THE "BREAKING-IN" OF LUBRICATED SURFACES*

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Summary

Steel cylinders were slid against flat steel surfaces, which were prepared to various surface roughness values, in order to determine the mechanisms of "break-in" of lubricated surfaces. It was found that proper break-in of a surface is effected when a film of Fe_3O_4 about 400 Å thick forms. The formation of Fe_2O_3 is to be avoided. In addition, it was found in laboratory experiments that proper breaking-in by sliding requires a specific initial surface roughness of about 0.1 μm center-line average. Smoother and rougher surfaces failed quickly. It appears that the optimum surface roughness was one in which the asperities plastically deformed at a rate that was too slow for fast progression to low cycle fatigue failure but at a rate sufficient to accelerate the formation of oxides.

1. Introduction

Lubricated machine surfaces will usually operate successfully from the newly manufactured state if the severity of contact (some combination of sliding speed and contact pressure) is restricted. A surface can, however, be made to sustain high severity loading if it is first operated at low severity loading for a short time. This early stage of operation is called breaking-in (or running-in or wearing-in) and it has been found in this work to involve some alteration of the surface topography together with the formation of protective films of Fe_3O_4 [1].

The consequence of high severity loading without breaking-in is that surfaces roughen very quickly, sometimes to the point of visible adhesion between the sliding members. This is called scuffing or scoring [2 - 7]. Frequently, as in the case of gasoline and diesel engines, specific compounds are added to lubricants to decrease the probability of scuffing. The mechanism whereby these additives protect the surfaces is not known. Their

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chemical reactions are evidently very complex as may be seen by the extensive discussions in the literature. This study had as its goal the measurement of the physical kinetics of the formation of protective films and the relation of these to scuff prevention performance. The main interest was in the formation of oxides when using mineral oil as the lubricant, but some tests using water as a lubricant produced some results that might explain the wear of water-lubricated steels.

2. Experimental details of tests

There are many wear tests described in the literature but there are few guidelines on how to study scuffing, or its obverse, breaking-in. Thus several test geometries and many experimental variables were tried before a reliable test was found. The final experimental device is shown in the line drawing in Fig. 1. The goal in designing the test was to achieve reproducible results for
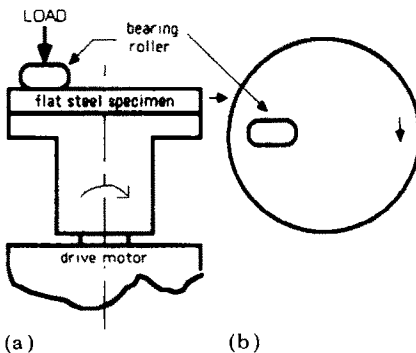

Fig. 1. Sketch of the cylinder-on-flat tribological apparatus: (a) side view; (b) top view.

conditions near those in some practical applications. The final device uses a rotating flat plate of type 1018 steel of hardness 10 HRC on which was loaded a stationary cylinder, oriented radially relative to the rotation of the plate, and both were immersed in liquid. With this geometry it is possible to calculate a fluid film thickness and thereby to approximate deliberately the conditions of inadequate lubrication. The sliding speed range of the apparatus is from 0.05 to 2.5 m s^{-1} and the load can be varied from 20 to 400 N. The stationary cylinder is a roller taken from a rolling element bearing (hardness, 60 HRC) 6.25 mm in diameter and 7.3 mm long. The friction force between the specimens is measured by strain gauges attached to a cantilever holding the stationary specimen.

At various stages in the tests the surface roughness was measured using a Bendix tracer and amplifier. The analog output from the amplifier was converted to digital form, from which several statistical parameters were calculated, including the asperity height distribution in terms of the arith-

metic average and r.m.s. height, the skew, the kurtosis and the correlation length.

The method used to observe the rate of formation of surface films was that of ellipsometry [8]. This method was considered to be the best available to observe the developing film in real time and in real atmospheres, *i.e.* as the film forms and as it is removed. Raman spectroscopy, IR spectroscopy, scanning electron microscopy and Auger electron spectroscopy (AES) were also used.

