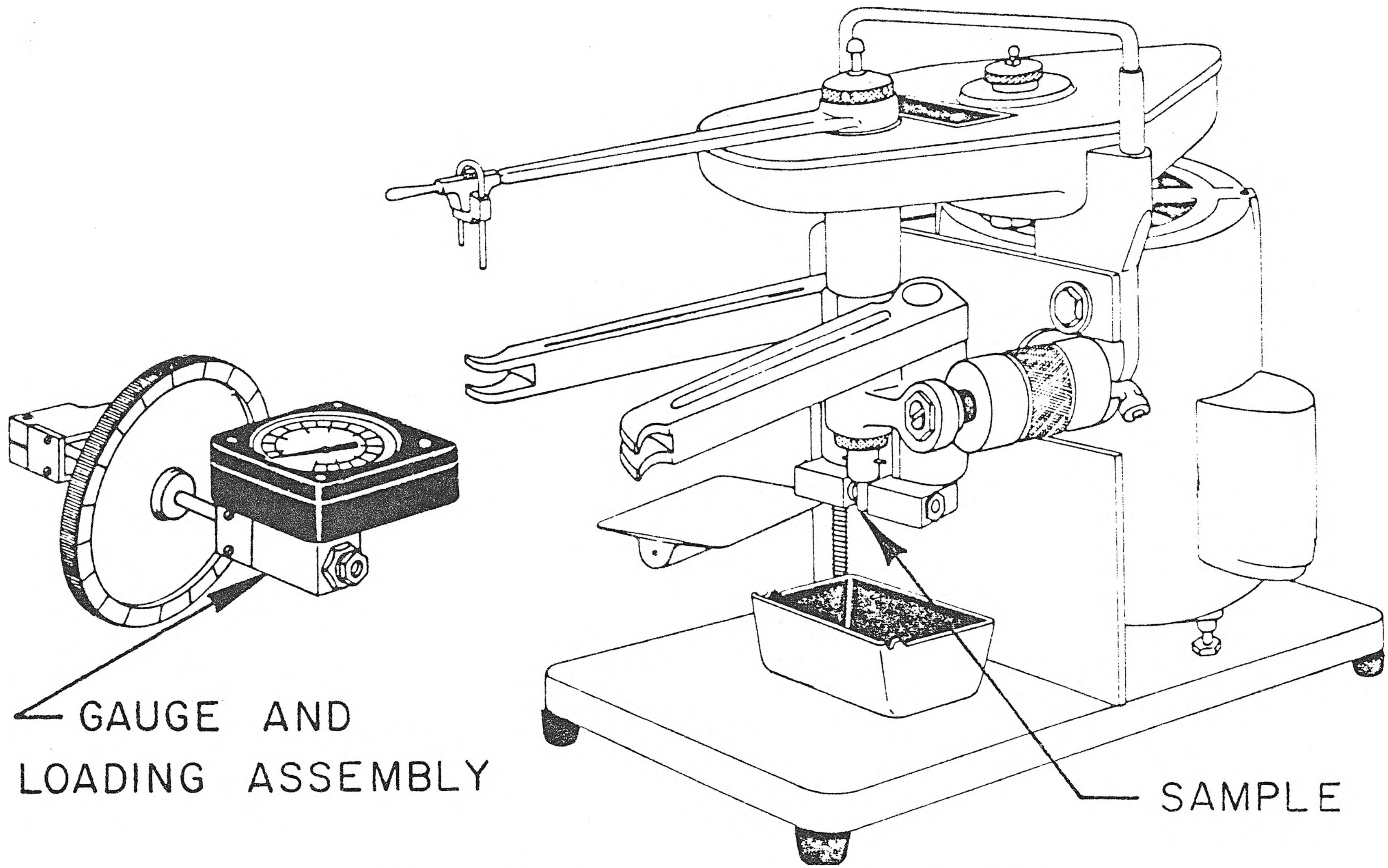


Figure 1. Falex Lubricant Testing Machine.

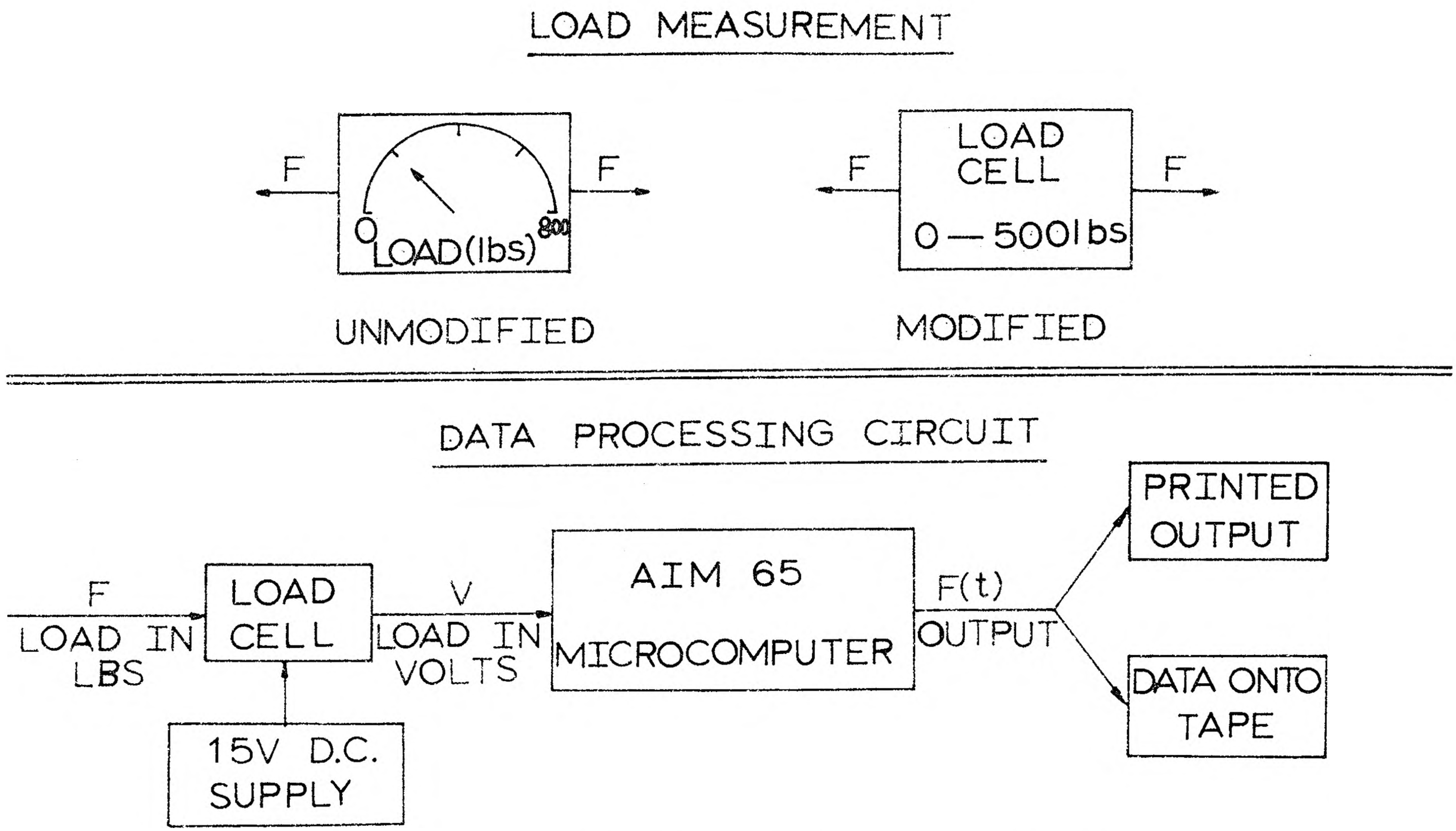


Figure 2. Modified load measurement and data processing flow chart.

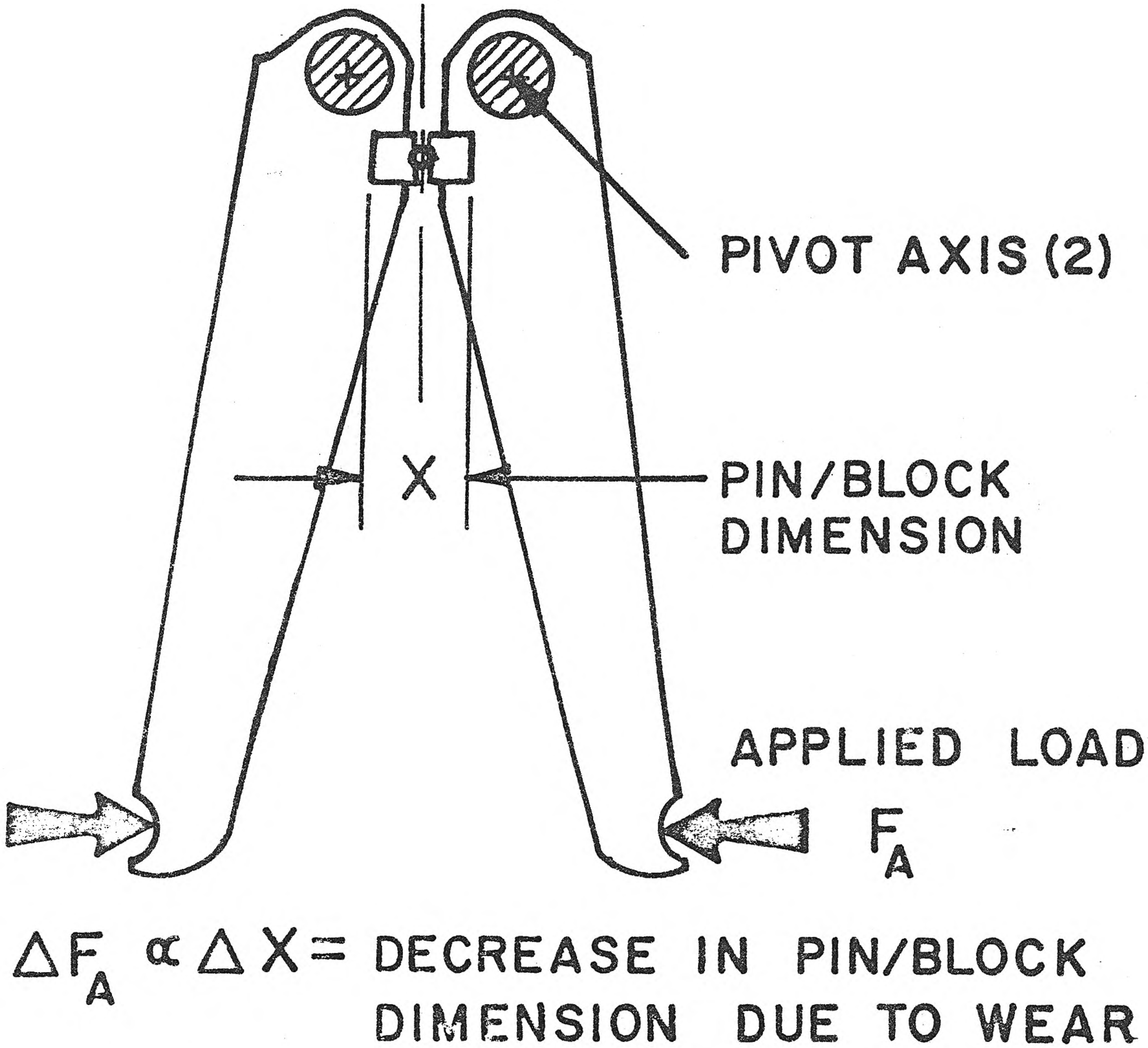


Figure 3. Loading configuration on the Falex Machine.

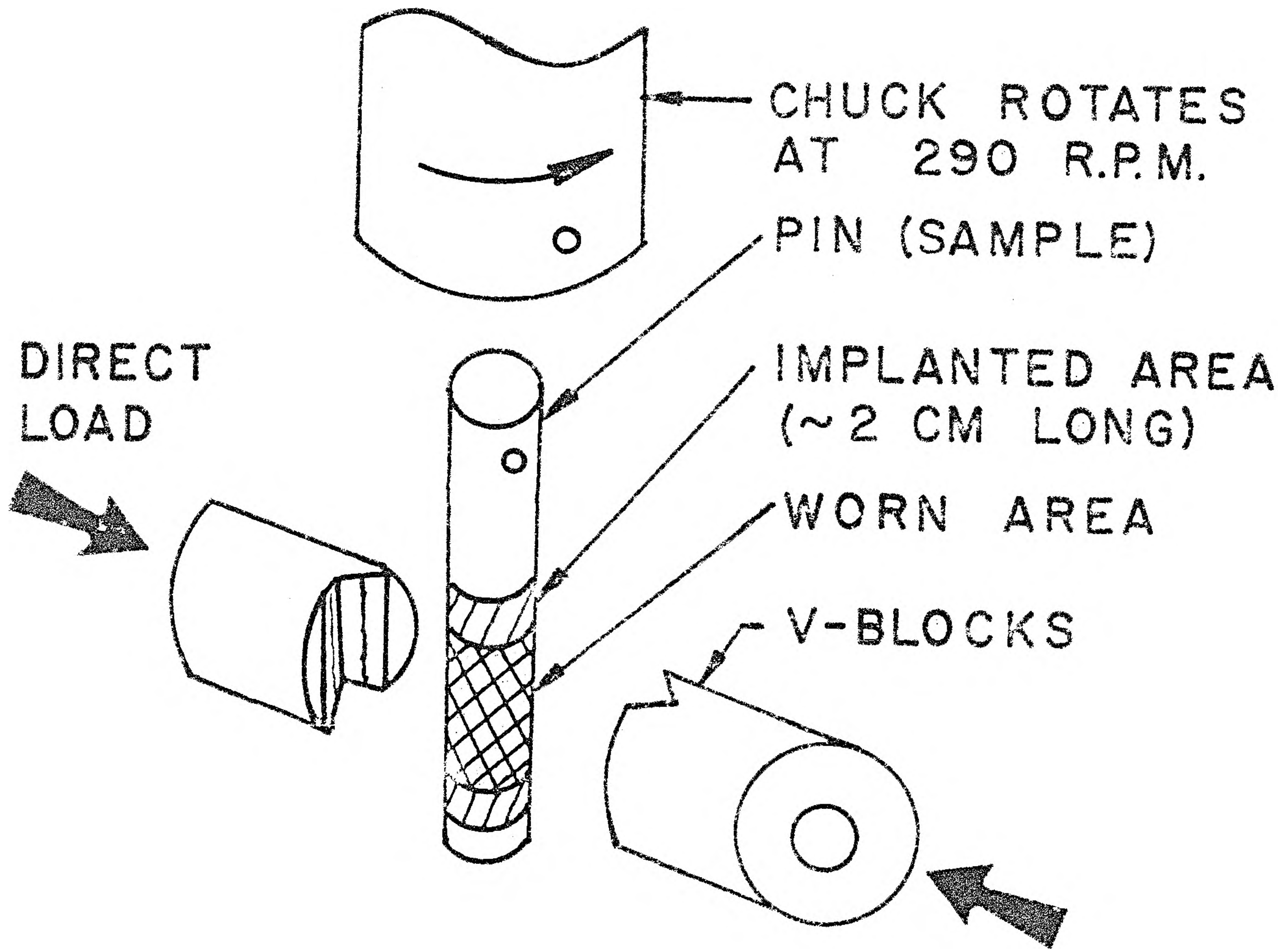


Figure 4. Details of test geometry on the Falex Machine.

Harvey, is a non-commercial accellerator specially constructed for use in research studies. It is a Cockcroft-Walton type accelerator capable of implanting ions with energies of 50 keV to 200 keV. Beam currents are typically 15 microamperes or less in a vacuum chamber held at 5 x 10^{-6} torr or less during implantation. The beam current is kept low so that heating of the sample does not influence the sample's mechanical properties. The samples are rotated in a rastered beam so that a uniform implantation dosage is received all around the pin.

Wear rates are determined by merely dividing the total mass lost by the pin by the time of test duration. Mass loss is determined by simply weighing each sample before and after the test. Each sample is weighed three times and an average value is used.

Scanning electron microscopy, Auger surface analysis and electron spectroscopy for surface analysis (ESCA) were performed at the Graduate Center for Materials Research.

IV. EXPERIMENTAL RESULTS

There has been an extensive amount of research into the effects of different implanted species on the surface mechanical properties of steels and other alloys, but little specifically devoted to an in-depth study of why. It was decided, therefore to begin an investigation into the wear mechanism involved in the wear testing of implanted and unimplanted pins using the Falex Lubricant Tester.

Figure 5 is a curve showing the load drop that occurs in the cylinder-in-groove wear test, showing that the drop in load, which corresponds to the wear rate, is more severe in the unimplanted case. The instrumentation provides a direct printout of the load vs. time in each test. There is a portion of the data, in the first few minutes, where there is considerable fluctuation of the load. This is interpreted to mean that the first few minutes of the test are the most severe and that the initial wear mechanism is set up in this time interval.

An unimplanted pin was selected and a series of tests was performed on it. Each test ran for a total of 30 minutes with new blocks being used in each test. The wear rates for each test in the series are shown on Figure 6. The figure shows that the incremental wear rates are essentially constant throughout the duration

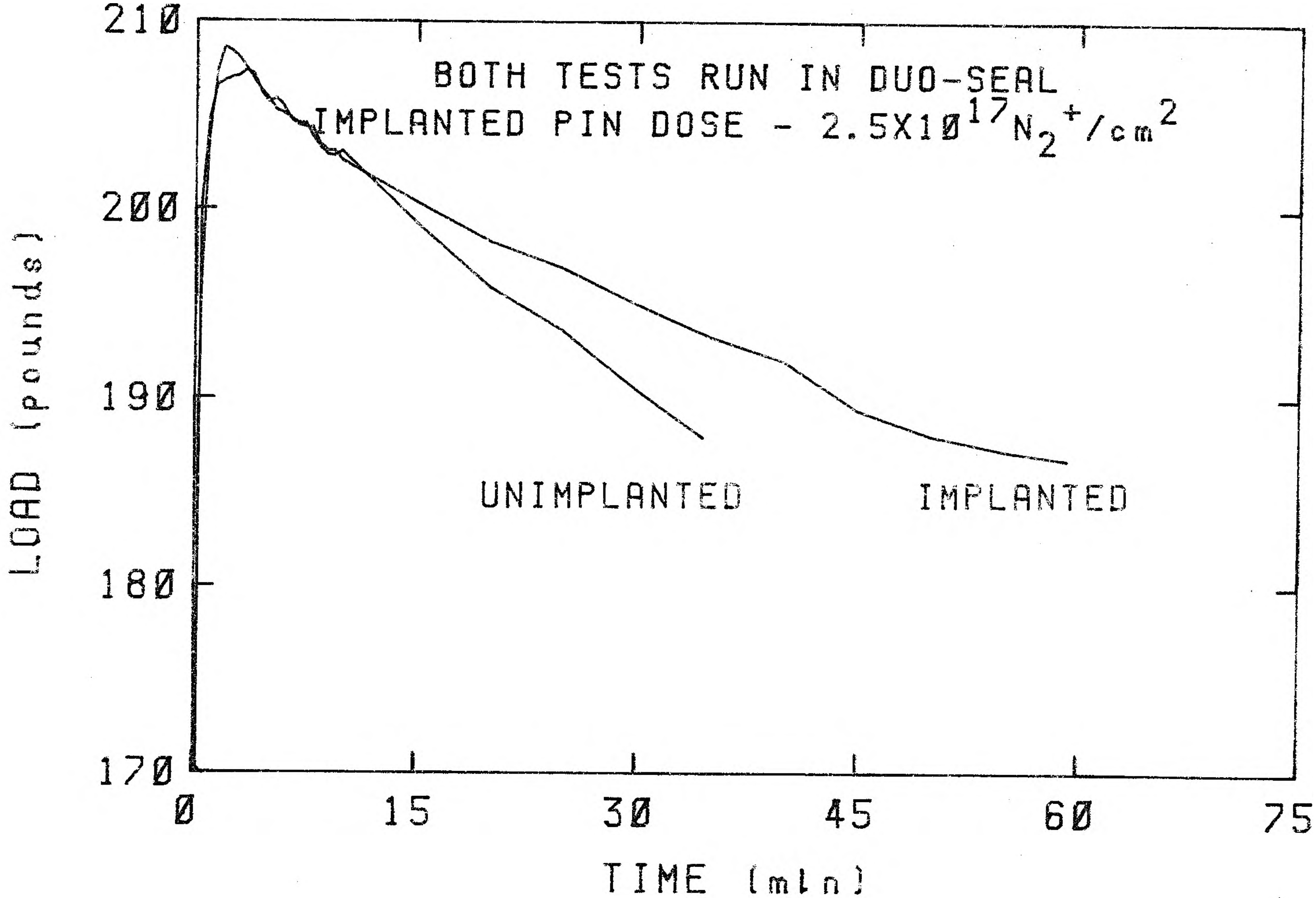


Figure 5. Typical load drop versus time curves

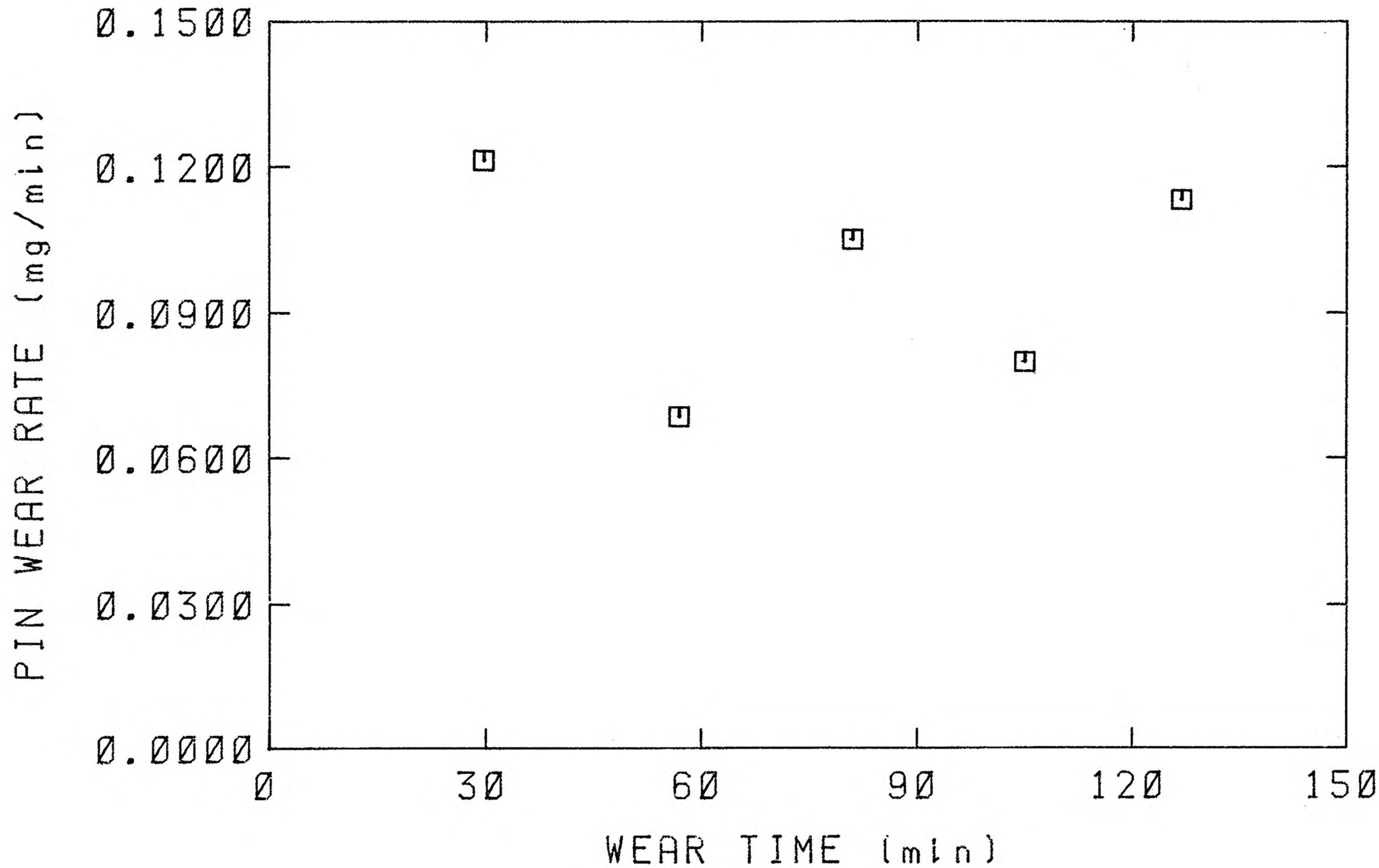


Figure 6. Unimplanted pin wear rate versus time

of the tests on unimplanted steel pins.

