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PROGRESS IN THE DEVELOPMENT AND UTILIZATION OF FERROGRAPHY

Foxboro/Trans-Sonics, Incorporated

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TECHNICAL REPORT

PROGRESS IN THE DEVELOPMENT AND UTILIZATION OF FERROGRAPHY

Sponsored by
ADVANCED RESEARCH PROJECTS AGENCY
CONTRACT NO. N00014-74-C-0135
ARPA Order No. 2532

FOXBORO/TRANS-SONICS, INC.
BURLINGTON, MASSACHUSETTS

FOR PERIOD ENDING
DECEMBER 31, 1975

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
Particles resulting from the wearing of machine surfaces are recovered with a Ferrograph analyzer. The particles have unique characteristics depending on the material of the wearing surfaces and the wear mode. The principal methods of identifying the materials of individual particles are discussed. Chemical reactions from the gaseous phase appear to offer the possibility of identifying materials on the basis of color without losing the particle morphology. (cont.)

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Six regimes of sliding steel wear have been identified. It appears that much research on friction and wear is concerned with regimes that do not obtain in normally running machines. Lubricants may be considered as shifting the load, temperature and speed at which a transition from one regime to another occurs. The combined effects of the physical and chemical properties of the oil may be rated by observing the regime boundaries.

The existance of a shear mixed layer is discussed. It is pointed out that the condition of a low sliding wear rate may be viewed as an equilibrium between the generation of the shear mixed layer and it's loss. If the shear mixed layer is lost faster than it is generated the surface becomes rougher and the much higher wear rates of regime 3 ensue.

This research was supported by the Advanced Research Projects Agency of the Department of Defense and was monitored by ONR under Contract No. N00014-74-C-0135.

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----- NOTE -----

The figures in this report are numbered using the section number plus a letter designating the order. Figure 5A is the first figure concerning Section 5 and Figure 5B is the second figure in Section 5.

Consequently the figure numbers are not consecutive and, for example, there are no Figures 1, 2, 3 and 4.

SUMMARY

The major efforts during the period 1 July through 31 December 75 were directed toward (1) the exploration of techniques which would permit rapid and simple identification of the material of which particles collected on a Ferrogram are composed and (2) the development methods for relating the type of particle generated by rubbing surfaces to the general conditions existing at the rubbing surfaces. Lesser efforts were devoted to a preliminary examination of the degree to which wear, in a particular rubbing wear situation, is influenced by the lubricant used and to further examination and interpretation of particles from the shear mixed (Bielby) layer.

Particle Identification

Earlier work had demonstrated that examination of Ferrograms in a high-quality optical microscope under a variety of illumination schemes (transmitted and/or reflected white light, bichromatic illumination, polarized white light) permits one to easily discriminate between metallic and non-metallic particles and to differentiate between some metals. Use of a scanning electron microscope permits examination of particles at higher magnification and thus provides additional information while x-ray microprobe analysis permits one to determine the composition of particular particles. While this latter technique provides a powerful tool for particle identification, its use is restricted primarily to research application.

Several chemical techniques were devised in which the particles are acted on in situ. With appropriate chemicals the reactions are such as to cause the surfaces of particles of particular materials to change color so that they can be identified. Although it was demonstrated that some particles could be identified in this way, the overall conclusion was that the utility of the technique would be confined to research applications. Similar reaction from the gaseous state appear to offer quicker and more practical techniques. A technique which proved both quick and practical and which permits one to distinguish between several important types of particles consists merely of heating the Ferrogram in air to 330°C (625°F) for 90 seconds. The technique causes particles of certain common materials to assume characteristic bright colors. For example, low alloy steels turn blue, aluminum - bright silvery-white, bronze to dark golden, etc.

Regimes of Wear

In order to further clarify the understanding of the wear process and the interpretation of Ferrograms a series of rubbing wear tests was run to determine whether conditions could be established under which essentially all of the wear particles generated were of one type. This work led to identification of the following six regimes under which rubbing wear of steel surfaces can be classified.

Regime	Condition	Particles Generated
1	Hydrodynamic lubrication	Low volume of small rubbing wear particles 1 to 15 µm
2	Boundary lubrication	1 to 15 µm rubbing wear particles
3	Partial breakdown of lubricant film	Free steel particles up to 150 µm
4	Mild oxidative wear	Hematite α Fe_2O_3 particles up to 150 µm
5	Severe oxidative wear - poor lubrication	Black oxide Fe_2O_3; Fe_3O_4; FeO particles up to 150 µm
6	Catastrophic wear	Free metal particles up to 1mm

As this work progressed it was found that the wear modes discussed by a number of other investigators belong to one or more of the above six regimes and that these constituted a convenient over-all framework for classifying rubbing wear modes.

The Influence of Lubricants

Lubricants

A set of preliminary tests was conducted to explore the antiwear properties of three lubricating oils and three hydraulic fluids. This work showed that under specific wear conditions the various lubricants could change the rubbing wear rate by more than fifty to one. Additional work will be required to relate these results to specific characteristics of the oils. Some oils were found to generate copious quantities of friction polymer which was precipitated on the Ferrograms. Two classes of polymer were observed, a loose amorphous deposit and a strong film which appeared in the oil as "rolling pins" of the polymer.

Rubbed or Sliding Surfaces

The final effort reported on here was a continuation of the investigation relating to the shear mixed layer. It was found, while manipulating the wear particles on a Ferrogram, that at least some of the particles from the shear mixed layer can easily be smeared on the surface of the Ferrogram. This apparent indication of the softness of the layer should be further investigated to clarify the role of the shear mixed layer in the wear process.

Sliding surfaces of machines usually operate in boundary lubrication ie, in regime 2. Exceptions are surfaces such as door hinges which are not required to slide very far. But many surfaces must slide long distances with boundary lubrication and very low wear. This is regime 2. Continued operation in regime 2 requires that the shear mixed layer be stable. If the layer is worn away faster than it is generated, the surface will become rougher and will enter regime 3 or a higher regime. Consequently, the maintenance of "normal" long life wear can be viewed as the establishment of an equilibrium of the shear mixed layer. That is, the generation rate of the layer must be greater than the loss rate otherwise the layer will be lost and the underlying crystal structure will generate large particles with a much increased wear rate. Materials and lubricants which are capable of generating stable shear mixed layer may be expected to yield long life in machine applications.

1. INTRODUCTION

The purpose of examining the wear debris in the lubricating oil of machinery is to assess the wear situation in that machine. To increase the significance of an examination it is desirable to be able to associate the particles produced with the wearing components. For this end further procedures have been investigated by which the particles on a Ferrogram may be identified as regard their mode of generation and composition.

Physical and chemical tests have been devised and used in conjunction with optical examination to aid the identification of the metallic content of the wear debris. Optical examination in itself can now yield a great deal of information on the composition and mode of generation of various particles.

To date tribological research has centered around the more extreme cases of wear, i.e., either unlubricated wear or some severe condition of load, speed or other parameters. A thorough understanding of the normal lubricated wear processes is still lacking. This work investigates these normal wear processes as well as the more severe wear modes.

Extensive experiments involving the sliding of a selection of steel surfaces on one another under varying conditions of lubrication, load and speed have been conducted. The resulting wear modes have been classified in terms of the particles produced. A strong correlation exists between the maximum size of the particles and the severity of the wear mode. The presence of oxides other than contamination being an indication of abnormal lubrication or no lubrication at all.

Previous work in the field has also shown the importance of the role played by the lubricants, shear mixed layer and the interaction between them. It has been shown for instance, that different lubricants to the same specification can display a 10 to 1 difference in wear rates when used in otherwise identical experiments. Work has been conducted to investigate the difference between similar lubricants for both sliding and rolling contact wear. The effective differences between the lubricants is more apparent in sliding wear and is frequently associated with the generation of various types of friction polymer.

Further work has been done to ascertain the characteristics of the shear mixed layer and again it is indicated that its metallurgical structure is fundamentally different from that of the original material.

2. PARTICLE IDENTIFICATION

2.1 Examination and identification of wear particle types is important for several reasons. By determining the mode of generation of a particle and its composition the severity of the wear situation and the wearing component may be identified. This makes the detection and repair of wearing components easier and less expensive.

The mode of generation of a particle can be ascertained by its size and appearance under a Ferroscope. Determining the composition of a wear particle can be more difficult. The following sub-sections discuss four techniques investigated for this purpose.

2.2 Optical Examination

2.2.1 Reflected White Light

The size, shape and color of the particle may be observed with this illumination. Copper based alloys appear as yellow or a reddish brown color while most remaining metals appear as a silver white. Steel particles, however, can display a yellow to blue color if during their formation they are subjected to any significant heating. These colors are interference effects due to slight surface oxidation. These colors on steel also give a good indication as to the severity of generation of the particles.

2.2.2 Transmitted White Light

Examination under transmitted white light ascertains whether a particle is transparent, translucent or opaque. The attenuation of light in free metal particles is such that they will be opaque even to sub-micron thicknesses. In consequence, free metal particles appear black when examined with this illumination. On the other hand, most other elements and all compounds appear translucent or transparent, the color displayed being characteristic of the material. Hematite - Fe_2O_3, for instance, will transmit red light through a thickness of several micrometers depending on the crystal size. It can often be ascertained whether an oxide particle has a free metal core or not because free metal is opaque.

2.2.3 Bichromatic Illumination

Here the particles are illuminated by reflected red light and transmitted green light. Free metal particles appear red while non-metals and all compounds appear green or yellow depending upon the degree of light attenuation. The red reflected light also allows examination of some of the particles' surface characteristics. This illumination arrangement combines some of the advantages of the previous two techniques and allows for easy detection of the more important metallic particles.

2.2.4 Low Aperture or Oblique Illumination

These illumination arrangements are variations on the standard reflected white light method. Either of these techniques will exaggerate the surface height of the particles giving an indication as to their mode of generation. With low aperture illumination, the angle of incidence of the light is such that rays reflected off of slant surfaces are not returned to the objective. Consequently such surfaces appear black. Oblique illumination achieves a similar effect by illuminating the particle from the side.

These techniques show up surface striations that result from a particle being generated by a sliding action as contrasted with a fatigue induced spall particle.

2.2.5 Polarized Light

The use of polarized light to examine particles has proven to be quite useful, quick and convenient as an aid in identifying the materials of particles particularly oxides, plastics and various solid contaminents in the oil.

The light is polarized and analyzed by oriented molecular films (Polaroid*) having high absorbtion of the electric field perpendicular to the polarization direction. Since the polarizer and analyzer are identical they will be referred to here as polars (Nichol prisms are obsolete for such applications).

If a Ferrogram is illuminated with white transmitted light through crossed polars, the background light is absorbed and the field of view is dark.

Metal particles appear dark but the edges are visible because of the distortion of the electromagnetic field by the edges.

Amorphous materials also remain dark. Occasionally optically active inclusions are seen.

Individual transparent or translucent crystals of the cubic system are dark. Crystals from all other crystal systems

*TM

are bright except for specific orientations where the light is extinguished. Changing the direction of polarization or rotating the stage causes light intensity to vary.