3. First observations

Many tests were run at various speeds and loads and using various types of flat surface preparation. Mineral oil of 125 - 135 cSt was usually used as a lubricant. Most tests began with smooth sliding and low friction, and they were continued to the point of high friction and considerable surface damage. Tests with low loads and moderately high speed would run a very long time (days) without surface failure. In the low load tests and in the early moments of the high load tests the coefficient of friction was 0.06. A series of tests was then run with loads that produced failure in the range from 5 to 120 min. In these tests a dark film appeared on the flat plate where sliding occurred. Scratches later appeared in the dark film and the dark film generally became thinner. Surface failure usually followed soon after.

It was first assumed that the dark film was the precursor to failure. A study of the scratches revealed that they were caused by metallic particles that developed by accumulation of very fine wear debris. At some point the particles accumulated in the interface between the cylinder and the flat plate, plowing a groove. Some scratches steadily broadened, others did not. Separation of the cylinder from the flat plate by large debris particles usually dislodged most of the debris and altered the scratch propagation sequence.

Later study showed that a dark film eventually appeared also at light loads where surface failure did not occur. These films became progressively darker with sliding. It then became obvious that the dark film accompanied breaking-in or, in the experiments using a high load from the start, that the dark film began to form but was removed by scratching. The dark film clearly produced unique effects. A major effort therefore was devoted to identify the composition of the film. It was dark in color and shiny, leading to the speculation that carbon and/or "friction polymer" may be its major constituent.

To check the above hypothesis some tests were run using water as the lubricant. The load for these tests was one-quarter that of the constant load tests using oil, to compensate for the lower viscosity of water. In these tests the cylindrical specimen wore away at a high rate and the coefficient of friction was about 0.5. Again, a film was formed but this film was not as dark or shiny as those in the tests using mineral oil. Surface films therefore are not necessarily composed of carbon or friction polymer.

4. Film identification

The first method used to identify the composition of the films was Raman spectroscopy. A strong spectrum for Fe_2O_3 was seen when irradiating the water-lubricated surfaces. No absorption peaks could be seen when irradiating the surfaces tested in oil.

The second method used was X-ray diffraction. Both the oil- and the water-lubricated surfaces appeared to be covered with oxide primarily. However, the coatings were not thick enough to obtain reliable X-ray results. These tests raised the possibility that the oil-lubricated surfaces may be covered by Fe_3O_4, which absorbs light and may thus make it impossible to obtain absorption peaks by the Raman method.

The third method used was IR spectroscopy. These tests confirmed the presence of Fe_2O_3 on the water-lubricated surfaces but could not identify the coating on the oil-lubricated surface. A search of the literature for an IR absorption spectrum for Fe_3O_4 produced none. Apparently Fe_3O_4 is an excellent absorber of IR radiation as well as of visible light.

It was necessary to resort to AES to identify the coatings. Before the specimens were placed in the AES instrument, the surfaces were washed in mild solvents. Figure 2 is a broad AES spectrum showing the presence of iron, oxygen and carbon on a surface lubricated in oil. The spectrum for the water-lubricated surface is difficult to distinguish from that shown in Fig. 2. Expanded spectra for iron are shown in Fig. 3. Figure 3, spectrum a, is for laboratory grade Fe_2O_3 on a copper screen and Fig. 3, spectrum b, is
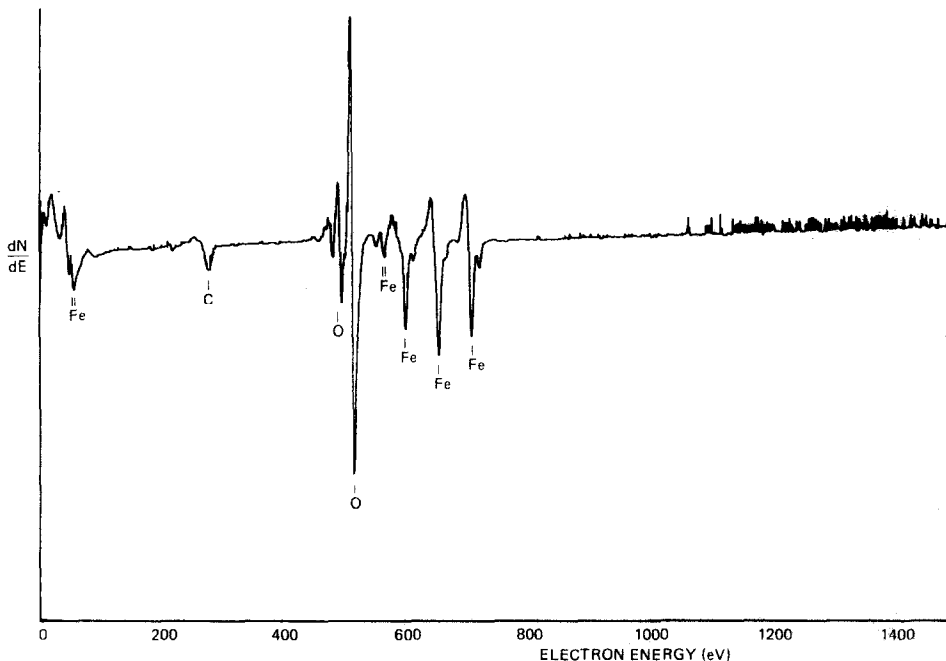

