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ABSTRACT

Fretting wear experiments were conducted on high purity iron, nickel and titanium in air under conditions of varied humidity and temperature, and in nitrogen. For iron and titanium, maximum fretting occurred at 10 and 50 percent relative humidity respectively. Nickel showed a minimum in fretting wear at about 10 percent relative humidity. With increasing temperature, all three metals initially showed reduced fretting wear, with increasing wear observed as temperatures increased beyond 200-300° C. For titanium, dramatically reduced fretting wear was observed at temperatures above 500° C, relatable to a change in oxidation kinetics. All three metals showed much less fretting wear in N_2 with the presence of moisture in N_2 having a proportionally stronger affect than in air.

INTRODUCTION

Fretting is a wear or surface damage phenomenon caused by low amplitude oscillatory motion between two contacting surfaces. Features that distinguish fretting from other modes of wear are the vibratory nature of the motion, and the long retention time of debris in the contact areas. Indeed, the nature and role of fretting wear debris has been the subject of considerable study, discussion and speculation.

Since fretting, after an initial "run-in" period, is a gradual process of material removal from the contact surfaces (1-3), very ample opportunity for interaction between environmental factors and fretting is present. The influence of environment on fretting is predominantly through corrosive effects. Therefore factors such as temperature, humidity, and presence or absence of corrosive constituents in the environment (e.g. O_2) would be expected to affect the extent of fretting damage.

Humidity effects on the fretting of mild steel were investigated by Feng and Uhlig (4), and Wright (5), both of whom identified a trend of decreasing fretting wear with increased humidity. Waterhouse (6) suggests that humidity might have two effects, both associated with the debris. First, moisture adsorbed onto the surface of debris particles might act as a lubricant, promoting more rapid debris dispersal and less abrasive wear. Second, a soft hydrated iron oxide may form in the presence of moisture, again resulting in less abrasive action.

Feng and Uhlig (4), and Hurricks (7,8) observed decreasing fretting wear with increasing temperature for mild steel. The trend is attributed to diminished metal-to-metal contact and adhesive damage because of the protective action of surface oxide films. Wright (9) suggests that above a transition temperature, specific for a given metal, rapid oxidation kinetics might promote enhanced fretting wear resistance; below the transition temperature the oxide film is mechanically disrupted on each fretting cycle.

Experiments conducted in N_2 (3,4) have generally shown reduced fretting wear compared to results obtained in air, despite the absence of surface protection effects attributable to oxide films. Thus, the importance of corrosion related mechanisms in accelerating fretting wear is emphasized.

The purpose of this investigation is to study the effects of humidity level, temperature, and inert environment (N_2) on the fretting wear of high purity iron, nickel, and titanium. The results are intended to provide a block of data, with associated microscopic observations, in which test geometry and other experimental parameters were all constant. The metals chosen for the investigation represent a range of behavior to be expected in various metallic systems, and at the same time are well characterized from the standpoint of oxidation kinetics. It is hoped that the roles of some of the fretting environmental variables will be clarified, and extended to metallic systems other than carbon steel, on which most of the work to date has been performed.

APPARATUS

A schematic diagram of the fretting rig is shown in figure 1. A linear oscillatory motion is provided by an electromagnetically driven vibrator with the frequency controlled by a variable oscillator. The load is applied to the specimens by placing precision weights on a pan which is hung from the load arm. The fretting specimens consist of an upper, stationary, 4.76-millimeter-radius, hemispherical tip in contact with a lower flat which is driven by the vibrator.

During high-temperature experiments, the specimens and grip assemblies were surrounded by a 310-stainless-steel susceptor which was heated by an induction coil. The temperature was monitored by a thermocouple probe mounted in the lower grip. The grips were specially designed so that they stored sufficient elastic energy to ensure that no slippage would occur as a result of differential thermal expansion.

A dry air environment was provided by flowing air through an absorption drier, then into the experimental chamber. In this way, moisture content was kept in the range of 10 to 100 parts per million. When a moisture-saturated environment was desired, the air was bubbled through a water-filled column, then led into the chamber. Intermediate levels of humidity were obtained by mixing dry air with moisture saturated air. A hygrometer was used to monitor the relative humidity of air flowing into the test chamber.

PROCEDURE

Before fretting, the flat specimens were lapped, mechanically polished with 0.05-micrometer alumina polishing compound, then rinsed in pure ethanol. The hemispherical tips, ground to a 0.1-micrometer (4-μin.) finish, were scrubbed with 0.05 micrometer alumina, and rinsed.

Following surface preparation, specimens were assembled into the grips in like-metal combinations. The test chamber was then purged with the desired gas, purge times being one hour for dry air and saturated air, and four hours for N_2. For elevated temperature experiments, all conducted in dry air, one hour was required for the specimens to reach the selected temperature and for the apparatus to reach thermal equilibrium, during which time the dry air purge was conducted.

With the vibrator now turned on, fretting was initiated by putting sufficient weight into the pan to just bring the specimens into contact. Additional weight was then added to apply the normal load to the rubbing surfaces. A normal load of 1.47 newtons was used in all cases. Other experimental conditions included a peak-to-peak amplitude of 50 micrometers (0.002 in.), a fretting frequency of 80±0.2 hertz, and a duration of 3×10^5 cycles.

Following each fretting experiment, the fretting scar on the flat surface was photographed to record the size and features of the wear scar and the debris accumulation around the scar. The loose debris was then rinsed off with pure ethanol, and a light section microscope was used to measure the wear volume on the flat specimen.

Specimens that were examined in the scanning electron microscope (SEM) were ultrasonically cleaned in methanol before viewing, to remove as much debris still adhering to the wear scar as possible.

MATERIALS

The titanium used in this investigation was of 99.8-percent purity. The principal impurities were carbon (150 ppm), oxygen (350 ppm), and silicon (150 ppm). In the as-machined condition, the hardness of the specimens was 74 on the Rockwell B scale. After exposure to 650° C for 1 hour, the hardness was measured to be 69 (Rockwell B). Further exposure to 650° C conditions in air resulted in no further reduction in hardness.

Iron of 99.95 percent purity was used in this fretting study, the principal impurities being carbon (30 ppm), oxygen (78 ppm), silicon (35 ppm), sulfur (30 ppm), molybdenum (50 ppm), copper (40 ppm) and tin (40 ppm). The hardness of the specimens was 21 on the Rockwell B scale.

The nickel used was of 99.99 percent purity, and the chief impurities were carbon (50 ppm), oxygen (20 ppm), and copper (15 ppm). Hardness of the nickel was 7 on the Rockwell B scale, 58 on the Rockwell F scale.

RESULTS AND DISCUSSION

As may be seen in figure 2, the effect of relative humidity level on the fretting wear of high purity iron is a complex one. A sharp increase in wear occurs as the humidity is increased from dry air conditions to about 10 percent relative humidity. Further increases in humidity result in a gradual reduction in fretting wear, with a minimum somewhere between 50 and 70 percent relative humidity, and a slight increase in wear as saturated air conditions are approached. These results compare trend-wise with those of Feng and Uhlig on mild steel (9) over the intermediate humidity range. Feng and Uhlig did not conduct tests, other than those in dry air, at humidity levels lower than 20 percent relative humidity, because of rusting of the mild steel they did not conduct tests in saturated air.

Optical micrographs shown in figure 3 indicate a striking effect of relative humidity on the distribution of fretting wear debris and nature of the fretting contact. Under dry air condition virtually all of the debris was retained in the contact area in the form of dark, oxidized material, as may be seen in figure 3(a). Fretting under conditions of 10 percent relative humidity resulted in dispersal of fretting debris from the contact area (fig. 3(b)), with exposure of metallic contact regions; the debris was reddish brown in color. Further increases in relative humidity did not bring about changes in the distribution of wear debris and nature of the fretting contact, until saturated air conditions were approached. After fretting in saturated air, the wear scar was virtually free of oxidized debris, all of the debris having been extruded from the contact area. The material comprising the contact area had a smeared metallic appearance.

Scanning electron microscopy (fig. 4) revealed a "leafy" wear scar surface after fretting in dry air. In reference 10, a similar surface morphology was reported for 9310 steel, and wear was described as proceeding by the gradual disintegration and oxidation of material comprising the edges of these layers or leaves. The mechanism was described as being a corrosion enhanced fatigue process. Similar fretting surfaces have been reported by Waterhouse for carbon steel (11) and by Hoeppner for Ti alloys (12). In contrast, the surface produced by fretting in saturated air is quite smooth as may be seen in figure 5. Only near the edges of the contact area where metallic material has apparently been extruded is there any evidence of layered morphology.

Fretting wear debris generated under dry air, 50 to 80 percent relative humidity (humidity varied during the long fretting exposure), and under saturated air conditions was examined using TEM, and electron diffraction patterns were obtained for the three debris samples. The electron diffraction patterns for the samples, shown in figure 6, were characteristic of α-Fe_2O_3. This observation was verified by X-ray diffraction studies performed on debris samples from saturated air fretting. Distinct diffraction spots were discernible in the patterns from the saturated air and intermediate humidity samples. A comparatively weak ring pattern, showing no spots was obtained from the dry air sample. The diffraction observations correlate with apparent crystallite size of the Fe_2O_3 comprising the debris. Under saturated air and intermediate humidity conditions, the debris is seen in figure 7 to consist of agglomerations of flat crystallites about 0.02 micrometers in diameter. Debris generated under dry air fretting conditions forms a layered structure with no clearly measurable crystallite size, thus giving rise to the diffuse diffraction pattern.

Together, the wear measurements, microscopic features of fretted areas, and analyses of debris are consistent with the suggestion, put forth by Waterhouse (6), that one of the effects of moisture presence might be the adsorption of H_2O onto debris particle surfaces allowing more rapid egress of debris from the contact area. Long debris retention times under dry air conditions leads to repeated working of particulates, promoting the very fine crystallite size observed. The long term presence of debris on the wear surface also affords some surface protection by inhibiting metal to metal contact. There is certainly no direct evidence that different oxides are formed under dry air and saturated air fretting conditions. Additional effects attributable to increased moisture content might include accelerated corrosive wear of the iron, and enhanced surface plasticity under wear conditions (13). The latter effect, known as the Rehbinder effect, is consistent with the fretted surface shown in figure 5.

Titanium showed the same general fretting wear trends with increased relative humidity as were observed for iron (fig. 8). Maximum fretting occurred at about 30 percent relative humidity, and the fretting wear under saturated air conditions was considerably higher than under dry air conditions. The appearance of the wear scars after fretting under various humidity conditions (fig. 9) did not change so dramatically as the wear scars on iron did. The dry air fretting contact surface was mostly smooth, with some pitting whereas fretting in saturated air resulted in a "leafy" heavily pitted surface as may be seen in figure 10. After 10^6 cycles of fretting in dry air, however, the contact surface on titanium began to show the degree of pitting and disruption seen after 3×10^5 fretting cycles in saturated air. From this observation it is concluded that part of the effect that moisture in the environment has on fretting of titanium is to accelerate surface fatigue. This is consistent with stress corrosion effects observed during the fatigue of titanium alloys in H_2O (14).