Polycrystalline clusters of small crystallites (<< 1 um) appear bright, snow like and do not change in intensity as the direction of polarization is changed. Polycrystalline clusters are made visible as the result of internal scattering of the light causing the light to be depolarized. These clusters have unusually strong colors considering how thin the particles often are. Multiple scattering of the light inside the particle causes the light to transverse back and forth through the particles so that the light path is longer than the particle thickness. This is one cause of the intense colors. Additionally, the elimination of the white light background allows the colors to be more saturated than would be the case if only transmitted white light were used.

Extruded plastics such as nylon fibers appear very bright and may be extinguished at certain angles.

Because they are too thin to show birefringent color effects all of the particles which shine out in polarized light exhibit the color of the substance. The colors are much more saturated than when the particles are viewed with unpolarized light permitting color distinctions which could not otherwise be made.

2.2.6 Location on the Ferrogram

The size of a particle and its location on the Ferrogram can give the viewer an estimate of the particles magnetic susceptibility. The difference between Ferromagnetic materials (Fe, Co, Ni) and other metals is easily distinguished. Ferromagnetic particles of 15 μm or larger are always deposited within 4 mm of the entry point of a Ferrogram and the smaller particles tend to form long strings. Conversely, paramagnetic or particles of low magnetic susceptibility, such as aluminum, show little tendency to form strings and large particles ($>$ 15 μm) may be found along the length of the Ferrogram.

2.3 Wet Chemistry

A Ferrogram may be submerged in a variety of liquids, including water, without loss of the particles. This has suggested that wet chemical techniques may be used to determine the composition of some of the particles.

For example, submerging the Ferrogram in a weak sodium hydroxide solution for five seconds dissolves aluminum particles.

The difficulty with submerging the Ferrogram in a liquid, however, is that all particles reacting with the liquid are dissolved or washed away so that interpretation involves either having two Ferrograms from the same oil or recording what is present on the Ferrogram before performing the chemical tests.

It is a simple procedure to place a Ferrogram in a scanning electron microscope, fitted with an x-ray dispersion apparatus and to determine the heavier elements present in individual particles. However, this technique is better suited to research than field application because of the complexity of the apparatus needed and the time required to analyze a population of particles, particle by particle.

What is needed is a technique which is simple, fast and allows the particle composition to be determined by inspection just as one distinguishes metal from wood or glass from plastic in daily life.

2.4 Electroless Plating

This technique involves the deposition of one metal on another without the influence of external electrolytic action. For the deposition to occur, the metal in solution has to be higher in the electrochemical series than the metal receiving the deposit.

The only common metals beneath tin in the electrochemical series are copper, lead and the noble metals. To assess the feasibility of using this technique as an identification procedure attempts were made to plate copper based alloy particles with tin.

A successful procedure that was developed involved immersing a Ferrogram containing copper based particles in a plating solution of thiourea, $SnCl_2$ and HCl for one minute. The Ferrogram is then dipped in distilled water several times to wash out the salts of the plating solution. Subsequent microscopic examination showed that the yellow bronze particles were coated with white tin indicating the presence of a metal below tin in the electrochemical series. The technique appears feasible but was not pursued to obtain a variety of solutions, plating at different levels in the electrochemical series because alternate methods appeared to be more advantageous.

2.5 Heating Ferrograms

By heating the Ferrograms to 330°C (625°F) for 90 seconds all low alloy steel particles turn a light blue color. This color results from interference effects due to surface oxidation. Bronze and other copper based alloys deepen in color to a dark brown and aluminum becomes a very bright silvery white. Organic materials such as friction polymer generally turn a deeper shade of brown but remain translucent.

Heating to 510°C (950°F) for 90 sec. causes copper based alloys to oxidize to a greyish green color. There is no effect on titanium, chromium and other oxidation resistant metals. Steel turns to hematite.

Low melting point alloys melt and are often oxidized. Some alloys develop a unique halo around the particle. It appears that a component sublimates and deposits very fine ($<<$ 1 um) oxide powder around the periphery of the particle. The appearance is distinctive and may be used for identification.

Organic materials such as friction polymers melt, evaporate or burn while being heated. Usually, the original particle morphology remains, the carbonaceous material burns away without changing the particle's shape.

2.6 Optical Examination

For most applications optical examination along with heating the Ferrograms yields sufficient information about particle composition for diagnostic purposes. Wet chemical tests and electroless plating may be used to confirm the composition of a suspected class of particles if the initial examination raises important questions about the source of the particles.

2.7 Chemical Reactions

This work and earlier investigations using acid vapor has shown that it is possible to obtain chemical reactions between the particles and gaseous vapor. The small size of the particles makes the slower reaction rate from the gaseous state desirable and has the advantage that the particles are left in situ so that their morphologies before and after the reaction may be observed.

3. WEAR REGIMES

"Characteristics of Particles Generated at the Interface Between Sliding Steel Surfaces"

A. A. Reda, R. Bowen and V. C. Westcott

To facilitate matters (color prints are involved) individual reprints of the above paper are attached to each copy of this report.

Wear, 34 (1975) 261 - 273

CHARACTERISTICS OF PARTICLES GENERATED AT THE INTERFACE BETWEEN SLIDING STEEL SURFACES*

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(Received May 6, 1975; in final form June 30, 1975)

Summary

This paper is an attempt to further clarify and classify the range of wear modes which can occur at the interface between steel surfaces subjected to sliding motion, a variety of speeds and loads, with and without lubrication. Previous researchers have been able, from examination of the wearing surfaces, to identify a number of distinct wear modes and to classify them according to operating conditions. The present paper shows that it is possible to arrive at a comparable classification of wear modes based on examination of the wear debris rather than the worn surfaces. Consequently, it is possible to determine the wear modes in a machine from observation of the debris in the lubricant without requiring access to the wear surfaces themselves.

The wear particles were generated by rotating a cylindrical sleeve of AISI 52100 steel in contact with three types of fixed wearing surfaces, crossed AISI 52100 steel cylinders, fixed spherical balls of AISI 52100 and AISI 1018 steel and a flat plate of AISI 1018 steel. Surface speeds ranged from 0.19 to 5.0 m/s and the load from 1 to 80 kg. These particles were collected and examined in both an optical and a scanning electron microscope. X-ray diffraction studies of selected particle types were also made.

These studies revealed six different wear regimes which depended on the test parameters. Each regime produced wear particles of characteristic morphology and composition. All of the types of particles studied here have been found in the lubricating oil of field operating machines.

Introduction

Considerable past work has been devoted to examination of the chemical, physical and mechanical phenomena responsible for various modes of wear. Burwell [1] suggested classification of wear modes according to cutting, adhesion, corrosion, abrasion and fatigue. More recently, Eyre and Maynard

*Paper presented at the 3rd International Tribology Conference, "Tribology for the Eighties", Paisley, 22 - 25 September, 1975.

-16-a

[2] and others [3, 4], experimenting with dry wearing surfaces, found that a corrosive mechanism operates at low sliding speeds while at higher speeds adhesion becomes the dominant wear process. Quinn and Wooley [5] were able to show that under mild conditions the oxide Fe_2O_3 was generated but that a transition to Fe_3O_4 occurred as the load or speed was increased above certain levels.

Begelinger and deGee [6, 7] and Czichos [8] investigated the wear processes associated with lubricated conditions and again found a series of wear modes with transitions from one mode to another as conditions were changed. Kragelskii [9] classified wear according to the various types of events which can occur between asperities in contact and defined five ways for the destruction of frictional bonds leading to the destruction of surface films and finally to the destruction of bulk material. In every wear situation, whether lubrication was provided or not, an operating mode which can be distinguished by its severity is identifiable. Hogmark *et al.* [3] in a study of adhesive, abrasive and corrosive wear state, "it proved possible to identify the different wear mechanisms from typical topographic features" of the worn surfaces.

Recently, Ferrographic techniques for the collection and examination of wear debris have been developed [10 - 13]. The objective of the present work was to conduct additional wear tests in which both the wear surfaces and the wear debris would be examined and thereby gather information to permit further clarification and classification of the wear regimes associated with sliding contacts. Identification of specific wear regimes in terms of the characteristics of the particles generated would provide information of use in *in situ* monitoring of the condition of a lubricated system.

This and other efforts have shown that a large number of combinations of load, surface speed, lubricant and temperature will result in the development of a specific wear regime.

As Eyre and Maynard [2] have pointed out "there are so many material and lubricant combinations that it is becoming increasingly difficult to predict the effect of even small variations in the operating conditions" on the resultant wear. The purpose of the experiments carried out during this study was not to attempt to establish boundaries and operating conditions at which a transition from one wear mode to another occurred but to show that a particular type of wear particle predominates if some prescribed set of conditions exists, and relate the various types of particles to the wearing conditions as indicated by examination of the surfaces in contact.

Experimental procedure

The wear experiments were carried out utilizing a lathe adapted to hold 100 mm diam. AISI 52100 steel sleeves between the head and tailstock. As shown in Fig. 1 a loading arm with a deadweight system was mounted on the carriage so that test samples held on the arm could be lowered onto and pressed against the sleeve. Jigs were made to hold

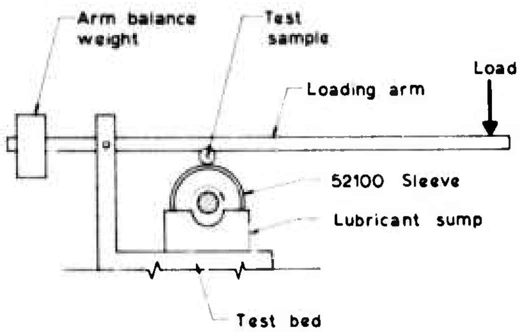


Fig. 1. Schematic arrangement of the test rig.

TABLE 1

Details of materials and specimens

Component	Material	Hardness	Surface finish (c.l.a. μm)
100 mm diam. sleeves	AISI 52100 (1.0% 1.5% Cr)	60 (Rc)	0.25
25.4 mm diam. spheres	AISI 52100 (1.0% 1.5% Cr)	65 (Rc)	0.125
12.7 mm diam. spheres	AISI 1018 (low C steel)	90 (Rb)	0.125
12.7 mm × 19 flats	AISI 1018 (low C steel)	90 (Rb)	0.20
Oriented with the long dimension perpendicular to sleeve axis.			

All parts and samples used in the tests were degreased with organic solvents.

12.7 mm and 25.4 mm diam. balls and 19 mm long flat plates of any width up to 19 mm. A stainless steel sump containing 100 ml of oil was located below the rotating sleeve to lubricate the wearing samples. The available lathe speeds gave the following selection of surface speeds — 0.19, 0.30, 0.47, 0.76, 1.30, 2.10, 3.20, 5.0 m/s.

Table 1 gives details of the materials and specimens used.

The oil used was a MIL-L-23699 synthetic polyester. All oil samples and solvents were passed through a 0.45 μm Millipore filter prior to use.

Approximately 120 tests were carried out under various conditions of load, speed, temperature, geometry, material and lubricant combination. Table 2 lists a selection of these experiments, together with the load, speed, duration and conditions of lubrication, for each test.

The condition of oil starvation was simulated by initially smearing the sleeve and test specimen with oil and conducting the tests with no further addition of lubricant.

