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SURFACE INTERACTIONS AND LUBRICATION RESPONSE OF SILICON NITRIDE BEARING ELEMENTS

H. Dalal, et al

SKF Industries, Incorporated

Prepared for:

Naval Air Systems Command

February 1974

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FINAL REPORT

#### **ON**

SURFACE INTERACTIONS AND LUBRICATION RESPONSE

OF SILICON NITRIDE BEARING ELEMENTS

February, 1974

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The need for a light-weight bearing material in clinical benefits of speed turbine applications is discussed and the potential benefits of silicon nitride in particular are reviewed. The wettability characteristics of six lubricants on a silicon nitride surface were measured to determine which lubricants are apt to be successful with silicon nitride bearing components in permitting the replenishment of lubricant onto the rolled-over tracks which is vital for the maintenance of an elastohydrodynamic (EHD) film and the consequent prevention of surface initiated fatigue.

Lubricant film thickness and traction force were measured with an BBF optical EHD apparatus. Friction and wear studies were conducted under subcontract by Prof. E. Rabinowicz of M.I.T. Seven endurance tests were run at BBF using different combinations of tool steel rollers and steel or Si<sub>3</sub>N<sub>A</sub> flats.

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Rolling Contacts							
Elastohydrodynami	ic Lubrication						
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### SURFACE INTERACTIONS AND LUBRICATION RESPONSE OF SILICON NITRIDE BEARING ELEMENTS

### 1. SUMMARY

This is the Final Report summarizing progress from November, 1972, through November, 1973, on a fundamental investigation conducted under Contract NOOO19-73-C-O150, of the suitability of silicon nitride as a rolling bearing material with emphasis on its interactive behavior with lubricating fluids.

This report contains the following sections:

<u>Background</u>. The need for a light-weight bearing material in critical high-speed turbine applications is discussed and the potential benefits of silicon nitride in particular is reviewed. There follows a discussion of the methods in use for producing silicon nitride, and a listing of the various physical properties of the hot pressed silicon nitride used in this program.

<u>Wettability</u>. The equipment and procedure, which were used to study the wettability characteristics of six lubricants against a silicon nitride surface, are described. These studies were made to determine which lubricants are apt to be successful with silicon nitride bearing components in permitting the replenishment of lubricant onto the rolled-over tracks which is vital for the maintenance of an elastohydrodynamic (EHD) film and the consequent prevention of surface initiated fatigue. The contact angle between the fluid and solid surface was used as an indicator of the wettability. The six lubricants used were: (1) Two synthetic paraffinic hydrocarbons with and without additions, (2) two ester oils with and without additions, (3) a mineral oil and (4) a polyphenyl ether.

Optical EHD Studies. A test rig developed by ESF for measurement of lubricant film thickness and traction force in elastohydrodynamic contacts of a ball or roller and a glass flat is described. The same six lubricants, that were used in the wettability study, were tested with the optical EHD apparatus. Fringe photographs of the contacts between a transparent glass flat and silicon nitride balls were obtained. In order to obtain clearly visible fringes the optical properties of the test element (1/2 inch diameter silicon nitride ball) and those of the

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coating of the glass flat must be well matched. The optimum reflectivity of the coating of the glass flat was determined theoretically and confirmed experimentally. Suitable coatings were selected. A series of fringe photographs were obtained on the optical EHD tester at two loads and rolling velocities in the 5 to 400 inch/sec. range with  $Si_3N_4$  - glass contacts.

<u>Traction Tests</u>. The optical EHD rig was also used to measure the traction force at the contact between the flat and the ball, each of which is connected to a driving spindle. The sliding rate is defined as the difference in surface speed of the two bodies. Tests were run to determine the traction coefficient as a function of sliding and rolling velocity, for the six test oils. Data were obtained for steel/steel, steel/silicon nitride and glass/silicon nitride contacts.

<u>Sliding Friction and Wear Studies</u>. A two-dimensional statistical description of a rough surface based on a paper written by Longuet-Higgins was presented and applied to the surface of a ground silicon nitride plate for comparison with similar measurements on ground steel surfaces. Friction and wear studies were conducted under subcontract by Professor E. Rabinowicz of MIT. In these tests a "pin on disk" apparatus was used. The top specimen is a 1/4 inch diameter rod which was pressed against the bottom specimen, a plate rotated at a speed of 0.3 cm/sec in the friction tests and 30 cm/sec. in the wear tests. A "Lapmaster" abrasive wear test apparatus was also used in some tests.

<u>Flat Washer Surface Interaction Studies</u>. The flat washer tester contains three rollers, guided by a plastic or metal cage, and rolling under load between two flat washers. In the tests under the present contract, the rollers were made of M50 or Rex 49 tool steel, the top washer was made of 52100 steel and the bottom washer of silicon nitride or 52100 steel. Seven endurance tests were run using different combinations of rollers and flats with an ester-base lubricant.

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#### CONCLUSIONS

1. There are considerable differences between the wetting behavior of the six test lubricants against a silicon nitride surface. The contact angle varies from 5° (for Mobil DTE Medium Heavy Mineral Oil) to 37° (for the five-ring polyphenyl ether OS124). The lowest contact angles were obtained with the DTE Medium Heavy oil and the Mobil Jet II. The contact angle was found to be larger in the direction perpendicular to the surface lay than parallel to it (for the same surface roughness in both directions). The lubricants, with one exception, give smaller contact angles for a smoother surface (1.5  $\mu$ in CLA surface roughness) than for a rougher surface (15  $\mu$  inch CLA surface roughness).

2. The optical EHL test rig at 2000 was found to be suitable for film thickness studies using silicon nitride/glass contacts. CeO and SiO coatings on the glass flat with a reflectivity of approximately 25% were found theoretically and experimentally, to give optimum results.

3. The plateau film thickness measured from the fringe photographs, at two loads and 9 rolling velocities in the 5-400 in/sec range is smaller for the silicon nitride contact than for the steel contact by 15 to 20%. The computed film thickness overestimates the measured film at high velocities.

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1. A study of the oil meniscus lines (indicating partial EHD starvation conditions) in the fringe photographs of steel-glass and silicon nitride-glass contacts shows that the steel-glass contact is somewhat less starved than the silicon nitride contact, especially at higher speeds. Nevertheless, the fringe photographs indicate that the silicon nitride contact has satisfactory film building behavior close to what is expected by current EHD theory.

5. The traction coefficient in a silicon nitride/glass contact was found to rise with <u>sliding</u> speed and then level off, as in previous tests with steel contacts. This shape of the curve was typical for 4 of the 6 oils tested. The other two curves reached a maximum and then decreased with <u>sliding</u> speed Test data also showed that the traction coefficient increases with load but decreases with <u>rolling</u> speed, a trend which is consistent with previous traction tests in steel contacts. The effect on traction of additives in paraffinic and ester oils is small.