The same series of tests was performed on an implanted pin. The pin was implanted with the standard dose implant of 2.5 x 10^{17} N_2^+ / cm^2 at 180 keV. The blocks were unimplanted. Figure 7 shows that for this implanted pin the incremental wear rates at the beginning of the test were greater than the values obtained later in the test. This again indicated that there was some phenomenon that occurred in the initial stages of the wear test that needed to be explored.

Several more implanted pins were tested at various time intervals and the wear rate of the pin was plotted versus time on Figure 8. This clearly shows that there is an initial period where the wear rate relates closer to the unimplanted case than to the implanted case. This may be explained by the fact that the greatest concentration of nitrogen occurs almost 1000 angstroms below the surface. We, therefore, theorized that we might easily show the depth dependance of the wear rate by implanting some pins with atomic nitrogen at the same energy level as the pins implanted with molecular nitrogen. This would effectively implant the nitrogen to a depth twice that of the previous tests. While the data is a little scattered, it shows a trend that the wear rate versus time curve definitely shifts upwards. This would indicate that you get shallower modification in surface properties if your implanted species is close

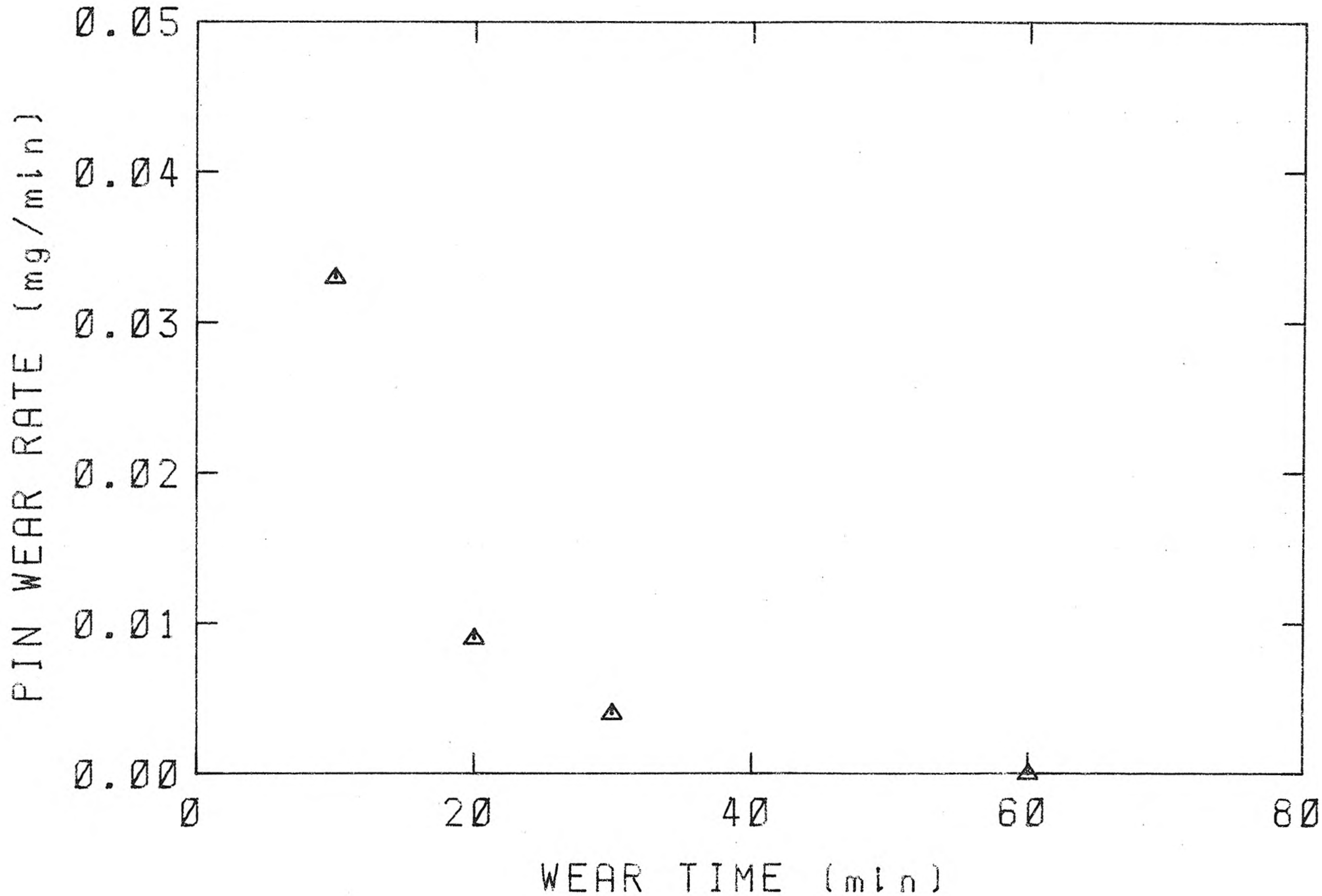


Figure 7. Implanted pin wear rate versus time, K-441-P

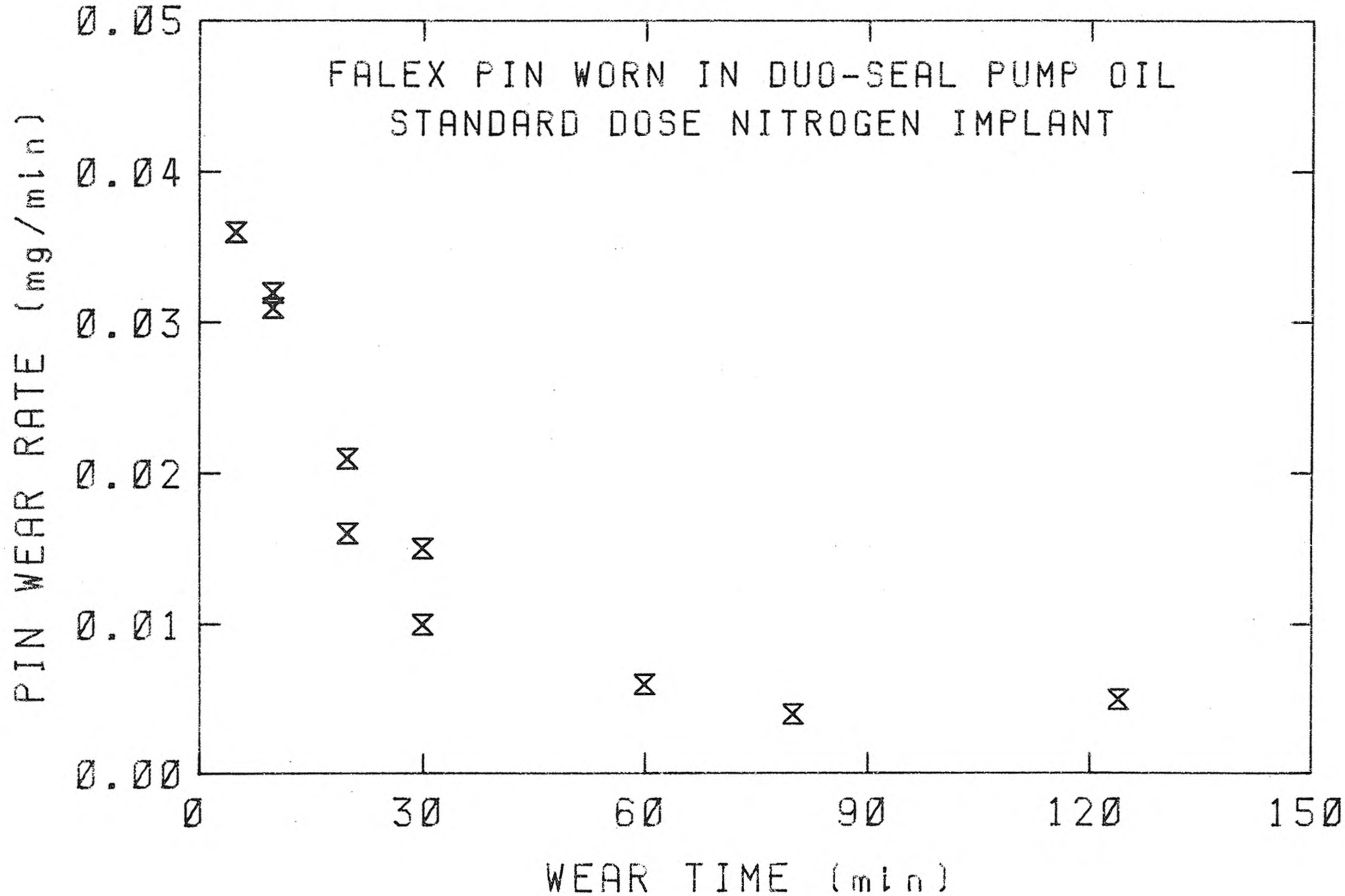


Figure 8. Implanted pin wear rate versus time

to the surface.

Auger surface analysis and scanning electron microscopy were performed in order to understand what happens at the surface on a microscopic scale. Figure 9 shows an Auger depth profile curve of a typical nitrogen-implanted steel pin. One of the more widely accepted explanations for why nitrogen implantation improves wear resistance is that the nitrogen forms a hard surface layer to resist wear and that the nitrogen diffuses ahead of the wear front. Figures 10 and 11 show that in our tests, conducted in both peanut oil and DuoSeal pump oil, that the level of nitrogen diminished rapidly in the wear tests and some slight broadening of the curves occurred indicating that some minor diffusion of the nitrogen occurred. Figures 12 and 13 show the peak intensities of the nitrogen Auger peak plotted versus wear test run time. These curves show that the level of nitrogen decreases rather uniformly until it is undetectable after approximately three hours.

One of the more visible differences between an implanted and unimplanted pin after the wear test is their appearance. The implanted pins have a smooth burnished appearance where the unimplanted pins are rough. This burnishing has been referred to in references as an oxide layer. Further Auger analysis, therefore, should help us to understand it better.

In the oxidative theory of wear, it is proposed

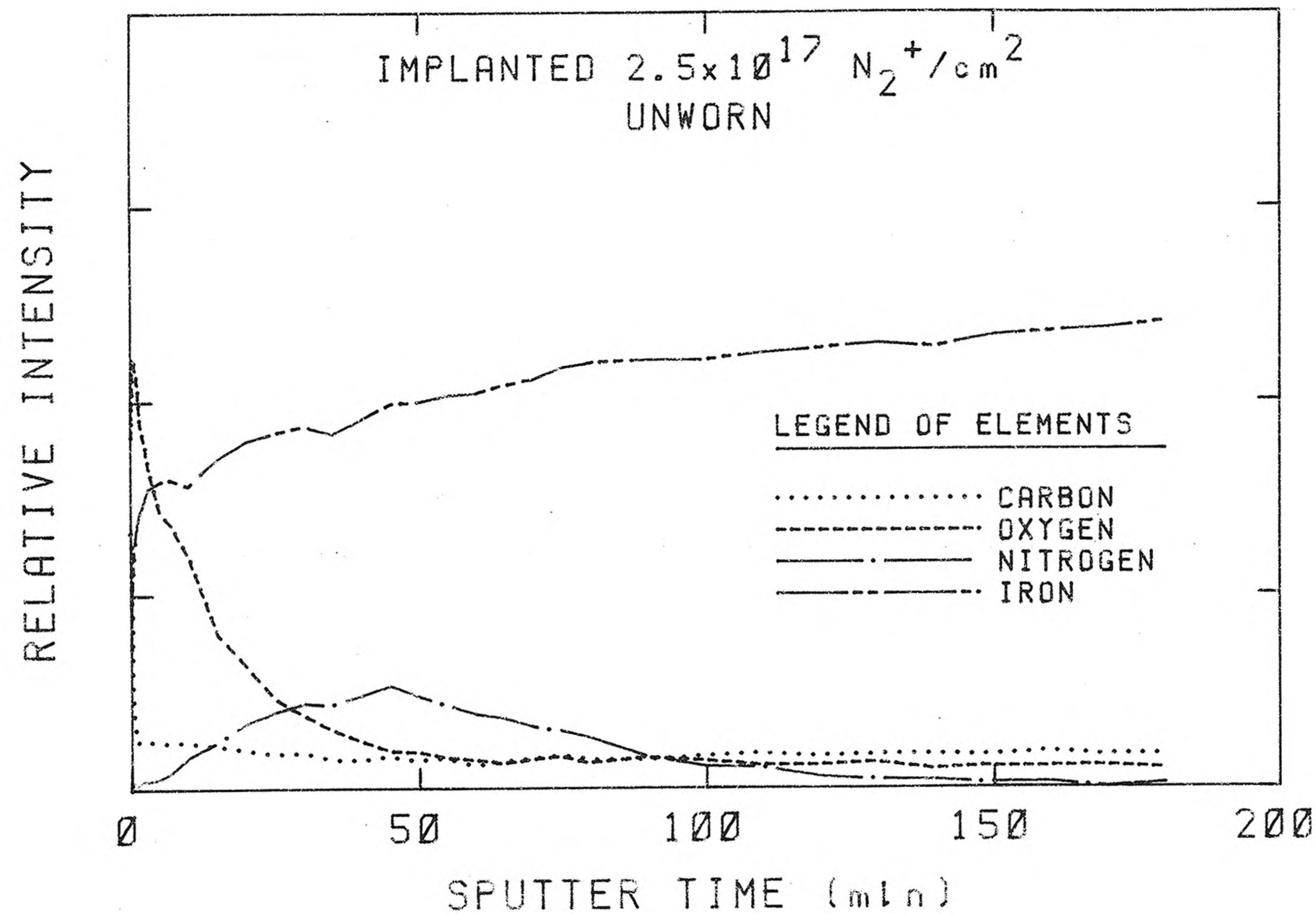


Figure 9. Auger depth profile of unworn, nitrogen-implanted pin

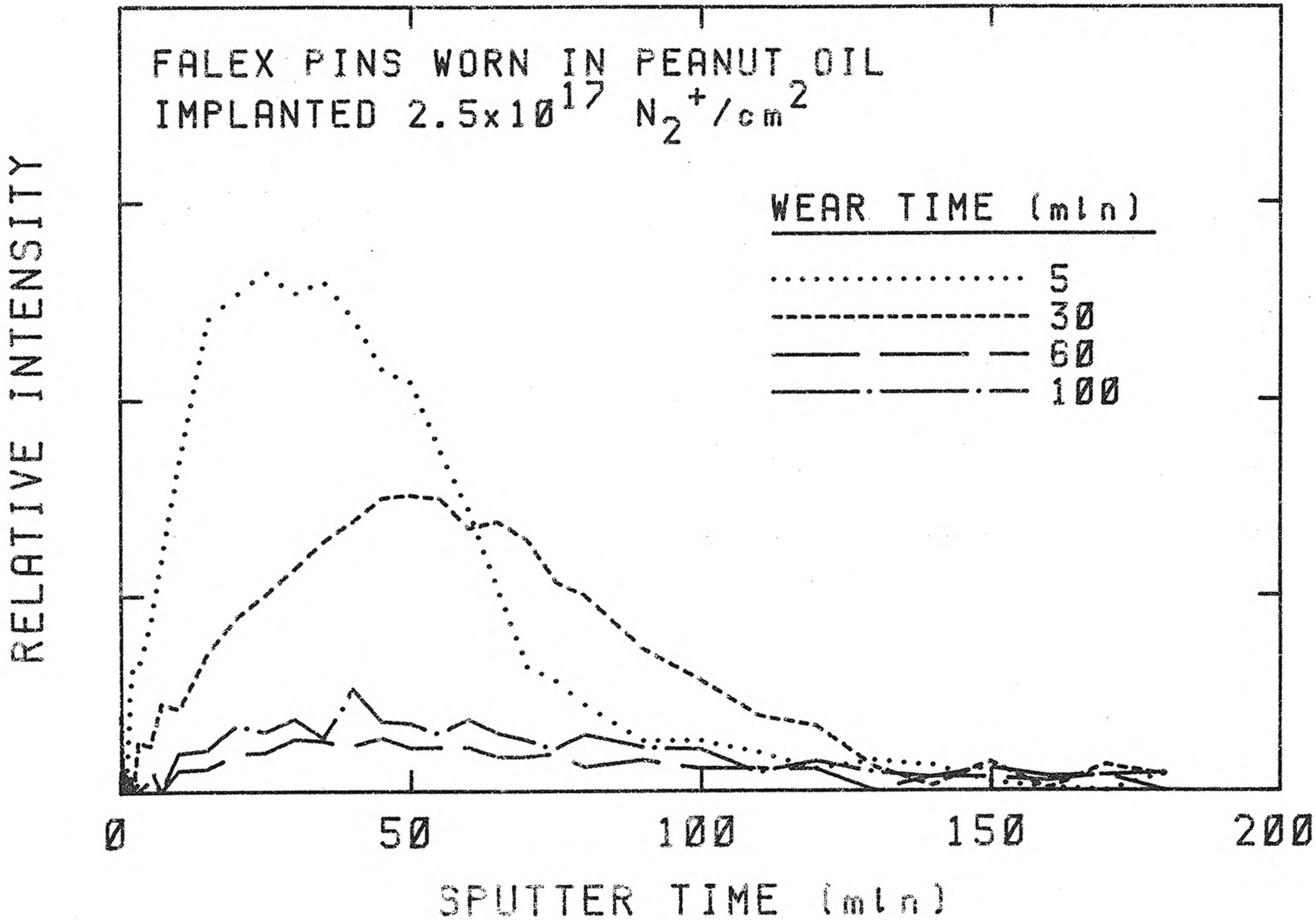


Figure 10. Nitrogen depth profiles versus wear time for implanted pins run in peanut oil