Fig. 2. Broad Auger spectrum for the surface of the oil-lubricated steel. (Chlorine and sulfur were also detected but are not shown on this spectrum.)
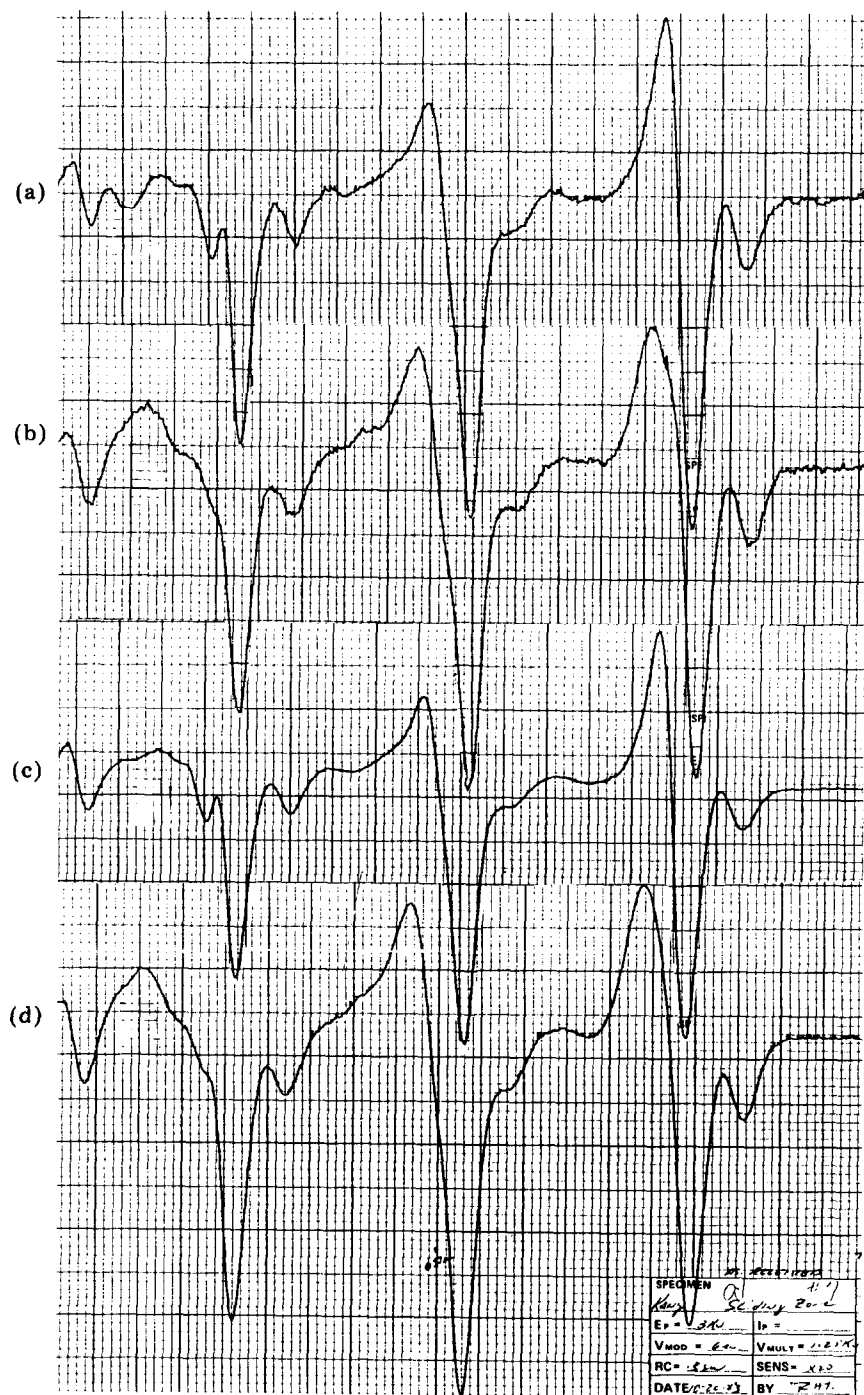
Fig. 3. Expanded Auger spectra for the iron content in four specimens: spectrum a, laboratory grade Fe_2O_3 on copper; spectrum b, laboratory grade Fe_3O_4 on copper; spectrum c, the film on the water-lubricated steel; spectrum d, the film on the oil-lubricated steel. It should be noted that the spectra were plotted with inadequate attention to the calibration relative to the position of the abscissa.