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### 2. CONCLUSIONS

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The two paraffinic oils give the lowest traction coefficients, while the mineral oil and the polyphenyl ether exhibit relatively high traction. Tests with steel/steel and steel/silicon nitride show that at the highest stress level, 271 ksi, the traction for the two types of contacts differ by no more than 8%. At lower stress levels, 216 and 247 ksi, the differences are larger, in one case by more than 20%. The traction coefficient in the silicon nitride contacts is generally lower than that prevailing in steel contacts.

6. Results of the "pin on disk" tests at MIT indicate that the friction coefficient on ground silicon nitride surfaces is lower when the pin traverses across the grinding grooves than when it runs parallel to the grooves. Teresso V78 and Turbojet 2380 lubricants gave the lowest friction coefficients with a slight margin. Some of the lubricants tested, and especially the silicone, give higher friction coefficients than the unlubricated conditions.

7. Tests on the "Lapmaster" abrasive wear test apparatus at MIT using 600 grit silicon carbide as abrasive showed that silicon mitride results in an order of magnitude greater abrasive wear coefficient than the three other ceramics tested. The wear resistance of silicon mitride under lubricated sliding contact (in the "pin-disk machine") is found to be somewhat higher than for M50 steel. The wear coefficient of 2.2 x 10<sup>-6</sup> is reasonable for a lubricated sliding syste: involving non-metallic materials.

8. Wear debris collected from the Lapmaster wear tests was examined at BIGF using a Scanning Electron Microscope. The SEM photograph of the debris shows the presence of faceted particles. This supports the contention of silicon nitride being brittle on the microscale, under the stresses created by lapping.

9. The very limited fatigue tests at dissip covered in this report, run with an ester lubricant in a heavily loaded roller/ flat washer configuration, have produced much shorter lives than predicted, both in baseline runs of ste'l/steel and in runs of silicon nitride/steel. (Further tests not reported here, under slightly modified conditions, gave normal lives). In the tests covered here, all fatigue was at the rollers, but there was wear at silicon nitride washers. Effects of lubrication, stress level, slide/roli ratio and surface roughness on fatigue life of silicon nitride all require further study. However, the tests reported do show that silicon nitride does not undergo catastrophic failure under rolling contact conditions.

#### 3. BACKGROUND

# 3.1 The Need for Ceramic Bearing Research

Enclosure 1 taken from  $(1)^*$  shows the estimates of six aircraft engine manufacturers of the industry's requirements up until 1975. It is seen that future aircraft engine bearings are expected to operate at speeds of 3 x 10<sup>6</sup> DN (DN = bearing bore diameter in mm times shaft speed in rpm) and temperatures in excess of 700°F. These requirements will pose a great challenge to proper bearing lubrication and life expectancy.

The predominant force on the balls in high speed engine bearings is the centrifugal force which induces such high outer ring contact forces that bearing life is reduced drastically as engine bearing speeds increase. A substantial portion of this loss in contact fatigue life can be regained if light-weight balls are used. Reducing the weight of steel balls by making them hollow introduces substantial manufacturing problems, such as the difficulty in achieving sufficient accuracy of the hole location. Endurance testing of hollow ball bearings has shown that the ball failure mode changes from contact fatigue to bending fatigue producing much shorter lives. Solid balls of low density bearing materials therefore are preferred.

Recent advances in ceramics technology have overcome many of the factors which have militated against the use of these materials as rolling bearing elements, notably porosity and excessive brittleness. The low density of ceramics coupled with their stability and high hardness levels at elevated temperatures have long made them appear desirable for use in bearings operating at extreme conditions. It can be exp^cted that the use of new, stronger ceramic materials will offer improvements in high temperature properties over steels, and due to low weight, be favorable where centrifugal loading is critical.

\*Numbers in parentheses refer to List of References at the end of this report.

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It has only been recently however, that ceramic materials have been developed which show signs that the problems of brittleness and poor fatigue properties have been overcome to the degree required for reliable, long-lived rolling bearings. One such material is a new silicon nitride ceramic as currently made by the Norton Co. under a license from the U.K., and it is projected that bearings or bearing parts such as falls and rollers can be successfully made from this material.

The complex interactions that exist between bearing elements, surface finishing methods, lubricants and the environment prevent the direct extrapolation of performance of steel bearings, to bearings made of ceramic materials. Specifically, the elastic moduli of ceramics are significantly higher than those of steels, and, therefore, the use of standard bearing designs and loading practices would result in extremely high Hertzian stresses at the contacts. Certain tensile stresses in the Hertzian field may be critical in ceramic bearings instead of the Hertz shear stresses used in the design of steel bearings. Modified designs and loading rules will be required in order to reduce these stresses to an acceptable level. This acceptable level is unknown for ceramics. New designs will be required to resolve cooling of the bearings in view of the low thermal conductivity of ceramics.

# 3.2 The Importance of Surface Interaction and Lubrication Effects

Surface initiated spalling is a principal cause of failure in aircraft jet engine mainshaft ball and roller bearings (and many other types of rolling bearings). Surface initiated spalling has been observed to be critically dependent upon lubrication conditions and surface characteristics of the bearing materials (2).

Previous work under Contracts N00010-68-C-0310 and N00019-71-C-0425 have shown that there is a reduction of film thickness around the shoulder of surface furrows. Also, intimate surface contact can take place between asperities if there is insufficient lubricant film thickness. The existence of these microscopic imperfections prevents idealistic film profiles from being obtained in their vicinity. However, there is reason to believe that the severity of stress concentration at the micro-defect is influenced by both the lubricant film pressure and the bearing material strength and ductility characteristics. This stress concentration affects the surface fatigue life through its influence on the onset of surface\_distress (3).

In addition to the above lubrication-related bearing failure modes from surface defects, high-speed bearings sometimes suffer a type of surface failure known as skid damage which is distinctly different from the above-mentioned surface distress. Skid damage apparently is related to the occurrence of abrupt load changes in the presence of substantial sliding at the Hertzian contacts in bearings, and thus the occurrence of this damage can be influenced by the traction or sliding friction characteristic of the lubricant and bearing material interface as it affects the bearing kinematics.

The differences in response between ceramics and steels to manufacturing processes will undoubtedly produce new features of surface microtopography. These features will affect both the traction forces at the contacts and skidding and wear damage when surface asperities penetrate the EHD film. Additionally. the differences in surface chemistry between ceramics and steels suggest that the wetting behavior of lubricants, the response to boundary lubricant additives, and the susceptibility to stress corrosion cracking in chemically active environments will be significantly different. Finally, the fatigue and galling response of ceramics in Hertzian contacts is poorly understood and needs to be evaluated. All these novel problems support the need to conduct experiments into the behavior of ceramic materials in lubricated rolling contacts and to obtain the knowledge necessary to predict the behavior to be expected in full scale bearings.