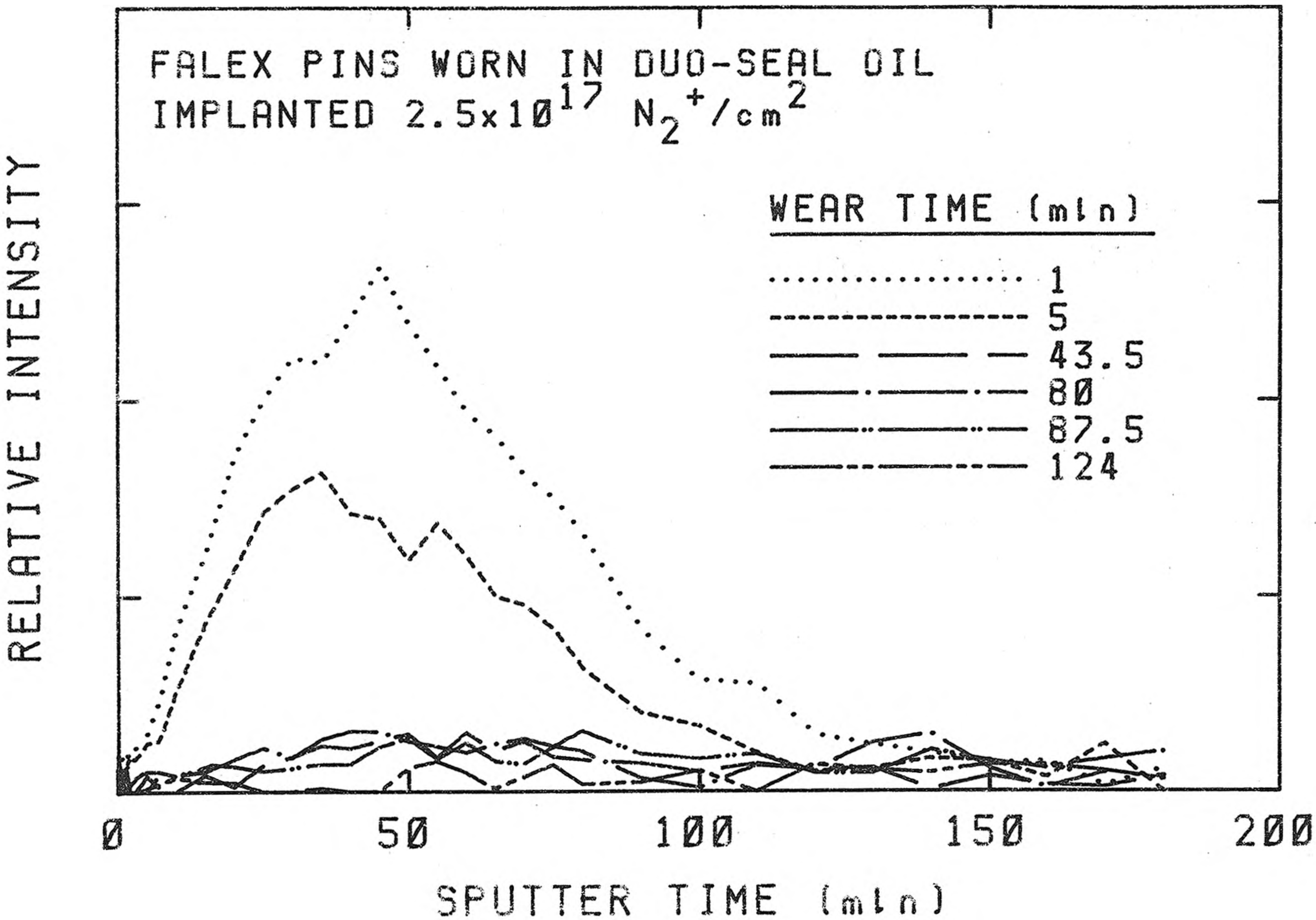


Figure 11. Nitrogen depth profiles versus wear time for implanted pins run in DuoSeal

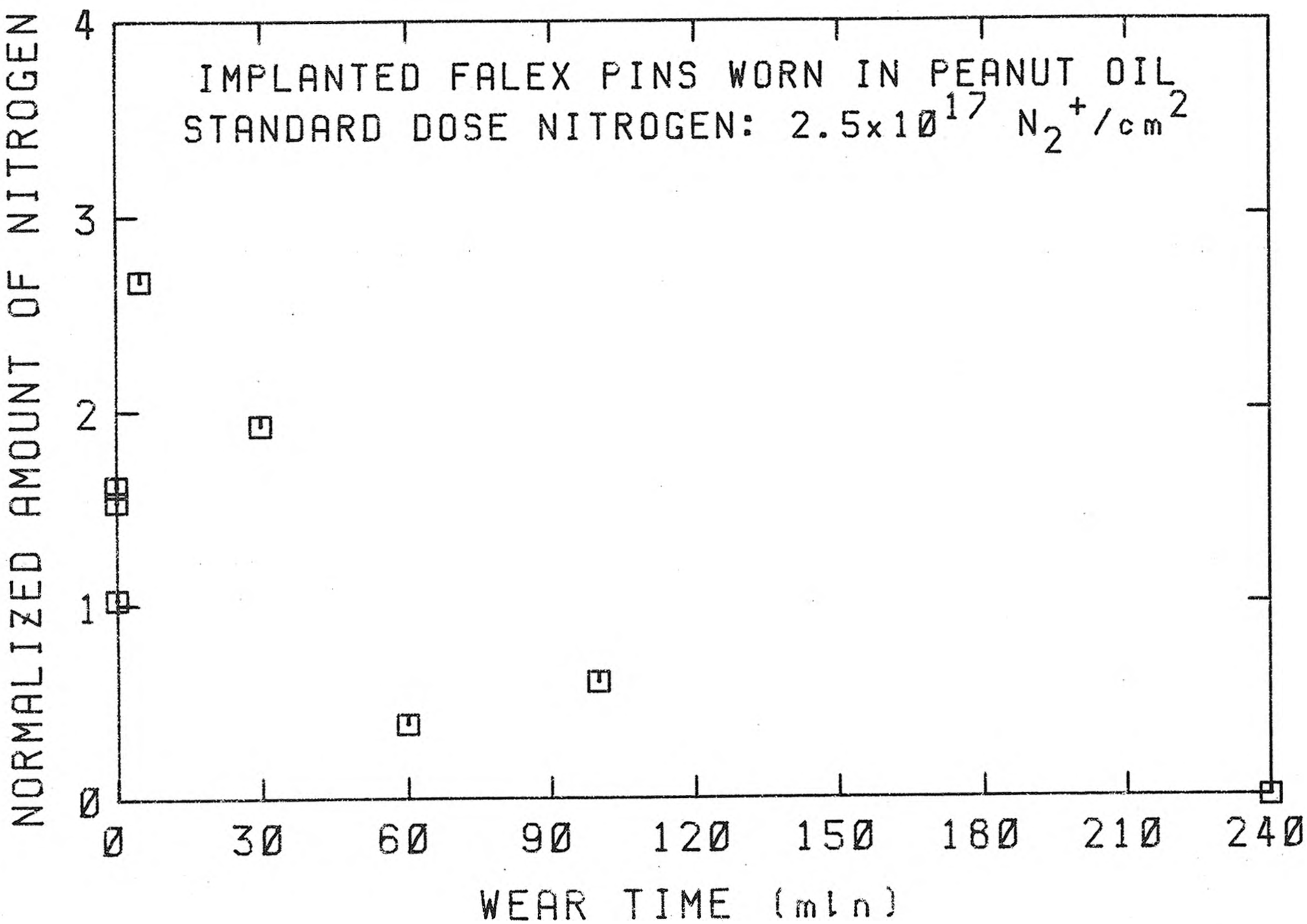


Figure 12. Implanted nitrogen remaining after wear test for implanted pins run in peanut oil

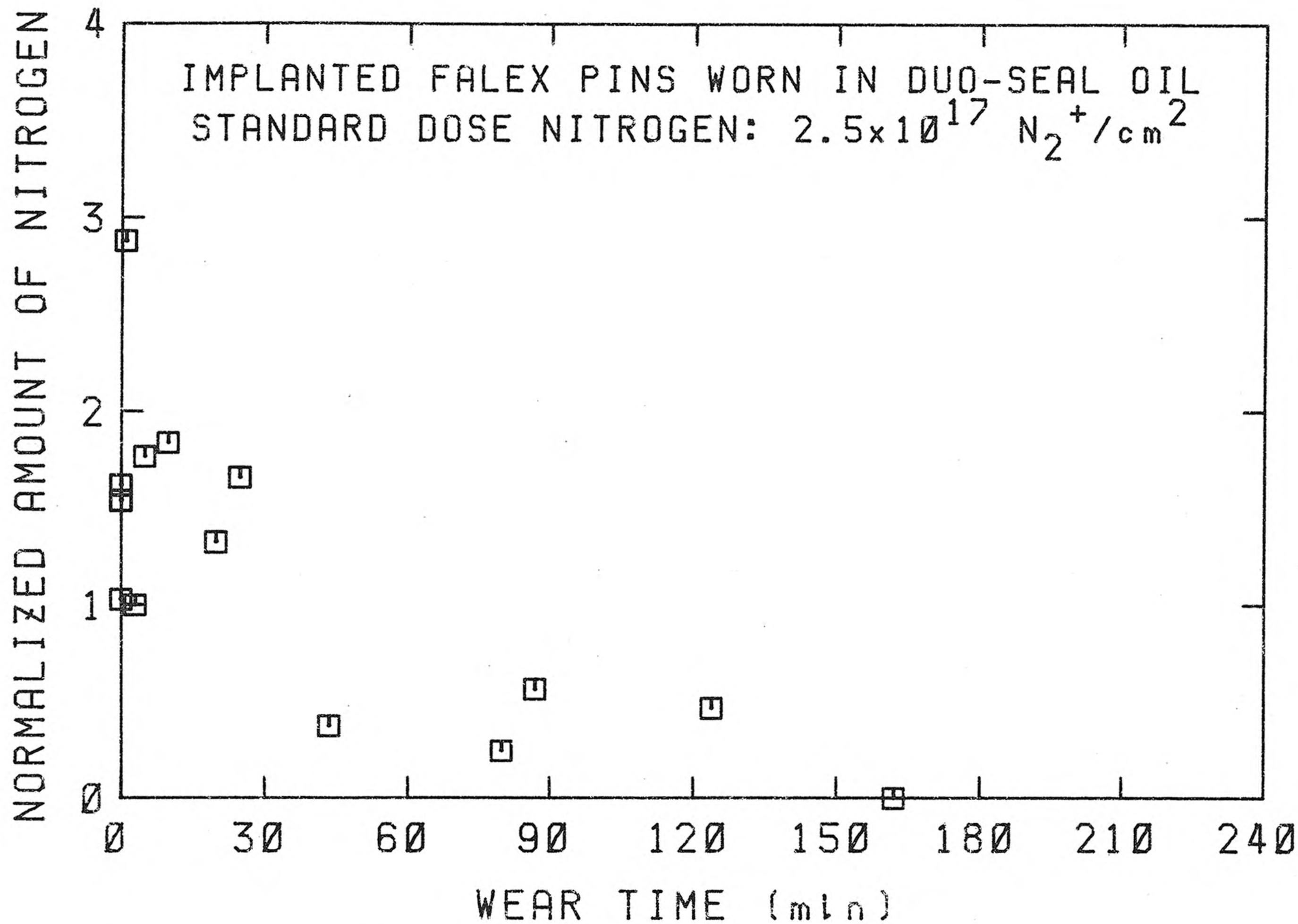


Figure 13. Implanted nitrogen remaining after wear test for implanted pins run in DuoSeal

that the surfaces heat, oxidize, and the oxide is removed. The formation of oxide is then the controlling factor in the wear rate. Figure 14 shows how the oxygen profile increases in a series of tests with unimplanted pins. The peak intensity is relatively constant, indicating that an iron oxide of fairly constant chemistry is formed. Figure 15 shows similar results for implanted pins run in DuoSeal pump oil. They show that the oxygen profiles do not grow as fast as in the unimplanted case. This would give strong evidence to the oxidative theory of wear and the effect of reduction of oxidation rates through ion implantation. Figures 16 and 17 show Auger curves of pins that were implanted with carbon to show that the same effect can be seen with a different implanted species. The worn pin shows almost none of the implanted carbon remaining but a substantial increase in the oxygen profile.

Scanning electron microscopy of the samples run for Auger analysis helps to further understand the wear mechanism. Figure 18 shows what the surface of a Falex pin looks like after it has been prepared for a wear test. There are some very shallow and irregular grooves present that are caused by the polishing procedure. Figures 19 through 25 show unimplanted pins worn for periods of 1, 2, 3, 5, 10, 20 and 28 minutes. They show that a gouging mechanism occurs almost immediately and that in two minutes there is nothing left of the

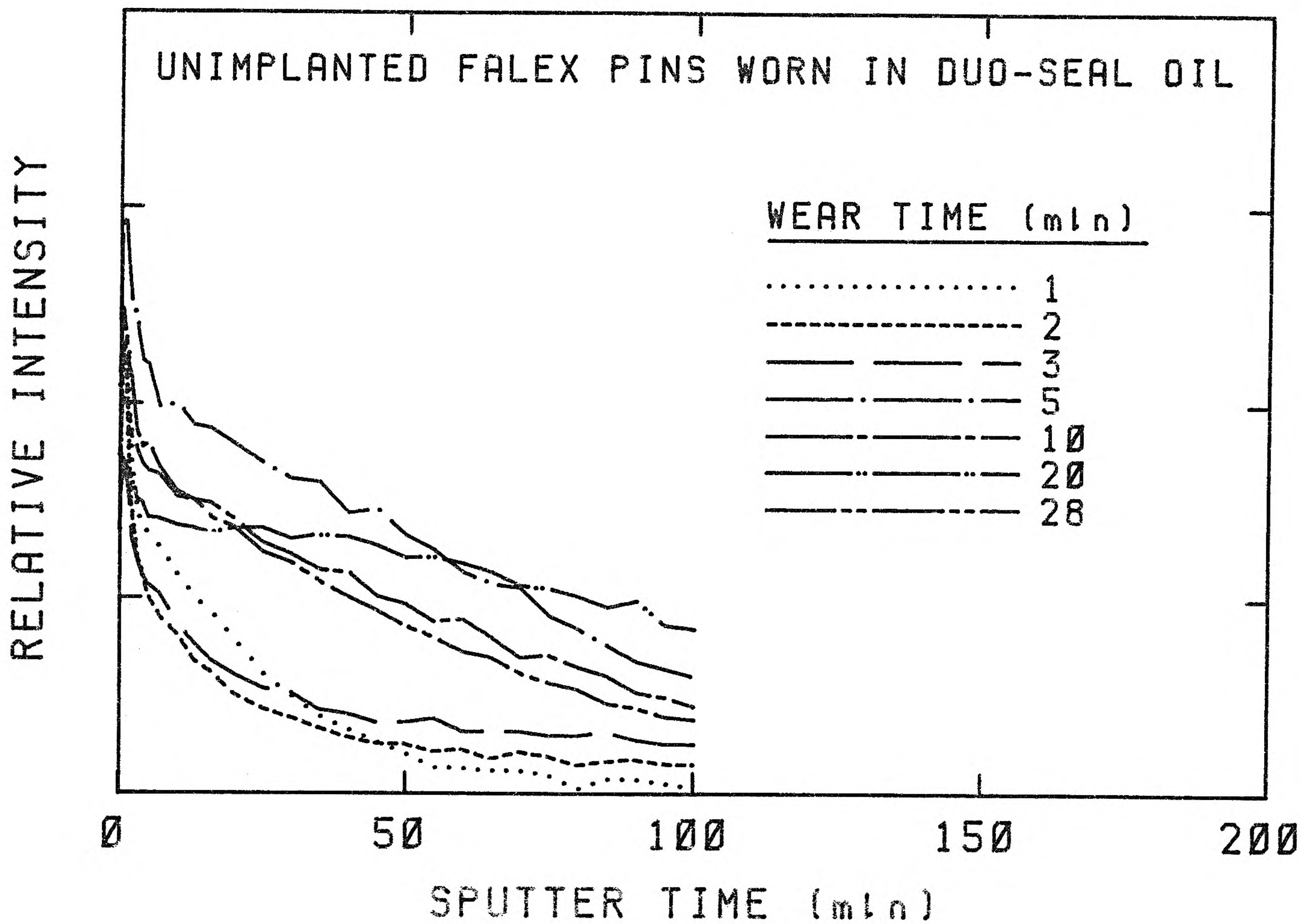


Figure 14. Oxygen depth profiles versus wear time for unimplanted pins run in DuoSeal

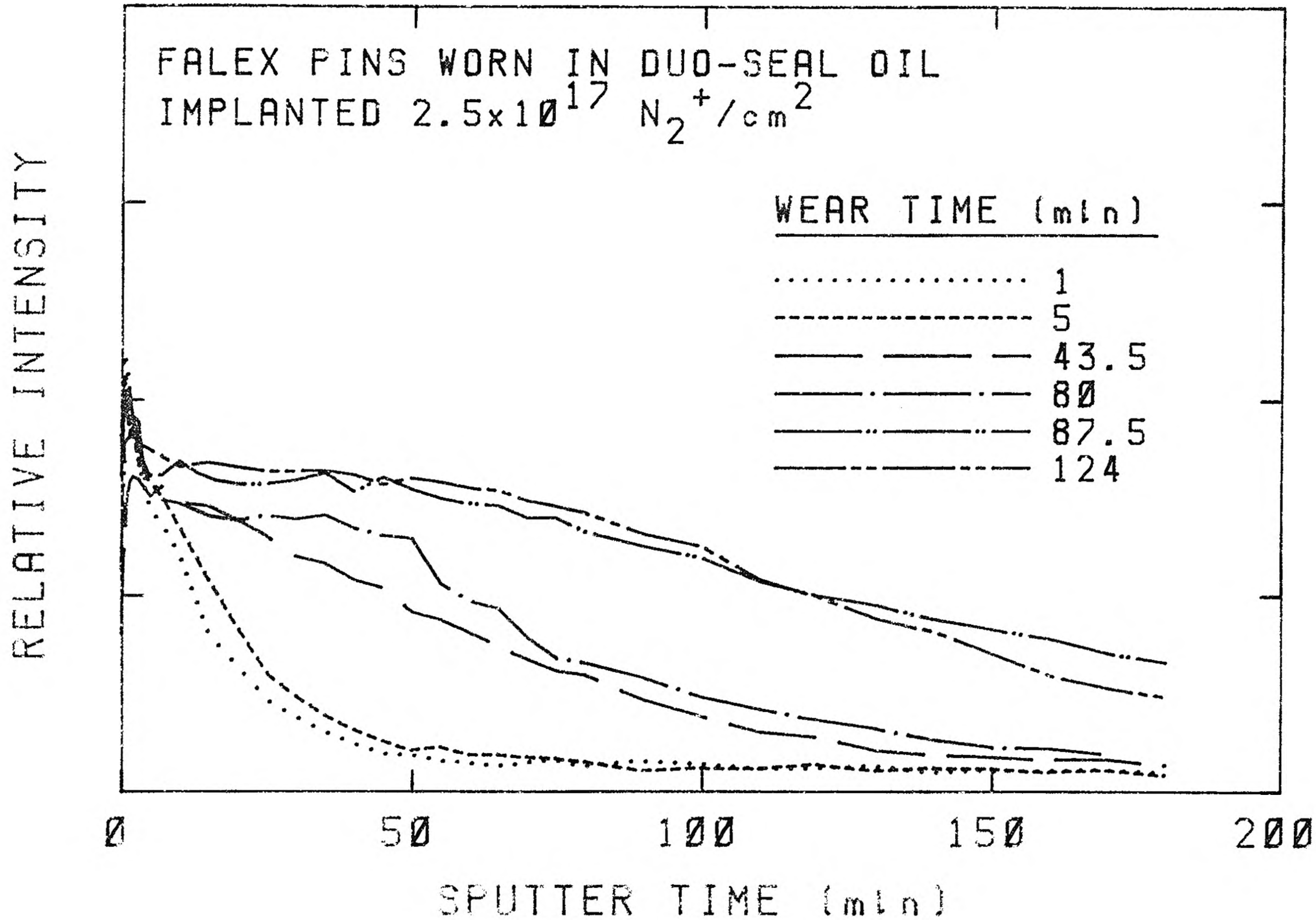


Figure 15. Oxygen depth profiles versus wear time for implanted pins run in DuoSeal