A minimum is observed at about 10 percent relative humidity in the fretting wear volume versus humidity curve for nickel, shown in figure 11. Slightly more fretting wear was observed under saturated air conditions than under dry air conditions. The wear scar on nickel after fretting in dry air was covered with dark oxidized debris, with only occasional spots showing evidence of metal to metal contact (fig. 12(a)), the situation resembling that observed on iron after fretting in 10 percent relative humidity air. Increasing the humidity resulted in a highly metallic appearing contact area, with extensive plastic working in evidence (fig. 12(b)), as was the case for iron under high humidity conditions.

Metallographic sections through fretting wear scars on nickel after exposure to fretting in dry air and saturated air are shown in figures 13(a) and (b) respectively. After fretting in dry air, surface pitting and roughening are in evidence with local regions of heavy plastic deformation and some areas of incipient spallation.

The results of fretting experiments conducted in a nitrogen environment are shown in figure 14, with the air results included for comparison. In all cases, for iron, nickel, and titanium, fretting wear in dry N_2 was about and order of magnitude lower than that in dry air. Introduction of moisture into the nitrogen environment resulted in a proportionally greater increase in fretting wear than did the introduction of moisture into air, especially for nickel and titanium.

The micrographs of figures 15 and 16 show the fretted surfaces resulting from experiments in nitrogen for nickel and titanium respectively. Comparing the two figures it is interesting to note that no loose debris was observed after fretting nickel in either dry or saturated nitrogen, whereas prodigious amounts of black debris were seen for titanium in both environments. Also, the extensively worked metallic appearance of the wear scar on nickel after fretting in saturated nitrogen (this type of wear scar was seen on iron as well) is very similar to that seen on iron and nickel after fretting in saturated air.

Fretting wear as a function of temperature is shown for iron, nickel, and titanium in figure 17. All three metals show a distinct reduction in wear as temperature is increased from 23° to 215° C. Increasing the temperature beyond 215° C for iron and titanium (330° C for nickel) results in increased fretting wear, expecially for titanium. As the temperature is increased beyond 500° C for titanium, however, a rapid reduction in fretting wear is observed.

The results obtained at 215° C are consistent with the observations of Hurricks (15) on the fretting of mild steel. Reductions in fretting wear are attributable to oxide film protection of the contacting surfaces, resulting in diminished metal-to-metal contact. Figure 18 shows the contact surface on nickel after fretting at 215° C. Similar features were seen on iron and titanium. Except for a rather large spalled region in the center of the wear scar, little material had actually been removed from the fretted surface.

The generally increased fretting wear observed to accompany further increases in temperature is not consistent with the overall results of Hurricks (15) for mild steel, nor with the observations of Feng and Uhlig (4). It is felt that the disagreements with Hurrick's results are due to two effects. First, the pure metals, particularly iron, used in the present study are susceptible to softening at elevated temperatures - more so than the mild steel used by Hurricks. Softening would lead to increased plastic flow of material under contact conditions, causing disruption of the oxide film. Further, the fretting frequencies employed in this study are more than 10 times as high as those used by Hurricks. The thickness attainable by a protective oxide film on a region of contact in the time interval between contact incidents would therefore be reduced. In fact, an oxidation wear mechanism as proposed by Quinn (16) would explain the increased rate of fretting wear as temperature is increased to 650° C for Ni and Fe, and to 500° C for titanium.

The dramatic decrease in fretting wear of titanium as temperature is increased beyond 500° C can be best understood in terms of the oxidation kinetics of titanium. Under static oxidation conditions, titanium undergoes a transition from relatively slow cubic kinetics to more rapid parabolic kinetics at a temperature somewhere between 500° and 600° C (17,18). In all cases the oxide formed is reported to be TiO_2. When rapid parabolic oxidation kinetics prevail, a thick dense oxide film fully capable of supporting the contact stresses is maintained in the contact area. Whatever wear occurs is completely within the oxide film - a significant departure from oxidative wear as proposed by Quinn (16). Indeed, the micrographs of figure 19 for titanium fretted surfaces generated at 650° C show almost a polished appearance, with the exception of cracks observed to form in the film. The metallographic section shown in figure 20 reveals no evidence of metal substrate distress under the fretting area.

It is concluded that Ni, which oxidizes slowly compared to titanium above 500° C, forming a soft easily worn oxide is unable to maintain a protective oxide film in the contact area under the fretting conditions imposed here. Iron, which begins fairly rapid parabolic oxidation above 325° C (19), forms a layered series of different oxides with considerable porosity; thus the oxide film is prone to spallation leading to oxidative wear as envisioned by Quinn. In contrast, titanium oxide film growth above 500° C is able to keep pace with fretting wear without spallation, resulting in essentially the low wear rate expected with TiO_2 surfaces in contact. Detailed considerations of this wear mechanism were developed in reference 20, and similar behavior was observed in the high temperature fretting of Ni-Cr-Al alloys (21).

CONCLUSIONS

Fretting wear measurements and experimental observations performed on iron, titanium, and nickel under varied test conditions support the following conclusions:

1. Under varied humidity, iron, and titanium showed maximum fretting wear at 10 and 30 percent relative humidity respectively. Nickel showed a minimum in fretting wear at about 10 percent relative humidity.

2. For iron and nickel, increased humidity had as its predominant influence increased debris mobility with some evidence of increased plasticity of the metal surface. For titanium, more rapid fatigue to the fretting surfaces was seen to accompany increased humidity levels.

3. All three metals showed substantially reduced fretting in dry N_2 compared to dry air. Admitting moisture to the N_2 environment resulted in a proportionally greater increase in fretting than was observed when fretting was conducted in saturated air, through under saturated conditions the amount of fretting wear was still less in N_2 than in air.

4. All three metals showed reductions in fretting wear as temperature was increased from 23° C to about 200-300° C. Further increases in temperature to 650° C however lead to increased fretting wear for iron and nickel. Titanium however showed increased fretting wear as temperature was increased to 500° C, beyond which sharply reduced fretting wear was observed.

5. Reduced metal to metal contact due to a thin oxide film formation is assumed to lead to the initial decrease in fretting with increased temperature. Thereafter, fretting wear is envisioned to proceed by wearing away and disruption of ever thicker oxide films, causing increased wear with temperature. Finally, for titanium, oxidation kinetics become rapid enough above 500° C for the oxide film to fully support the contact and fretting takes place entirely on a TiO_2 surface of good integrity.

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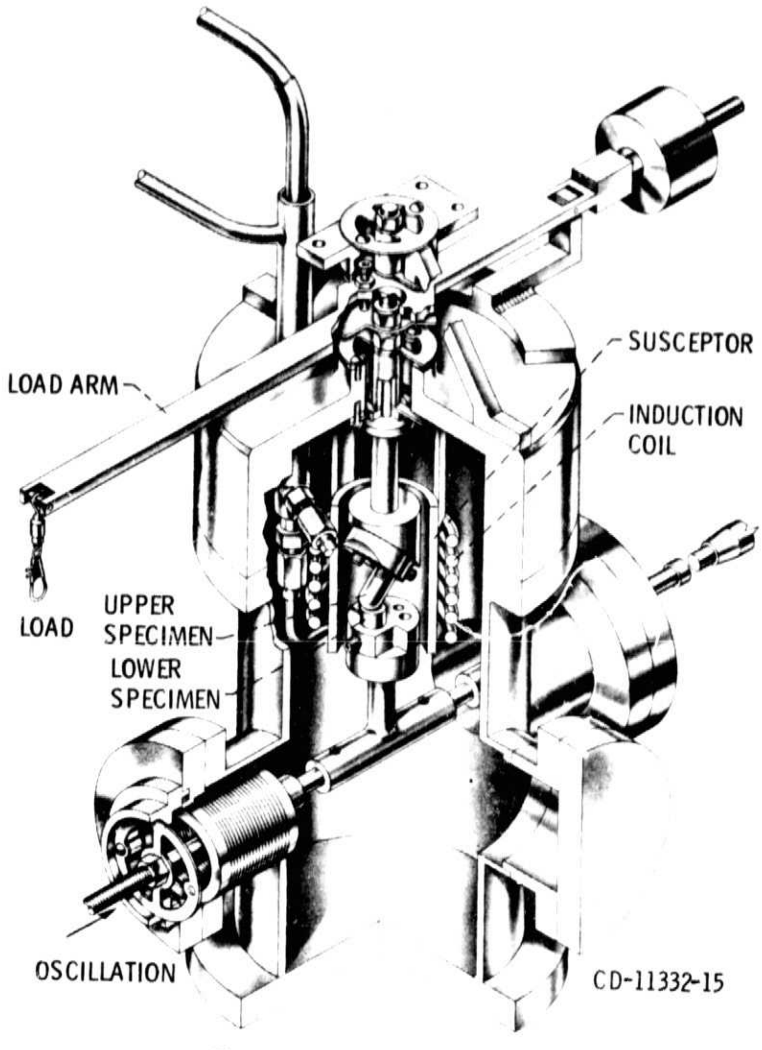


Figure 1. - Fretting apparatus.

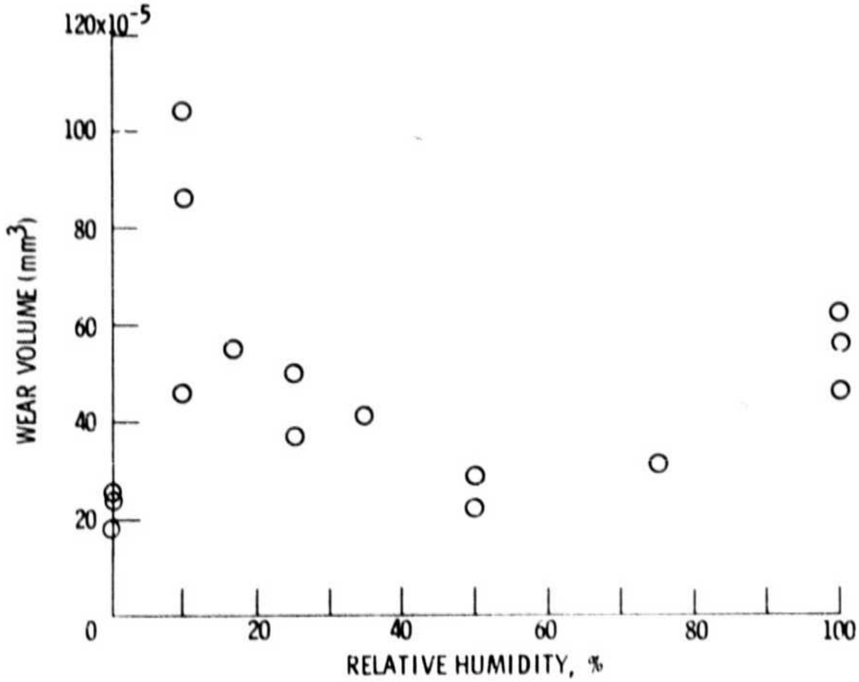
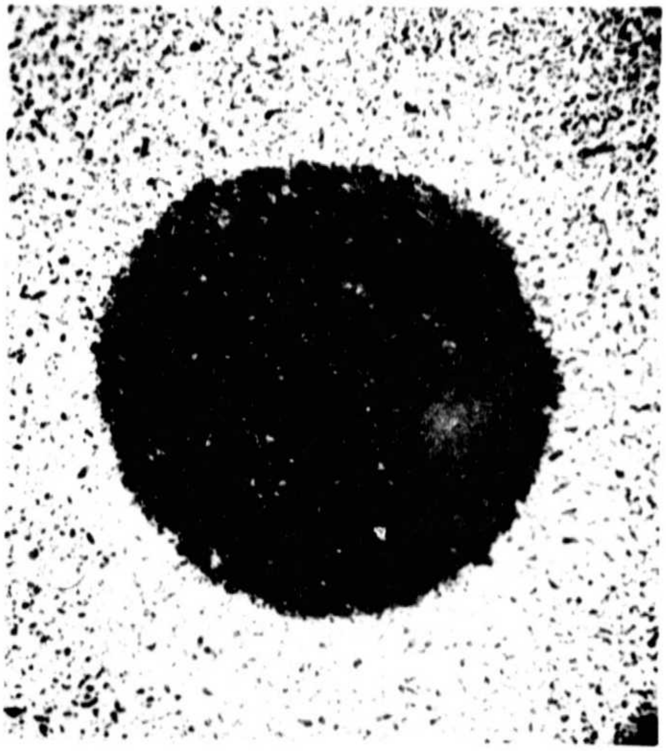