# 3.3 Progress in the Application of Ceramics For Bearings

Early efforts at screening a wide variety of available ceramic materials for bearing applications met with only limited success due to the inherent porosity and lack of homogeneity of the then available materials (4,5). A comprehensive study of the application of ceramics technology to rolling bearings about ten years ago resulted in a bearing with the best available materials which gave remarkably promising performance (6). This bearing is illustrated in Enclosure 2 showing its good condition after successful operation at high speed (8000 rpm) and temperature (1500°F). This bearing was equipped with cemented carbide rings, ceramic balls and molybdenum cage and lubricated with  $MoS_2$  powder in argon carrier and thrust loaded to 300,000 psi contact stress. Some deterioration of the contact surfaces in the raceway tracks was evident after this testing. These poor surface fatigue characteristics (presumably from poor structural integrity of the then currently available commercial compositions) have limited rolling-bearing use of ceramics.

The advantage of using ceramics for rolling bearing components stem from their following characteristics:

- a) high strength and hardness at room as well as at elevated temperatures
- b) low density
- c) low material cost, and
- d) excellent corrosion resistance.

The main disadvantages of ceramics are

- a) brittleness.
- b) high notch sensitivity,
- c) low thermal conductivity, and
- d) high finithing costs to accurate bearing tolerances.

The high modulus of elasticity of most ceramics results in high bearing stiffness but also in high contact stresses causing increasing severity of lubrication conditions.

Of the various ceramics tested for structural applications silicon nitride has shown the best performance (13, 14). It has out-performed other ceramics in terms of thermal shock resistance and resistance to wear. Silicon nitride therefore is an obvious candidate material for bearing applications.

Silicon nitride has two allotropic forms. The low temperature d -phase transforms to the high temperature Ø -phase between 2700°F and 2900°F. Both forms have a hexagonal crystal structure.

Structural components of Si N can be manufactured in one of two ways. 34

a) Reaction Bonding: This method tends to leave 15-20% porosity in the part. It is, therefore, suitable only for the manufacture of stationary parts that are not subjected to high stresses or impacts.

b) Hot Pressing: Fully dense parts are produced by this method.

Rolling bearings require materials of construction which have superior performance characteristics from several different points of view. The desirable low density characteristics of silicon nitride must be complemented by other superior properties for it to become a viable rolling bearing material. The most essential properties for ball or roller and ring materials are

- 1. High hardness under operating conditions
- 2. Adequate contact fatigue strength
- 3. Adequate ductility

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- 4. Adequate impact strength
- 5. Dimensional stability
- 6. Low friction and wear
- 7. Wettability by lubricants 8. Manufacturability
- 8. Manufacturability to precise tolerances.

Properties of silicon nitride are compared with those of typical bearing steels in Enclosure 3. These properties have only recently been improved to the point where silicon nitride can be considered for use in rolling bearings. However, some properties, such as elastic modulus and thermal expansion coefficient, are sufficiently different from those of steel that modifications of standard bearing design practices will be required to utilize silicon nitride effectively in rolling bearings. Other properties, such as the high hardness, will require development of special manufacturing techniques to produce bearings. Some important properties of hot pressed silicon nitride produced by the Norton Co. are listed in

More details on the properties and manufacturing of silicon nitride specimens are given in the First Quarterly Report (8) on this contract.

To date very little is known about the behavior of Si  $N_4$ in rolling contact. A few preliminary tests have been performed at NASA by Parker and Zaretsky (7). The tests were conducted in a five-ball tester under the following conditions:

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Max. Hertz stress (psi) Shaft speed (rpm) Contact angle (degrees) Race temperature (°F)

The above tests were performed on a group of 20 balls. The results of the tests are given below:

L <sub>10</sub> , stress cycles	$2.5 \times 10^{6}$
L <sub>50</sub> , stress cycles	17 x 106
Weibull slope	0.99
Failure index	19 out of 20

A few of the spalled balls produced in the above tests were studied in a scanning electron microscope at  ${\mathbb E} {\mathbb E} {\mathbb F}$  (see Enclosures 5 and 6. It is clear from Enclosure 5 that the morphology of the spalls on a  ${\rm Si}_3{\mathbb N}_4$  ball is similar to those found on steel balls. Enclosure 6 shows a lubrication distress zone at the end of the spall. The severity of lubrication distress around the spall seems to be greater than that normally observed on steel balls.

The fatigue life of  $Si_3N_4$  rolling elements has been found to be very sensitive to the processing technique used (23). Work at Norton has indicated that proper choice of processing steps can make a significant difference in the fatigue life of rolling elements. Enclosure 7 shows the surface of an unrun  $Si_3N_4$  ball. The surface is seen to be pitted. The presence of such pits can cause a collapse of the lubricant film and lead to failure due to lubrication distress. It is therefore imperative that proper surface finishing techniques be found to exploit the full potential of  $Si_3N_4$  for rolling contacts.

Other studies with the more advanced silicon nitride ceramics also have shown promising rolling contact fatigue life in element tests (23) with apparently some variability with surface condition and lubrication of the ceramic elements.

Principally under Navy contract, 2013 P has developed and operated an optical interferometric apparatus to define the parameters affecting elastohydrodynamic lubrication of ball and roller bearings. Theories of partial and full EHD behavior and of rolling contact fatigue have been developed and published by the 2023 P Laboratory (3, 15 and 17).

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## 4. DETAILS

# 4.1 Wettability Study

One of the requirements for good lubrication and heat transfer is that the lubricant wet the bearing elements under all intended operating conditions. Oil wetting of ceramic surfaces is not as certain as with metal surfaces, and the wetting behavior of six specified oils on a silicon nitride specimen was therefore studied.

The six lubricants used in these tests consist of the followina:

# Mobil Jet II (Mil-L-23699)

This is Mobil Jet II as formulated with XRM 234 plus a proprietary additive package.

#### XRM 177F 2.

The synthetic hydrocarbon designated XRM109F and described below has been modified by the addition of an additive package consisting of an oxidation inhibitor and anti-wear additive, to see what difference in surface properties may be induced by the use of these additives. This material is designated by the supplier (Mobil Oil Co.) as XRM 177F, and it has been tested extensively as a candidate advanced turbine engine mainshaft bearing lubricant.

# 3. DTE Medium Heavy

This is a conventional high quality Mobil turbine oil, refined from mineral oil and having added to it rust, oxidation and foam inhibitors. Its viscosity index is 95 and viscosities are 63.5 cs @ 100°F (295 SUS) and 8.2 cs @ 210°F (52.8 SUS).