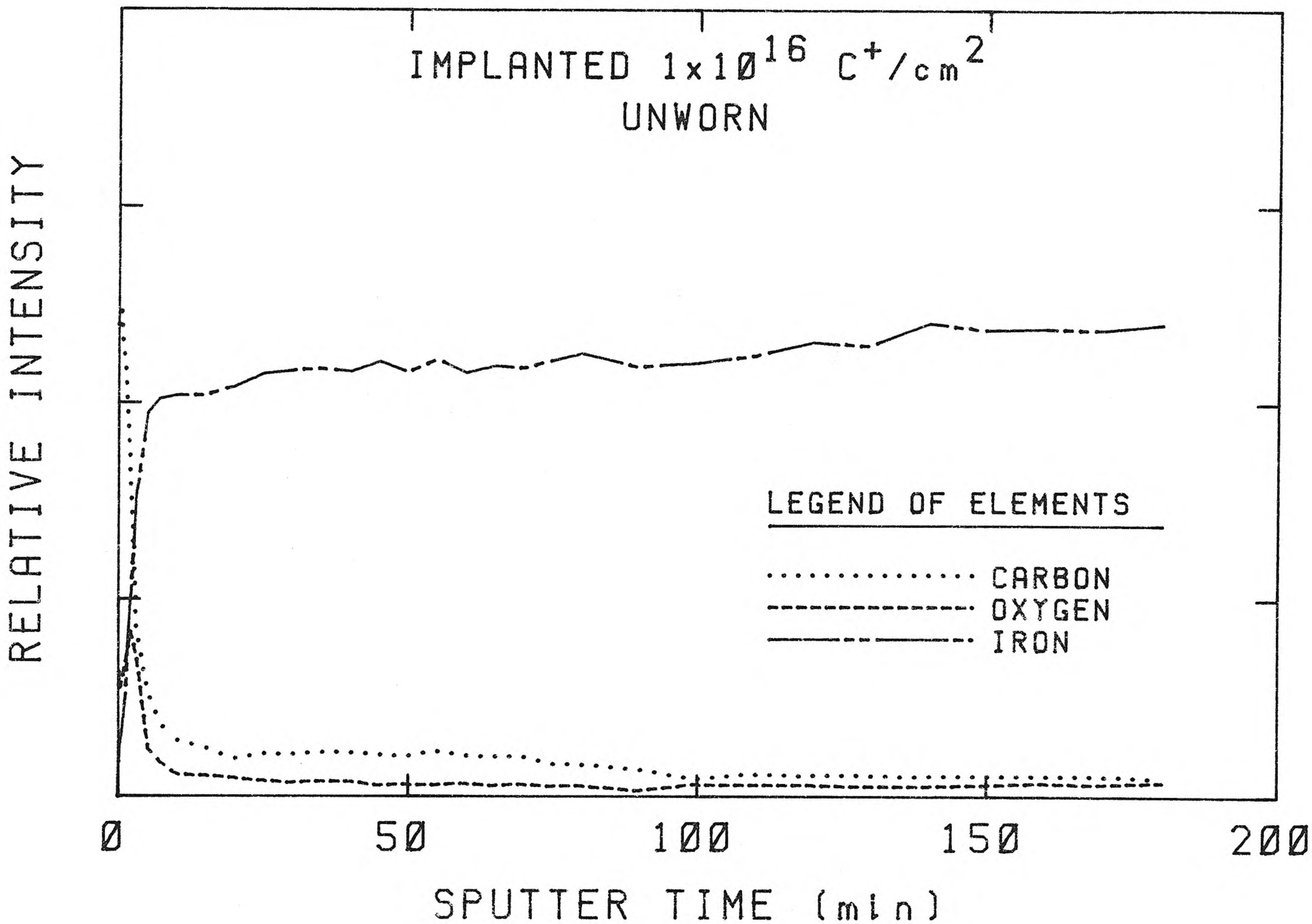


Figure 16. Auger depth profile of unworn, carbon-implanted pin

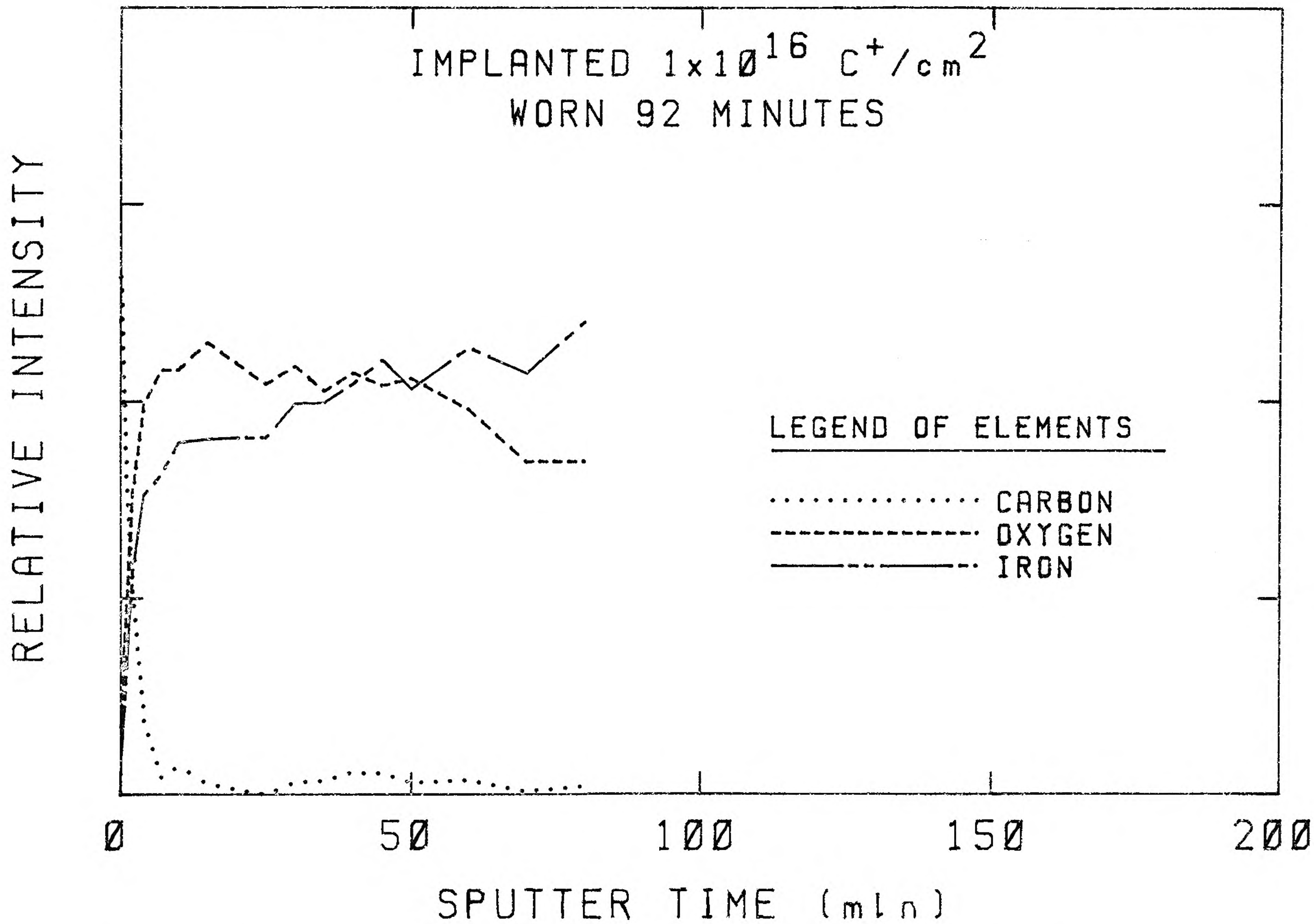
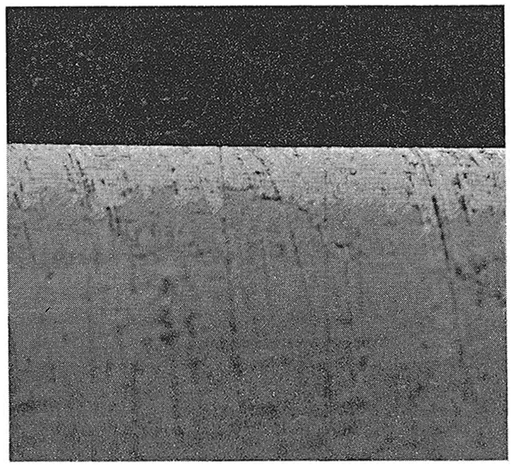


Figure 17. Auger depth profile of worn, carbon-implanted pin



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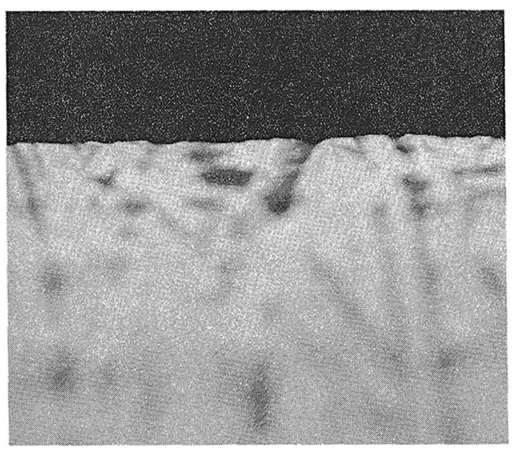

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Figure 18. Scanning electron photomicrographs
of Falex pin - as-polished

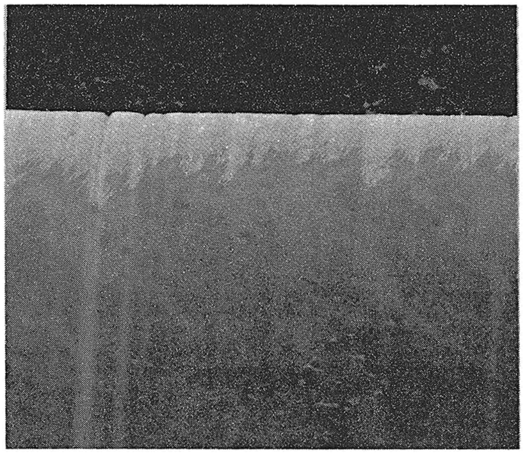

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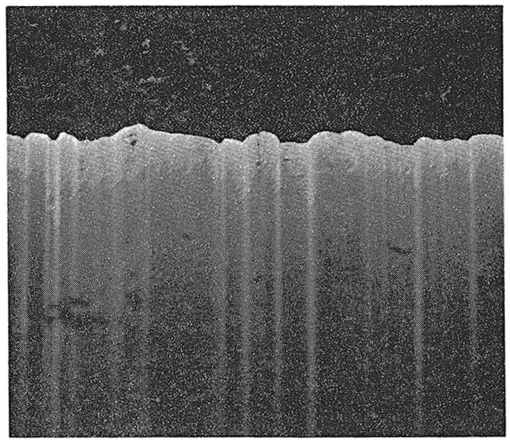
Figure 19. Scanning electron photomicrographs of unimplanted Falex pin - worn one minute



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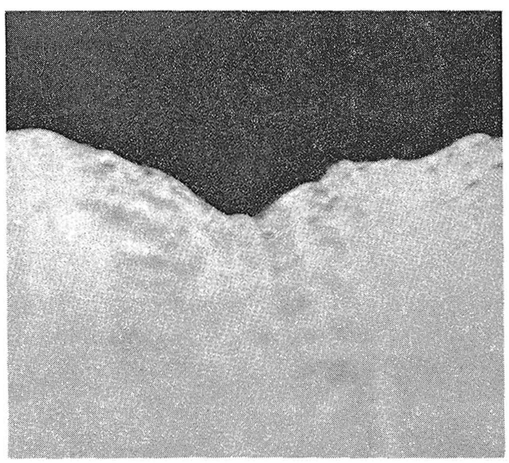

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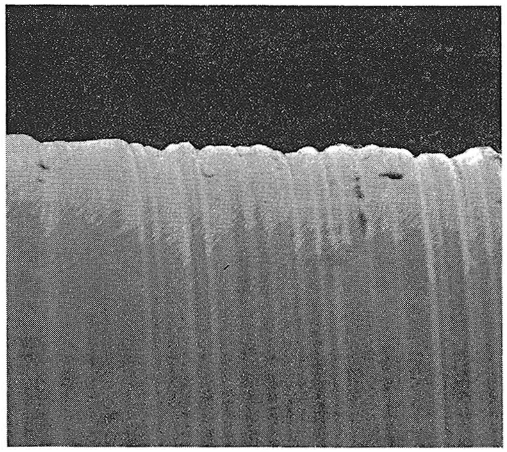
Figure 20. Scanning electron photomicrographs of unimplanted Falex pin - worn two minutes



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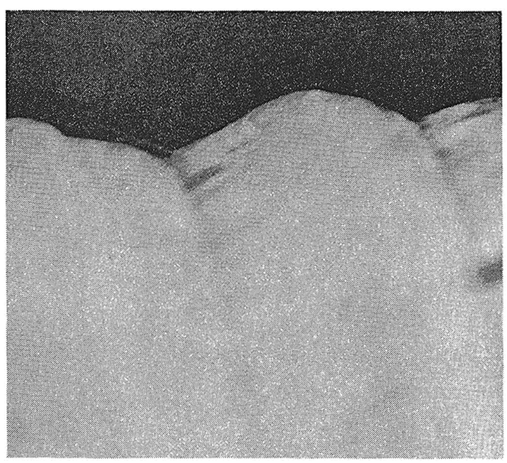

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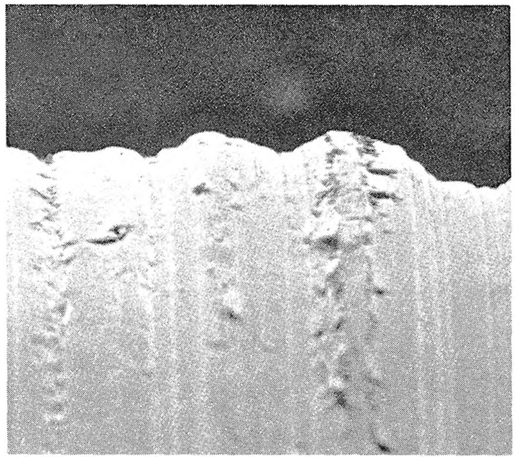
Figure 21. Scanning electron photomicrographs of unimplanted Falex pin - worn three minutes.



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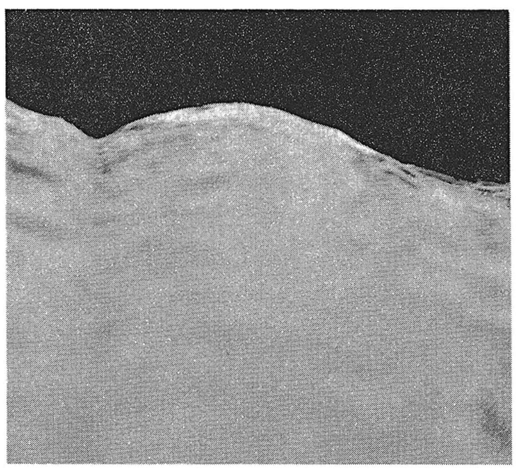

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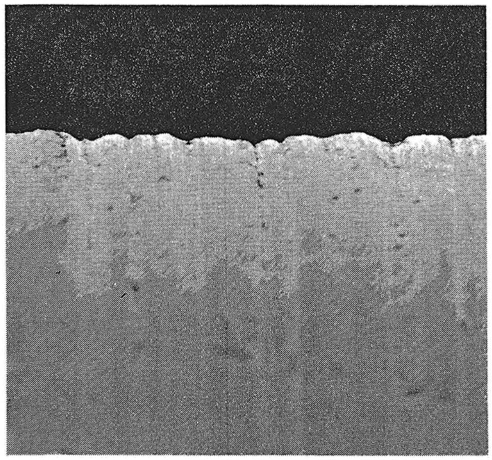
Figure 22. Scanning electron photomicrographs of unimplanted Falex pin - worn five minutes



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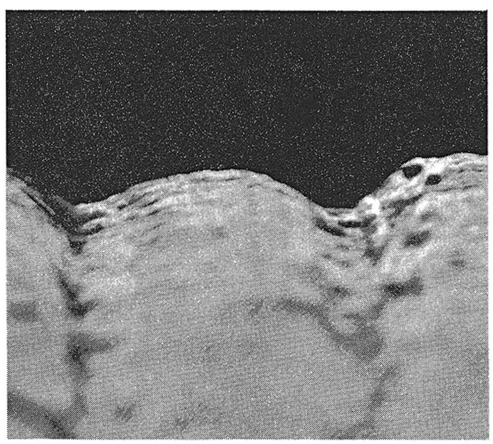

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Figure 23. Scanning electron photomicrographs of unimplanted Falex pin - worn ten minutes

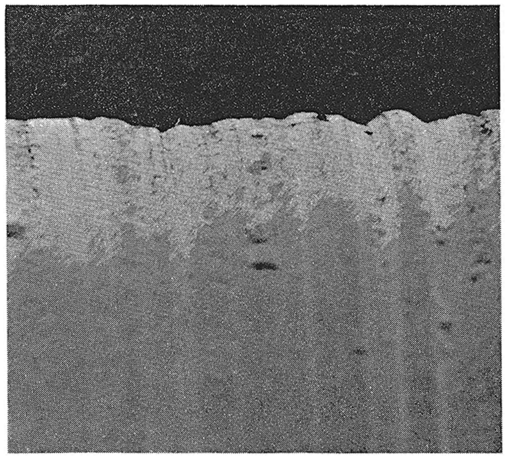

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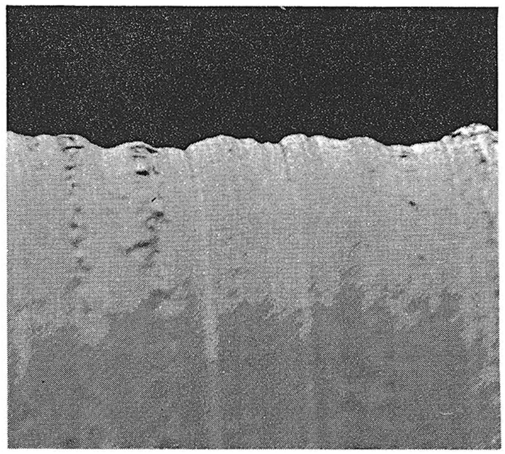
Figure 24. Scanning electron photomicrographs of unimplanted Falex pin - worn twenty minutes



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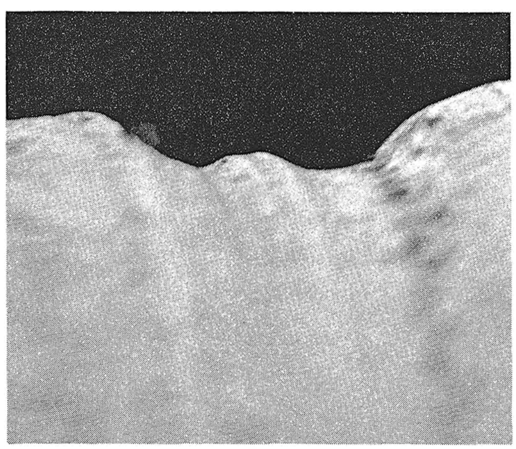

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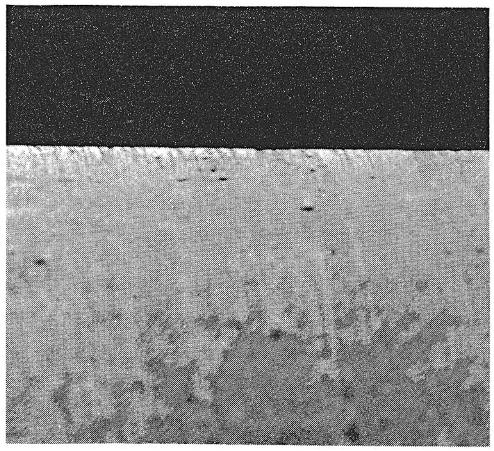
Figure 25. Scanning electron photomicrographs of unimplanted Falex pin - worn twenty-eight minutes

original surface. The series of S.E.M. photographs in Figures 26 through 31 show that in the tests of an implanted pin on unimplanted blocks there is virtually no visible severe wear occurring. Figures 32 through 34 show a better view of the apparent smoothing that occurs in the implanted cases, where small imperfections are healed on the surface in the early stages of wear and some more severe wear after longer wear times, but nothing compared to the unimplanted cases.

Figures 35 and 36 show a comparison of the blocks worn against the pins in the 5 and 20 minute runs. There is severe adhesion occurring in the unimplanted cases but there is no evidence of adhesive wear in the implanted case. The mechanism appears to have changed to mild abrasion.

The prior research at U.M.R. had all been conducted with peanut oil as the lubricant. The justification for this decision was that peanut oil was readily available and did not contain any additives. However, during previous summer months there had occurred a phenomenon referred to in our group as the "stuck pin" mode. What occurred was that a test could be run in peanut oil and an unimplanted Falex pin would run for over four hours with very little load drop and yield a wear rate very near that of an implanted pin.

This caused us to inquire into the makeup of the peanut oil lubricant that we were using for our



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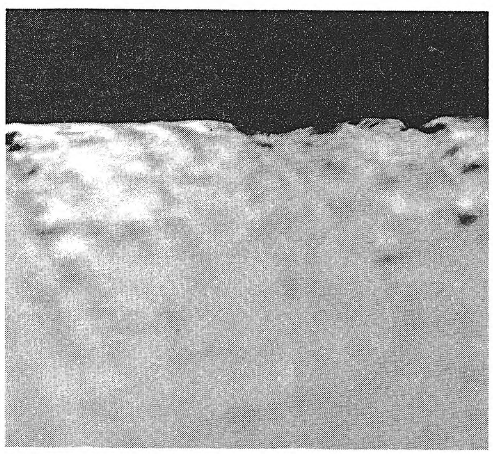

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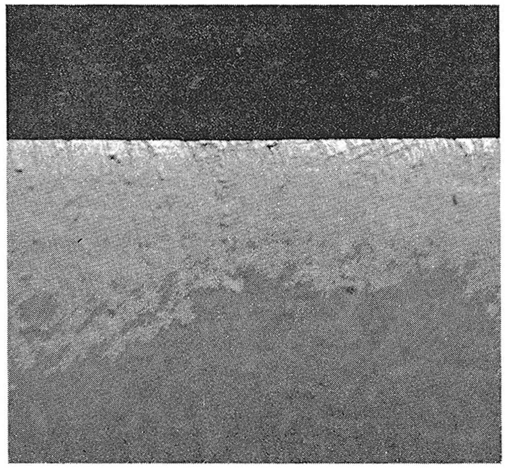
Figure 26. Scanning electron photomicrographs of implanted Falex pin - worn one minute



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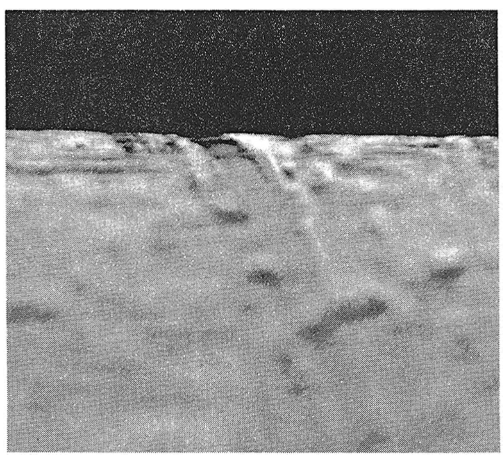

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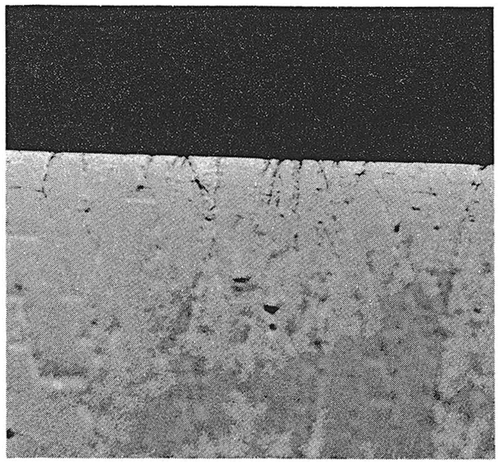
Figure 27. Scanning electron photomicrographs of implanted Falex pin - worn two minutes



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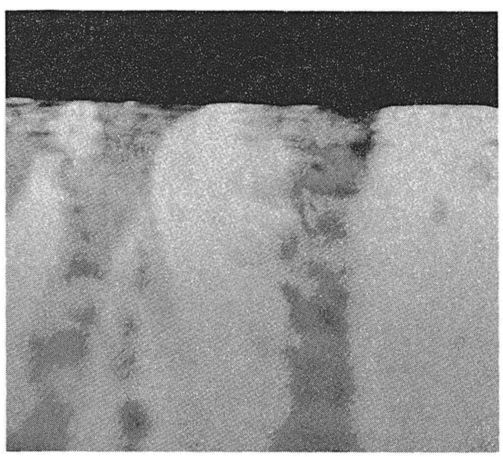