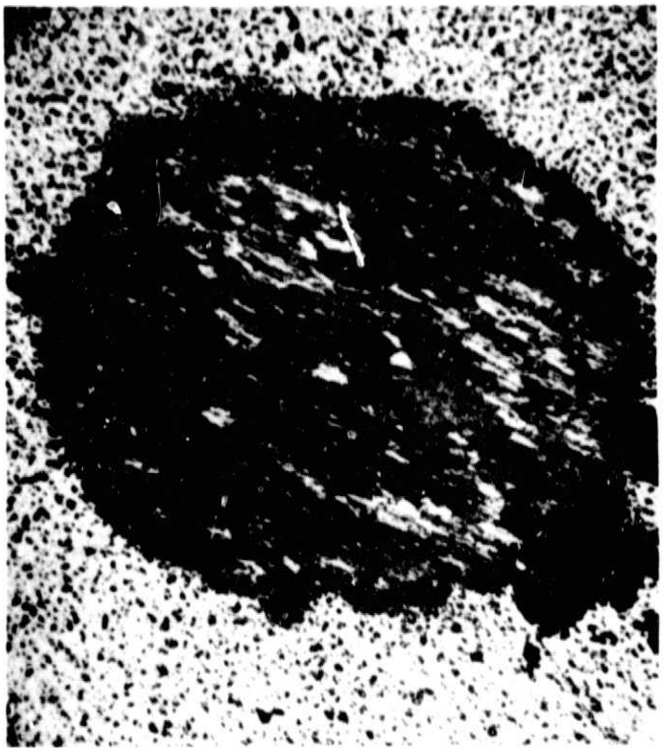
Figure 2. - Fretting wear volume as a function of relative humidity for 99.9% iron.



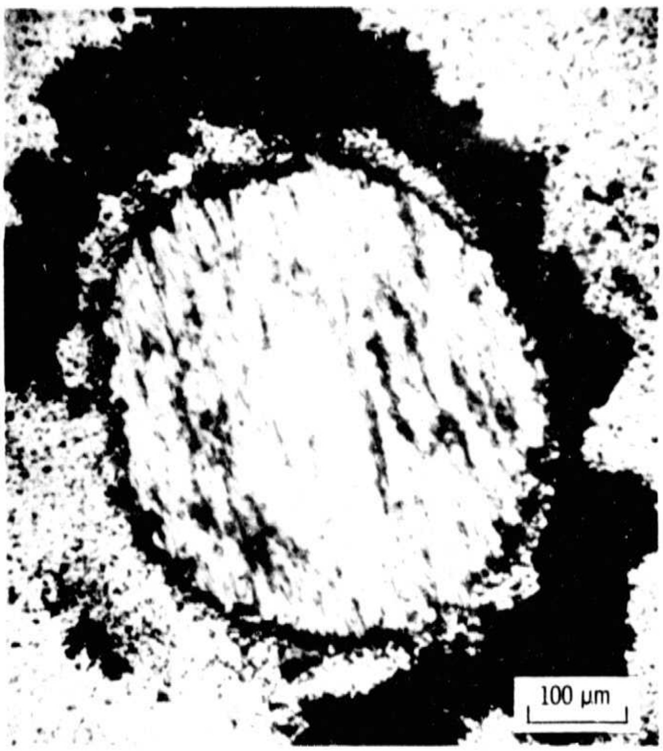
(a) DRY AIR.

(b) 10 PERCENT RELATIVE HUMIDITY.

Figure 3. - Fretting wear scars on high purity iron after $3x10^5$ fretting cycles in air under indicated humidity conditions.

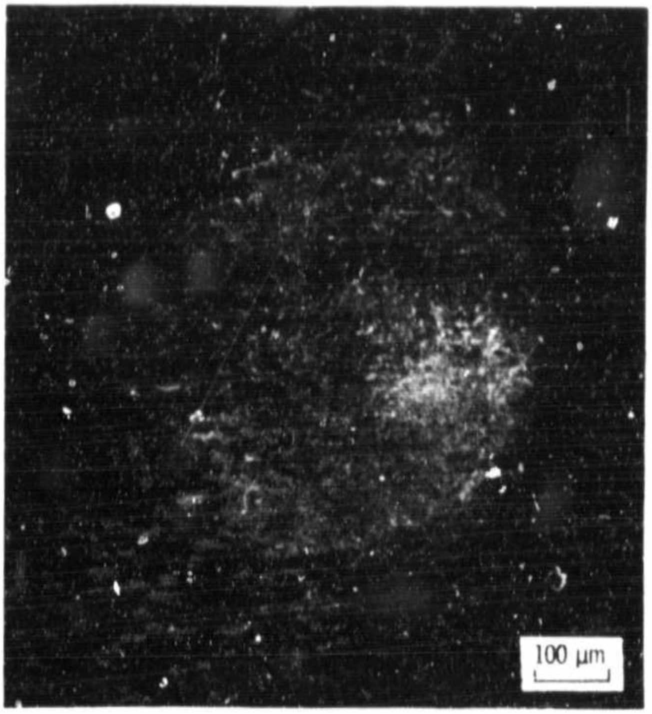


(c) 35 PERCENT RELATIVE HUMIDITY.

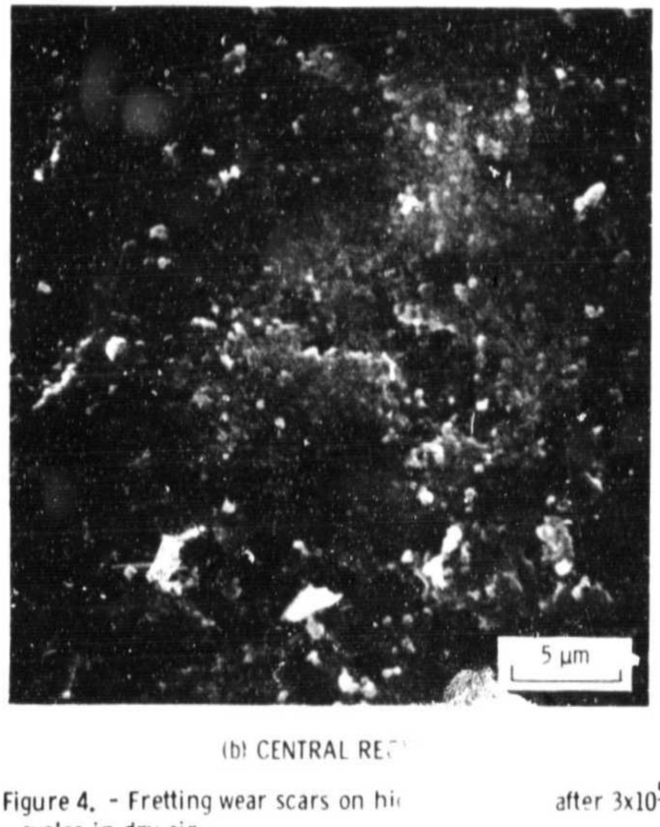


(d) SATURATED AIR.

Figure 3. - Concluded.

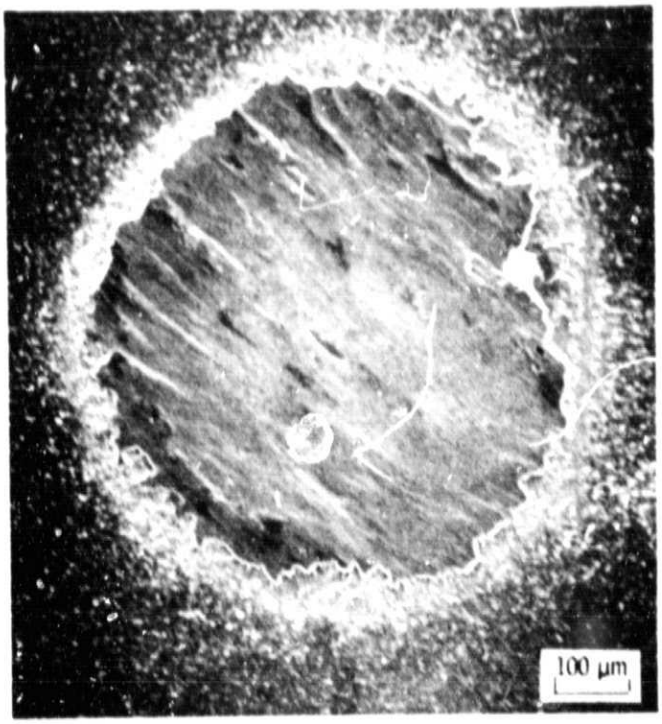


(a) OVERVIEW.

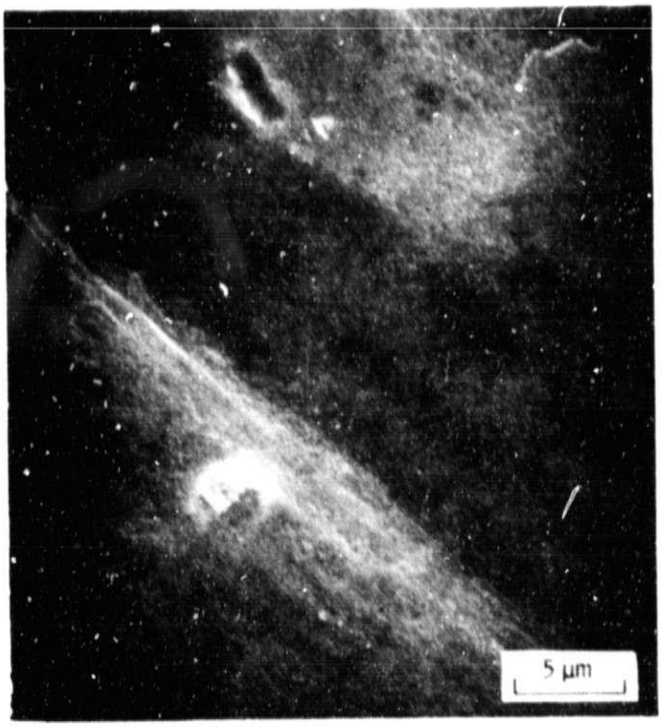


(b) CENTRAL RE

Figure 4. - Fretting wear scars on hi after 3×10^5 cycles in dry air.