# 4. XRM 109F

An unmodified synthetic hydrocarbon (paraffinic) which is made by polymerization of an alpha-decene. The particular material used is designated XRM 109F by Mobil. It has a viscosity of about 445 centistokes at 100°F and can be considered a quite pure fluid in the sense that the only constituents of the fluid are the products of the polymeric reaction with some removal of the light ends.

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#### 5. 0S124

This is a five-ring polyphenylether which has a viscosity of about 35 centistokes at 100°F. Without an additive package, it is supplied by Monsanto Chemical Co. under the designation OS124, and it is the basestock for an advanced military turbine engine lubricant.

#### 6. XRM 234

This chemical type of fluid tested is the base fluid of lubricants which are formulated to meet the specification MIL-L-23699 for aviation turbine engines. These fluids are mixed esters of polybasic and/or monobasic alcohols, the exact composition of which is generally proprietary with the particular supplier. The material selected for these studies is that which serves as the base of the Mobil Oil Co. product, Jet II. It has a viscosity of about 28 centistokes at 100°F.

A list of various properties of these six oils is given in Encl. 8.

The contact angle between the liquid surface and the solid specimen is commonly used as an indicator of the wettability of the liquid-solid interface.

An attempt was first made to use Adam and Jessop's (11) tilting plate method to measure the contact angle between the liquid surface and a silicon nitride specimen. This attempt did not, however, meet with much success due to the interference of the meniscus at the lubricant/glass interface on the sides of the container. Due to this interference, the angle between the plate and the oil surface cannot be clearly observed, making it difficult to obtain accurate and consistent readings. An Eberbach cathetometer was also tried without success. A modified drop method was finally used to obtain quantitative data on contact angles and is described below.

The procedure consists of projecting the shadow of a lubricant drop applied to the substrate, onto a photographic paper by means of a magnifying lens, developing the print, and measuring the contact angle of the image of the drop on the image of the plane surface of the substrate. Illustrative examples are shown in Encl. 9.

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The plates were prepared by cleaning with a nonionic detergent and rinsed with water and acetone between each measurement.

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The drops of oil, of approximately 0.01 mm dia., were delivered to the surface of the substrate from a fine hypodermic needle. The hypodermic needles and syringes were thoroughly cleaned before each use.

The drops of oil were carefully placed on the surface of the substrate and allowed to equilibrate for 30 seconds before projecting the image. After 30 seconds, the flow of the oil on the substrate appeared to stop and the image of the drop remained stable.

Two kinds of substrates were used for this investigation. A silicon nitride plate was surface ground to 15 microinch finish, having a uni-directional lay on the surface. The surface of the second silicon nitride plate substrate was diamond polished using 6 and 0.25  $\mu$ m diamond paste successively. The surface thus produced had a rcughness of 1.5 microinch with practically no directionality.

The flow of lubricant on the ground substrate was greater in the direction of the lay than across the lay, thus resulting in an oval drop as shown in more detail in the Second Quarterly Report (9). The oil flow was found to be uniform in all directions on the polished surface, resulting in a round drop.

The results of the measurements are shown in Encl. 10.

It appears from Encl. 10 that there are considerable differences in the wetting behavior of the lubricants tested. Mobil Jet II and the DTE Medium Heavy lubricants give the best overall wettability (the lowest contact angles).

Encl. 10 also shows that, in all cases except DTE Medium Heavy oil, the contact angle on the polished surface is lower than the highest reading obtained on the rougher surface.

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## 4.2 Optical EHD Studies

# 4.2.1 Description of EHD Test Rig - Mechanical System

A test rig, developed by 話 ほ伊 for measurement of lubricant film thickness and traction force in a lubricated elastohydrodynamic (EHD) contact of a ball or roller and a glass flat, is described below.

The basic test configuration used in this study is shown in Encl. 11, and consists of the contact of a roller or 1/2" diameter ball and a rotating transparent disk. The optical system, situated above the disk, is used to photograph the interference fringes formed by lubricated contact between the ball and disk.

In order to control the rate of sliding between the roller or ball and the disk, the ball is driven by a quill through a flexible coupling, and the disk is connected to a vertical spindle. Figure (a) in Encl. 11 shows that the test ball is supported below by a disk which is actually a ring of a thrust ball bearing. The load is applied through this support bearing.

Figure (b) is a plan view of the test assembly. Three balls or rollers, equally spaced on a 1.5" pitch circle, are used to support the disk, one of the balls being the test ball. The test ball is restrained by a spring cage. The lateral force acting between the ball and cage is measured by a strain gage mounted on a leaf spring beam, and the torque in the driving

quill is measured by a torque meter. The strain gage and torque sensor outputs are used to compute the traction in the contacts (15).

Encl. 12 is a photograph of the entire assembly. The device which loads the three balls (or rollers) against the transparent plate consists of a flexible diaphram, covering a cylinder into which controlled air pressure is introduced. The diaphram exerts a force on the lower thrust bearing washer, through an adapter. The load applied to the observed contact is controlled by means of a calibrated mercury manometer.

The test lubricant is gravity fed to the Teflon cage by a simple once-through system. The oil flow rate can be varied. The test lubricant temperature is measured by a thermocouple probe installed approximately 1/16" from the inlet of the contact.

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Both the vertical and horizontal spindles are driven from one variable speed electric motor. A variable-ratio positive drive between the motor and the vertical spindle allows rolling with a controlled magnitude of sliding between ball or roller and plate. The degree of sliding can be changed if desired while viewing the contact.

#### 4.2.2 Description of EHD Test Rig - Optical System

The layout of the optical system, consisting of microscope, light source and flats, is shown in Encl. 13.

Glass flats of high optical quality and low  $(0.2 \ \mu \text{in rms})$ surface micro-finish were obtained. A high efficiency antireflective coating is applied to the upper surface of the glass flats giving approximately 0.2% reflectivity.

A high intensity pulsed Xenon lamp of short pulse duration (1.25-7  $\mu$  sec) is used as the light source for taking the fringe photographs. The maximum pulse rate is 1000 Hz, and the intensity of the lamp is 20-300 x 10<sup>6</sup> beam candles. An external capacitor is used in the circuitry to increase the lamp intensity to 10<sup>9</sup> beam candles and the pulse duration to 9  $\mu$  sec.

#### 4.2.3 Test Lubricants

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A total of six lubricants were selected for testing in the optical EHD rig. These are the same lubricants as those used in the wettability tests (Section 4.1). The oil trade names and their properties are listed in Encl. 8. Encl. 14 is an ASTM standard viscosity temperature chart for the six oils. As the chart shows, the oils cover a wide range of viscosities.