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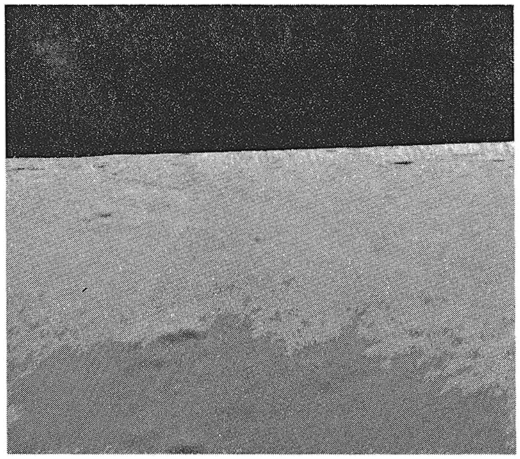
Figure 28. Scanning electron photomicrographs of implanted Falex pin - worn five minutes



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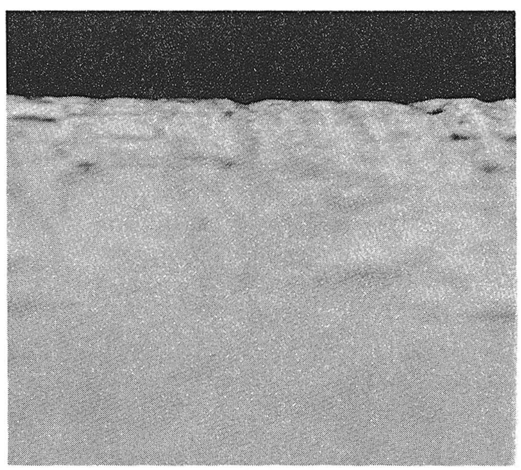

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Figure 29. Scanning electron photomicrographs of implanted Falex pin - worn twenty-five minutes

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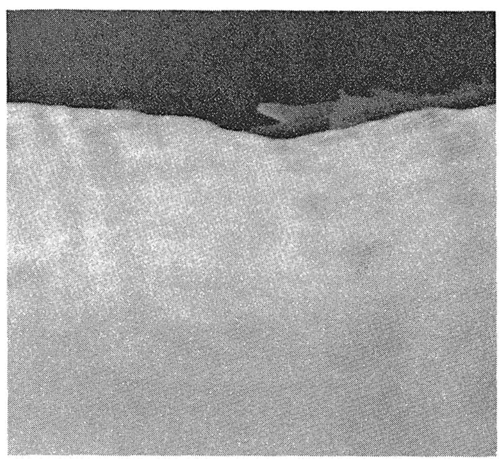

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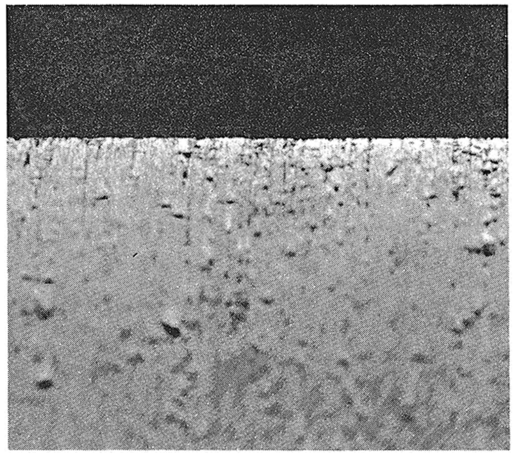
Figure 30. Scanning electron photomicrographs of implanted Falex pin - worn forty-three and one-half minutes



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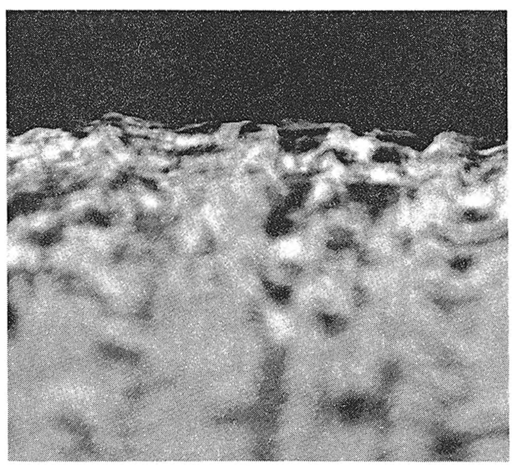

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Figure 31. Scanning electron photomicrographs of implanted Falex pin - worn eighty-seven and one-half minutes

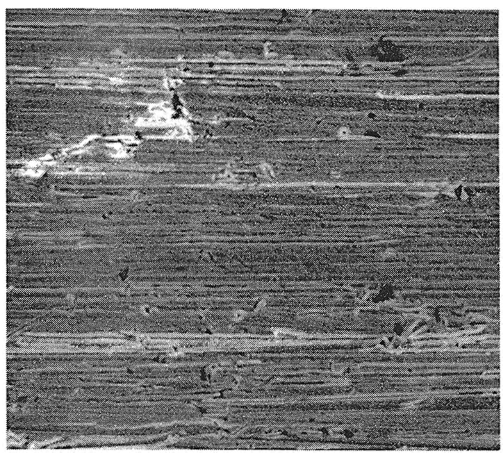

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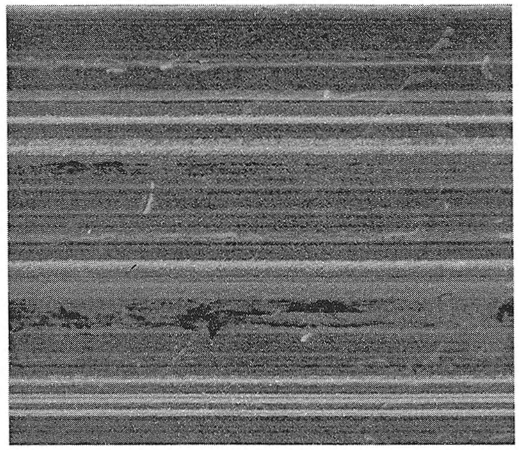
Figure 32. Scanning electron photomicrograph of Falex pin - as-polished



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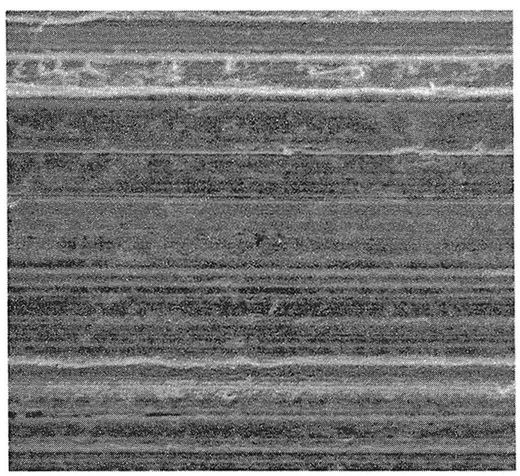
Worn five minutes



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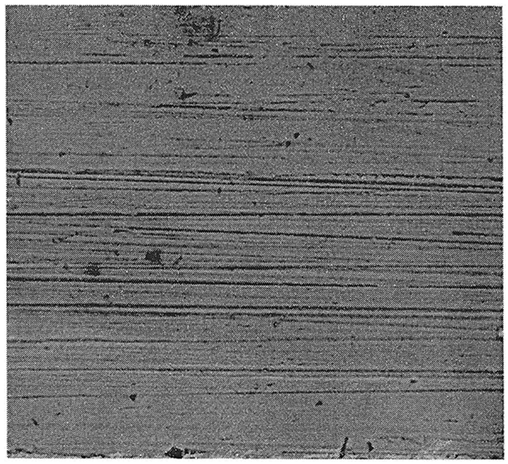
Worn twenty minutes

Figure 33. Scanning electron photomicrographs of unimplanted Falex pins worn for five and twenty minutes



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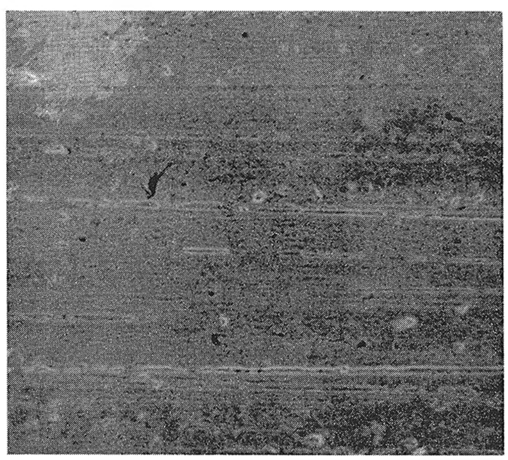
Worn five minutes



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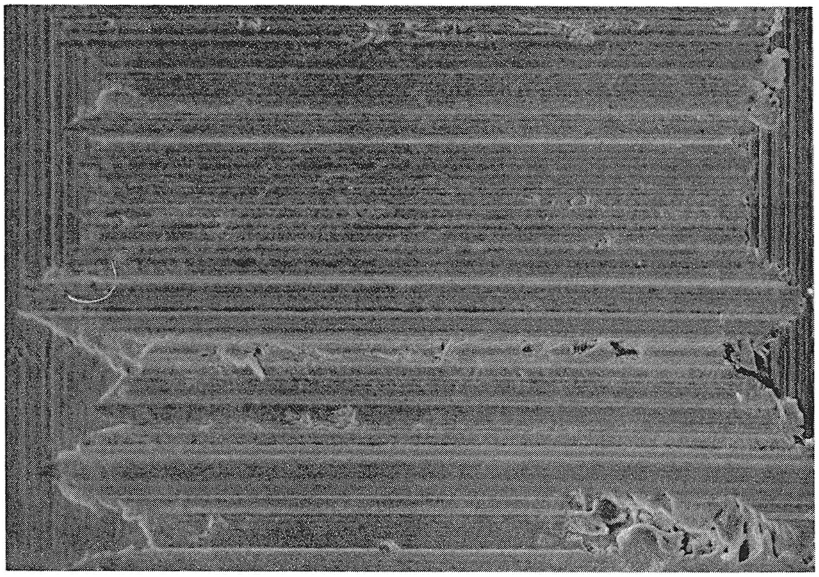
Worn twenty minutes

Figure 34. Scanning electron photomicrographs of implanted Falex pins worn for five and twenty minutes



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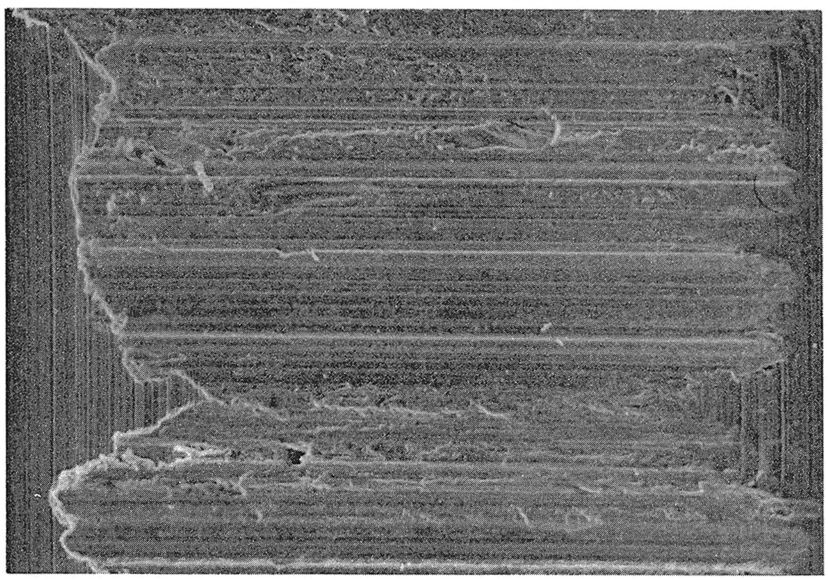
Worn five minutes



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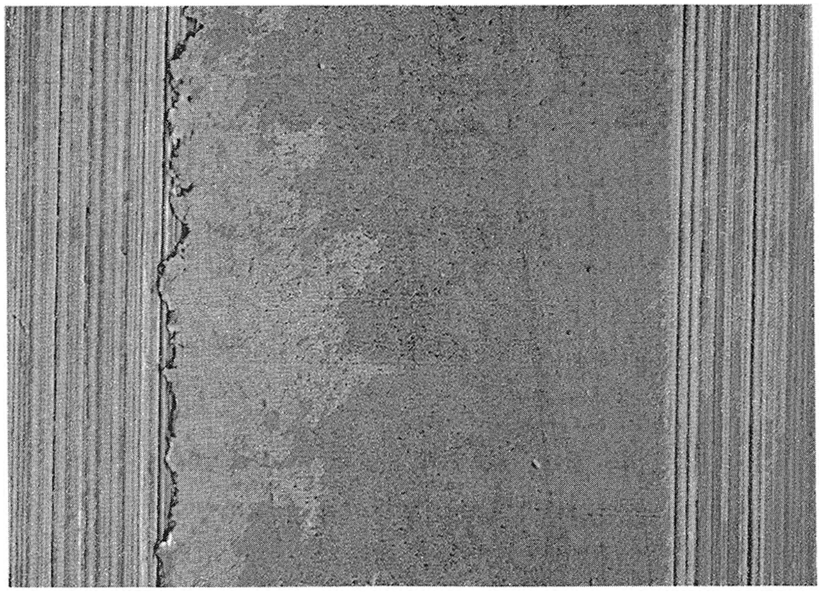
Worn twenty minutes

Figure 35. Scanning electron photomicrographs of wear scars of blocks worn against unimplanted Falex pins for five and twenty minutes



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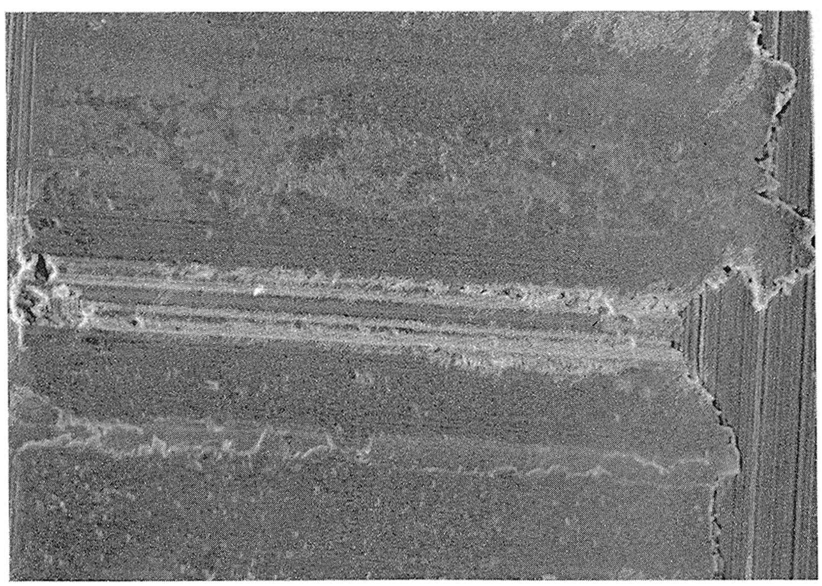
Worn five minutes



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Worn twenty minutes

Figure 36. Scanning electron photomicrographs of wear scars of blocks worn against implanted Falex pins for five and twenty minutes

experiments. A phone call to the manufacturer revealed that peanut oil is a very complex mixture of fatty acids. Some of these organic acids are similar to the additives that go into motor oils. In order to determine if there was an effect of the fatty acids on the wear rates of our pins, we ran some tests in non-detergent motor oil and some in a mixture of motor oil and oleic acid, a major constituent of peanut oil.

In the previously reported work, (4), (21), (35), (34), the wear rates of unimplanted pins run in peanut oil that had not displayed the "stuck pin" phenomenon were typically 0.25 mg/min. We ran six baseline tests in a non-detergent SAE 30 motor oil and had an average wear rate of 0.146 mg/min (see appendix). Then several tests were conducted with the same motor oil with additions of 1% and 5% oleic acid. A 1% addition of the oleic acid reduced the wear rate by an average of 75% and a 5% addition of oleic acid reduced the wear rate to an average of 0.0075 mg/min (see appendix).

Another series of tests were run to see if there was any difference between different lot numbers of peanut oil. A case of peanut oil was purchased with each bottle being stamped with a number that signifies the batch, year and day that it was produced. We labeled each bottle and ran several unimplanted pins using oil from bottles showing different lot numbers. The results were that, in the same case there were several bottles that resulted in normal, expected, values for the wear rate and several bottles that yielded the "stuck pin" phenomenon. With these results we decided that a change should be made in the lubricant used for our experiments.

We decided to examine non-detergent motor oil and DuoSeal pump oil as possibile lubricants in our subsequent experiments. They would be compared against the results obtained with the peanut oil. We wanted a lubricant with a wear rate nearly equal to the past results obtained with the peanut oil and one that had as little spread from an average value as possible. Figure 37 shows the results obtained using peanut oil. The average value was acceptable, but there is a large amount of scatter in the wear rates. Figure 38 shows the data using SAE 30 motor oil, and Figure 39 displays the data using DuoSeal pump oil. The DuoSeal pump oil yields an average wear rate in a desireable range and the best statistical spread of values.

Some work had begun on investigating the effects of implanted Group IV elements on the wear properties of steels when the "stuck pin" phenomenon, and equipment malfunction caused a delay in testing. The preliminary results on silicon and tin are represented on the dose curves in Figures 40 and 41. The curves show that for Si and Sn there is a similar dose dependence to that shown previously with nitrogen. In both cases the dose