Ferrograms from the nonlubricated and starved tests were made from clean oil used to wash down the test specimen, sleeve and dry sump after

TABLE 2

Representative test details

Regime	Test* identification	Specimen	Speed (m/s)	Load (kg)	Duration	Lubrication
1**	3411	52100[2]	0.47	14	120 min	Rotating sleeve partially immersed
1	3531	52100[2]	0.76	11.5	90 min	Rotating sleeve partially immersed
1	3197	52100[1]	0.47	80	60 min	Rotating sleeve partially immersed
1	3514	1018[3]	2.1	4.5	90 min	Rotating sleeve partially immersed
2	3364	52100[2]	0.47	35	45 min	Rotating sleeve partially immersed
2	3535	52100[2]	0.76	35	30 min	Rotating sleeve partially immersed
2	3503	1018[3]	0.76	18	60 min	Rotating sleeve partially immersed
3	3522	52100[2]	0.47	180	10 min	Rotating sleeve partially immersed
3	3530	52100[2]	0.76	55	30 min	Rotating sleeve partially immersed
3	3519	1018[2]	0.76	180	10 min	Rotating sleeve partially immersed
3	3516	1018[3]	1.3	180	10 min	Rotating sleeve partially immersed
4	2400	52100[2]	0.47	1.4	20 min	Unlubricated
4	42	52100[2]	0.47	140	180 min	Rotating sleeve partially immersed Lubricant heated to 150 °C
4	34	52100[2]	0.3	11.5	60 min	Lubricant starvation
4	3533	1018[3]	0.19	1.0	90 min	Unlubricated
5	24	52100[2]	0.47	35	10 min	Unlubricated
5	3418	52100[2]	0.76	35	5 min	Unlubricated
5	32	52100[2]	0.3	90	30 min	Lubricant starvation
5	3515	1018[3]	0.76	9	10 min	Unlubricated
6	1G	52100[2]	1.3	180	5 se	Rotating sleeve partially immersed
6	3590	52100[2]	0.76	180	5 s	Rotating sleeve partially immersed
6	2G	52100[2]	0.76	70	5 s	Rotating sleeve partially immersed
6	3G	52100[2]	2.1	1.4	5 s	Unlubricated
6	3596	1018[2]	1.3	180	5 s	Rotating sleeve partially immersed
6	3501	1018[3]	3.2	9	5 s	Unlubricated

*All 2 and 4 digit numbers have Ferrograms with corresponding number.

**Hydrodynamic lubrication refers to a condition in which the surfaces are completely separated by a fluid film. We can infer that a hydrodynamic lubrication condition exists in the Regime 1 tests because (1) the wear rates approached zero after the initial break-in period, (2) the wear scars quickly reached a steady state size, (3) as the load was increased within the bands of Regime 1 the area of the wear scar increased in direct proportion so that the unit loading remained constant. The existence of circumferential grinding grooves in the rotating sleeve apparently served to supply oil to the load bearing area and thus maintain the hydrodynamic film.

(1) Fixed crossed cylinder against rotating cylinder.
(2) Fixed sphere against rotating cylinder.
(3) Fixed flat against rotating cylinder.

completion of the test. Ferrograms from the lubricated tests were made from samples of the oil taken at completion of the test.

The Ferrograms were examined with a Bichromatic microscope and, when necessary, with a scanning electron microscope fitted with X-ray energy dispersion analysis equipment. X-ray diffraction was also used to identify the oxides.

Results

Examination of the worn surfaces of test specimens and the particles generated during test indicated six distinct wear regimes, Table 3, each being clearly identified by characteristic wear particles and surface features revealed by examination in an optical microscope.

TABLE 3

Wear regime

Regime	Particle description and major dimension	Surface description	Wear rate
1	Free metal particles usually less than 5 μm	Varies between polished and very rough. One surface can be polished while the opposing surface remains as generated.	Approaches zero
2	Free metal particles usually less than 15 μm	Stable, smooth, shear mixed layer with a few grooves depending on the number of particles in the oil.	Low
3	Free metal particles usually less than 150 μm	Ploughed with evidence of plastic flow and surface cracking.	High
4	Red oxide particles as clusters or individually up to 150 μm	Ploughed with areas of oxides on the surface.	High
5	Black oxide particles as clusters or individually up to 150 μm	Ploughed with areas of oxides on the surface.	High
6	Free metal particles up to 1 mm	Severely ploughed, gross plastic flow and smearing	Catastrophic

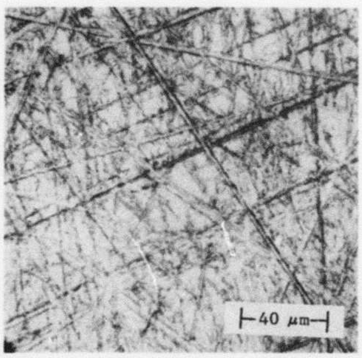


Fig. 2. Typical surface finish of flat 1018 steel specimen.

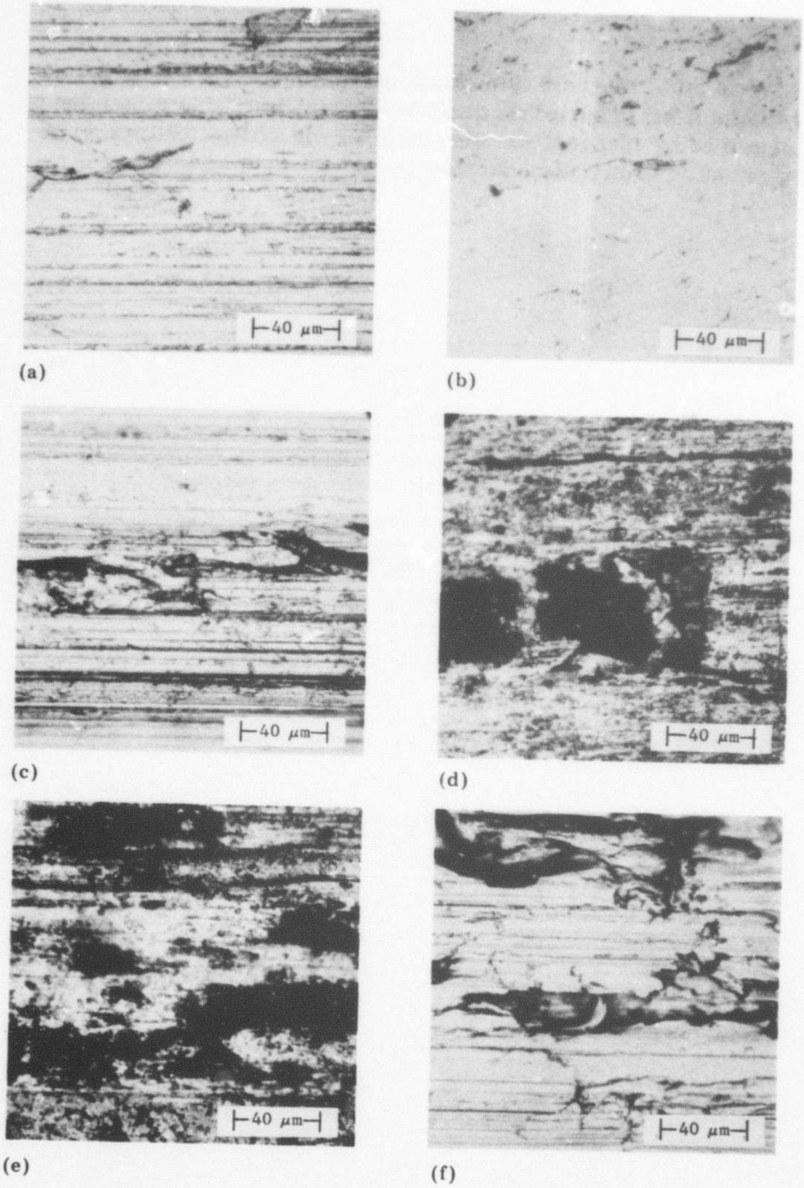


Fig. 3. Typical surfaces produced by each wear regime. (a) Regime 1 — Shear mixed layer in process of formation. The low wear rate results in slow formation of the shear mixed layer. (b) Regime 2 — Fully developed shear mixed layer. (c) Regime 3 — Breakdown of shear mixed layer. (d) Regime 4 — Surface roughening associated with production of red oxide. (e) Regime 5 — Surface roughening associated with production of black oxide. (f) Regime 6 — Complete breakdown of surface.

-16-f

Regime 1

In this regime wear rates are very low and it is inferred that the surfaces are separated by a hydrodynamic lubricant film. It is generally agreed that in practice much of the wear associated with this regime is confined to the starting and stopping of the machine. The majority of particles are of the rubbing wear type as described in Regime 2 and Fig. 2 shows the typical surface finish of flat 1018 steel specimens and Figs. 3(a) and 4(a) show the worn surface and the wear particles, respectively, typical of this regime. However, a few large linear particles are occasionally found. Presumably such particles break off the grinding marks left from surface finishing and are characteristic of the "break-in" period which, in this regime, may be extremely long.

Regime 2

This is a normal low-wear-rate regime associated with boundary lubrication. The process is similar to that of Regime 1 but the high surface shear forces associated with a very thin lubricant film or actual contact cause wear at a continual low rate.

The wear particles are generated during the formation of, and subsequent wearing of, a smooth 1 μm thick shear mixed (Beilby) layer of short crystalline order (<300 Å) as a result of the sliding shear. The shear mixed layer prevents the shear stresses from penetrating deeply into the material but suffers itself from continual removal of material in the form of thin rubbing wear particles. These particles may be formed as the result of delamination or by breaking off of parts of the shear mixed layers that are cantilevered over cracks or grooves on the surface [14, 15].

The flat rubbing wear particles are free metal, are usually less than 1 μm thick, and less than 15 μm in major dimension. Under the so-called boundary lubrication mode, the oil film is so thin that it may be penetrated by irregularities on the surfaces or by contaminants in the oil. Therefore, the shear mixed layers may be slightly grooved due to abrasion (Figs. 3(b), 4(b)). None the less, in Regime 2, as in Regime 1, a stable shear mixed layer developes and wear is confined to it. Hard steels exhibit a lower wear rate than soft steel in these regimes.

Regime 3

Regime 3 characterizes a wear mode in which breakdown of the boundary lubricant film occurs. Here the shear mixed layer becomes unstable and localized adhesion and severe plastic flow of the surfaces results.

The wear particles are of free steel varying in size from sub μm to 150 μm. Recirculation of the larger wear particles often generates grooves in the metal surfaces to depths of 25 μm depending on the running conditions. (Figs. 3(c), 4(c)). Soft steels exhibit deeper grooves and higher wear rates than hard steels.

Regime 4

In Regime 4 a mild form of oxidative wear dominates. The majority of the particles are hematite, αFe_2O_3 and are of two types. The first type is polycrystalline and when viewed on a Ferrogram appears orange in reflected white light and is bright red in transmitted polarized light with a crossed analyzer. The second type appears gray in white reflected light and red in white transmitted light. The gray appearance in reflected light is caused by the particles having a smooth flat upper surface which reflects all colors equally.

The surfaces of specimens tested under Regime 4 contained grooves to depths of 20 μm. Sometimes the bottom of the grooves were oxidized and areas of hematite may be found on the surface. (Figs. 3(d), 4(d)).

Regime 5

This regime is dominated by the generation of black oxides which by X-ray diffraction were identified as γFe_2O_3, Fe_3O_4 and FeO. It represents a more severe form of oxidative wear and can result as a transition from Regime 4 when the load is increased.