#### 4.2.4 Film Measurement Techniques

The fringe photographs in Encl. 15 show typical contacts between a steel ball and the transparent plate under MIL-L-23699 lubricated conditions, consisting of a relatively flat plateau and a slender constriction at the back and side edges of the contact circle. The film thickness at the plateau varies with the operating and lubricant parameters. Because of the relative uniformity of the plateau film thickness, the

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fringegrams when viewed through the eye piece of the optical system, exhibit a dominant color which can be related to film thickness by means of a calibration process. The fringe order of the color can be readily identified by increasing the speed gradually from zero.

Encl. 16 is a calibration chart relating color to film thickness for a standard fluid (quinoline) with atmospheric pressure refractive index  $n_0 = 1.625$ . For other fluids under high pressure having an effective refractive index  $n_p$ , the film thickness obtained from this calibration chart is multiplied by the factor  $(1.625/n_p)$ .

The refractive index of oil in a high pressure Hertzian contact  $(n_p)$  is greater than that measured at atmospheric pressure. The high pressure refractive index can be calculated using the Lorentz-Lorentz equation, together with hartung's equation for estimating density at high pressure. A computer program was used to calculate the effective refractive indices using ambient viscosity and atmospheric refractive index as input. The values of the refractive index of the six lubricants listed in Encl. 8 are plotted in Encl. 17 at a function of

# 4.2.5 Surface Preparation Studies

Twelve silicon nitride 1/2" diameter balls, manufactured by Industrial Tectonics, Inc. from Norton Grade 110 silicon nitride, were purchased for the optical elastohydrodynamic tests. Diameter and weight measurements on these balls yield a density of  $3.17 \text{ g/cm}^3$ , which is 99.7% of the theoretical density. These ball specimens were finished by the manufacturer, using an unidentified manufacturing process. Initial trial runs in the optical EHD apparatus demonstrated that the surface condition of the balls was such that no optical interference fringes could be observed. Scanning electron micrographs (Encl. 7) show that the surface is covered with pits of approximately one  $\mu$ m diameter, which are left by the final manufac-

The unusual appearance of the finished ball suggests that silicon nitride does not respond to manufacturing procedures in the same manner as steel (assuming that standard ball manufacturing procedures were used to produce the balls). It was reported (12) that silicon nitride specimens prepared by grinding with successively finer grit wheels, followed by lapping with diamond paste, had far greater fatigue life than specimens which were made by coarse grinding directly followed by diamond lapping, even though both specimens had the same surface finish. This information suggests that coarse grinding initiates microcracks in the surface, which can be removed by finer grinding, but that lapping does not remove sufficient material to eliminate the cracks. Such an interpretation implies that the silicon nitride surface is brittle on a microscale. Further evidence of microbrittleness is the pitted surface of the balls. Such pits can be produced if segments of material crack out of the surface during finishing processes. Experiments on lapping of silicon nitride by Professor Rabinowicz reveal that material removal under lapping with alumina abrasive occurs at a rate which is ten times faster than predicted by theory or occurs with other ceramics. One explanation for the faster lapping is that silicon nitride particles which are considerably larger than the abrasive particles are torn from the surface.

An attempt to produce a better silicon nitride surface than the one shown in Encl. 7 has been accomplished by polishing the surface by spinning the balls at high speed in a wood lap using ferric oxide as the ablasive. Scanning electron micrographs to determine the effect of this processing on surface texture are shown in Encl. 18. Optical interferograms with these balls show some weak colors which possibly can be used for EHD film thickness measurements. Encl. 19 is a black and white static fringe photograph of this silicon nitride ball contact showing a few interference fringes.

In order to obtain more clearly visible fringes than those shown in Encl. 19, the optical properties of the test element (1/2" diameter silicon nitride ball in the present case) and those of the coating on the glass flat must be well matched. In the case of a steel ball, a coating of cerium oxide with a reflectivity of 30% and absorption less than 1% is found to be adequate. Due to the lower reflectivity of the surface of the silicon nitride ball, a different coating must be used.

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The intensities of light reflected from the surface of the silicon nitride ball and the coated (CeO with 30% reflectivity) glass flat are not well matched to produce clearly visible interference fringes.

Therefore, an investigation was made on the optical visibility of the contact considering reflectivity and absorption of the ball and various coatings.

The value of the reflectivity of the silicon nitride ball surface was obtained using a Pentax light-meter. A beam of light was reflected off the surface of a steel ball, and the intensity was measured on a relative scale on the light-meter to be 5.9. The steel ball was replaced with a silicon nitride ball, and the light-meter reading was found to be 5.3. According to the Pentax manual, the relationship between a pair of relative scale readings ( $r_1$  and  $r_2$ ) and the corresponding intensities  $I_1$  and  $I_2$  is as follows

$$\frac{I_1}{I_2} = 2^{(r_1 - r_2)}$$

Since the reflectivity of steel ball surfaces in oil is known to be 60% (16), the reflectivity of the silicon nitride ball is calculated to be 44%.

In optical interferometry, the visibility of fringes is governed by the intensities of the two interfering light beams,  $(I_1 \text{ and } I_2)$ . In the optical system shown in Encl. 20,  $I_0$  is the intensity of the collinated beam from the objective lens.  $I_1$  is the intensity of the beam reflected from the layer and  $I_2$  is the intensity of the beam reflected back from the contact and through the coating (twice). The visibility is defined as the ratio of the geometric mean to the arithmetic mean of the two beam intensities  $I_1$  and  $I_2$  i.e. (16, 18)

$$V = 2(I_1I_2)^{1/2}/(I_1+I_2)$$

Maximum visibility (V=1) occurs when  $I_1 = I_2$ .

Knowing the reflectivity and absorption coefficients of the layer and the ball, it is possible to compute the relative intensity of the two interfering beams  $I_1$  and  $I_2$  considering

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two ball materials (steel and silicon nitride) and various coating materials. Encl. 21 gives the properties of coatings investigated for this program as obtained from the supplier. The choice of coatings was made such that maximum visibility would result when used with the  $Si_3N_4$  ball. Since the reflectivity of the  $Si_3N_4$  ball surface was found to be 44%, one can compute the reflectivity of the coating required for visibility of 100% by equating  $I_1$  and  $I_2$ . From Encl. 20 we have

$$I_{1} = R_{1}I_{0} = I_{2} = R_{2} \left[ 1 - (R_{1} + A_{1}) \right]^{2} I_{0}$$
 (1)

where  $I_0$  = intensity of incident beam

 $A_1 = absorption coefficient of the coating$ 

 $R_1$  = reflectivity of the coating

and  $R_2$  = reflectivity of the ball surface = 0.44

Since the absorption coefficient of CeO is small it can be neglected. Therefore for CeO

 $R_1 = 0.4(1-R_1)^2$  from which  $R_1 = 22.5\%$ 

Due to experimental limitations, it was not possible to get a coating with R = 22.5% exactly. The cerium oxide coatings obtained all had R values between 20 and 25%. These values are considered close enough to the computed value.