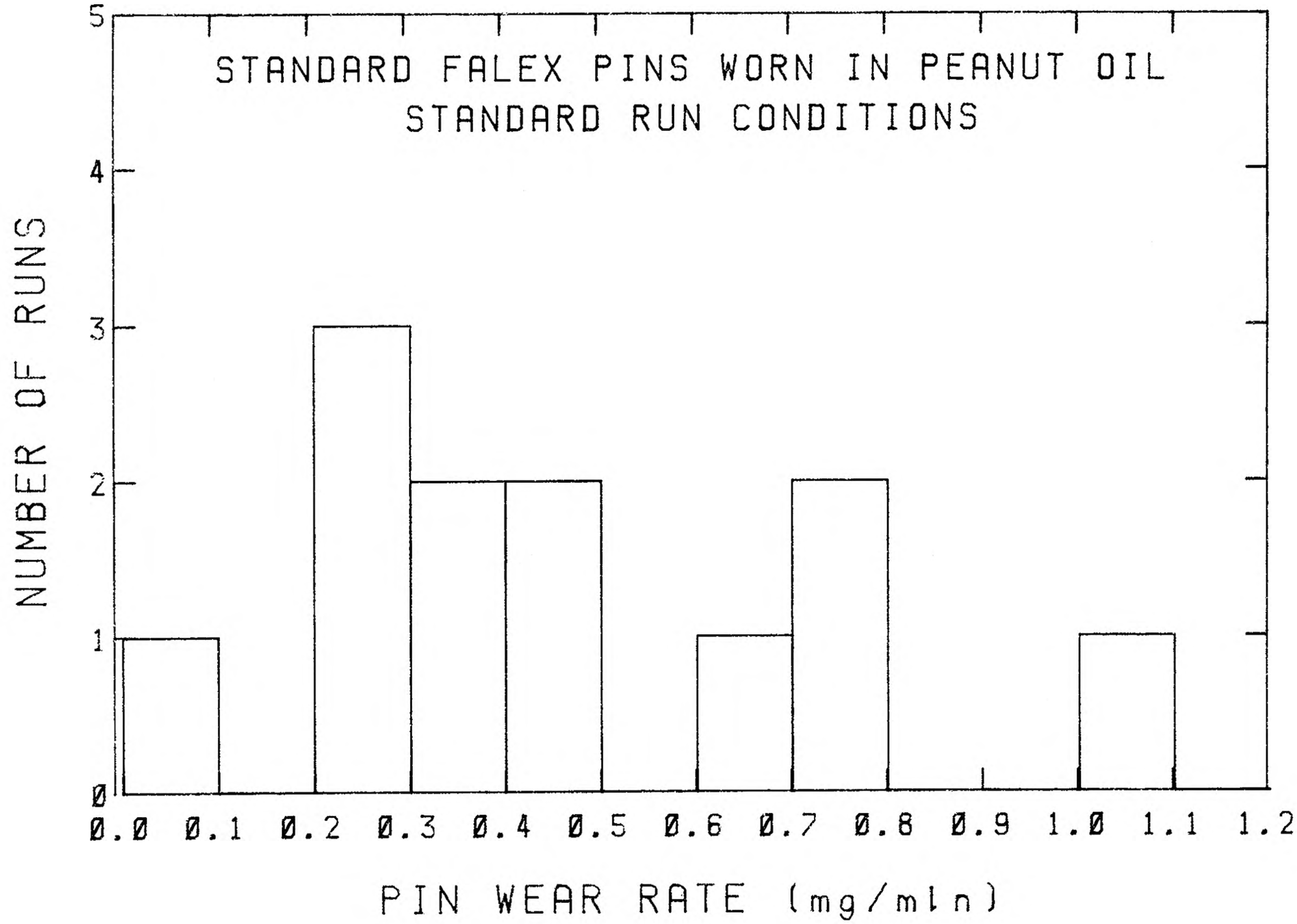

Figure 37. Baseline wear rates for unimplanted pins worn in peanut oil

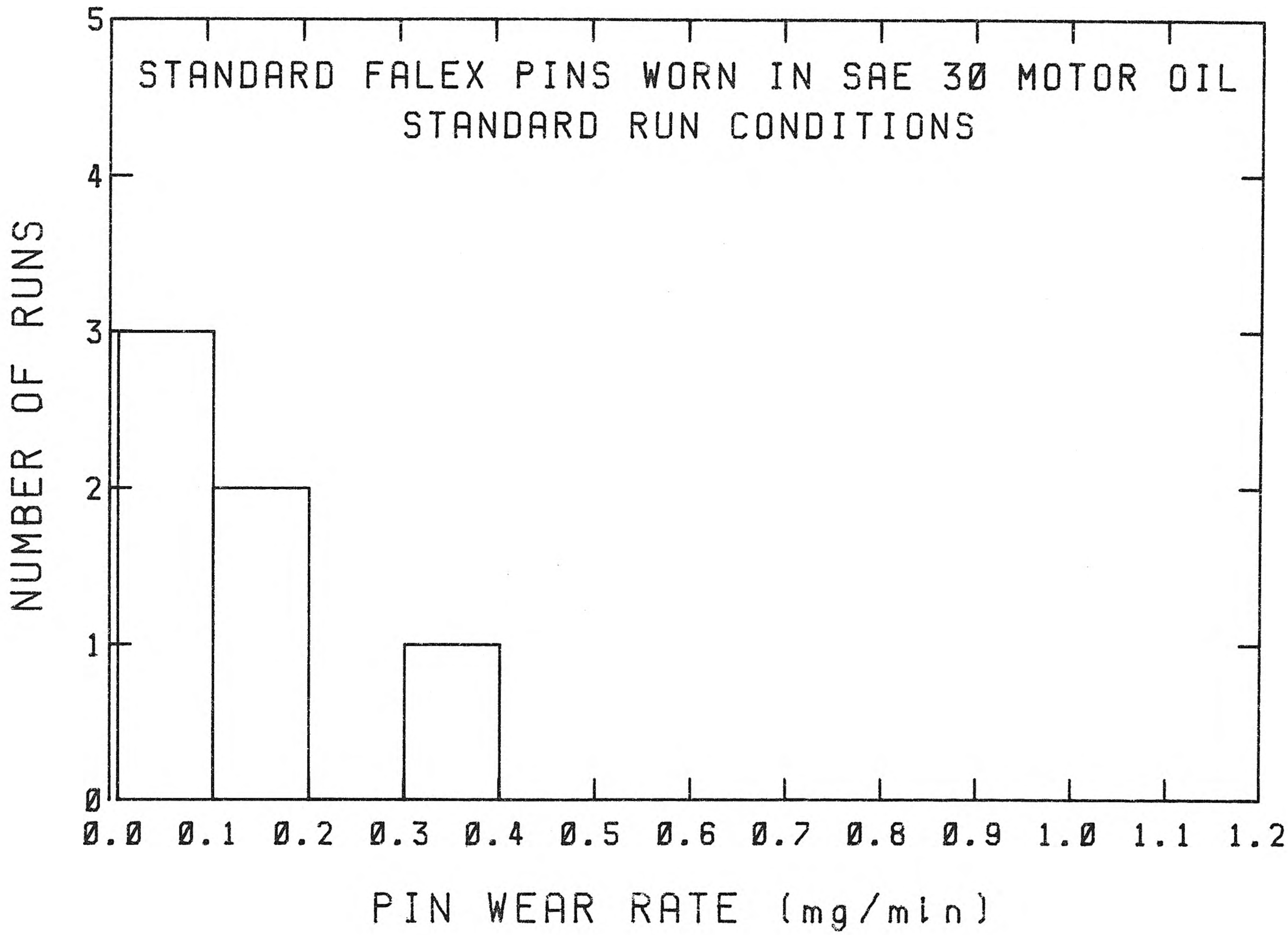

Figure 38. Baseline wear rates for unimplanted pins worn in motor oil

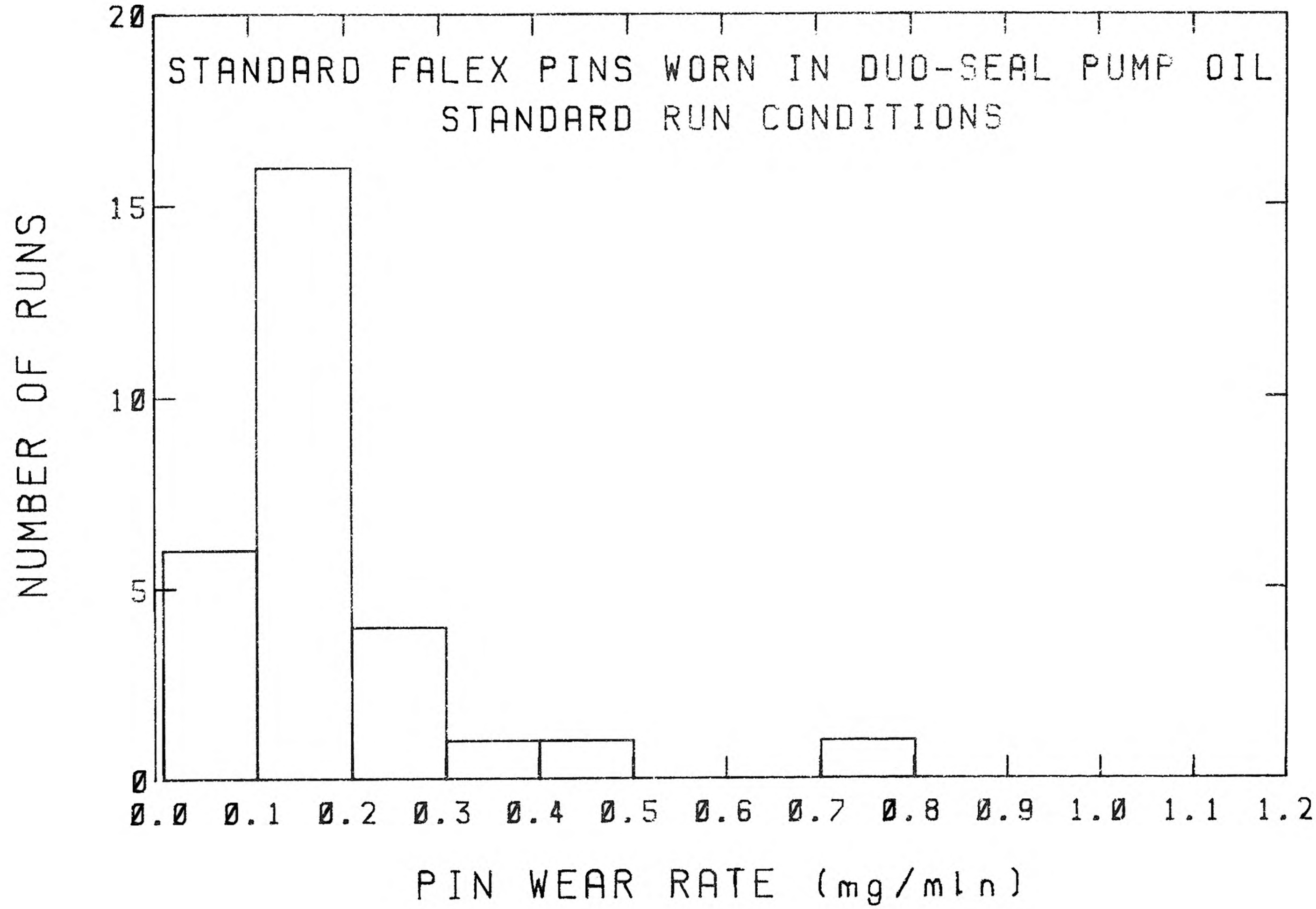

Figure 39. Baseline wear rates for unimplanted pins worn in DuoSeal

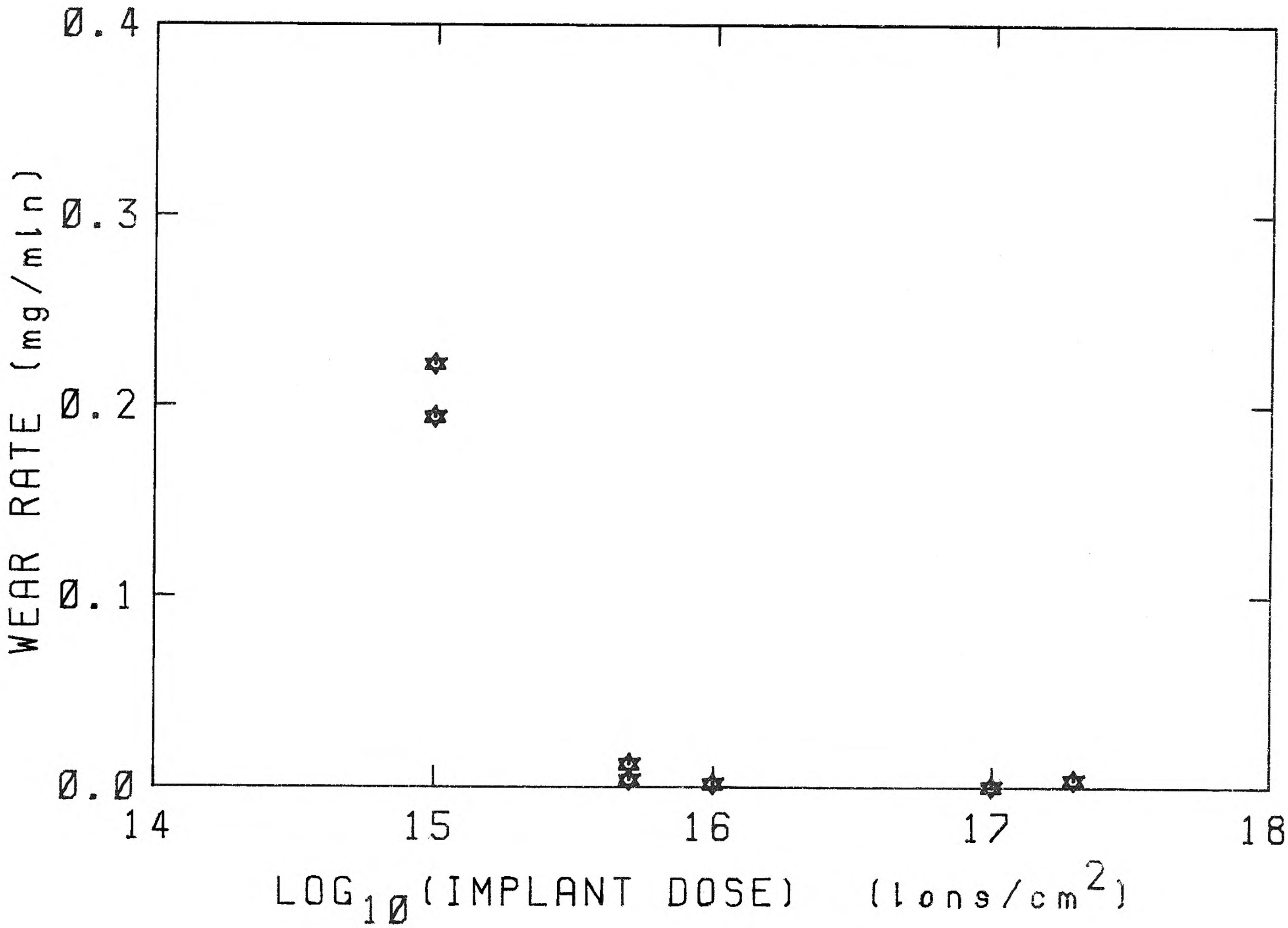

Figure 40. Tin dose curve in peanut oil

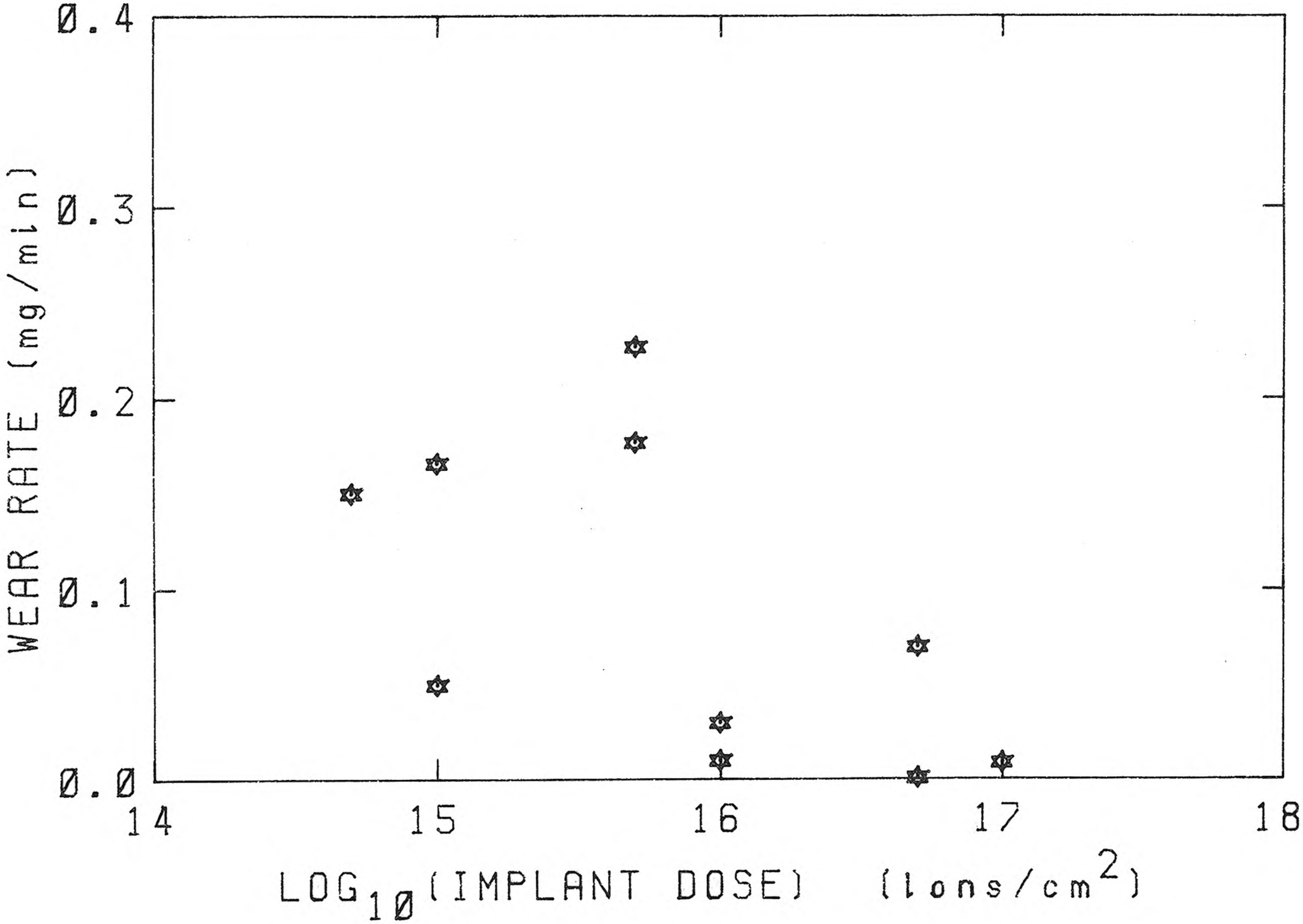

Figure 41. Silicon dose curve in peanut oil

necessary to cause a decrease in wear rate is less than with nitrogen. However, the beam currents obtainable with these heavier elements is much less and the time to implant a sample is much longer than with nitrogen.

It was necessary to develop a dose curve for nitrogen in DuoSeal pump oil in order to select a dose for the aging experiments. Figure 42 shows the dose dependence for nitrogen is virtually the same as the relationship as that recorded in peanut oil. The only difference is that the upper plateau for low dose and unimplanted pins is lower than the values with peanut oil.

During the delay in testing we performed tests on pins that had been implanted earlier and not yet tested. There were some pins that had been implanted with carbon and not yet tested. These pins had been implanted with relatively low doses, but showed reduced wear rates. When the accelerator was functional again, more samples were implanted in order to generate a dose curve for carbon. When the pins were run the resultant dose curve was significantly above the data points that were run initially. This data is shown pictorally on Figure 43. When the two original points were examined it was learned that they had been implanted 4 and 5 months prior to the wear tests. This provided the incentive to implant some more pins and perform aging experiments in order to determine if the aging effect that had been

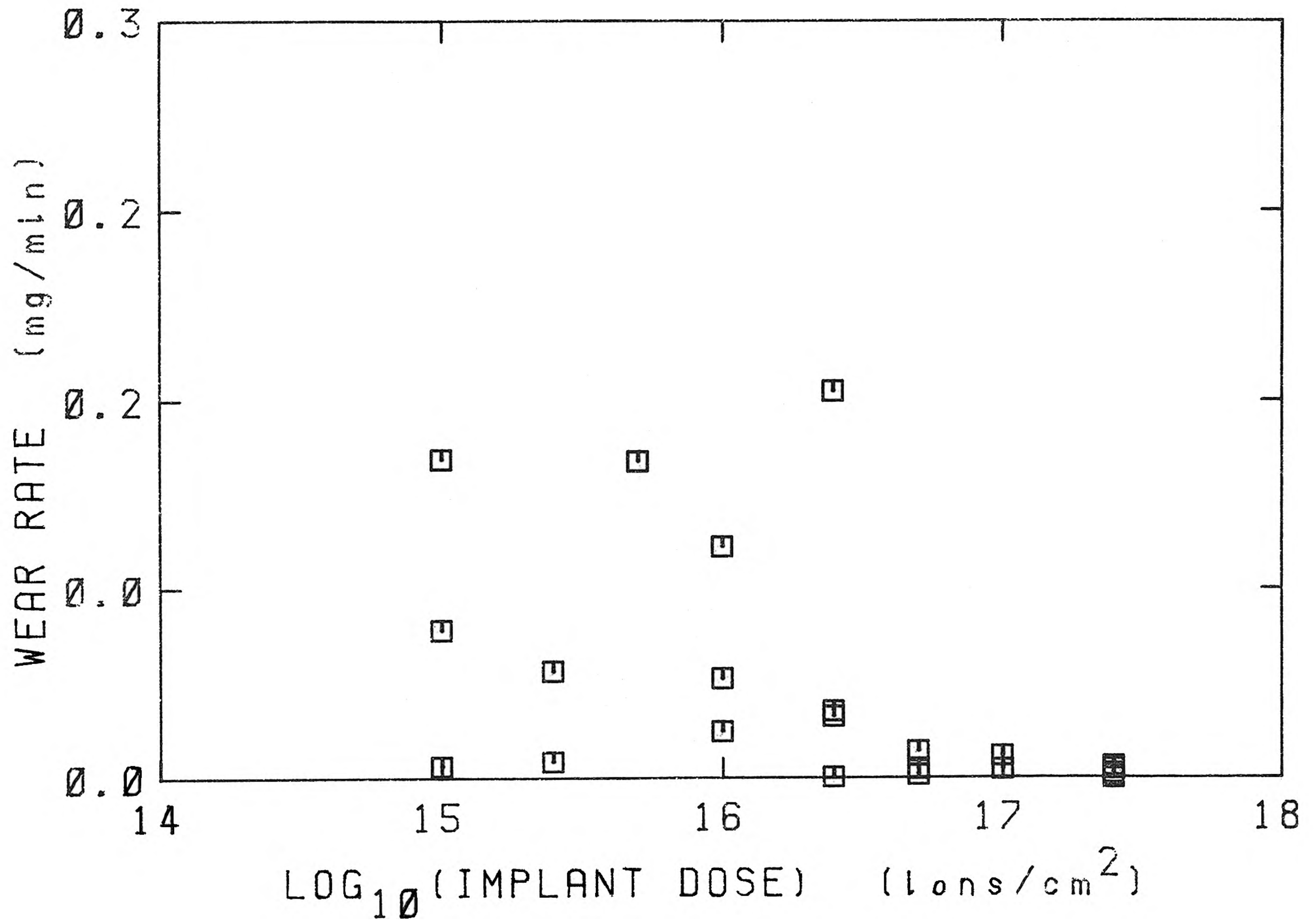

Figure 42. Nitrogen dose curve in DuoSeal

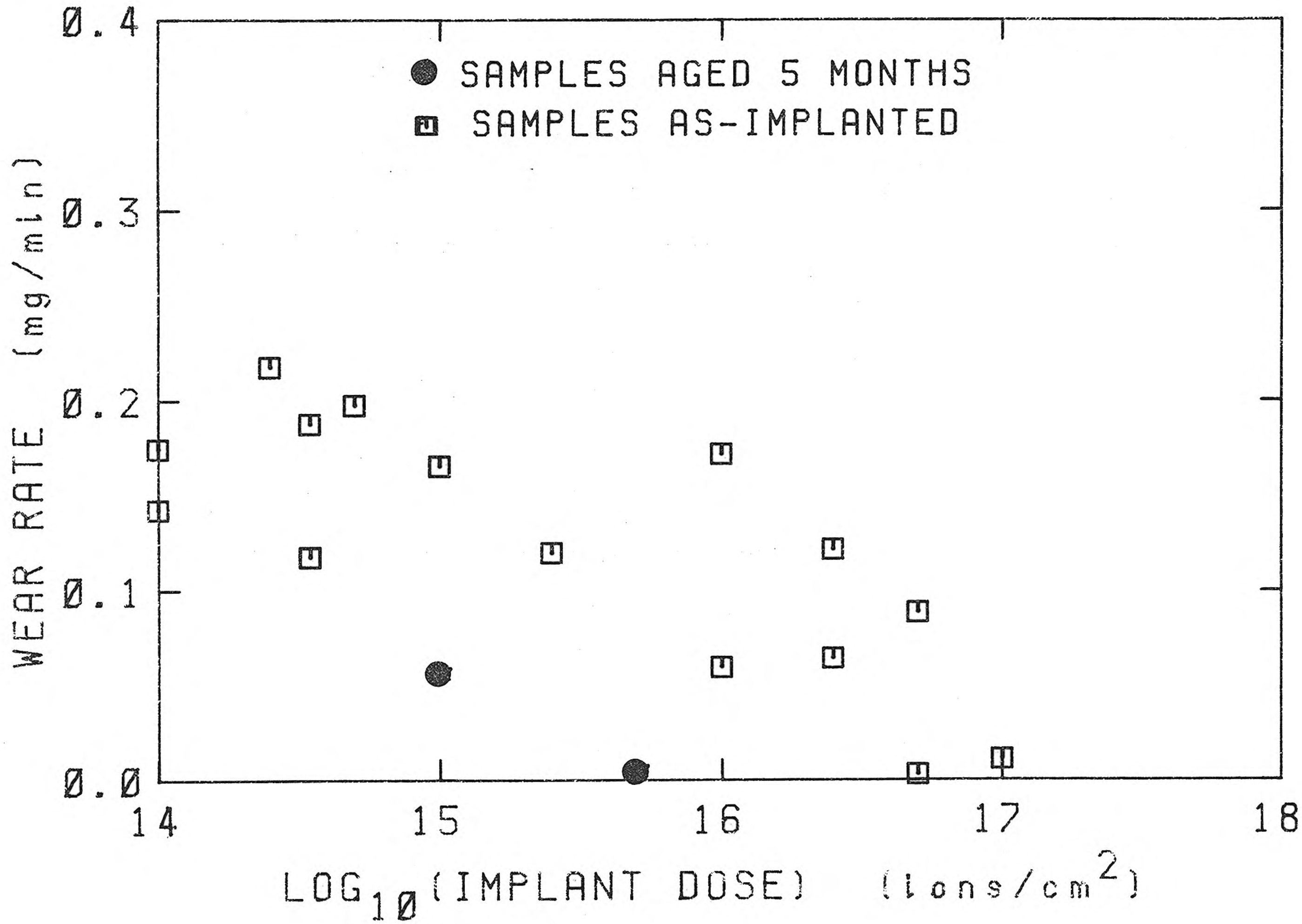

Figure 43. Carbon dose curve in DuoSeal

discovered by Hu et al.(26) on fatigue might be seen in the wear properties of steel. Hu's results are depicted on Figure 44.