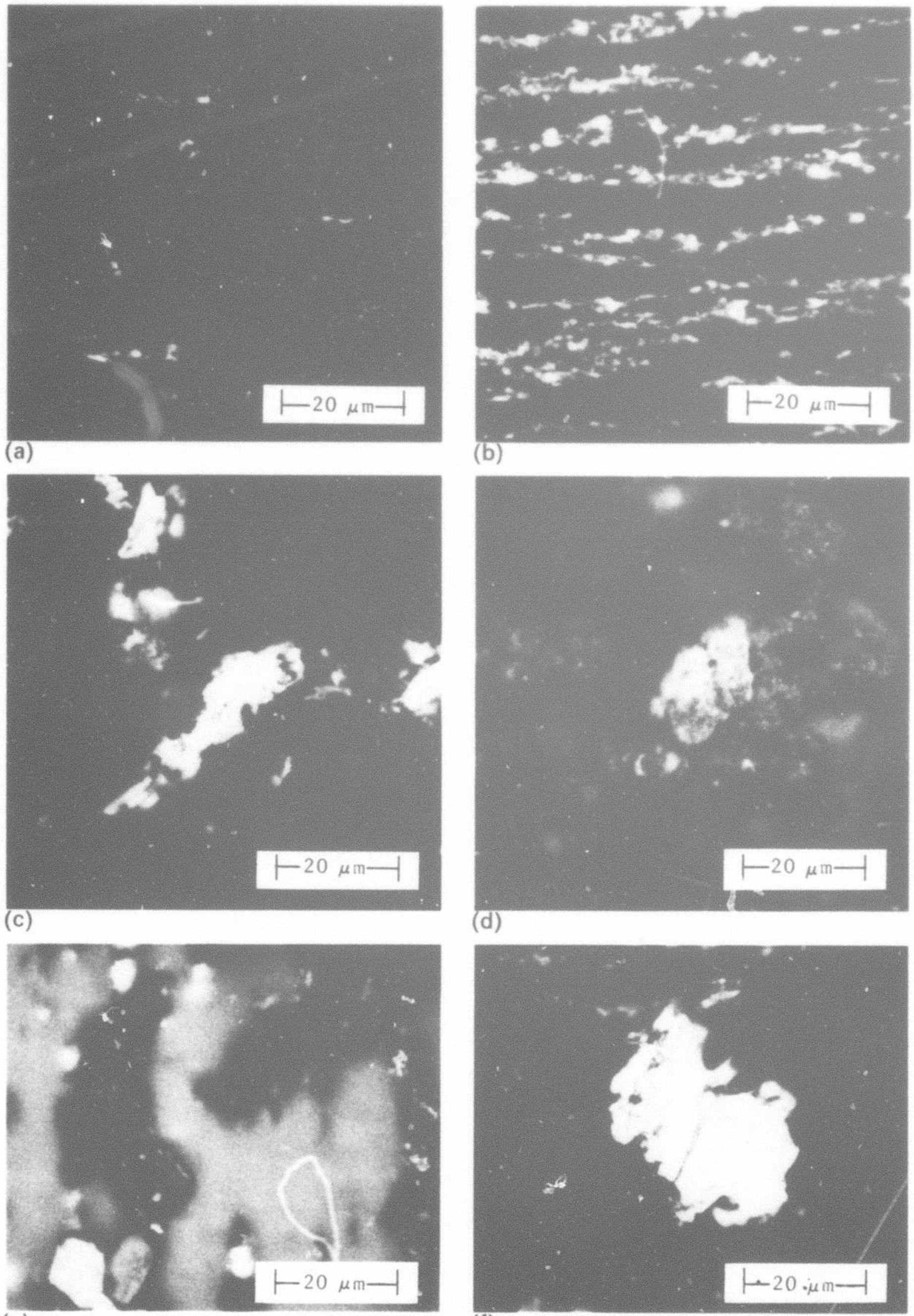
The wear particles vary in size up to 150 μm but most are less than 5 μm in major dimension and appear both as clusters and as individual particles. Some of the larger particles contain free iron. The particles may appear as smooth opaque black pebbles or as gray particles similar to those characteristic of Regime 4. However, these gray particles can be distinguished from those of Regime 4 because they are opaque. (Figs. 3(e), 4(e)).

The surfaces of specimens tested in this regime may be grooved to depths of 100 μm and areas of black oxide may be observed on the surface. As in Regime 4, under lubricated conditions, the presence of these oxides is indicative of poor lubrication.

Regime 6

This is catastrophic sliding wear mode which is indicative of failure of the components involved. Excessive surface shear stresses cause the complete breakdown of one or both surfaces and the generation of free metal wear particles having dimensions ranging up to 1 mm (Figs. 3(f), 4(f)). Smearing and macro-adhesion further roughen the contacting surfaces and escalate the wear process by ploughing action. This results in deep grooves (20 - 200 μm)

Fig. 4. Particles typical of each wear regime. (a) Regime 1 — Rubbing wear particles in combination with dark particles resulting from surface break-in. (b) Regime 2 — Rubbing wear particles, thin platelets arranged in strings by the magnetic field. (c) Regime 3 — Large free metal wear particles following breakdown of shear mixed layer. (d) Regime 4 — Large particles of iron oxide, α hematite (Fe_2O_3). The red particles are polycrystalline. The grey particles have flat surfaces which reflect the incident light before it enters the material. (e) Regime 5 — Large particles of iron oxide, γ hematite or magnetite (Fe_3O_4). Free iron and FeO may also be present. The grey particles reflect the incident light. (f) Regime 6 — Large free metal particles resulting from complete surface breakdown. The larger particles cannot be photographed at this high magnification.



(a) (b) (c) (d) (e) (f)

in the surface of specimens in continuous contact. With mating materials of different hardnesses, the softer surface tends to become grooved while the harder surface has large particles of the soft material adhering to it.

The surface shear stresses are a function of the sliding speed and the frictional force (and hence the normal force). Consequently, lubrication of any form helps to prevent the onset of this regime. However, it has been observed that in the lubricated case the transition to this regime from a lower regime is sudden. A surprising feature of this regime is that a higher load is required to enter this regime with a soft steel such as 1018 than for the case of a harder steel such as 52100. Whether this is a function of hardness or material composition is not known. The high loads necessary to produce Regime 6 result in considerable plastic flow of the metal.

Discussion

This work has shown that specific regimes of wear can be classified by the nature of the particles produced when steel surfaces are in sliding contact. This knowledge of wear particles, together with the considerable information accumulated by studies of wearing surfaces, enables a more comprehensive understanding of the mechanisms by which wear occurs.

This series of tests has identified six wear regimes, each of which generates characteristic particles as summarized in Table 3.

Exploratory unlubricated tests were carried out to ascertain the kinds of particles that might be produced by a machine in which the lubrication distribution system has failed. Interestingly, under loads and speeds in excess of those normally encountered in service, lubricated surfaces can generate the same types of particles as dry rubbing surfaces.

Regimes 1 and 2 represent normal wear conditions corresponding to hydrodynamic and boundary lubrication. Evidence of one or more of the higher regimes (3, 4 or 5) indicates that some parameter of the system has changed unfavorably. The occurrence of Regime 6 indicates immediate catastrophic failure.

Transition from one regime to another is affected by changes in the operating parameters of the system. The complexity of the wear phenomena is such that a progressive transition through Regimes 1 to 6 cannot be achieved in a particular system by the gradual change of any individual parameter.

Transition between Regimes 1, 2, 3 and 6 can be achieved by changing running speed and load. Under unlubricated conditions the red oxide Regime 4 is favored by low speed and load while the black oxide Regime 5 is favored by low speed and high load. Under lubrication, Regimes 4 and 5 are difficult to obtain but with inadequate lubrication or excessive temperatures Regimes 4 and 5 may appear. When operating in Regimes 4 and 5 a sufficient increase in speed will cause a transition to Regime 6. Also, environmental conditions such as temperature or the presence or lack of oxygen may determine which regime dominates under a given set of circumstances. Regimes 4 and 5 can be considered as environmental or special regimes because their existence not only depends on the geometry, material, speed and load of the sliding surfaces as do other regimes, but also depends on the available oxygen, temperature, oxygen transfer characteristic of the lubricant and other factors.

Free metal particles are produced in Regimes 1, 2, 3 and 6. These regimes may be identified by the particle size. Regimes 1 and 2 are characterized by rubbing wear particles in the form of flakes which are usually less than 15 μm across and about 0.75 μm thick. In Regime 3 the particles range in size up to 150 μm while in Regime 6 the particles may exceed 1 mm. The distinction between Regime 3 and 6 may be lost if the oil sampling technique is not designed to recover the macro particles.

Except for adhesive ploughing of Regime 6, grooving of the surfaces is believed to be the result of abrasive recirculation of wear debris and abrasion during the removal of particles from the contact area. This recirculation of wear particles can roughen a surface operating in Regime 2 sufficiently to break down the lubricant boundary layer and cause the onset of Regime 3. This should summarize briefly the idea that the type of particle generated has a correspondence to the conditions (stress, etc.) on the surfaces.

Further study to clarify the transition from regime to regime and to establish the dominant factors in sliding contact is needed. Other factors such as oil contaminants, fatigue, abrasive wear, rolling contact fatigue, while not considered in this investigation are known to play a most important part in the wear of specific mechanisms. These too have their characteristic particles which are associated with these modes. Further investigation may show additional regimes or sub classes in the regimes given above. It is the correspondence between the particles generated and the surface conditions of wear which make such investigations of interest in machine diagnostics.

Acknowledgements

The authors are indebted to Dr. E. C. Van Rueth and the Advanced Research Project Agency, U.S. Dept. of Defense, for technical and contract support under contract N000-14-74-C-0135. They also express appreciation to Dr. R. Miller of the Office of Naval Research, who administered the contract, to Mr. Douglas Scott and the National Engineering Laboratory, East Kilbride, for valuable assistance, and to Dr. W. Ruff and Mr. C. J. Bechtoldt of the National Bureau of Standards for electron and X-ray diffraction data.

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4. THE INFLUENCE OF LUBRICANTS ON WEAR RATES

The recognition of the six regimes of rubbing wear for steel sliding on steel has led to a new viewpoint on the function of lubricants. A lubricant has the effect of moving the boundaries between the regimes and in some cases even supressing a regime.

The boundaries between the regimes are not sharp and a pair of surfaces may operate in a mixed regime. Nevertheless, lubricants influence the load, speed, temperature and other environmental parameters necessary to obtain a given regime.

For example, an E.H.P. additive has the effect of raising the load that may be tolerated before regime 6 (catastrophic wear) is entered. The additive does this by promoting oxidation or other chemical reactions at the surfaces. The additive, therefore, moves the boundary between a lower regime such as 5 and regime 6 in the direction of higher load.

On the other hand, such an additive can have a detrimental effect on the lubricant in terms of the maximum load which may be supported without a transition between regimes 2 and 3; from boundary lubrication to a failing surfece wear mode. This explains why E.H.P. additives are used only when required instead of universally.

The concept that a lubricant influences the position of the regime boundary suggests that lubricants (at least in so far as they are to be used to lubricate rubbing surfaces) can be evaluated and rated by their effect on the boundaries of the six regimes. The concept encompasses the physical (viscosity, viscosity index) as well as the chemical properties of the lubricant and can be related to operating parameters of actual machines.