In addition, silicon monoxide coatings were found to be available which offered high calculated visibility values. A few glass flats coated with SiO were ordered for trial. Their wear characteristics were found to be equivalent to those of CeO. Encl. 22 gives the computed values of the fringe visibilities for various combinations of ball and coating materials.

Encl. 23 is a comparison of static black and white fringegrams of the contact at 5 lb load, taken with glass flats coated with the three different coatings indicated in Encl. 21. The fringegrams (b) and (c) of Encl. 23 produced from the silicon

cerium oxide combination, and from the silicon nitride-silicon monoxide combination, show sharp fringes for both combinations and confirm the results of the calculated visibility.

Encl. 24 also shows the very satisfactory fringes obtained in a dynamic silicon nitride-glass contact at a rolling speed of 40 in/sec and a maximum pressure of 113 ksi with a silicon monoxide coating.

On the basis of the results above, both the CeO and SiO coatings on the glass flat produce satisfactory fringes in tests with Si $_3N_4$  balls.

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#### 4.2.6 Plateau Film Thickness Measurement

Due to the relative uniformity of the plateau film thickness, the contact when viewed through the eyepiece of the optical system exhibits a dominant color which corresponds to a certain film thickness. The correlation between film thickness and color can be obtained by means of a calibration process explained in Section 4.2.4 of this report. The fringe order of the color can be readily identified by increasing the speed gradually from zero.

Film thickness measurements have been completed for Mobil Jet II (Lubricant No. 1 in Enclosure 8) at a multitude of rolling speeds and two loads (5 lbs. and 15 lbs.). For comparison, both a silicon-nitride ball and steel ball were run.

The elastic modulus of  $Si_3N_4$  is  $45 \times 10^6$  psi against  $30 \times 10^6$  psi for steel. The maximum Hertzian stresses under 5 and 15 lb.. loads are respectively 113 and 161 ksi for the  $Si_3N_4$  contacts and 100 ksi and 144 ksi for the steel contacts.

In Enclosure 25 the measured plateau film thickness is plotted as a function of rolling speed at two loads for the two material combinations.

The curves show that the  $\rm Si_3N_4$  contact EHD film thickness is less than the steel contact at the same load by about 15-20%.

Enclosure 26 shows the measured film thickness  $h_0$ , given in Enclosure 25 divided by the theoretical unstarved film thickness  $h_0$  as a function of rolling speed V, where  $h_0$  was empirically deduced from optically measured data given in (17) for ester oils as follows:

$$A_{of} = 0.87 R \left(\frac{\gamma V \alpha}{R}\right)^{0.70} \left(\frac{Q}{E'R^2}\right)^{-0.05}$$
(2)

where Q = load (lbs)

 $\eta$  = absolute viscosity (lb-sec/in<sup>2</sup>)

 $\sigma$  = pressure viscosity coefficient (in<sup>2</sup>/lb)

R = radius of ball (in)

E' = reduced Young's modulus (1b/in<sup>2</sup>)

Enclosure 26 shows that the prediction formula fits both data sets at low 'elocities. As V increases  $A_{o}/A_{o}$  decreases indicating that the formula of Eq. (2) <u>overestimates</u> the film thickness at high velocity. This behavior is typical of lubricant film starvation wherein the amount of oil present in the inlet is insufficient

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to maintain the theoretical plateau film thickness. This explanation is confirmed by measurements made from fringegrams of the contacts of the meniscus location i.e. the distance from the contact center to the oil/air interface in the inlet region. The appearance of the curves in Enclosure 26 suggests that starvation begins at somewhat lower speeds for  $Si_3N_4$  contacts.

The coincidence of the curves for 5 and 15 lbs, loads on this plot suggests that Eq. (2) is quite adequate in explaining the effect of load on film thickness for  $Si_3N_4$  as well as for steel.

# 4.2.7 Fringegrams of Glass-Si<sub>3</sub>N<sub>4</sub> Contacts

Using the ester (No. 1) oil at 5 and 15 lbs. loads, a series of fringegrams have been taken using Ansco-500 high speed color film at speeds of 5, 10, 20, 40, 60, 100, 200, 300, and 400 in/sec.

Enclosure 27 shows a selected subset of the fringegrams of the glass/Si3N4 contacts at two loads and three speeds. The pictures show that the remiscus location in the contact inlet moves progressively closer to the contact as speed increases. Comparison of the original color fringegrams at two loads taken at 100 in/sec (pictures C and E) show that the plateau color is the same, indicative of the low dependence of (plateau) film thickness on load. (A common color does not imply an identical film thickness since refractive index varies with pressure). An interesting feature in pictures B and C is the symmetry and sharp leading edge of the meniscus line. Meniscus lines observed with stecl contacts are relatively more rounded.

The distance from the contact center to the meniscus  $(r^*)$ measured from the fringegrams and scaled by the Hertz contact radius a is plotted as the lower curve in Enclosure 28 for the Si $_3N_4$  contacts. The upper curve is a plot of the measured  $r^*/a$  values for the same oil measured in a previous contract (15) for steel contacts. It is seen that the steel-glass contact is somewhat less starved than the  $Si_3N_A$ -glass contact, with the difference being small at low rolling speed. The greater evident starvation for the  $Si_3N_4$ contact can partially account for lower observed film thichnesses in Si<sub>3</sub>N<sub>4</sub> contact.

A previous investigation of starvation (17) indicates that insufficient replenishment of oil between successive contacts is a cause of starvation.

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In the film replenishment model, the relevant parameters are  $P_{\bullet}/E'$  and  $a/h_{o}$  where  $p_{o}$  is the maximum pressure, a is the contact radius and  $h_{o}$  is the ambient layer height. Under a given load, both  $p_{o}$  and a depend on the reduced Young's modulus E'. So there is possibly some effect of E' on film replenishment rate. It is also known that increasing  $h_{o}$  corresponds to an increase in the replenishment rate. The quantity  $h_{o}$  can be dependent on the wetting characteristics of the solid surface.

In spite of the experimental evidence that the Si $_3N_4$  contact exhibits a somewhat earlier occurrence (i.e. at lower speed) of starvation than the glass-steel contact, the optical film thickness measurements show that a lubricated Si $_3N_4$  contact has satisfactory film building behavior close to what is expected by current EHD theory.

### 4.2.8 Traction Tests

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As described in (8, 15) the traction force at the test contact in the EHD test rig is obtained from the torque measured at the ball or roller driving quill and the lateral force in the beam spring which constrains the ball or roller.

The test contact is the circular contact between the flat and the ball each of which is connected to a driving spindle. The sliding rate is defined as the difference in surface speed of the two bodies.