The doses selected for the aging experiments were chosen to be slightly above the upper knee of the dose curves so that a definite change could be observed if it truly exists. All the pins that were implanted were stored in a desiccator at room temperature. The pins that were aged did not exhibit any significantly lower wear rates after aging up to twenty weeks than the pins that had similar implant doses and were tested immediately after implantation. Figures 45 and 46 show the wear rate versus aging time for carbon and nitrogen implanted pins.

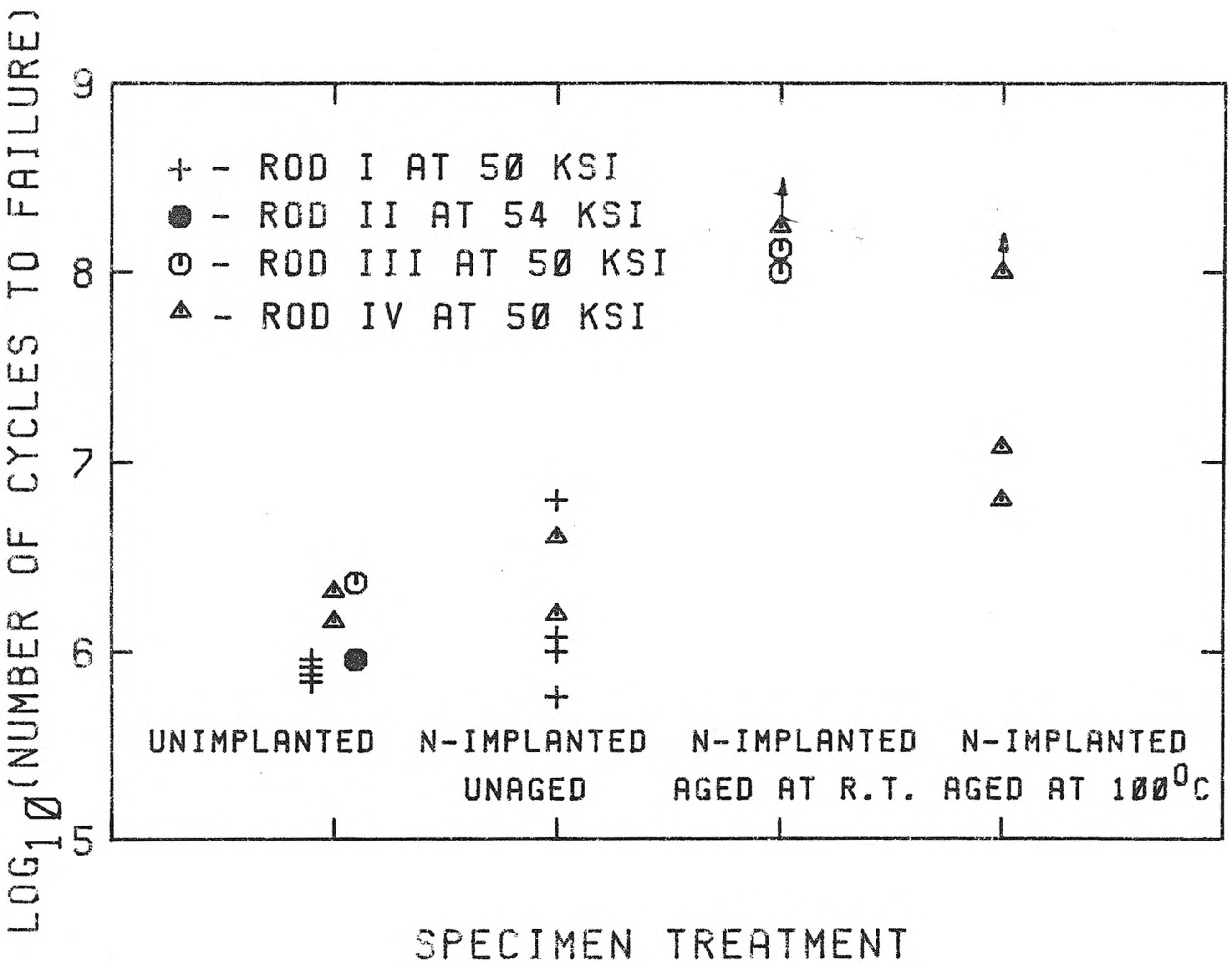

Figure 44. Age dependence of fatigue life

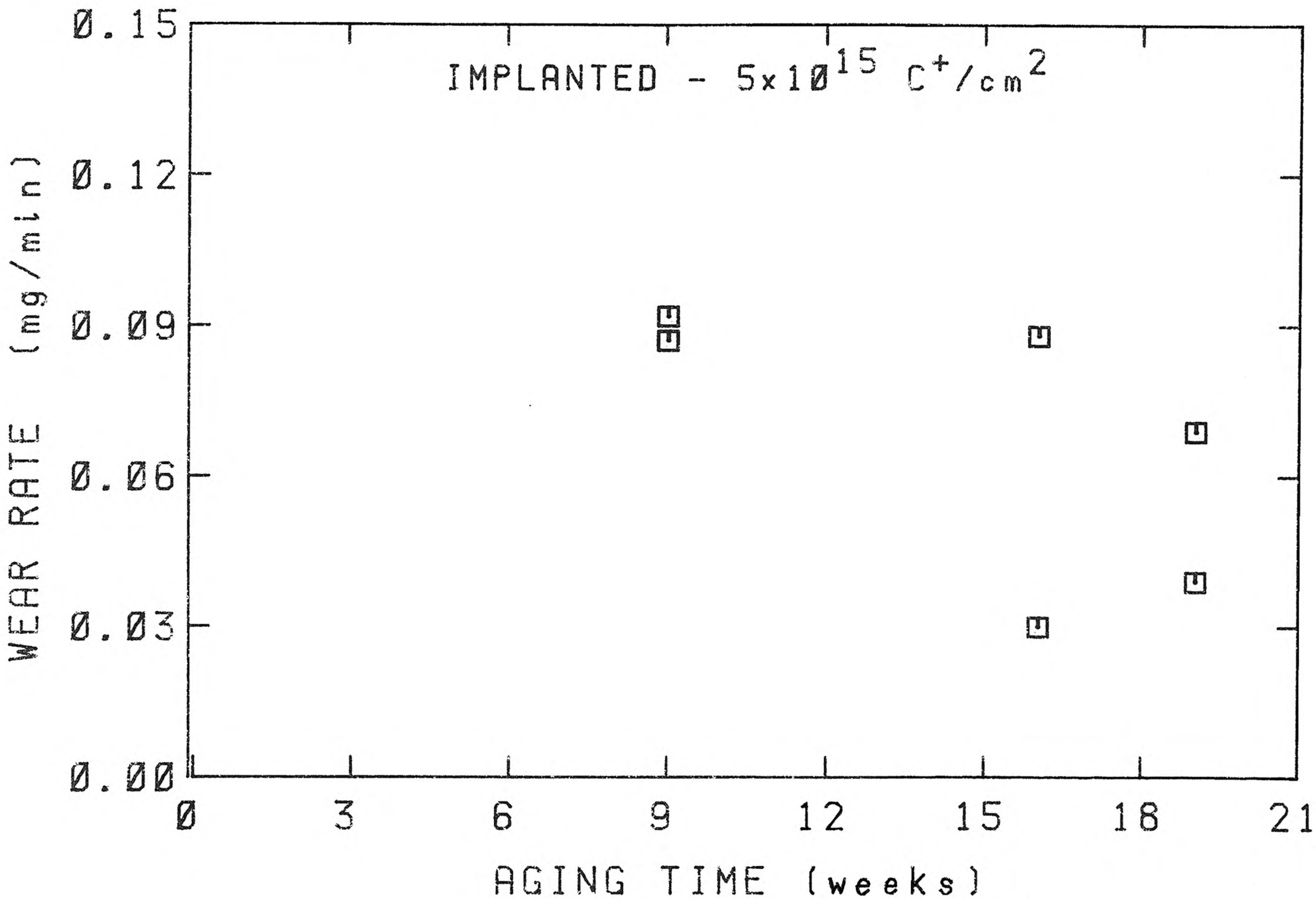

Figure 45. Age curve for carbon-implanted pins

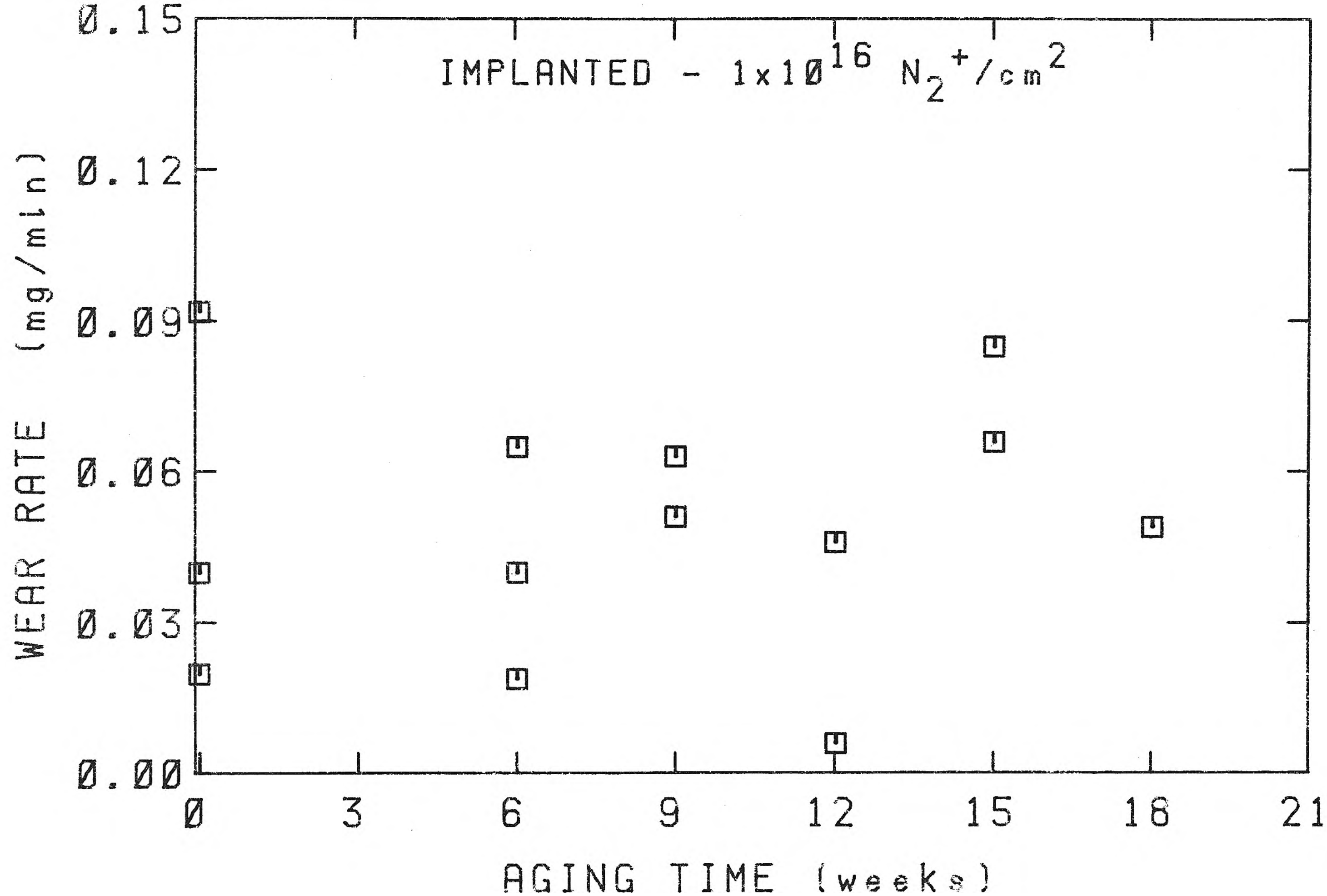

Figure 46. Age curve for nitrogen-implanted pins

V. DISCUSSION

Since the main thrust of the work at U.M.R. has been on wear, it was decided to determine what actually occurs in our wear tests. There are basically two theories on why ion implantation improves the wear properties of metals. The first, and the older of the two, is that the implanted species (ie. Nitrogen) forms a hardened surface layer that is resistant to wear (22), (23). The implanted species diffuses ahead of the wear front through a network of dislocations and essentially forms a resistant barrier directly below the surface that is self-perpetuating. The second theory includes the oxidative theory of wear which introduces the premise that surface asperities come into contact, heat, oxidize and that the subsequent oxide grows to a critical thickness at which it becomes unstable and wears away (15), (16).

Hartley et al. (36) have lent additional support to the importance of the oxide film by indicating that ductile oxide films can aid in reduction of wear rates by their ability to repair damage done to themselves.

Our work at U.M.R. tends to support the oxidative theory of wear, but makes some additional and interesting observations. In Figure 8 there are definitely two distinct zones. The initial portion of the curve indicates that there is some sort of initiation phase that is taking place where the wear rate is falling to some steady state level. Once this steady state is reached the wear rate remains at a very low value, even though the Auger results, as depicted on Figures 12 and 13, show that the nitrogen has been reduced and is undetectable after a finite length of time. Here is where we begin to differ from the standard theory of ion implantation and oxidative wear. If the implantation reduces the wear rate by reducing the oxidation of the surface, then how does the wear rate remain so low after the nitrogen is gone?

There is great deal of information to be obtained from the scanning electron photomicrographs of the worn pin surfaces in conjuction with the Auger results. In the photographs of the unimplanted pins there are very deep ruts or gouges and small transverse cracks along the surface. Occasionally there is found what appears to be a small flake as shown on Figure 47. This flake does not appear to be the remnant of an adhesive wear mode, but does appear to be the result of an oxide delamination. The thickness of the flake corresponds very closely to the depth of oxide film found from our Auger study.

The scanning electron photomicrographs of the implanted pins show that in the early minutes of the wear test there is virtually no wear. However, on

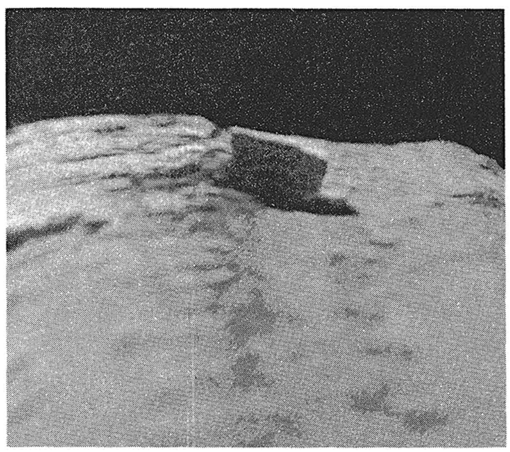

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Figure 47. Scanning elctron photomicrograph of oxide flake

closer examination it is seen that there is a polishing effect that is occuring. This polishing is evidenced by the subtle disappearance of the original polishing scratches that are at an angle to the axis of the pin. Once the polishing out of the original scratches is over, there is a continued smooth wear until at some point we see the introduction of the transverse cracks that we believe to be delamination of the oxide layer. The oxide thickness of the worn implanted pin corresponds to the thickness of the worn unimplanted pin at the time intervals where the delamination is seen to occur. In the unimplanted pins this effect was seen in the pin worn for two minutes, but did not become evident in the implanted pin until a wear time of approximately forty-five minutes. This optical interpretation of the wear modes is diplayed on Figure 48.

One major difference in the surface of the pins at the point that delamination occurs is the roughness of the pin surface. The implanted pin has gouge depths that are on the order of one-tenth of the one seen on the unimplanted pins. These gouge depths are tabulated in Tables 3 and 4. The gouge depth is taken directly from the scanning electron photomicrographs and the average wear depth is calculated from the pin mass loss with the assumption that the mass is lost as a thin sheet of steel off the pin surface.

This would mean that the smoother surface would be

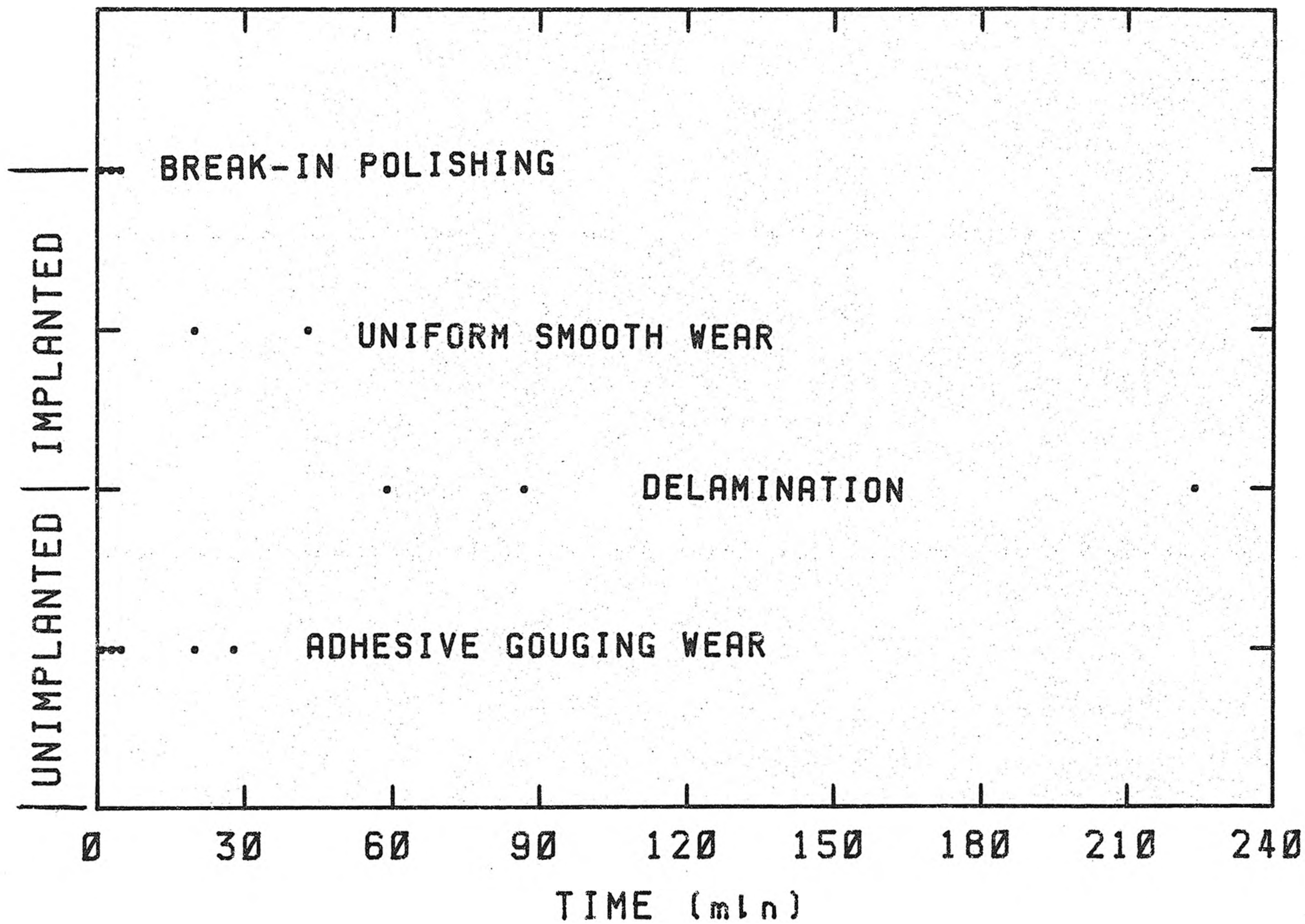

Figure 48. Optical characterization of wear modes

TABLE III

TYPICAL WEAR DEPTH DATA FOR UNIMPLANTED PINS

WEAR TIME (min)	GOUGE DEPTH (microns)	AVERAGE WEAR DEPTH (microns)
0	0.14- 0.28	
1	1.4 - 1.8	0.06
2	4.0 - 4.8	0.12
3	1.6 - 3.2	0.18
5	10.4 -20.8	0.32
10	1.8 - 2.4	0.61
20	2.6 - 6.6	0.46
28	4.8 -10.4	1.69

TABLE IV

TYPICAL WEAR DEPTH DATA FOR IMPLANTED PINS

WEAR TIME (min)	GOUGE DEPTH (microns)	AVERAGE WEAR DEPTH (microns)
0	0.14- 0.28	
1	0.4 - 0.5	
2	0.26	
5	0.26- 0.52	0.11
10		0.20
20		0.19
25	0.26	
43.5	0.5 - 1.3	0.06
87.5	0.5 - 1.0	0.04
224	0.26- 0.52	0.50

more likely to develop a lubricating film in an elasto-hydrodynamic mode than the rough surface of the unimplanted case (37). In the unimplanted case there would also be higher stresses at the peaks and more severe deformation would continue to occur. In this contrasting situation the implanted pin would have a uniform surface structure with an oxide layer growing at a steady state and small delaminations would occur with a correspondingly low wear rate. On the other hand the unimplanted pins have a rougher surface with correspondingly higher stresses at the asperity contact points. The higher stresses would cause faster oxidation corresponding to a larger wear rate.