The relative merits of an oil in preventing rolling fatigue may differ for rolling fatigue versus rubbing wear. In the rolling fatigue case the effect of the lubricant on oxide formation, its viscosity and other properties are of importance.

The speed and ease with which the regime boundaries can be determined suggests that the concept will be a practical tool for rating oils and other lubricants.

In the course of the investigation, rubbing wear tests were carried out using two polyester lubricants both of which conform to specification MIL-L-23699. Lubricant A contained a so called light additive package and was designed for use as a jet engine lubricant. The other, oil E, had a high load additive package and is intended for application on jets when the gear loads may be high. Ferrograms from heavy rubbing wear tests with oil A contained a structureless, amorphous polymer which was deposited on the Ferrogram because it embodied many small steel wear particles.

Ferrograms from rubbing wear tests with oil E, on the other hand, contained thousands of tiny rolling pins, often 5 to 10 μm in diameter, and 10 to 30 μm long. Such rolling pins had been observed previously during tests of polyphenyl ether lubricants.*

For the tests of oils A and E the wearing parts consisted of a 1/4" dia 1018 steel cylinder turning against a fixed 1018 steel plate. After the test with oil E the plate was examined to ascertain that it was free of particles. A clean microscope slide was rubbed against the fixed plate near the contact surface. Microscopic examination of this slide revealed numerous rolling pins similar to those found on Ferrograms prepared from the test lubricant.

It is postulated that under the conditions of the test, friction polymer is formed from the lubricant in the contact area as the result of the localized conditions in the contact area. This friction polymer forms a film between the opposing surfaces. If the film is weak it sluffs off and results in an amorphous appearing polymer on the Ferrogram. If, however, the film is strong and tenacious it is rolled up to produce the rolling pins. Sliding of the surfaces rolls up the film.

Spiral markings are seen on the surface of the pins. These marks are the edges of the rolled up film. Often the rolling pins have conical ends just as a rolled rug when the rolling axis is not in line with the direction of rolling.

*Reference - Jones-NASA-Lewis, ASLE Trans. (1974), A. Reda - Wear, 32 (1975)

During these tests a sufficient quantity of friction polymer from oil A was deposited on the Ferrogram so that it was practical to scrape it off and place it in an IR transparent pill for infra red analysis.

It is interesting to note that the three oils A, E, and the base stock exhibited little difference in the accelerated fatigue life testing at N.E.L. while exhibiting marked differences in their rubbing wear properties.

4.1 Scanning Electron Microscopical Examination - Jet Engine Wear Debris

Hollow spheres of a compound were observed on a Ferrogram made from oil taken from a jet engine which was in the course of failing from a pump roller bearing.

The following report is an analysis of these spheres. Such organic materials are increasingly seen and may be associated with high acid number for the oil with the presence of tramp polymers in the oil and with the presence of an immisable liquid in the oil system.

4.2 NEL Report - "Scanning Electron Microscopical Examination of Ferrogram F2917" -- See next page --

NATIONAL ENGINEERING LABORATORY

STRUCTURES AND MATERIALS GROUP
Creep and Tribology Division

REPORT ON

SCANNING ELECTRON MICROSCOPICAL EXAMINATION OF FERROGRAM F2917

for

Trans-Sonics Inc, Burlington, Massachusetts, USA

AUTHORITY FOR EXAMINATION

Trans-Sonics Inc Purchase Order No 31068, Contract No N00014-74-C-0135 dated 11 July 1974: "Prepare electron micrographs of hollow spherical particles on Ferrogram F2917. Determine, if possible, composition and information relative to their manner of formation and source".

INTRODUCTION

Ferrogram F2917 was prepared from a sample of additive fortified ester-based lubricant from the sump of a jet engine on test. It contained numerous rough spherical-shaped particles within and outwith the strings of magnetic particles. Many of the spherical particles appeared to contain holes.

SPECIMEN PREPARATION FOR SCANNING ELECTRON MICROSCOPY

The Ferrogram was microscopically examined and carefully cut with a diamond cutting tool into sections of a size suitable for accommodation in the specimen chamber of the scanning electron microscope. Selected sections were coated in vacuo with gold-40% palladium.

A. I. Smith
A I SMITH
Head of Division
For D H MALLINSON
Director

DATE January 1975

REFERENCE Z3/2/75
Z3/TRC/2

SCANNING ELECTRON MICROSCOPICAL EXAMINATION

Fig. 1 shows a general view of strings of particles near the entry deposit of the Ferrogram. Fig. 2 shows typical spherical-shaped particles, containing holes and of rough surface appearance, in the strings of magnetic particles.

The spherical-shaped particles were generally of rough surface appearance and contained one or more holes, Fig. 3. The larger holes were more defined and tended to have a characteristic hexagonal shape, Fig. 4, possibly indicative of preferential chemical attack. The rough surface appearance of the spherical-shaped particles, shown in greater detail in Figs 5 and 6, could be due to some form of chemical attack or the incomplete balling-up of many small particles. The size of the holes in the spherical-shaped particles varied, Figs 7 and 8, indicative of different degrees of chemical attack, erosion or completeness of balling-up of smaller particles. Some spherical-shaped particles appeared to have partially disintegrated, Fig 9; these particles appeared to have contained a large single cavity and to have been almost hollow spheres before disintegration.

Several spherical particles in the entry deposit appeared intact and had a comparatively smooth surface appearance, Fig. 10.

X-ray energy analyses were carried out in the scanning electron microscope on selected spherical particles. All spherical particles analysed were found to be of similar composition. Figs 11 and 12 show typical spherical particles and their corresponding analysis spectra. A background analysis, that is the analysis of an area on the gold/palladium-coated Ferrogram devoid of wear particles, is shown in Fig. 13. There is little difference between the analysis spectrum for the background substrate devoid of wear particles, and those from the spherical particles, except that the latter contain iron. Analyses spectra from plate-like wear particles in the entry deposit show that such particles contain principally iron with traces of steel alloying elements and lubricant residue, the main elements present in the background and the spherical particles apart from iron. Thus by a process of elimination the spherical-shaped particles appear to be principally carbonaceous and/or organic, possibly 'friction polymer' with about a 10% content of small ferrous particles.

It was considered worthwhile to prepare scanning electron micrographs of several other specific types of particle present on the Ferrogram.

Fig. 14 shows a large piece of metal, possibly a surface spall. The markings may be evidence of rubbing and smearing on the surface. Figs 15 and 16 show typical cutting wear particles. It is interesting to note that X-ray energy analysis of the cutting-type wear particles indicated that they are of similar composition to the spherical particles and may thus be friction polymer cut from the rubbing surface. Fig. 17 shows a collection of unusual particles which appear to have suffered some working.

CONCLUSIONS

The spherical-shaped particles appear to be composed of carbonaceous and/ or organic material containing a small percentage of ferrous particles. They may be formed by coalescence of lubricant and organic additive degradation products intermixed with small ferrous particles. Friction polymer detached from the rubbing surface may contribute to nucleation and build-up of the particles. Incomplete coalescence, insufficient working and highly localized chemical attack of active sites in highly stressed material may all contribute to hole formation.

LIST OF FIGURES

1 Strings of particles near the entry deposit. (x 250)

2 Typical spherical-shaped particles. (x 8,500)

3 Rough surface appearance of a spherical particle containing holes. (x 18,000)

4 Well-defined large, hexagonal-shaped hole in a spherical particle. (x 21,000)

5 Rough surface appearance of a spherical particle. (x 16,500)

6 Rough surface appearance of a spherical particle. (x 18,000)

7 Small hole in apparently hollow spherical particle. (x 22,000)

8 Large hole in a spherical particle. (x 18,000)

9 Broken or partially disintegrated spherical particle. (x 16,500)

10 Comparatively smooth surfaced spherical particles in the entry deposit. (x 1,250)

11 X-ray energy analysis spectrum of the spherical particle. (x 9,000)

12 X-ray energy analysis spectrum of the spherical particle. (x 19,500)

13 X-ray energy analysis spectrum of the Ferrogram background.

14 Large piece of metal with surface markings. (x 3,500)

15 Cutting wear particles. (x 8,500)

16 Cutting wear particles. (x 8,500)

17 Unusual particles. (x 24,500)

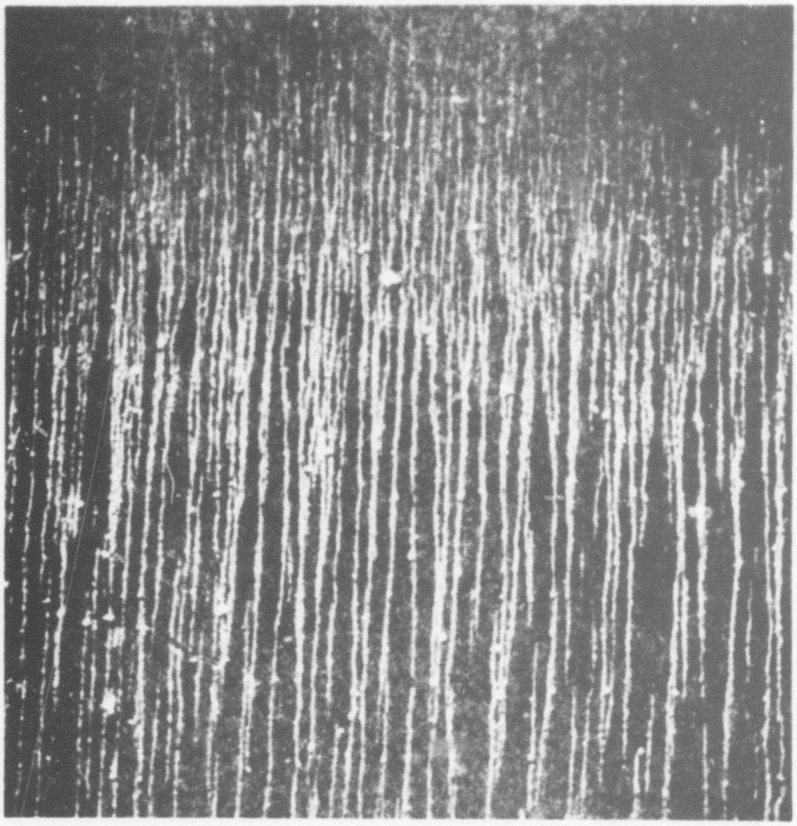


FIG. 1 (x 250)