for laboratory grade Fe_3O_4 on a copper screen. Some small but consistent differences can be seen betweeen the two. Figure 3, spectrum c, is for a water-lubricated surface. It is seen to be more similar to spectrum a than to spectrum b. Again, Fig. 3, spectrum d, is for the oil-lubricated surface. It is more similar to spectrum b than to spectrum a. These analyses were repeated several times confirming a view that the film on the oil-lubricated surface is primarily Fe_3O_4 whereas that on the water-lubricated surface is primarily Fe_2O_3. Very little carbon was found in the surface film. An Auger analysis of the debris from the oil-lubricated system showed considerable carbon, however.

Figures 4 and 5 are derived from Auger spectra at various depths into the surfaces. The curves are plots of the intensity of three elemental peaks for $C(272 eV)$, $O(510 eV)$ and $Fe(703 eV)$ as a function of depth into the film and substrate. Penetration was achieved by ion milling at about 100 Å min^{-1}. Figure 4 is for the oil-lubricated surface and Fig. 5 is for the water-lubricated surface. Figure 6 shows the ratios of oxygen to iron taken from Figs. 4 and 5, plotted against the time of ion milling. From Fig. 6 it would be difficult to identify readily the oxides, but it is clear that the film on the
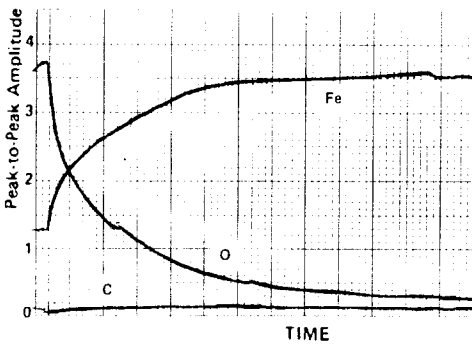

Fig. 4. The intensity variation in the peaks for carbon, oxygen and iron in the Auger spectra at various depths into the surface film from the oil-lubricated surface (time axis scale, 10 min per division).
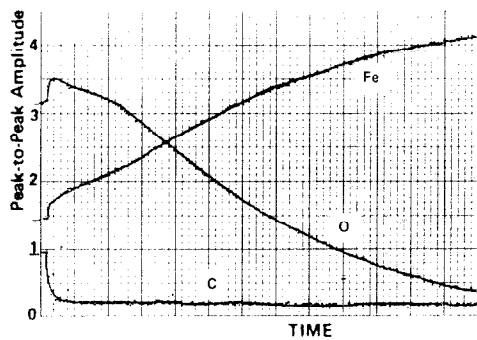
Fig. 5. The intensity variation in the peaks for carbon, oxygen and iron in the Auger spectra at various depths into the surface film from the water-lubricated surface (time axis scale, 20 min per division).
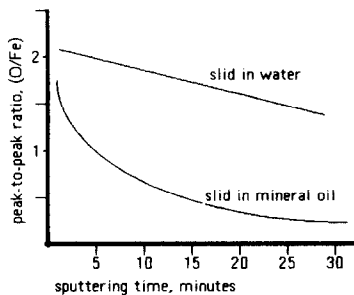

Fig. 6. The ratio of Auger spectra for $O(510 eV)$:$Fe(703 eV)$.

oil-lubricated surface is thin compared with the Fe_2O_3 film thickness. We estimate that the effective film thickness of Fe_3O_4 is between 250 and 500 Å whereas the Fe_2O_3 film is very much thicker.