The following traction tests were run:

- 1. Typical plots of traction coefficient (defined as the sliding traction divided by normal load) against sliding speed are shown in Enclosure 29 for oil No. 6 at two loads and two rolling speeds. A glass flat and silicon nitride balls were used. It is seen that the traction rises with sliding rate and then levels off as in many previous tests conducted with steel contacts. This shape is typical for all oils except Nos. 3 and 5 which reached a maximum and then decreased with sliding speed.
- 2. A series of traction tests were run at three loads i.e. 5, 10 and 15 lbs. corresponding to 131, and 146 and 161 ksi maximum Hertz stress respectively. The entrainment speeds used were 40, 100 and 400 in/sec. A glass flat and silicon nitride balls were used. In Enclosure 30 the maximum traction coefficients are tabulated for the

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6 lubricants at three rolling speeds and three loads. The data show that traction coefficient increases with load but decreases with rolling speed, a trend which is consistent with previous traction tests in steel contacts. The data also shows that the effect on traction of additives in paraffinic and ester oils is small as characterized by the relatively small difference in traction between oils (2) and (4) and between (1) and (6). It is found that the two ester oils (1) and (6) have higher traction than the two paraffinic oils (2) and (4). The DTE mineral oil (3) and polyphenyi ether (5) exhibit relatively high traction. The two paraffinic oils (2) and (4) have the lowest traction coefficients.

- 3. Enclosure 31 shows traction force data for steel/silicon nitride and steel/steel contacts. Lubricant No. 1 (Ester oil with additive) was used. These tests were run in order to assess the differences in traction coefficients in real bearings, where the contacts are silicon nitride/steel or steel/steel. In order to obtain these data a st el washer with 5 μ in surface roughness was used instead of the glass flat used in the previous investigations. It is seen from Enclosure 32 that at the highest stress level, 271 ksi, the traction force for steel/steel and steel/silicon nitride differ by no more than 8%. At the lower stress levels, 216 ksi and 247 ksi, the differences between steel/steel and steel/silicon nitride are generally larger in one case by more than 20%.
  - 4. Enclosure 32 is a plot of the measured maximum traction coefficient at a rolling speed of 40 in/sec with an ester oil (lubricant No. 1) as a function of maximum contact pressure for three values of the axis ratio of the contact and for five material combinations. The axis ratio is 1:1 for a ball against flat contact, and differs from 1:1 for a roller against flat contact. The data points show that the traction coefficient in the silicon nitride contact is comparable or lower than that prevailing in steel contact.

# 4.3 Flat Washer Surface Interaction Studies

# 4.3.1 Description of Flat Washer Test Rig

A "Flat Washer" test rig provides a rapid and inexpensive means

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of obtaining fatigue data with the use of relatively simple test equipment and simple shaped specimens. The effects of parameters such as surface topography, wettability, wear resistance, and ability to generate EHD films on endurance in rolling/sliding contact may be studied with this tester.

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In the 🖺 🔀 牙 "Flat Washer" test rig shown in Enclosure 33, three rollers, guided by a plastic or metal cage. roll under load in a circular path between two flat washers, as shown in detail in Enclosure 34. The upper washer is stationary and the lower one rotates. The rollers are of vacuum melted M50 tool steel, hardened to Rockwell C61-64. They are 5 mm in diameter and 6 mm long, and are crowned as shown in Enclosure 35. The rollers are guided by the cage in the "aligned" position, such that the axis of the rollers intersects the common axis of the two washers. Some sliding occurs between the rollers and washers due to the curvature of the rolling path on the washers. The greatest amount of sliding occurs near the ends of the rollers, at which points the slideto-roll ratio, may be nearly 10% with the specimen dimensions used in this test rig, and there is one point usually near the middle of each roller-to-washer contact line at which almost pure rolling (zero sliding) occurs. Lubricant is supplied to the roller/flat washer assembly at a rate of approximately 1 pint/min. from an overhead reservoir through a hollow shaft on which the upper washer is mounted. Prior to each test, the oil is filtered through a 5 µm Millipor filter. The bulk temperature of the upper washer is monitored during the tests with a thermocouple. The output of this thermocouple is monitored continuously either on a chart recorder or by a digital computer test monitoring system.

The design of the flat washer specimens of silicon nitride tested in the lower position in the flat washer tester is shown in Enclosure 36. A photograph of several flat washer testers is shown in Enclosure 37.

# 4.3.2 Flat Washer Test Results with Steel Specimens Only

Half-inch diameter steel balls are sometimes used in place of the rollers in the flat washer tester, as shown in the lefthand side of Enclosure 33. Endurance tests with AISI 52100 steel flat washers of different hardnesses and Hofors AOH 52100 steel balls conducted previously at EDCS F show that the L<sub>10</sub> life deviates most drastically from predicted behavior in a critical stress range depending on hardness (life drops by a factor of 5 to 10 below that extrapolated from either higher or lower stress data),

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the lower hardness washers having a reduced life region in a lower load range. Rockwell 63C and 61C hardness washers were tested, as shown in Enclosure 38, on which each point represents about thirty flat washer fatigue tests. Similar results were obtained with Rockwell 64.5C balls rolling together in a rolling 4-ball tester, also shown plotted in Enclosure 38.

Indications are that the life of rolling contacts is decreased below expected values in critical contact stress ranges where the onset of gross plastic deformation is allowed to take place probably resulting in sub-surface residual stresses formed in such a manner that the amplitude of the maximum sub-surface reversing orthogonal shear stress is increased. (This stress is considered the critical stress in rolling fatigue failure.)

It was observed that considerable plastic grooving occurred in the ball track of the flat washers tested with balls and that the groove radius increased with decreasing load. In order to minimize this variable of grooving on life, testing has been conducted on a flat washer against cylindrical roller configuration as shown in Enclosure 34. The  $L_{10}$  life obtained from tests conducted previously at  $\mathfrak{B} \otimes \mathfrak{F}$  on 52100 steel flat washers of 61 Rc in the roller-flat washer configuration exceeds the theoretical life calculated by the 4th power method (24) in contrast with results obtained in ballflat washer tests. This is shown graphically in Enclosure 39 where a comparison is given between the theoretical load-life behavior and the experimental load-life for both the  $L_{10}$  (10%) life and the  $L_{50}$  (50%) life. The deviation from a linear relationship produced at the lower load can be related to the use of rollers with excessive crown.

Endurance data on the rollers in a flat washer/roller configuration has been collected previously at 25 25 on manufacturing lot of 5 x 6 mm "triple crowned" (crown drop of 2-2.5 µm at the measuring point on one side equal to ~3.3 times the Lundberg crown drop for 1200 lbs. washer load) rollers made from consumable electrode vacuum melted (CVM) M-50 tool steel. Flat washer testers (Enclosure 37) with circulating Mobil DTE medium heavy lubricant were used.