Strings of particles near the entry deposit

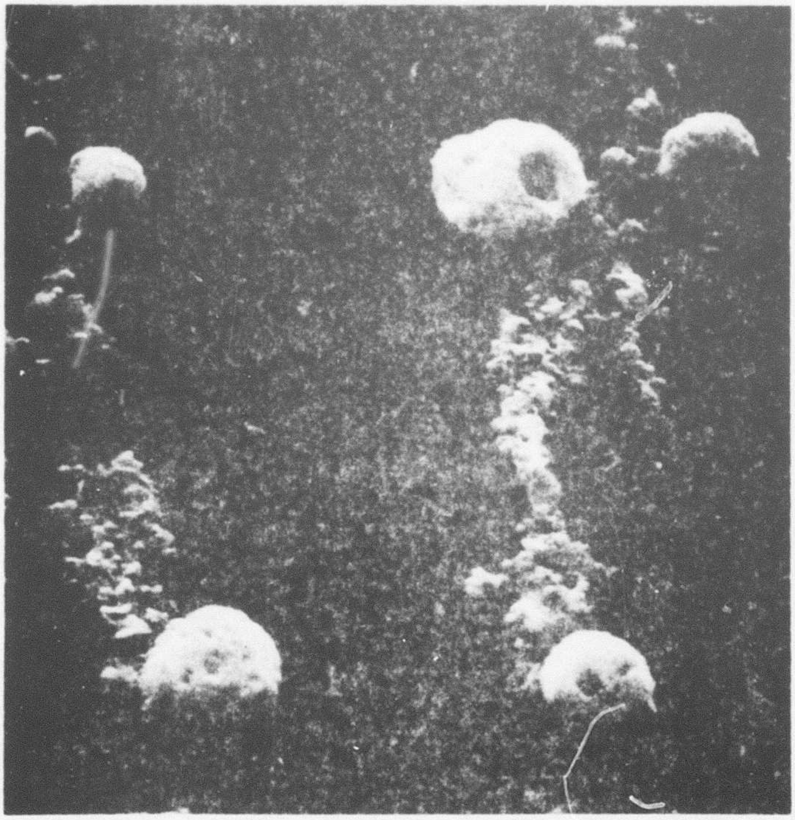


FIG. 2 (x 8,500)

Typical spherical shaped particles

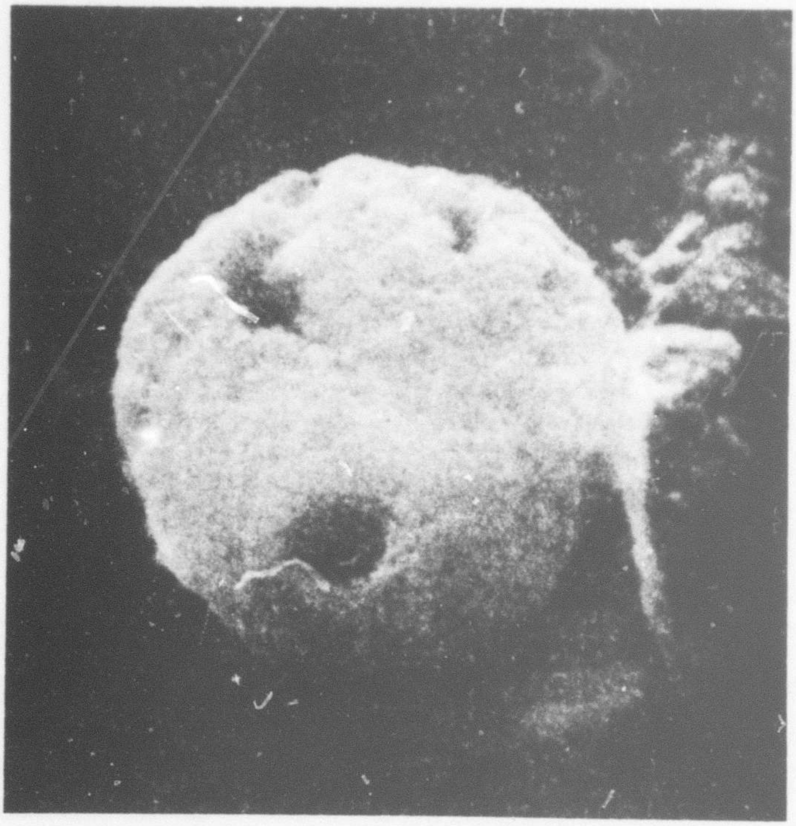


FIG. 3 (x 18,000)

Rough surface appearance of a spherical particle containing holes

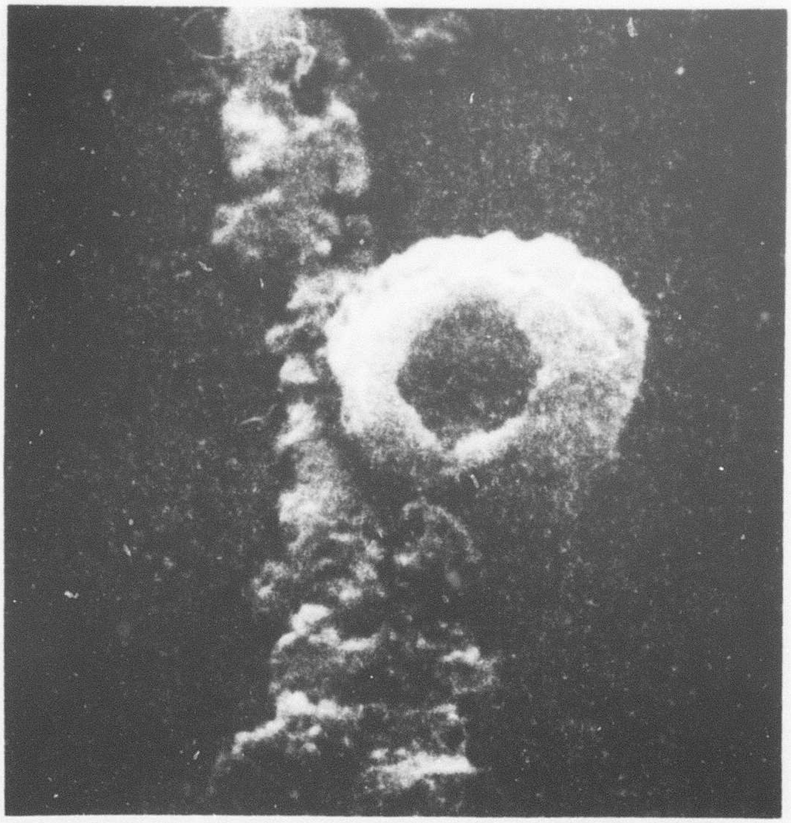


FIG. 4 (x 21,000)

Well defined large, hexagonal shaped hole in a spherical particle

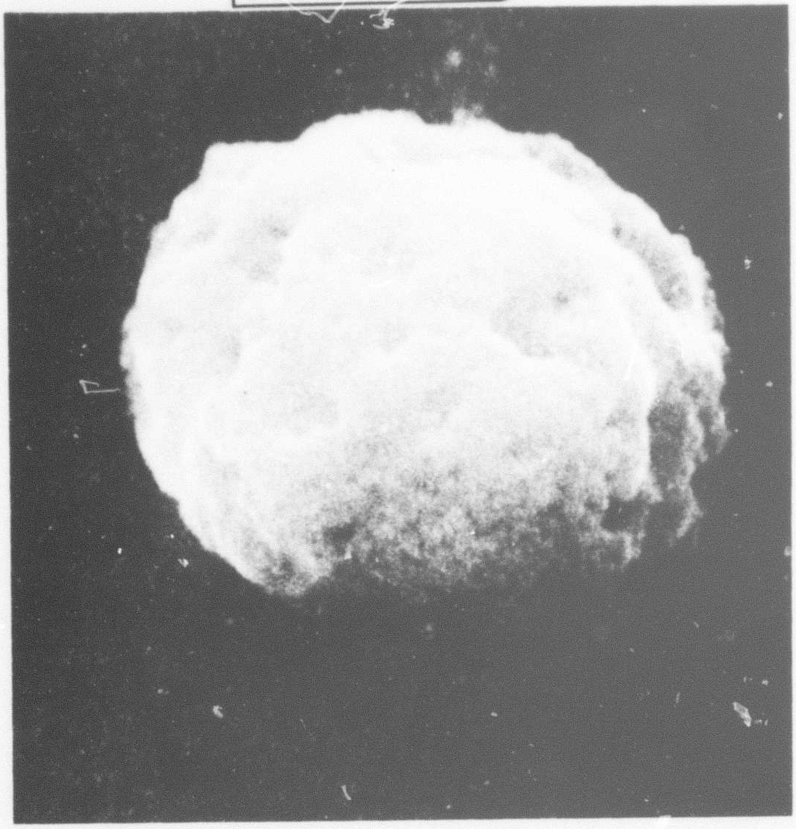


FIG. 5 (x 16,500)

Rough surface
appearance of a
spherical particle

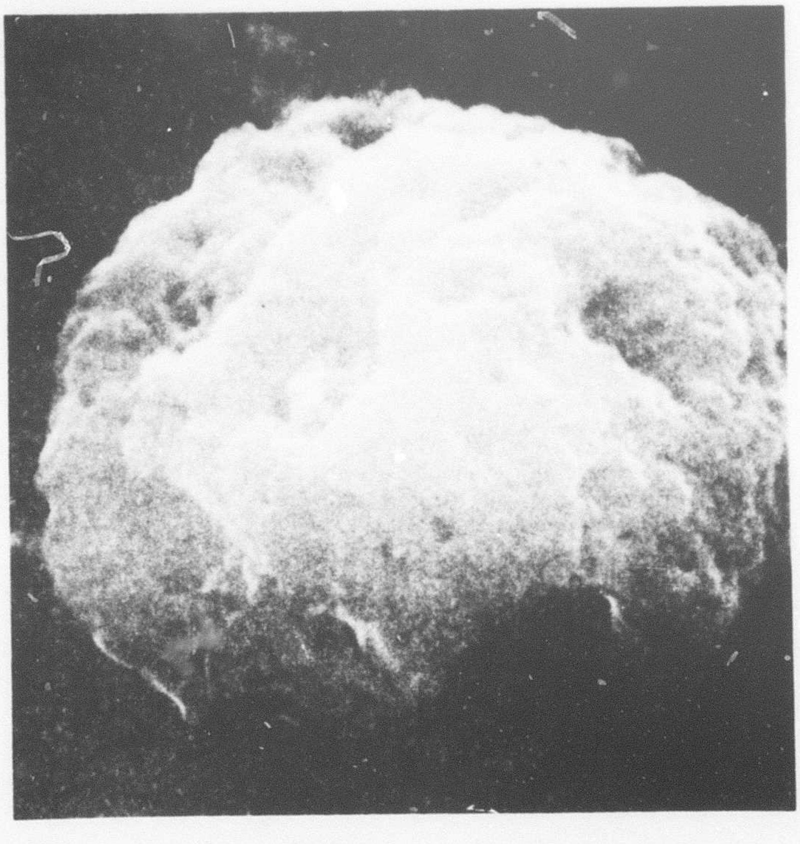


FIG. 6 (x 18,000)

Rough surface
appearance of a
spherical particle

FIG. 7 (x 22,000)