Both films were photographed using a Francon polarizing interference microscope objective (25X). The original photographs were in color, but the black-and-white copy of the Fe_3O_4 film is shown in Fig. 7(a) and that of the Fe_2O_3 film is shown in Fig. 7(b). Both photographs were taken in the vicinity of Tukon hardness indentations. It should be noted that the Fe_3O_4 film appears to be very ductile since it conforms to the indented surface. The Fe_2O_3 film is brittle because it breaks up into fragments, both in the indentation and in the region around. The region of chipping provided an opportunity to measure the film thickness using Tolansky interference microscopy. It is estimated to be between 1200 and 1800 Å, a range of uncertainty that results from the great difference in optical properties of Fe_2O_3 and the steel substrate.

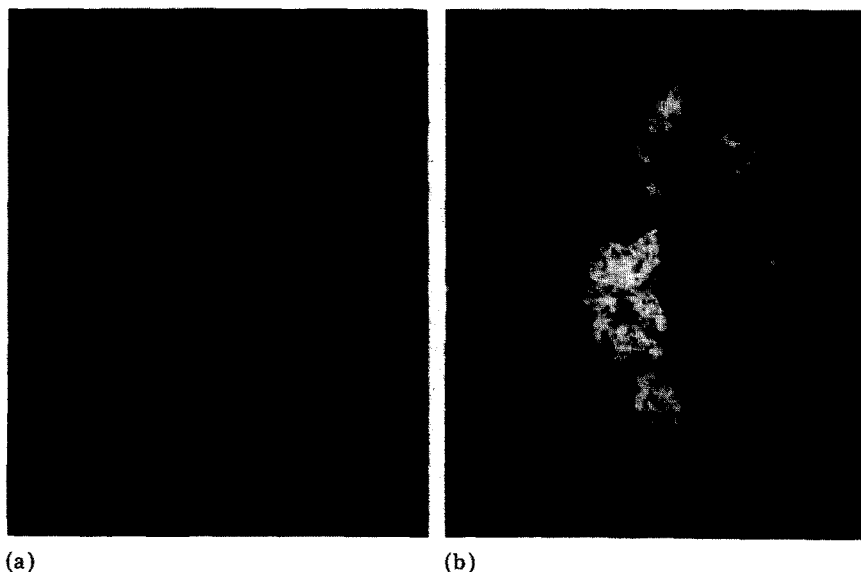(a)   (b)

Fig. 7. Photographs of oxide surfaces, using a Francon polarizing objective, showing the effect of a Tukon indenter on the oxide behavior: (a) Fe_3O_4; (b) Fe_2O_3. (Magnifications, 550X.)

5. Ellipsometry

The final instrument used to compare the films was the manual null ellipsometer. At one angle of incidence, and with a single wavelength of light, this instrument produces data that can be used to calculate either a film thickness or the optical properties of a film of known thickness but not both. Assuming a (real) index of refraction n of 2.60 and an index of absorption k of 0.3 for Fe_3O_4 and values of $n = 2.65$ and $k = 1.3$ for the steel

substrate [9], the film thickness of oxide was determined for various test conditions as detailed below. This method proved to be very convenient in that specimens require little preparation for the measurement and data can be obtained in about 15 min. The absolute accuracy of the results is, among other things, dependent on the values of the optical properties used in the calculation. The method has been found in this work, at least, to yield highly reproducible results, and the method is most useful for comparison of thicknesses of films formed under various loads, speeds and other experimental conditions.

Two series of tests were performed using step loading. The specimens were removed from the test apparatus and placed in the nearby manual ellipsometer. Within about 20 min the specimens were returned to the wear test apparatus and testing was resumed. The results of these interrupted tests are not identical with the results of uninterrupted step-load tests. The reason is not known at this time. However, the results reported below are instructive, both for understanding breaking-in and for showing the value of ellipsometry as a tool in such studies.