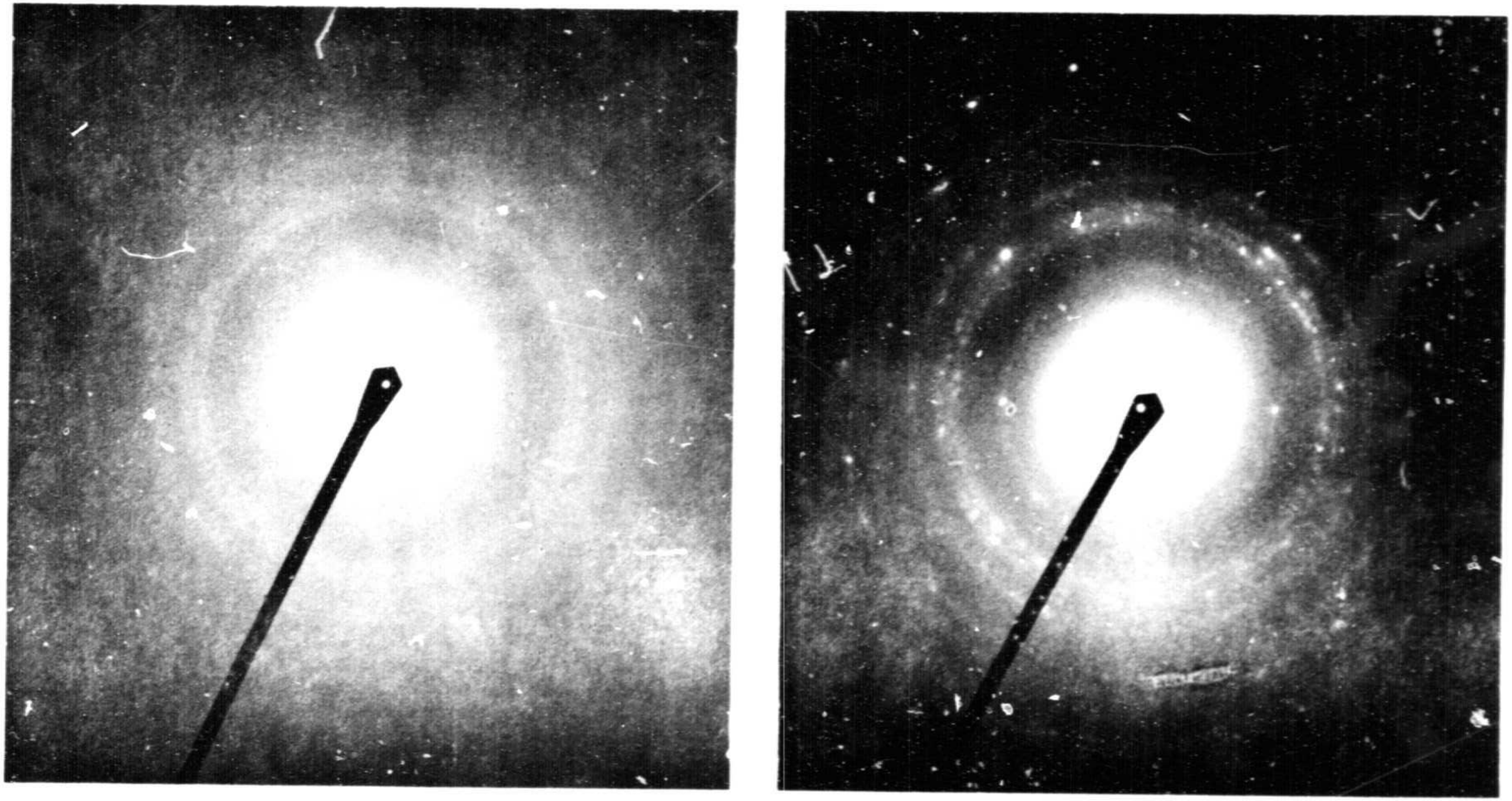


(a) OVERVIEW.



(b) CENTRAL REGION.

Figure 5. - Fretting wear scars on high purity iron after $3x10^5$ cycles in saturated air.

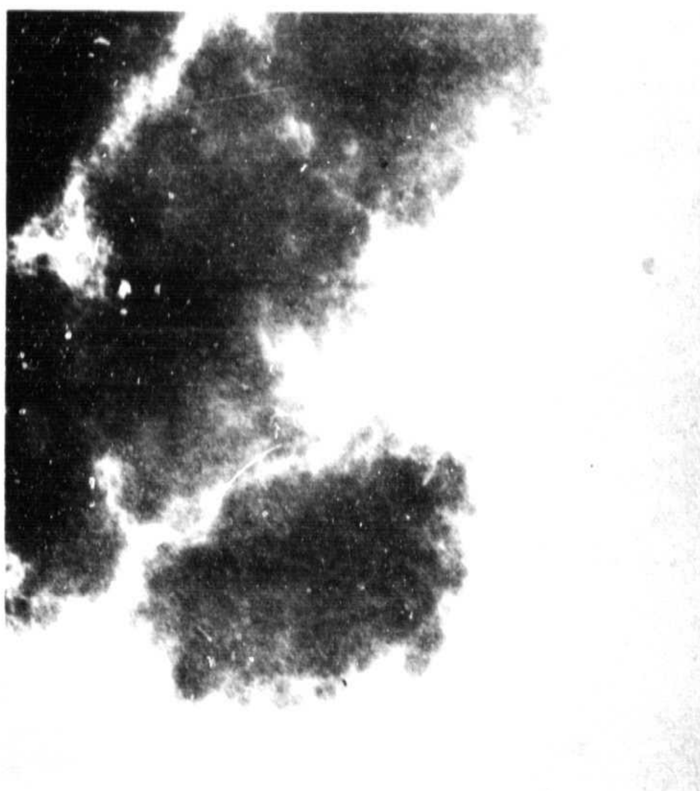


(a) DRY AIR.

(b) SATURATED AIR (AND 50-80 PERCENT RELATIVE HUMIDITY).

Figure 6. - Electron diffraction patterns of fretting debris resulting from fretting of iron in dry air and saturated air (pattern for 50-80 percent relative humidity was like that for saturated air).

(a) DRY AIR.



(b) SATURATED AIR.

Figure 7. - TEM micrographs of fretting debris resulting from

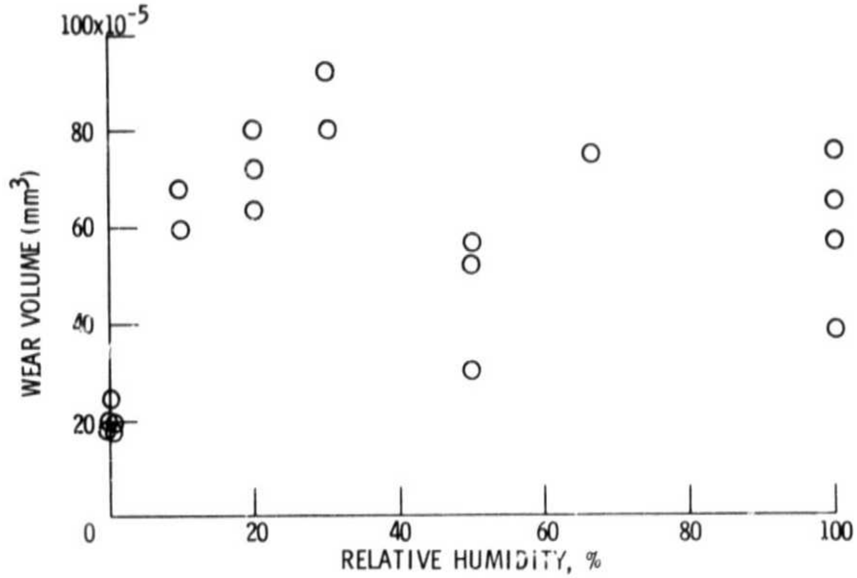


Figure 8. - Fretting wear volume as a function of relative humidity for 99.9% titanium.

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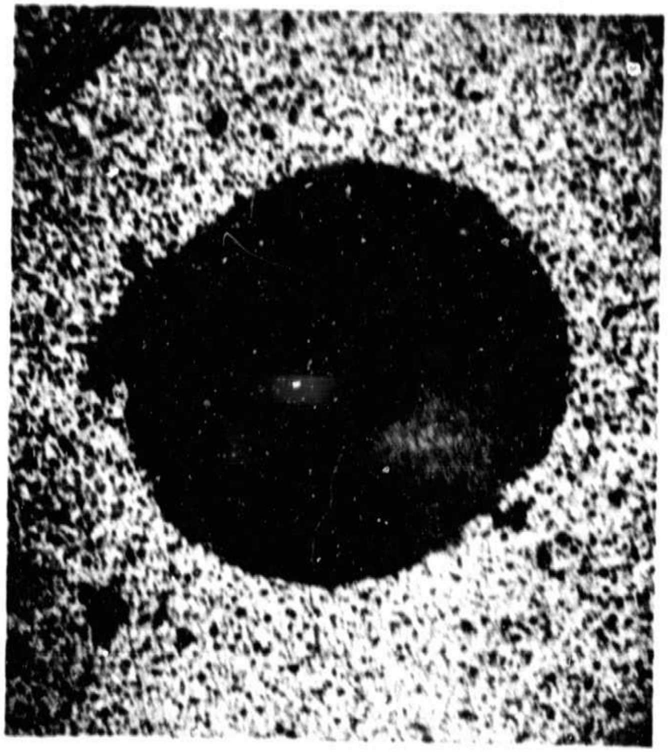
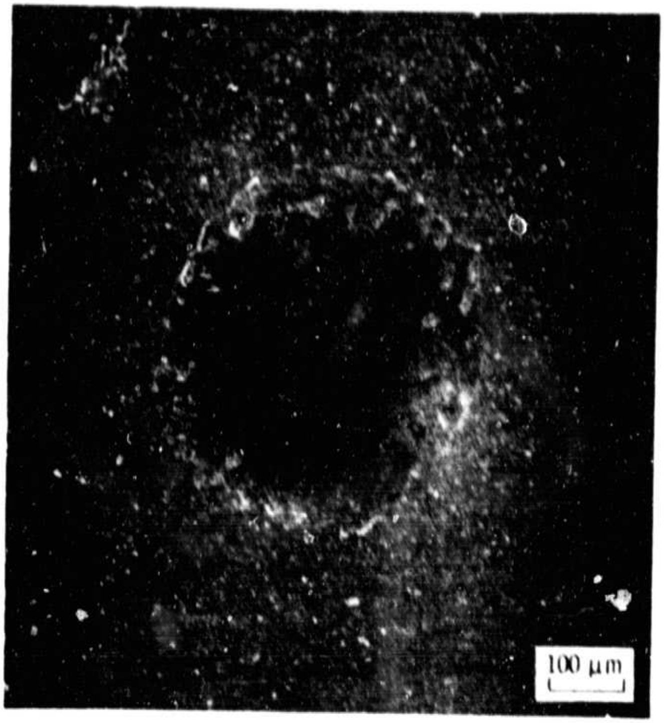