Small hole in apparently hollow spherical particle

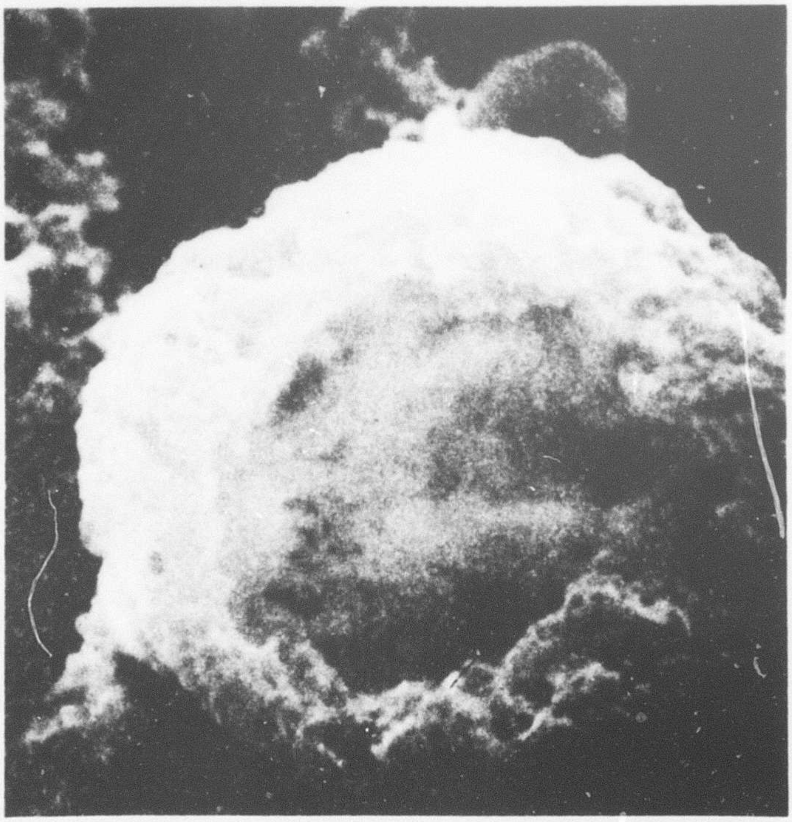


FIG. 8 (x 18,000)

Large hole in a spherical particle

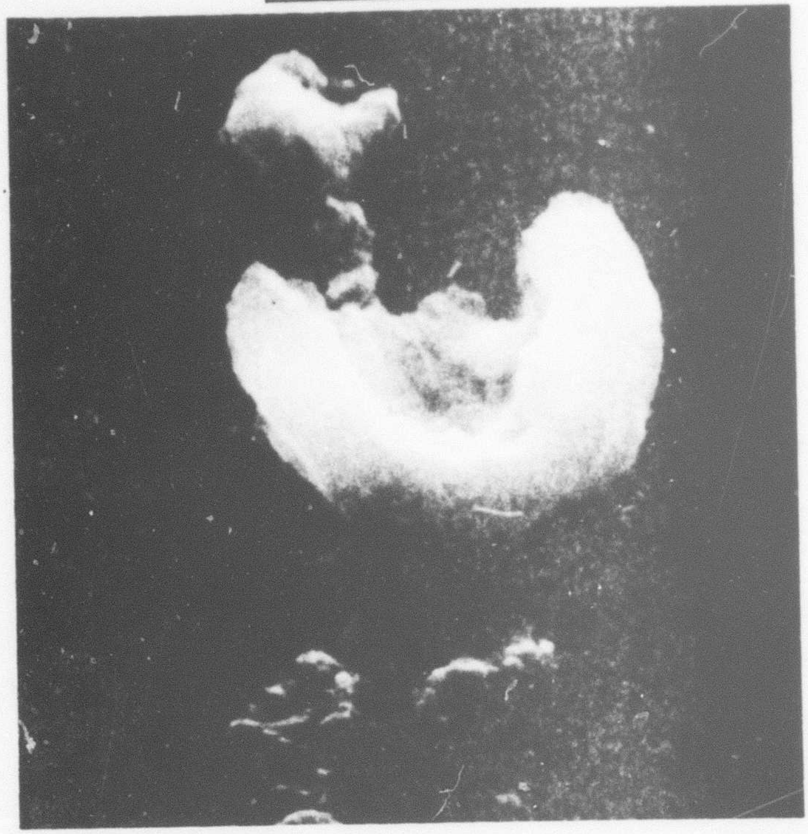


FIG. 9 (x 16,500)

Broken or partially disintegrated spherical particle

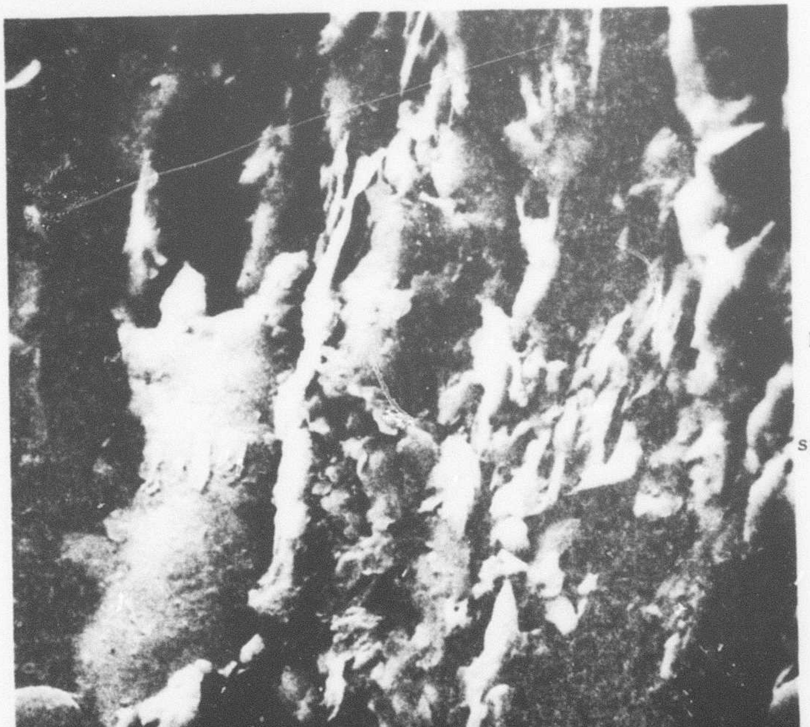
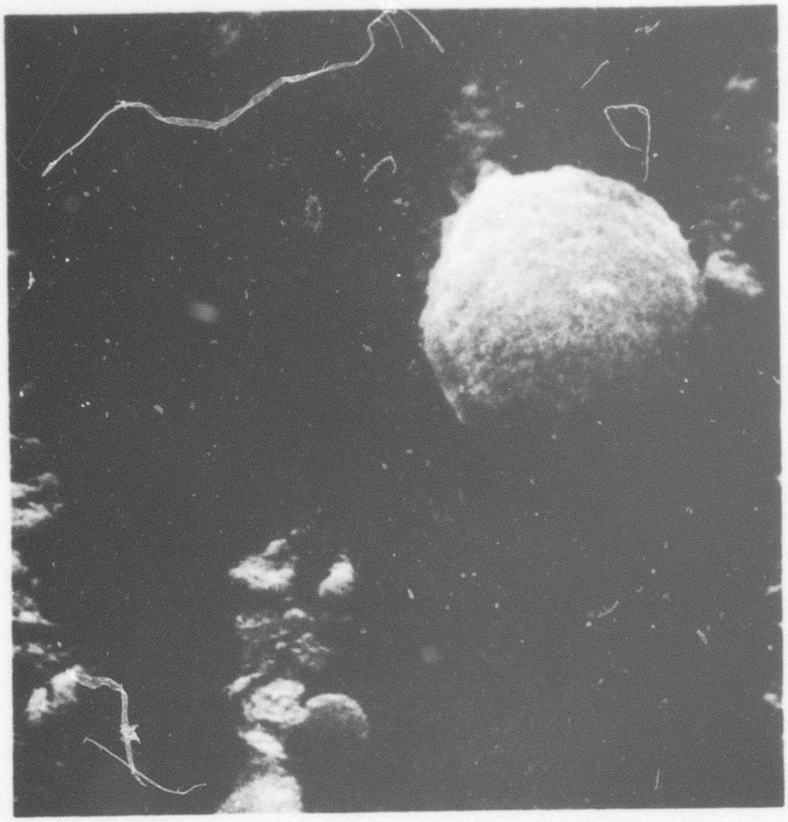


FIG. 10 (x 1,250)

Comparatively smooth surfaced spherical particles in the entry deposit



(x 9,000)

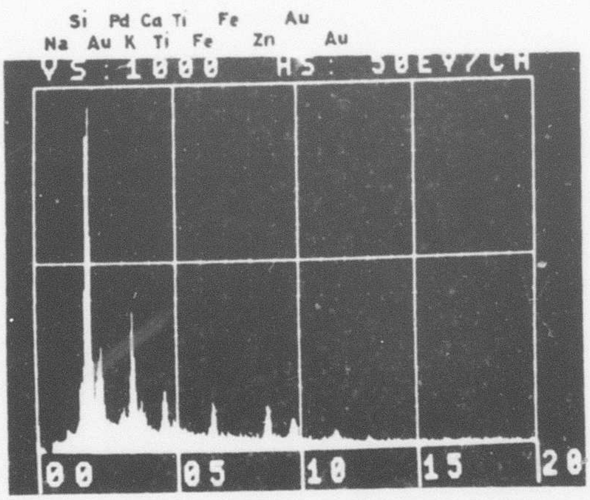


FIG. 11 X-ray energy analysis spectrum of the spherical particle

(x 19,500)

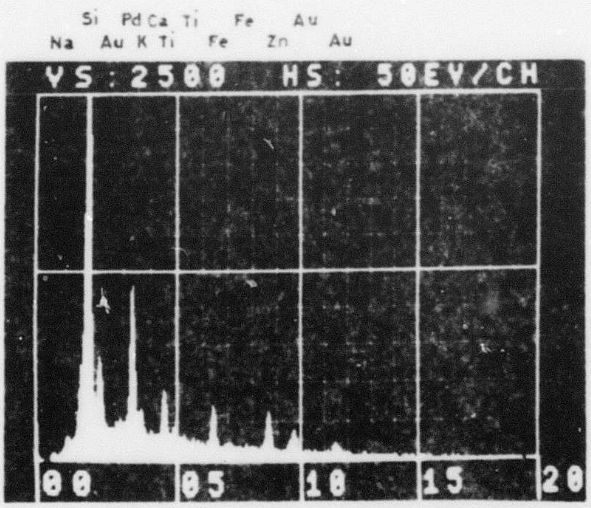


FIG. 12 X-ray energy analysis spectrum of the spherical particle

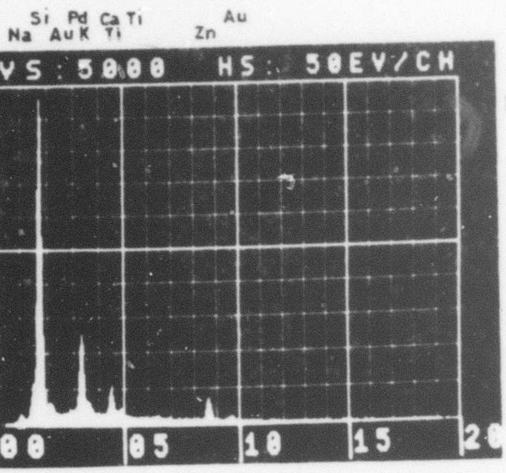


FIG. 13 X-ray energy analysis spectrum of the Ferrogram background

FIG. 14 (x 3,500)

Large piece of metal with surface markings

FIG. 15 (x 8,500)