P.L. Hurrick (38) performed some experiments on the effect of oxide films on fretting wear that also relates to this study. By heating samples and forming surface oxides prior to fretting wear test, he was able to decrease the wear rate. In the fretting wear test there are very small displacements so the surface becomes very smooth. His introduction of an oxide film prior to the destructive fretting mechanism indicates the importance of the oxide film in the wear mechanism.

We have characterized what happens on the wear surface but not yet on the reason why. In order to support our reasoning in the upcoming discussion we need to return to our experience with testing of unimplanted pins in peanut oil. We experienced a phenomenon which we termed the 'stuck pin' mode. These unimplanted pins displayed wear rates that rivaled those that had been implanted. Schey (39) has alluded to the fact that as organic oils age they release more of the organic acids and increase the lubricity of the oil.

It is our contention that the implantation of nitrogen causes the coeffícent of friction to be reduced, which in turn reduces the tangential frictional forces. These reduced frictional forces would reduce the resultant stress state and more importantly reduce the frictional heating. With reduced heating there would be decreased oxidation, which has been observed, and the surface oxide would grow in a uniform manner reaching a steady state value. Once the surface oxide reaches a critical value it would begin to delaminate and thus establish a steady state wear rate. An important note in this steady state situation is the surface morphology. The surface smoothness of the implanted pin is an important variable as it reduces the surface stresses at the asperities by increasing the actual amount of surface area that carries the load.

Our results on the aging effect on wear properties are not very conclusive. This, in itself, is consistent with other research. Some researchers have found that no aging effect exists where others have found that it does exist. Difference in the beam current has been the main difference in the previous work on fatigue. We did have some slight evidence that there was an effect on wear, but it is too slight to draw any conclusions.

The results that have been obtained by the research at U.M.R. with peanut oil have been very perplexing. It has caused us to alter the course of our testing by changing the lubricant. We did some investigation into the makeup of the peanut oil and found it to be a very complex mixture of organic fatty acids. One of the cautions on the labels of some vegetable and peanut oils is that when exposed to extremes of temperature they can either condense fats or become rancid or spoil. These cautions in themselves indicate the instability of these substances.

A comment is found on the use of organic lubricants by Schey in the book "Tribology in Metalworking" (39). It doesn't explain in full detail, but it indicates that as organic oils age there is a decomposition that occurs and releases more organic acids into the system and they become better lubricants.

VI. CONCLUSIONS

The reduction in retained nitrogen versus wear time would tend to dispute the theory that the nitrogen forms a hard surface layer and diffuses ahead of the wear front. The increase in oxygen levels versus wear time would indicate that there is an oxidative wear mode in effect. The lower rate of growth of oxygen in the implanted pins with respect to the unimplanted pins certainly indicate that the implantation causes the oxidation rate to be decreased and the wear rate is correspondingly reduced.

The S.E.M. photographs do show that the wear mode and surface morphology of the implanted and unimplanted pins are drastically different. There is severe adhesive wear in the unimplanted case along with a very rough surface. The mode of wear changes to mild abrasion in the implanted case coupled with a very smooth surface.

There is sufficient evidence in the published literature coupled with our experimental results to suggest that a combination of reduced oxidation, increased surface hardness, reduced coefficient of friction and other parameters not yet investigated all contribute to the reduction of wear through ion implantation.

There appears to be some effect of aging on the wear properties of the Falex pins, but the data is very inconclusive. There was enough reduction in wear rate of the first two carbon implanted pins to warrant a further investigation, and there were a few pins that did achieve a reduction after aging. The amount of substantiating evidence is not enough to make any claims, but further investigation would certainly be worthwhile.

There was a need to change the lubricant used in the wear tests, as there does seem to exist a connection between the wear rate of the Falex pins and the lubricant used. The organic acids in the peanut oil seems to be at least partially responsible. Apparently there is a difference in lubricity for different bottles of peanut oil that makes it too variable to use in further tests. It is for this reason that the change to DuoSeal pump oil was made.

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VITA

Kenneth William Burris was born on November 2, 1953 in Peoria, Illinois. He attended elementary school at Robein Elementary School in Robein, Illinois. He received his secondary education at Marceline R-5 High School in Marceline, Missouri. He graduated as Salutatorian of the Class of 1971. He subsequently received his Bachelor of Science in Metallurgical Engineering from the University of Missouri-Rolla, graduating Magna Cum Laude in 1975.

He attended graduate school from August 1975 to December 1977, at which time he took employment with Caterpillar Tractor Company in Peoria, Illinois. He returned to finish the requirements for his Masters degree in August 1983 and has held a Research Assistantship for the period August 1983 to June 1984.

APPENDIX: Table of data

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
1	240	0.26	0.0011	None	Peanut Oil
2	28.25	3.12	0.110	None	SAE 30
3	58.75	3.58	0.061	None	SAE 30
4	47.75	0.00	0.000	None	99% SAE 30 1% Oleic acid
5	53.75	3.90	0.076	None	99% SAE 30 1% Oleic acid
6	51.5	0.59	0.012	None	95% SAE 30 5% Oleic acid
7	30.25	0.11	0.004	None	95% SAE 30 5% Oleic acid
8	21	7.51	0.358	None	SAE 30
9	57.75	5.53	0.096	None	SAE 30
10	85.75	8.38	0.098	None	SAE 30
11	78.5	3.91	0.050	$2.5 \times 10^{17} N_2^+/cm^2$	SAE 30
12	61	8.97	0.147	None	SAE 30

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
13	18	6.98	0.388	$5 \times 10^{15} Si^+/cm^2$	Recirculating Peanut oil
14	33	5.65	0.171	None	Recirculating Peanut oil
15	34	7.03	0.207	$1 \times 10^{15} Si^+/cm^2$	Recirculating Peanut oil
16	35.25	4.76	0.135	$1 \times 10^{16} Si^+/cm^2$	Recirculating Peanut oil
17	20	7.01	0.351	$5 \times 10^{16} Si^+/cm^2$	Recirculating Peanut oil
18	16.25	7.13	0.439	None	Recirculating Peanut Oil @ 120 ^{o}F
19	35.25	7.17	0.203	None	Peanut oil
20	Machine malfunctioned - no values obtained				
21	91.5	5.54	0.061	None	Peanut oil Code A-3159
22	29	7.71	0.266	None	Peanut oil

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
23	240	0.67	0.003	$5 \times 10^{16} Si^{+}/cm^2$	Peanut oil Code A-3159
24	120	4.28	0.036	None	Peanut oil
25	83.75	1.64	0.020	None	Peanut oil
26	15	6.54	0.436	None	Peanut oil Code A-3159
27	24.5	7.02	0.287	None	Peanut oil
28	13	8.20	0.631	None	Peanut oil
29	240	1.47	0.006	$1 \times 10^{16} Si^{+}/cm^2$	Peanut oil
30	8	8.68	1.085	None	Peanut oil
31	240	0.80	0.003	$1 \times 10^{15} Si^{+}/cm^2$	Peanut oil
32	36.5	11.19	0.307	None	Peanut oil
33	18.5	8.61	0.465	None	Peanut oil
34	240	6.05	0.025	$1 \times 10^{14} Sn^{+}/cm^2$	Peanut oil
35	40.25	8.94	0.222	$1 \times 10^{15} Sn^{+}/cm^2$	Peanut oil

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
36	14.75	10.34	0.701	None	Peanut oil
37	37.5	8.91	0.238	$5 \times 10^{15} Si^{+}/cm^{2}$	Peanut oil
38	10.75	7.83	0.728	None	Peanut oil
39	18.75	7.95	0.424	$2.5 \times 10^{15} Sn^{+}/cm^{2}$	Peanut oil
40	23.75	8.41	0.354	None	Peanut oil
41	20.75	7.92	0.382	$5 \times 10^{15} Sn^{+}/cm^{2}$	Peanut oil
42	31	7.33	0.237	None	DuoSeal
43	26.5	3.79	0.143	None	DuoSeal
44	33.5	7.17	0.214	None	DuoSeal
45	16	6.90	0.431	None	DuoSeal
46	16.5	11.59	0.702	None	DuoSeal
47	30.5	3.12	0.102	None	DuoSeal
48	69.25	0.81	0.012	$2.5 \times 10^{17} N_{2}^{+}/cm^{2}$	DuoSeal
49	26	3.15	0.121	None	DuoSeal

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
50	30.25	3.09	0.102	None	DuoSeal
51	61	0.72	0.012	$2.5 \times 10^{17} N_2^+/cm^2$	DuoSeal
52	27.5	3.04	0.111	None	DuoSeal
53	115.75	0.62	0.005	$5 \times 10^{15} C^+/cm^2$	DuoSeal
54	31.25	3.92	0.125	None	DuoSeal
55	52.75	2.99	0.057	$1 \times 10^{15} C^+/cm^2$	DuoSeal
56	66	2.38	0.036	$5 \times 10^{12} Pb^+/cm^2$	DuoSeal
57	25.75	3.25	0.126	$5 \times 10^{15} N_2^+/cm^2$	DuoSeal
58	27.75	3.53	0.127	$1 \times 10^{15} N_2^+/cm^2$	DuoSeal
59	103.75	2.33	0.022	$1 \times 10^{16} Ar^+/cm^2$	DuoSeal
60	32.5	2.54	0.078	None	DuoSeal
61	18.25	2.16	0.118	$3.5 \times 10^{14} C^+/cm^2$	DuoSeal
62	23	3.82	0.116	$1 \times 10^{15} C^+/cm^2$	DuoSeal
63	16.25	3.22	0.198	$5 \times 10^{14} C^+/cm^2$	DuoSeal

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
64	26.25	4.59	0.175	$1x10^{14}C^+/cm^2$	DuoSeal
65	23.5	4.43	0.188	$3.5x10^{15}C^+/cm^2$	DuoSeal
66	42.5	2.53	0.060	$1x10^{16}C^+/cm^2$	DuoSeal
67	32.75	3.44	0.105	None	DuoSeal
68	27	3.21	0.119	None	DuoSeal
69	17.75	4.15	0.234	None	DuoSeal
70	21.5	3.85	0.179	None	DuoSeal
71	16	2.70	0.169	None	DuoSeal
72	26.25	3.31	0.126	None	DuoSeal
73	21.25	3.33	0.157	None	DuoSeal
74	21.75	4.09	0.188	None	DuoSeal
75	20.5	2.85	0.139	None	DuoSeal
76	240	2.90	0.012	None	Peanut oil
77	32.5	4.65	0.143	$1x10^{14}C^+/cm^2$	DuoSeal

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
78	23	5.02	0.218	$2.5 \times 10^{14} C^+/cm^2$	DuoSeal
79	23.5	8.80	0.374	$5 \times 10^{14} C^+/cm^2$	DuoSeal
80	27.75	3.33	0.12	$2.5 \times 10^{15} C^+/cm^2$	DuoSeal
81	23	3.96	0.172	$1 \times 10^{16} C^+/cm^2$	DuoSeal
82	32	2.86	0.089	$5 \times 10^{16} C^+/cm^2$	DuoSeal
83	33.5	4.09	0.122	$2.5 \times 10^{16} C^+/cm^2$	DuoSeal
84	33	3.04	0.092	$1 \times 10^{16} N_2^+/cm^2$	DuoSeal
85	34	3.74	0.11	$1.5 \times 10^{16} N_2^+/cm^2$	DuoSeal
86	30	4.63	0.154	$2.5 \times 10^{16} N_2^+/cm^2$	DuoSeal
87	129.25	1.41	0.011	$5 \times 10^{16} N_2^+/cm^2$	DuoSeal
88	29	3.54	0.122	$1 \times 10^{12} Pb^+/cm^2$	DuoSeal
89	30.5	3.41	0.11	$5 \times 10^{11} Pb^+/cm^2$	DuoSeal
90	27.25	4.46	0.16	$1 \times 10^{13} Pb^+/cm^2$	DuoSeal
91	25	4.37	0.175	$1 \times 10^{14} Pb^+/cm^2$	DuoSeal

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant
92	39.25	1.87	0.048	$5 \times 10^{13} Pb^{+}/cm^{2}$	DuoSeal
93	147.5	0.88	0.88	Vapor Deposited Aluminum	DuoSeal
94	30.25	5.17	0.17	$5 \times 10^{14} Pb^{+}/cm^{2}$	DuoSeal
95	40	3.51	0.088	$1 \times 10^{15} Pb^{+}/cm^{2}$	DuoSeal
96	24.5	2.89	0.12	$2.5 \times 10^{15} Pb^{+}/cm^{2}$	DuoSeal
97	24	4.25	0.18	$5 \times 10^{12} Pb^{+}/cm^{2}$	DuoSeal
98	33.75	4.20	0.12	Implanted Blocks $2.5 \times 10^{17} N_2^{+}/cm^{2}$	DuoSeal
99	38	4.23	0.11	Vapor Deposited Aluminum	DuoSeal
100	47.25	3.19	0.068	Vapor Deposited Aluminum	DuoSeal

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
101	30	0.30	0.010	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	1st run K-438-P
102	30	0.08	0.003	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	2nd run K-438-P
103	30	0.15	0.005	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	3rd run K-438-P
104	30	0.14	0.005	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	4th run K-438-P
105	30	0.24	0.008	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	5th run K-438-P
106	30	0.00	0.000	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	6th run K-438-P
107	30	0.05	0.002	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	7th run K-438-P
108	30	0.18	0.006	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	8th run K-438-P
109	30	0.00	0.000	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	9th run K-438-P
110	10	0.34	0.034	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	1st run K-433-P
111	20	0.31	0.016	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	1st run K-439-P
112	5	0.19	0.038	$2.5x10^{17}N_2^+/cm^2$	DuoSeal	1st run K-440-P
113	5	0.54	0.108	None	DuoSeal	
114	10	1.02	0.102	None	DuoSeal	

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
115	20	0.78	0.039	None	DuoSeal	
116	89.5	0.43	0.005	$1x10^{15} N_2^+/cm^2$	DuoSeal	
117	40	1.73	0.043	$5x10^{15} N_2^+/cm^2$	DuoSeal	
118	49	0.91	0.019	$1x10^{16} N_2^+/cm^2$	DuoSeal	
119	30	0.15	0.005	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	10th run K-438-P
120	30	0.02	0.001	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	11th run K-438-P
121	30	0.21	0.007	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	12th run K-438-P
122	10	0.33	0.033	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	1st run K-441-P
123	10	0.09	0.009	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	2nd run K-441-P
124	10	0.04	0.004	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	3rd run K-441-P
125	30	0.00	0.000	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	4th run K-441-P
126	42.75	2.52	0.059	$1x10^{15} N_2^+/cm^2$	DuoSeal	
127	61.5	0.46	0.007	$5x10^{15} N_2^+/cm^2$	DuoSeal	
128	44.75	1.80	0.040	$1x10^{16} N_2^+/cm^2$	DuoSeal	

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
129	121.75	0.009	0.001	$2.5x10^{16} N_2^+/cm^2$	DuoSeal	
130	53.5	1.43	0.027	$2.5x10^{16} N_2^+/cm^2$	DuoSeal	
131	30	0.14	0.005	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	13th run K-438-P
132	30	0.00	0.000	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	14th run K-438-P
133	30	0.20	0.007	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	15th run K-438-P
134	30	0.33	0.011	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	16th run K-438-P
135	171.5	0.69	0.004	$5x10^{16} N_2^+/cm^2$	DuoSeal	
136	162.75	0.25	0.002	$5x10^{16} N_2^+/cm^2$	DuoSeal	
137	30	0.45	0.015	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	17th run K-438-P
138	30	0.00	0.000	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	18th run K-438-P
139	30	0.04	0.001	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	19th run K-438-P
140	30	0.00	0.000	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	20th run K-438-P
141	59.25	0.56	0.009	$1x10^{17} N_2^+/cm^2$	DuoSeal	
142	224.25	0.83	0.004	$1x10^{17} N_2^+/cm^2$	DuoSeal	

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
143	87.5	0.06	0.001	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	
144	30	0.01	0.0003	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	21st run K-438-P
145	30	0.05	0.002	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	22nd run K-438-P
146	60	0.24	0.004	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	23rd run K-438-P
147	60	0.03	0.0005	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	24th run K-438-P
148	60	0.37	0.006	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	25th run K-438-P
149	60	0.31	0.005	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	26th run K-438-P
150	28	2.82	0.101	None	DuoSeal	
151	34.5	3.23	0.094	None	DuoSeal	
152	51.25	0.97	0.019	$1x10^{16} N_2^+/cm^2$	DuoSeal	Aged 6 weeks
153	34	1.36	0.04	$1x10^{16} N_2^+/cm^2$	DuoSeal	Aged 6 weeks
154	46	3.01	0.065	$1x10^{16} N_2^+/cm^2$	DuoSeal	Aged 6 weeks
155	43.5	0.10	0.002	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	
156	124	0.60	0.005	$2.5x10^{17} N_2^+/cm^2$	DuoSeal	

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
157	25	0.00	0.000	$2.5 \times 10^{17} N_2^+/cm^2$	DuoSeal	
158	32.75	2.10	0.064	None	DuoSeal	Tempered 30 minutes at 500^oF
159	35	2.44	0.070	None	DuoSeal	Tempered 30 minutes at 500^oF
160	39.5	2.24	0.057	None	DuoSeal	Tempered 30 minutes at 500^oF
161	27.75	0.70	0.025	$2.5 \times 10^{16} N_2^+/cm^2$	DuoSeal	
162	80	0.31	0.004	$2.5 \times 10^{17} N_2^+/cm^2$	DuoSeal	
163	40.25	2.62	0.065	$2.5 \times 10^{16} C^+/cm^2$	DuoSeal	
164	32.75	2.85	0.087	$5 \times 10^{15} C^+/cm^2$	DuoSeal	Aged 9 weeks
165	30	2.76	0.092	$5 \times 10^{15} C^+/cm^2$	DuoSeal	Aged 9 weeks
166	92	0.31	0.003	$5 \times 10^{16} C^+/cm^2$	DuoSeal	
167	82.75	0.96	0.012	$1 \times 10^{17} C^+/cm^2$	DuoSeal	
168	32.5	1.65	0.51	$1 \times 10^{16} N_2^+/cm^2$	DuoSeal	Aged 9 weeks
169	34	2.16	0.063	$1 \times 10^{16} N_2^+/cm^2$	DuoSeal	Aged 9 weeks

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
170	5	1.16	0.032	$5x10^{17}N^+/cm^2$	DuoSeal	1st run K-487-P
171	5	0.00	0.000	$5x10^{17}N^+/cm^2$	DuoSeal	2nd run K-487-P
172	5	0.21	0.042	$5x10^{17}N^+/cm^2$	DuoSeal	3rd run K-487-P
173	5	0.04	0.008	$5x10^{17}N^+/cm^2$	DuoSeal	4th run K-487-P
174	10	0.05	0.005	$5x10^{17}N^+/cm^2$	DuoSeal	5th run K-487-P
175	10	0.15	0.015	$5x10^{17}N^+/cm^2$	DuoSeal	6th run K-487-P
176	10	0.00	0.000	$5x10^{17}N^+/cm^2$	DuoSeal	7th run K-487-P
177	30	0.37	0.0123	$5x10^{17}N^+/cm^2$	DuoSeal	8th run K-487-P
178	30	0.00	0.000	$5x10^{17}N^+/cm^2$	DuoSeal	9th run K-487-P
179	30	0.81	0.027	$5x10^{17}N^+/cm^2$	DuoSeal	10th run K-487-P
180	5	0.37	0.074	$5x10^{17}N^+/cm^2$	DuoSeal	1st run K-488-P
181	10	No data taken - Scale malfunction				
182	5	0.82	0.164	$5x10^{17}N^+/cm^2$	DuoSeal	1st run K-489-P
183	5	0.16	0.032	$5x10^{17}N^+/cm^2$	DuoSeal	2nd run K-489-P

APPENDIX (continued)

Run #	T_{stop} (min)	δm_{pin} (mg)	ω_{pin} (mg/min)	Implantation Dose	Lubricant	Comments
184	10	0.00	0.000	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	3rd run K-489-P
185	10	0.01	0.001	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	4th run K-489-P
186	20	0.44	0.022	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	5th run K-489-P
187	20	0.41	0.021	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	6th run K-489-P
188	43.25	1.99	0.046	$1x10^{16}N_{2}^{+}/cm^{2}$	DuoSeal	Aged 12 weeks
189	20	0.47	0.023	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	7th run K-489-P
190	161.25	0.98	0.006	$1x10^{16}N_{2}^{+}/cm^{2}$	DuoSeal	Aged 12 weeks
191	5	0.00	0.000	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	1st run K-490-P
192	5	0.20	0.040	$5x10^{17}N^{+}/cm^{2}$	DuoSeal	2nd run K-490-P
193	28.75	2.54	0.088	$5x10^{15}C^{+}/cm^{2}$	DuoSeal	Aged 16 weeks
194	83	2.53	0.030	$5x10^{15}C^{+}/cm^{2}$	DuoSeal	Aged 16 weeks
195	30.5	2.01	0.66	$1x10^{16}N_{2}^{+}/cm^{2}$	DuoSeal	Aged 15 weeks
196	35.25	3.00	0.085	$1x10^{16}N_{2}^{+}/cm^{2}$	DuoSeal	Aged 15 weeks