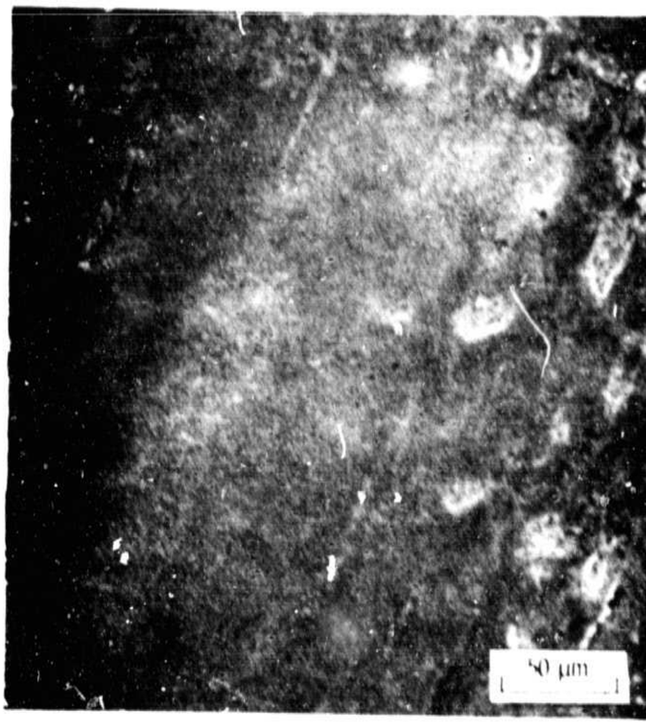
(a) DRY AIR.

(b) SATURATED AIR.

Figure 9. - Fretting wear scars on high purity titanium after 3×10^5 fretting cycles in dry air and in saturated air.



(a) DRY AIR, OVERVIEW.



(b) DRY AIR, CENTRAL REGION.

Figure 10. - SEM photographs of fretting wear scars on high purity titanium after 3×10^5 cycles in dry air and saturated air.

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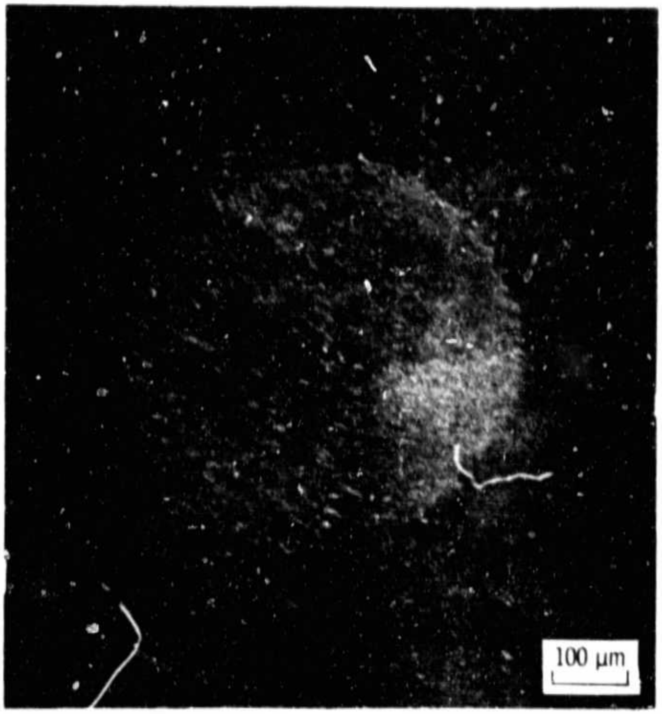
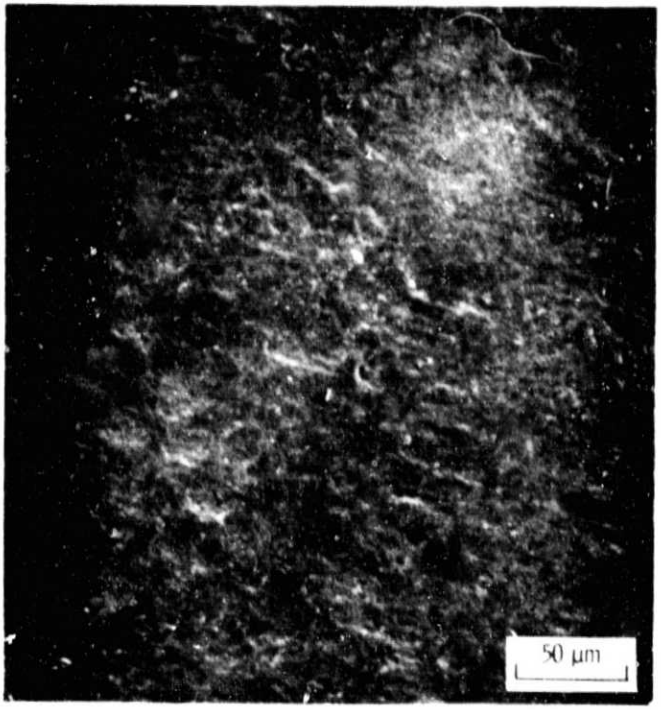


(c) SATURATED AIR, OVERVIEW.



(d) SATURATED AIR, CENTRAL REGION.

Figure 10. - Concluded.

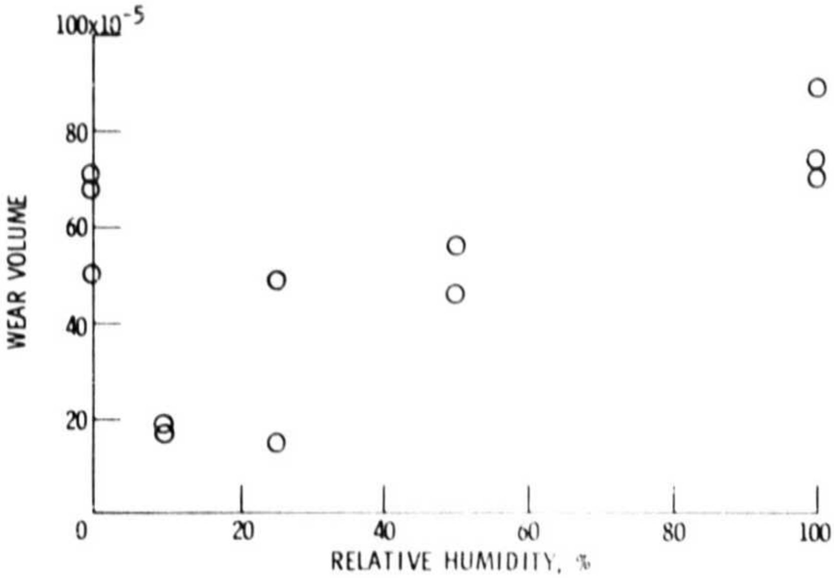
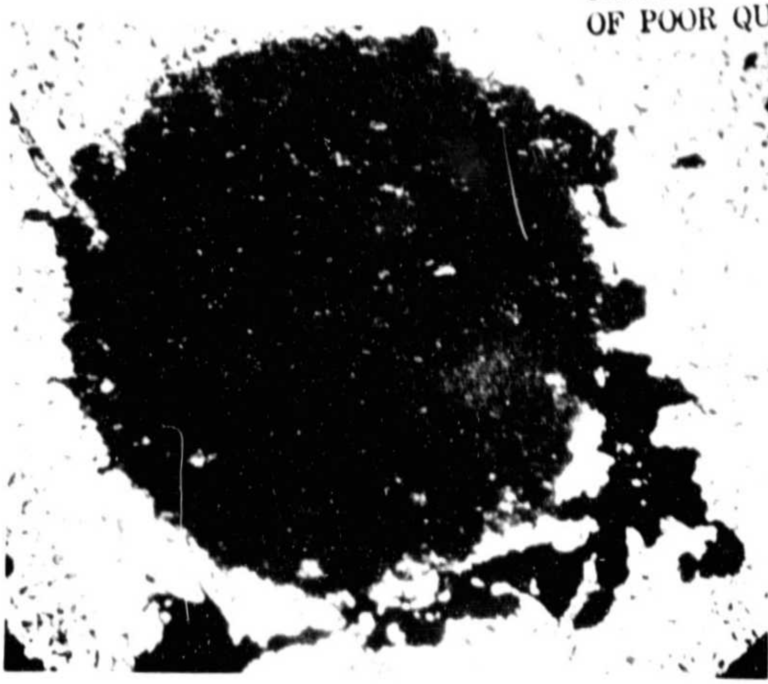
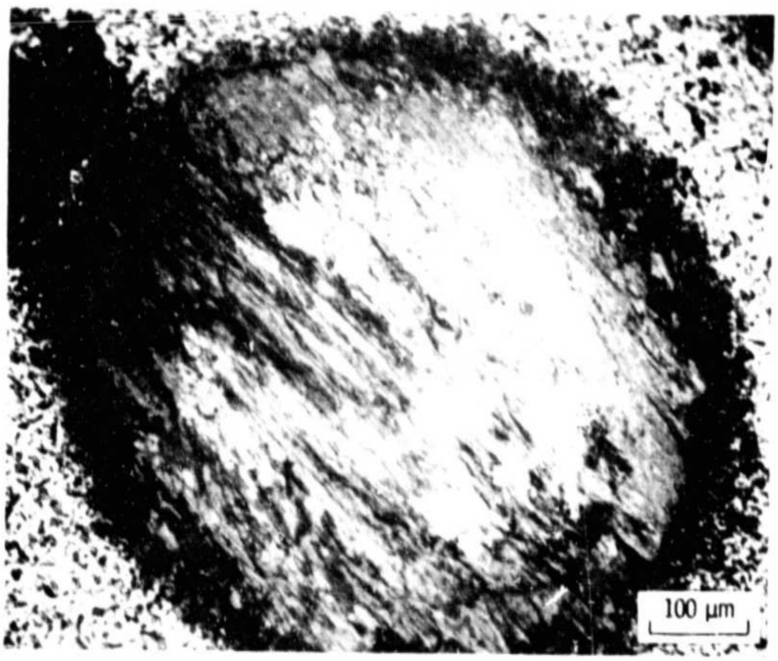


Figure 11. - Fretting wear volume as a function of relative humidity for high purity nickel.