In the first test series flat specimens with various original surface finishes were tested, using steps of 15 min duration and load increments of 22 N. The interesting specimens were those with original surface roughnesses of 0.05, 0.1 and 0.3 μm. Figure 8 shows the oxide film thickness as a function of the accumulated time of sliding with increasing load. The smooth surface oxidized quickly but the film was also removed quickly and surface failure occurred in about 50 min. The roughest specimen also oxidized quickly at first but more slowly later. The intermediate roughness surface oxidized slowly and steadily to a greater thickness than did the others. Figure 9 shows the change in surface roughness during the tests. The intermediate roughness surface became smoother during sliding while the other two did not.
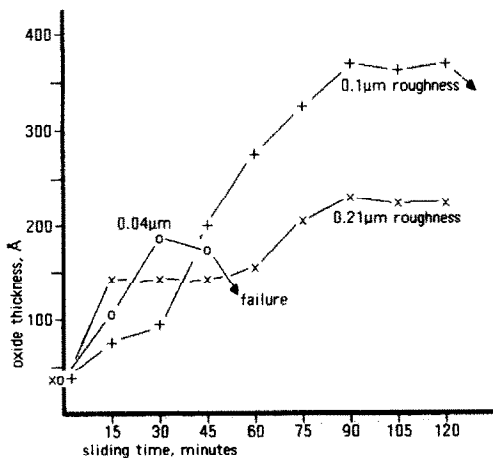

Fig. 8. The effect of the initial surface roughness of the flat steel surface on the rate of growth of oxide.
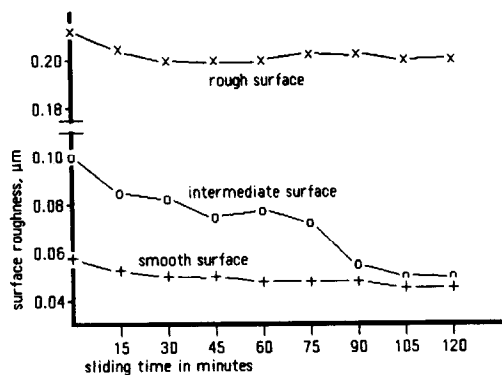
Fig. 9. The surface roughness change using the step-load method.

The second series of tests was done to determine whether the arbitrarily selected 15 min step-load interval was reasonable. Tests were therefore done at 5 min intervals and 25 min intervals as well as at 15 min intervals. Figure 10 shows the results. It may be noted that the oxide thickness is not simply proportional to the accumulated time of sliding because the film thickness in the 25 min interval test reaches a maximum of 425 Å whereas in the 5 min interval test it reaches a maximum of 225 Å.
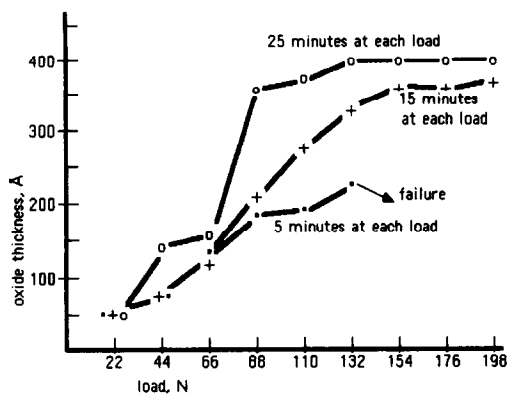

Fig. 10. The effect of the step-loading time on the rate of growth of oxide.

6. Discussion and conclusions

The sliding conditions in all the tests were in the severe range in that the calculated elastohydrodynamic "film thickness" was much less than the measured surface roughness of the specimens. At the beginning of sliding, in the step-load test as well as in the constant load test, metallic wear debris formed quickly. This may have been due to low cycle or plastic fatigue. The material loss rate was relatively high but the coefficient of friction was low, about 0.06. From the beginning of sliding in oil a dark film began to form on the flat surface and the oil became darkened. The dark film was found to be

a composition approximating Fe_3O_4 more nearly than other iron oxides or hydroxides and is apparently soft and ductile. When a sufficient film of Fe_3O_4 forms, the metallic wear debris ceases to form. However, Fe_3O_4 is removed from the upper surface of the film, some of which accumulates on the stationary slider and some of which darkens the oil. This is then a second mechanism of material loss from the flat specimen but the loss rate is low.

The soft and ductile Fe_3O_4 film, if thick enough, apparently prevents the formation of local (asperity dimension) high shear stresses that could produce plastic flow and low cycle fatigue. At the beginning of sliding there appears to be a competition between the rate of oxide formation and the rate of oxide removal, particularly by the metallic debris particles. If the metallic particles prevail the surface fails. If the oxide prevails the surface survives, or has been broken-in.

Acknowledgments

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