Cutting wear particles

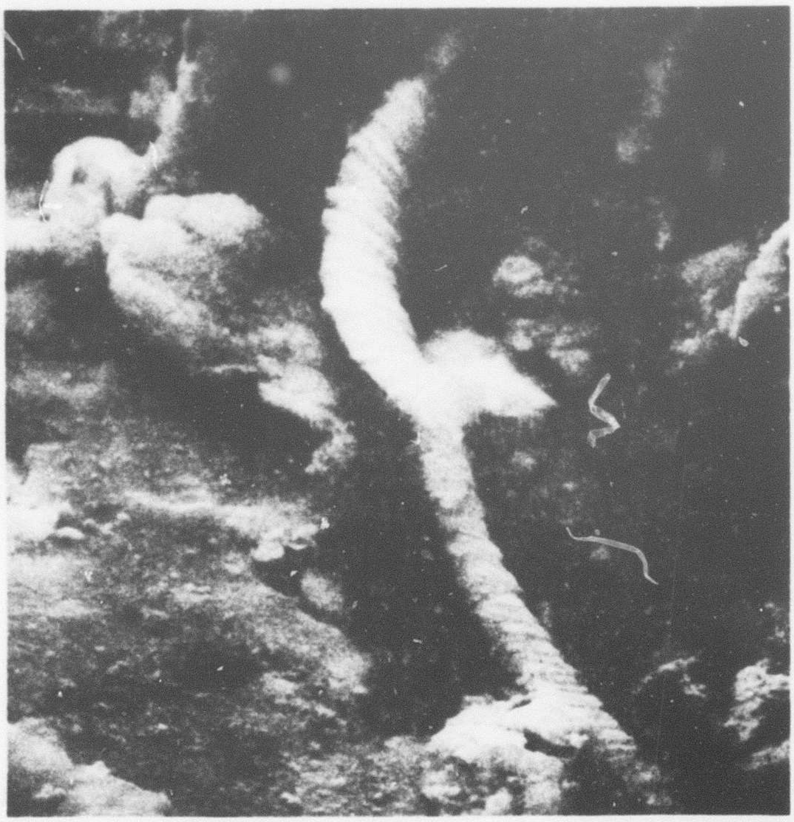


FIG. 16 (8,500)

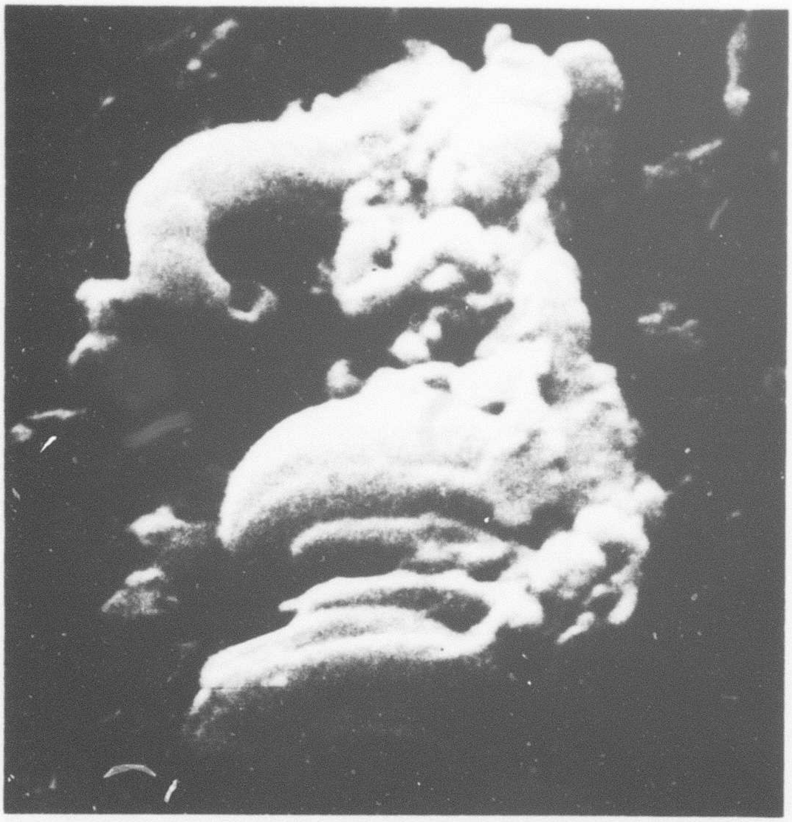


FIG. 17 (x 24,500) Unusual particles

5. THE SHEAR MIXED LAYER

During the study of particles from regimes 1-3, certain rubbing wear particles were found to be very soft. While manipulating the wear particles on Ferrograms it was noted that some particles could be smeared onto the glass surface of the Ferrogram (see Fig. 5A) by pressing a rod against them. Others do not smear. The ease with which the shear mixed layer moves along the worn surface also suggests that the layer may be either temporarily or permanently soft.

The particles that smeared were generated from 52100 steel with a hardness of 63 Rockwell C. An unused 52100 steel ball was rubbed against a glass slide and no material was removed. Thus it is concluded that the formation of the shear mixed layer may result in a softer material on the surface.

Since the layer is thin, often less than 1 μm in steel, there are no accepted methods of assessing the hardness of this material. It should be possible to estimate the layers' hardness by rubbing particles against materials of known hardness and observing the degree of smearing that results. It is felt that further work in this area is desirable as a knowledge of the properties of this layer is fundamental to the understanding of mild sliding wear.

FIG. 5A   Smeared Shear Mixed Layer

The studies of the regimes of wear reported here indicate that long life operations with boundary lubrication requires a stable shear mixed layer in which the generation rate equals the loss rate. If the loss rate is higher than the generation rate the layer is abraded away and a transition to a higher wear mode results. Consequently, an understanding of the physical and chemical properties of this layer is of significance to the reduction and stabilization of the wear process.

During the break in period, characteristic large particles are generated. As a machined surface breaks in, a shear mixed layer forms on the peaks of parallel grinding ridges. This layer flattens out to form cornices or overhangs along the ridges, (see Fig. 5B). These cornices subsequently break off forming long thin flat particles typically 15 μm X 3 μm and 3/4 μm thick, (see Fig. 5C). These particles will be generated in decreasing quantity until the original machining grooves are worn away or covered over.

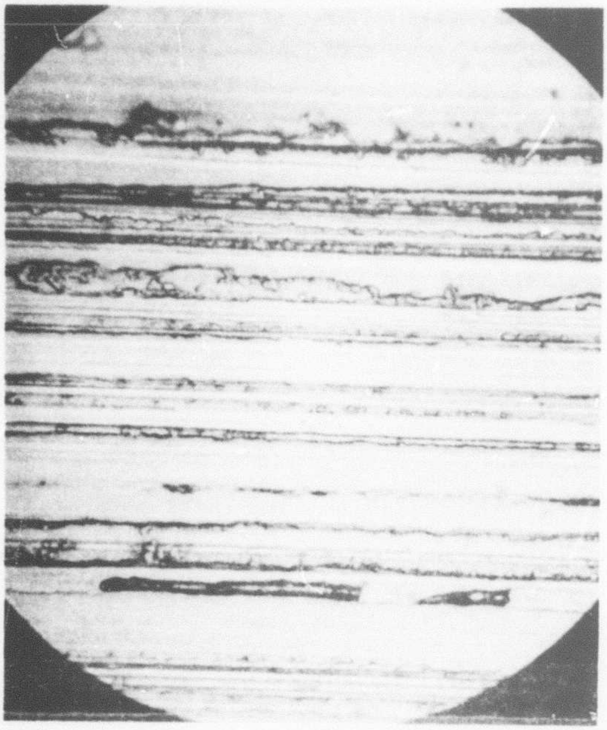


FIG. 5B

Partially broken in wear surface. Originally a ground finish.

FIG. 5C
Break in particles formed during the wearing of a ground surface.

6.0 CONCLUSIONS

6.1 Identification

The identification of the materials of which wear particles are composed is extremely useful in localizing a failure mode and is helpful in evaluating the severity of the wear process. Visual examination of Ferrograms in the microscope permits direct identification of most of the common engineering materials such as steel, bronze, aluminum and several others. In addition, a variety of oxides, such as hematite Fe_2O_3 may be identified by simple inspection.

However, on occasion, particles of foreign or unexpected materials are seen and it is most useful to be able to identify such particles quickly. Heating a Ferrogram to 330°C (625°F) for 90 seconds has proven a quick and reliable method for distinguishing low alloy steels from high alloy steels and as a method of confirming visual observation of brass, bronze, copper alloys and aluminum.

Polarized light is useful in identifying quartz, polycrystalline hematite and certain oriented plastics such as nylon (as distinguished from glass fibres) etc.

Wet chemistry is practical without losing the particles. However, wet chemistry methods are not presently considered for field applications, largely because other simpler and faster methods appear to be possible.

Individual particles may be removed from Ferrograms and analyzed. Because of the large number of particles present, such methods are too tedious for ordinary machine diagnostics. However, such methods are useful for research and may be very important when dealing with tests on new engineering materials.

The use of a scanning electron microscope with energy dispersion X-ray analysis permits direct analysis of individual particles on a Ferrogram. It is quick and practical to determine the percentage composition of various heavy elements including most engineering metals. The technique may be used with particles as small as 1 X 1 X 1 μm. The technique is particularly useful for engineering tests of machines or materials where the answer to specific questions such as "what is the source of that particle" may be answered.

For general field work, faster techniques in which all of the particles on a Ferrogram are analyzed at once, are indicated. Causing the particles to take on specific colors as the results of interference colors, or by converting the metals to compounds having specific colors, appears to be practical particularly if the chemical reactions can be carried out from the gaseous phase. Preliminary tests indicate that such reactions are possible and may be simple and definitive.

6.2 The Regimes of Wear

As a result of a series of tests to determine the relationship between the running condition and the type of particle generated, it was recognized that the rubbing wear of steel surfaces could be classified into six regimes. Each regime produces a typical type of particle and the wearing surfaces for each regime have certain properties which are characteristic of the regime. By considering these regimes, much of the divergence in the results of tribological research can be understood. Further, it is noted that while the stable running of sliding surfaces on machines involves running in regimes 1 and 2 much of the reported tribological research is concerned with the operation of surfaces in regimes 3, 4, 5 and 6. While such knowledge is certainly of great importance in a scientific and general sense it is, nevertheless, true that a great deal of tribological research reported to date is concerned with modes of wear which occur only as a machine is failing.

6.3 Friction Polymers

During this program, a number of tests were conducted to determine what types of wear particles are generated by various wearing situations. It was noted that friction polymer could be recovered from the oil as the result of steel wear particles which were embedded in the polymer. The ability to recover the polymer in milligram quantities has made it possible to study the physical and chemical properties of the substance.

Two general type classes of polymer were observed. The first is gel like, has no definite shape and has been found in MIL-L-23699 oil having a "light" additive package. The second type obtained from MIL-L-3699 oil, with a heavy load additive package, forms a tenacious film which is rolled up by the relative motion of the wearing surfaces to form rolling pins, often 5 μm in diameter by 20 μm long. The oil forming such rolling pins exhibited a Rider gear test rating 1-1/2 times higher than a similar oil that did not generate the pins. It is believed that the pins are indicative of a tough friction polymer film which, in turn, seems to prevent adhesion between surfaces.

6.4 "Break In"

During the course of this work, numerous tests were carried out and in each case it was observed that during "break in" large particles were produced initially. As the surfaces adjusted to each other the maximum size of the wear particles decreased, often to less than 15 μm. When, however, the wearing surfaces started to fail and become rougher with time, the maximum size of the particles increased by as much as one or two orders of magnitude.