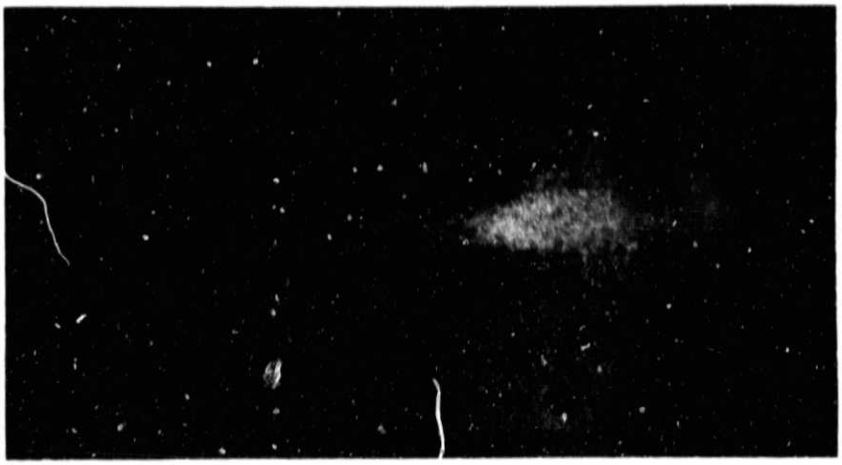


(a) DRY AIR.

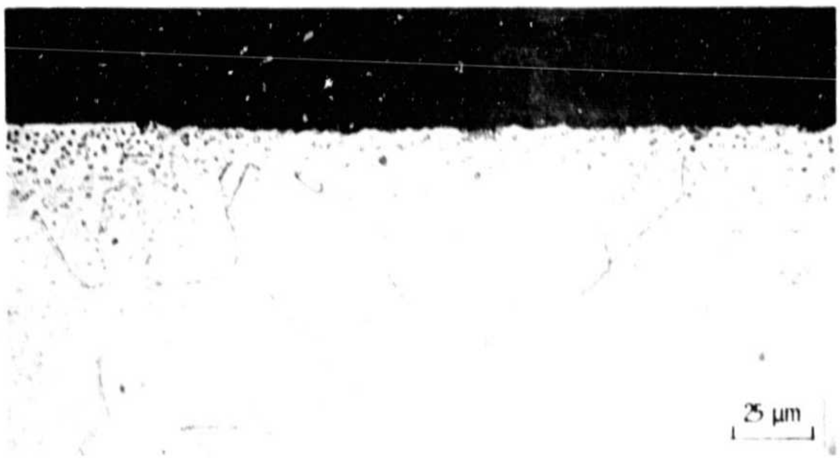


(b) SATURATED AIR.

Figure 12. - Fretting wear scars on high purity nickel after $3x10^5$ cycles.



(a) DRY AIR.



(b) SATURATED AIR.

Figure 13. - Metallographic sections through fretting wear scars on high purity nickel after 3×10^5 cycles in dry air and saturated air.

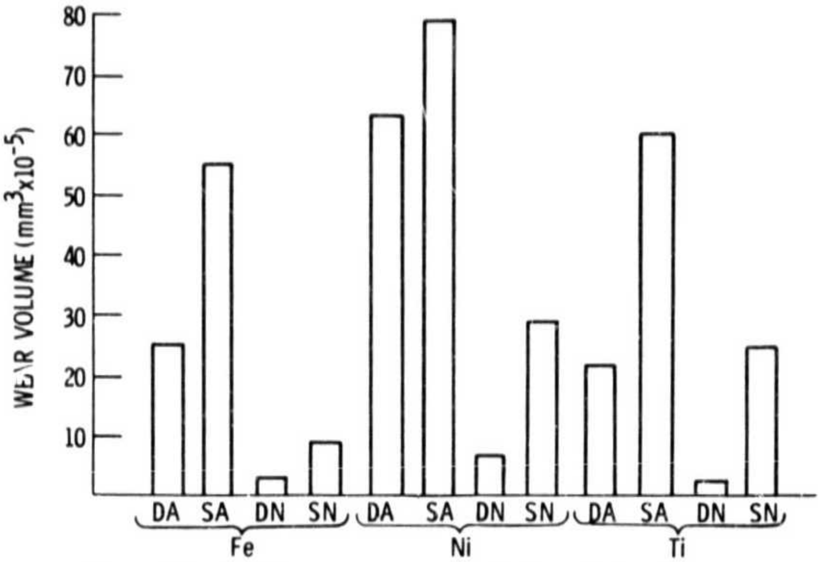
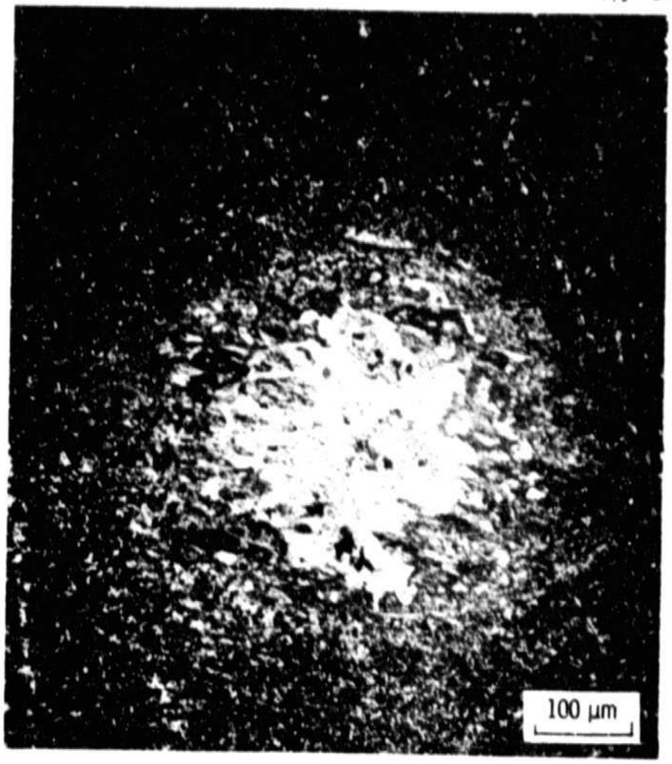
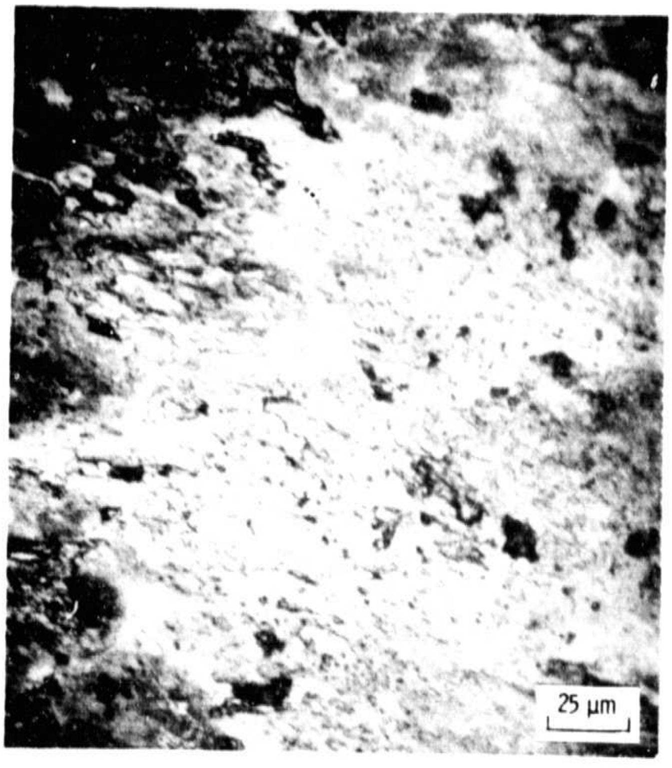


Figure 14. - Fretting wear volume for high purity Fe, Ni, and Ti after 3×10^5 fretting cycles in dry air (DA), saturated air (SA), dry nitrogen (DN), and saturated nitrogen (SN).



(a) DRY NITROGEN, OVERVIEW.



(b) UPPER LEFT QUADRANT OF (a).

Figure 15. - Fretting wear scars on high purity nickel after $3x10^5$ fretting cycles in dry nitrogen and saturated nitrogen.

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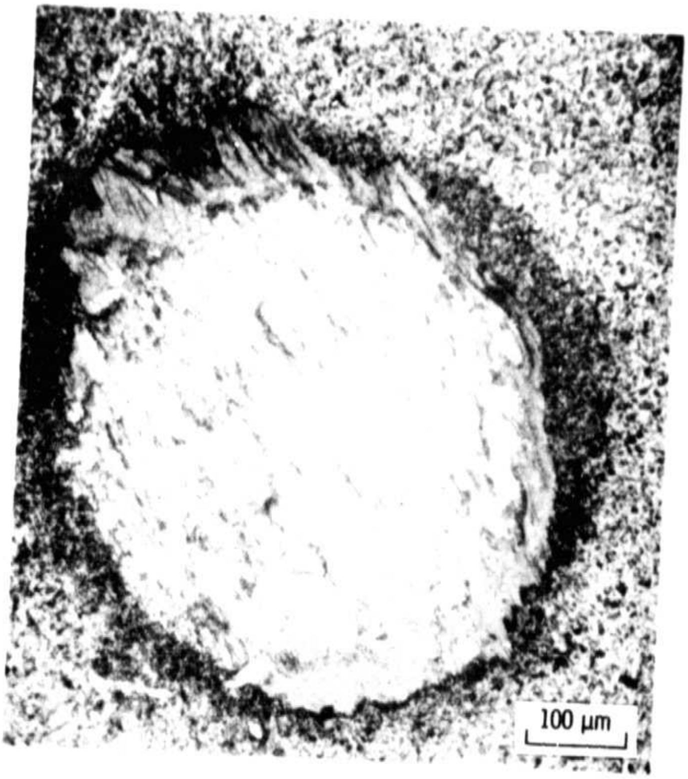
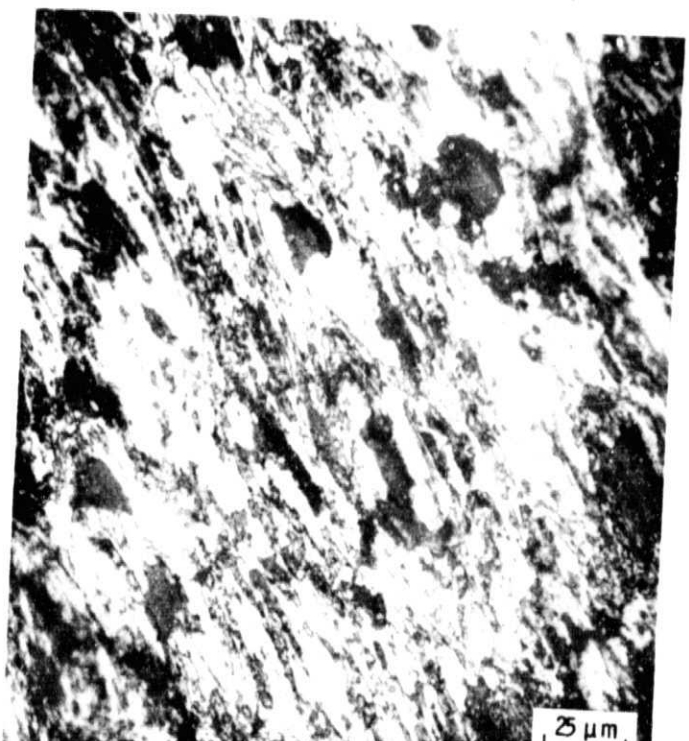
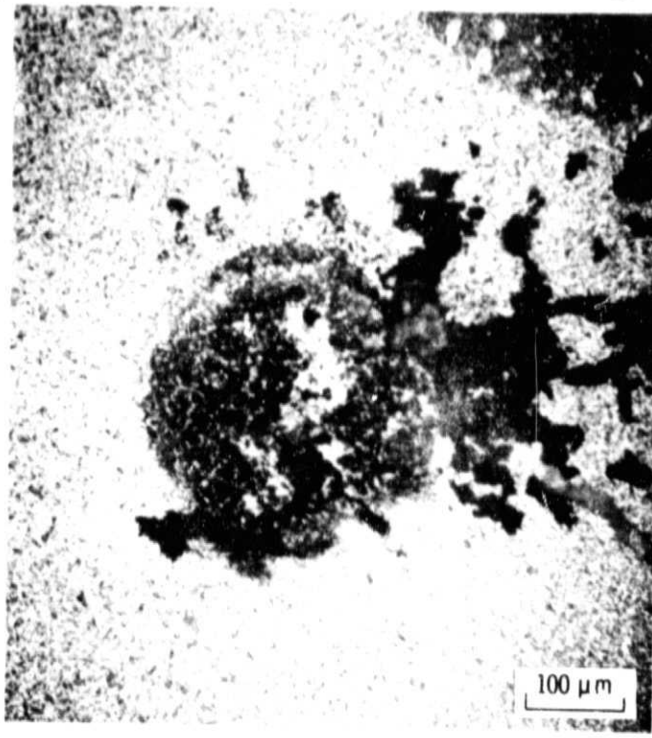


(c) SATURATED NITROGEN, OVERVIEW.

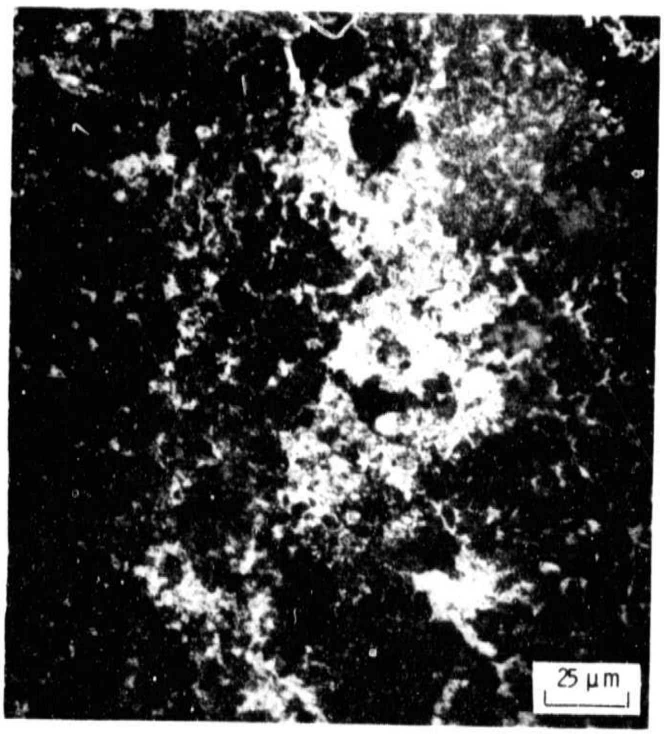


(d) CENTRAL REGION OF (c).

Figure 15. - Concluded.

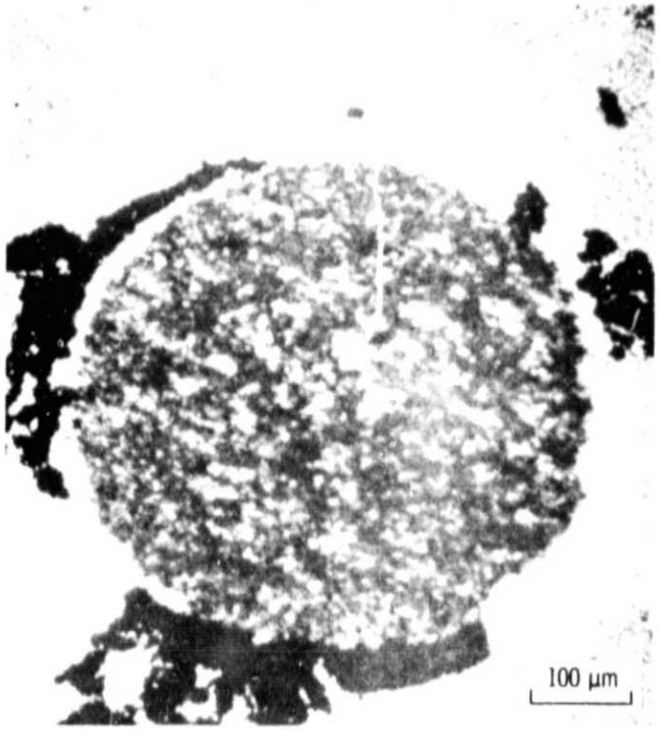


(a) DRY NITROGEN, OVERVIEW.

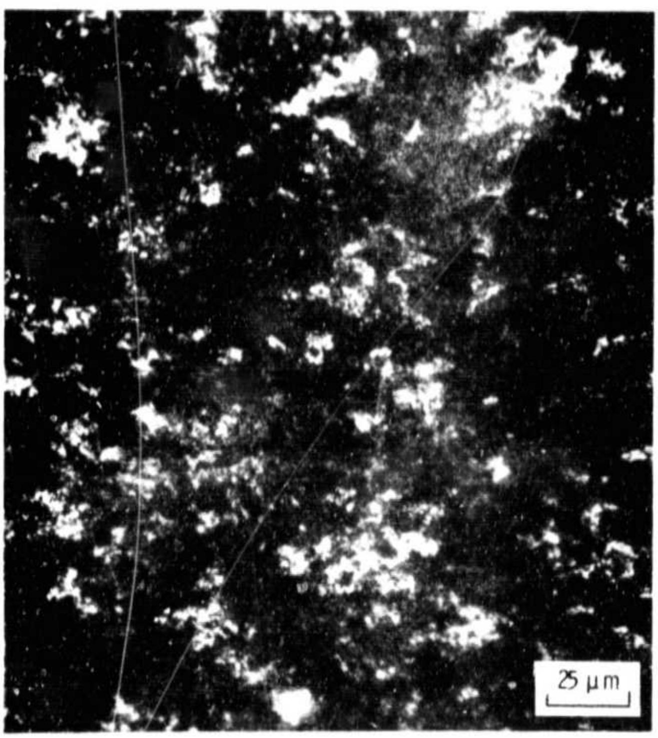


(b) CENTRAL REGION OF (a).

Figure 16. - Fretting wear scars on high purity titanium after $3x10^5$ fretting cycles in dry nitrogen and saturated nitrogen.



(c) SATURATED NITROGEN, OVERVIEW.



(d) CENTRAL REGION OF (c).

Figure 16. - Concluded.

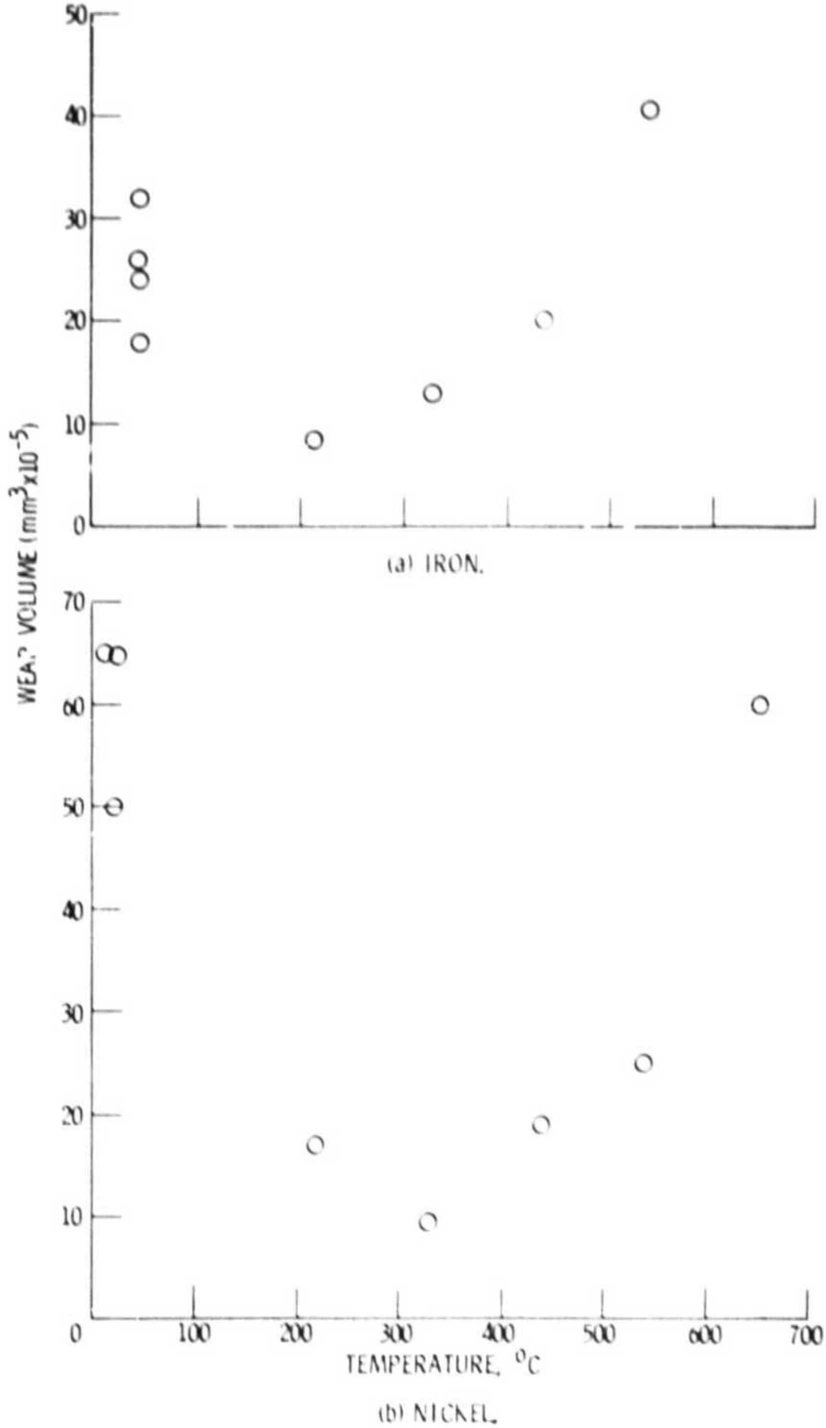
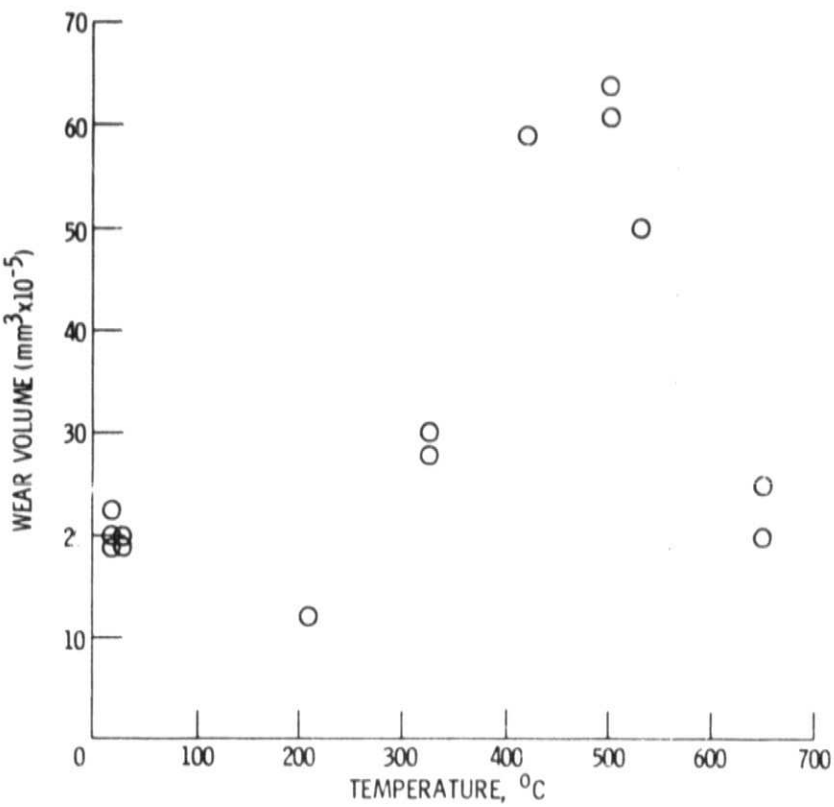
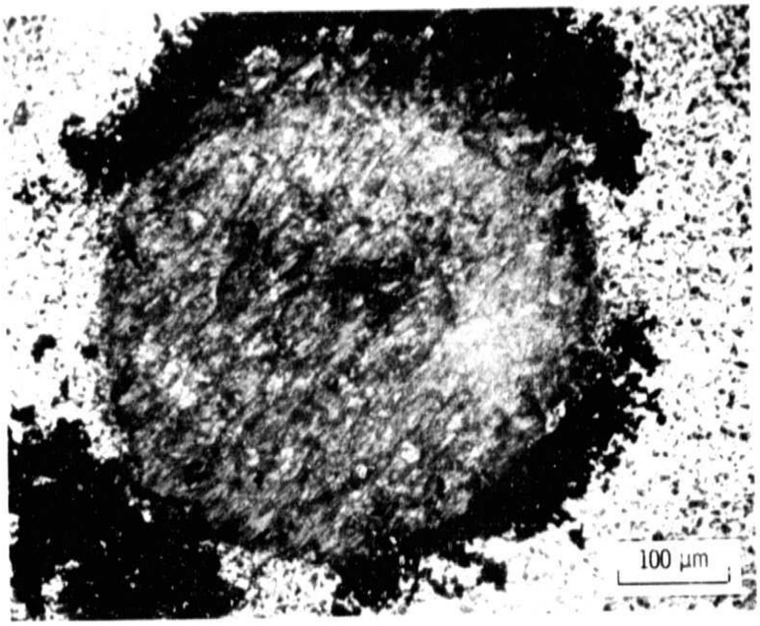


Figure 17. - Fretting wear volume as a function of temperature for high purity iron, nickel, and titanium, after $3x10^5$ fretting cycles in dry air.

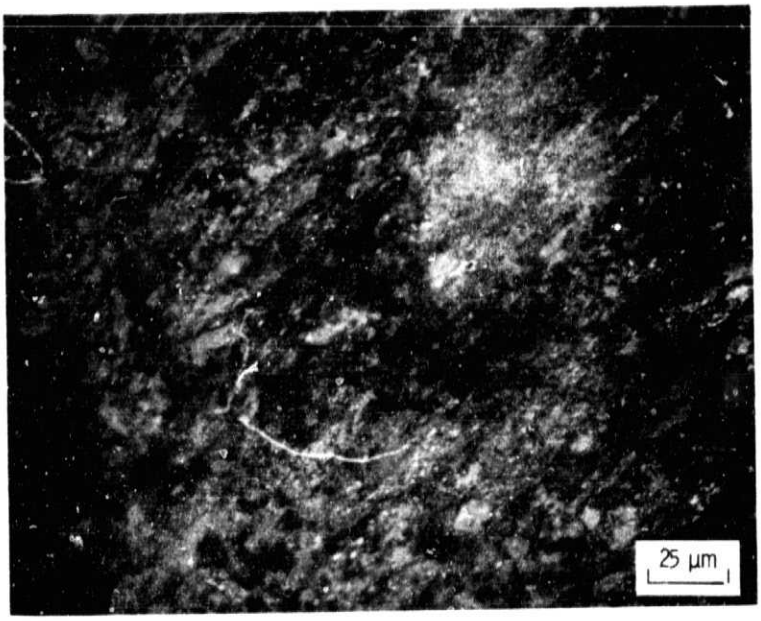


(c) TITANIUM.

Figure 17. - Concluded.

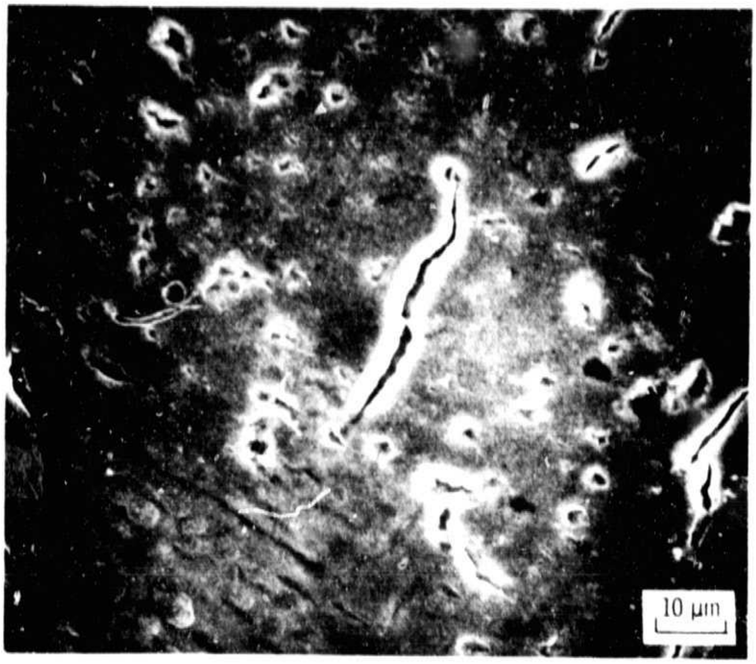


(a) OVERVIEW.

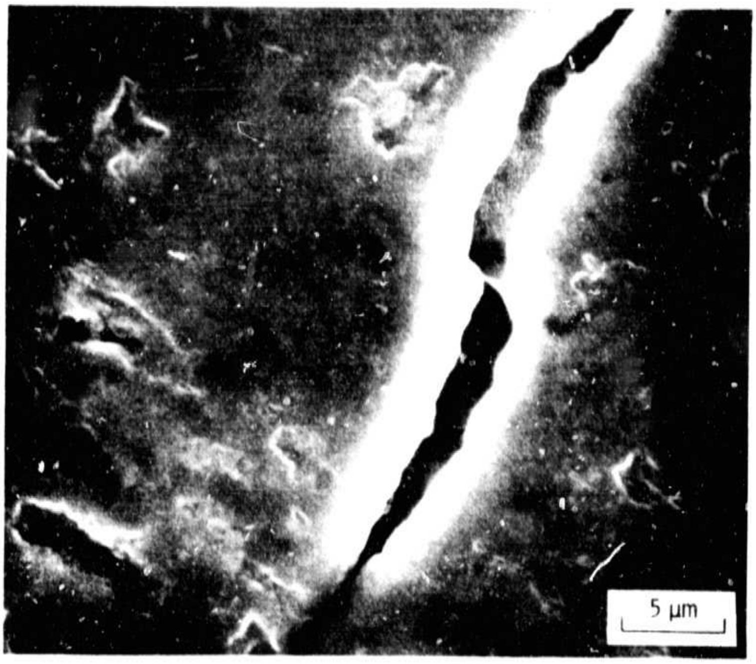


(b) CENTRAL REGION SHOWING SPALL PIT.

Figure 18. - Fretting wear scar on high purity nickel after 3×10^5 cycles at 215^0 C.



(a) OVERVIEW.



(b) CENTRAL REGION OF (a).

Figure 19. - SEM photograph of fretting wear scar on high purity titanium after 3×10^5 cycles at 650^0 C.

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OF POOR QUALITY

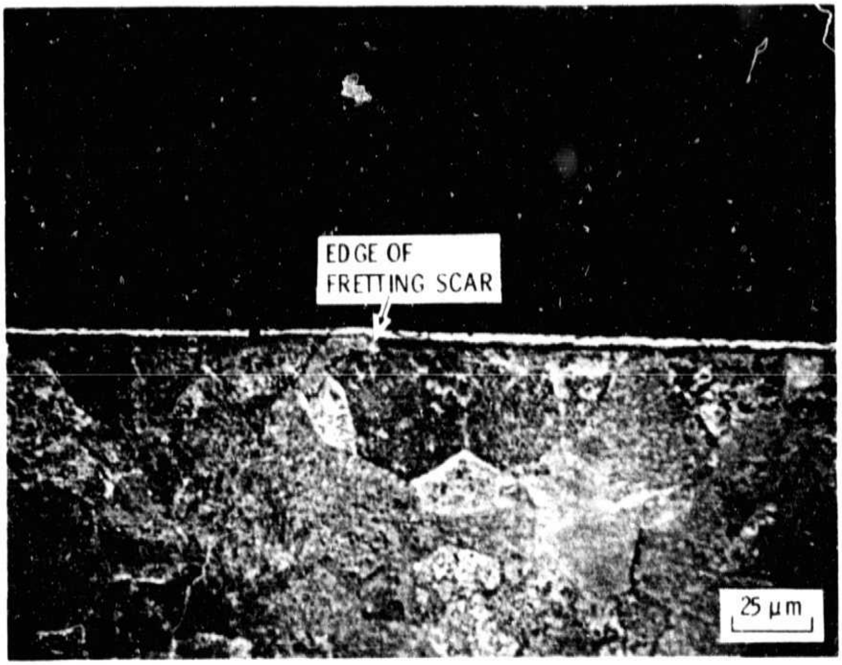

Figure 20. - Section through the fretting wear scar on high purity titanium, resulting from $3x10^5$ fretting cycles at 650^0 C.