Test conditions were 3400 rpm, 250°F, and three load values. Summarized in the table below are the detailed life data obtained:

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	Теат	MAX, HERTZ	THEORETICAL LIFE	THEORETICAL LIFE® OF ROLLER SET LIO MILL, REVE.	NUMBER OF TESTED ROLLER SETS	NUMBER OF FAILED ROLLER SET	MAX. LIKELIHOOD ESTIMATED IS LID MILL, REVS.
GROUP	LOAD	STRESS, KEI	L	2.8	45	,	8.4
3141, 3142	1200	815	1,12	2,0 2,0	67		18,2
3143 1751 1755	1000	280	2,44	4.10	20	1	•• '
31.66	600/	255	5,9	12,4	80	•	

The experimental lives of CVM M-50 rollers run against steel flat-washers are in excess of 3-1/2 times the theoretical 4th power roller set life. These lives are not significantly different from the 5 times calculated life often quoted for CVM M-50 rings. If one considers the severe operating conditions in a flat washer test (spin, low speed, low C/P) the results support the life prediction of 4 to 5 times calculated life for CVM M-50 steel under application conditions.

# 4.3.3 Test Results with Silicon Nitride Washers

Enclosure 40 shows endurance test results on seven flat washer assemblies run on the present program. The top washer was made of 52100 steel in all tests. The bottom washer was made of 52100 steel in two of the tests and of silicon nitride having as-ground surface finish (5 microinches, AA) in the remaining five. The rollers were made of CVM M-50 tool steel in five tests and Rex 49 tool steel in two tests. The speed was 3330 rpm and the load 1000 lbs. except for one test where the load was 500 lbs. The lubricant was MIL-L-23699. It is seen that every failure was due to spalled or distressed rollers. Enclosure 41 is a Weibull plot of the test data in Enclosure 40, excluding Test 3, because of the lower load in this test. The Weibull slope of the data in Enclosure 41 is 1.36. This value is within the range usually obtained in bearing endurance testing and indicates that the life variations are not necessarily due to differences in flat and roller materials, but rather to expected random variations within the sample of six washer assemblies.

Life estimates calculated and bias corrected per (25).
 Life in excess of 80 M.R.

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The estimated L<sub>10</sub> life of this group shown in Enclosure 41 is about 0.2 million revolutions, or about a factor of 100 below the estimated L<sub>10</sub> of 18.3 million revolutions for 62 rotler sets tested previously in these testers with DTE medium heavy mineral oil lubrication. As shown in the above tabulatin. It was noted in the recent tests that the roller and steel visuor track surfaces, where steel washers were used, suffered lubrication distress. The bilicon nitride flats had a groove about 20 microinches deep worn in their tracks and the rollers against which they ran were especially distressed. Subsequent testing of steel washers in this rig with DTE medium heavy mineral oil lubrication has produced significantly longer lives, thus indicating that the low endurance lives of Tests No. 1 and 2 in Enclosure 40 with steel washers were the result of a life reduction due to excessive lubrication distress with the MIL-L-23699 lubricant in this tester operating under the severe conditions of high sliding, low speed and high load. Apparently under these severe conditions significant wear of the silicon nitride occurs, causing very early spalling failure of the tool steel surface of the rollers against which it is run. Since these contact conditions are typical of the high-spinning high-load critical inner-ring contacts of ultra-high-speed turbine-engine mainshaft angular-contact ball bearings, further studies of these phenomena is needed and is currently in progress.

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## 4.4 Sliding Friction and Wear Studies

## 4.4.1 Characterization of Surface Finish

The random microgeometry of surfaces in rolling contact has been recognized in recent years as a major determinant of the contact's fatigue, wear and traction behavior. Measurements of the center line average (CLA) or root mean square (RMS) height of a profile trace are standard methods to characterize the degree of roughness of the surfaces. The ratio  $h/\sigma$  of EHD film thickness h to the composite RMS surface roughness height **G**, with  $\sigma_2^2 - \sigma_1^2 + \sigma_2^2$  and  $\sigma_1 + \sigma_2$  the RMS roughness amplitudes of two mating lubricated surfaces, has become a widely accepted measure of the efficacy of the lubrication conditions in rolling contact.

In unlubricated conditions or when less thar full film exists ( $h/\sigma < 3.9$ ), asperities interact and the degree of plastic working that results has been observed to depend on the slopes of the asperities and not just upon the CLA or RMS height.

Accordingly, the EDESOF Kesearch Laboratory developed a device which operates in conjunction with a Talysurf instrument to yield RMS values of the profile slope in the direction of tracing.

A problem in the characterization of surfaces by means of one-dimensional profile data is the fact that a biased impression is thereby obtained of the summit heights and slopes on the two-dimensional surface. Both are understated in profiles. This is particularly true of surfaces having strong directionality

such as the one shown in Enclosure 42. Since the sliding friction coefficient, and hence the wear rate, are different for motion along and across the furrows it becomes necessary to characterize the surface finish more completely.

Recent theoretical developments permit the deduction of twodimensional surface characteristics from profile measurements of RMS height and RMS slope, the latter being taken in at least three known directions. It is possible, using such a group of RMS slope measurements, to characterize the degree of anisotropy of the surface by finding the pair of orthogonal directions along which the RMS profile slope is maximum and minimum and the value of these extreme RMS slopes.

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## 4.4.2 Theory of Longuet-Higgins on Surface Microgeometry Characterization

A surface whose statistical description is independent of the direction of (profile) measurement is called an isotropic surface. Due to the directionality of proces ing operations, most real surfaces have statistics that dependent the direction of profile measurement. Surfaces that have direction-dependent statistical descriptions are called an isotropic.

Williamson (19) has shown that a large variety of surfaces encountered in engineering practice are Gaussian, i.e. the distribution of surface heights follows a Gaussian or normal distribution. A Gaussian assumption will be made here.

Surface statistics are most usually deduced from analyzing profile traces made by a displacement sensitive stylus traversing the surface. This is the principle of operation of the Talysurf instrument widely used for this purpose. The assumption is made that the tip radius of the stylus is small compared to the features to be traced.

One-dimensional profile traces yield distorted information regarding the actual two-dimensional roughness process, but until Nayak (20) unearthed the little-known work of Longuet-Higgins (21), there was n) commonly known method for compensating for this distortion.

Longuet-Higgins derived the relationship between the onedimensional spectral moments of a profile and the two-dimensional spectral moments of a surface.

A description of a portion of the Longuet-Higgins paper applicable to this study is given in Appendix I.

The profile statistics method by Longuet-Higgins was applied to a flat silicon nitride specimen. The necessary measurements were obtained on a Talysurf instrument (model No. 4). The value of  $\sigma^2 = m_{co}$  was obtained directly on a CLA meter. Values of  $m_{co}$ and  $m_{20}$  were obtained at 11 different angles of measurement equally spaced at 15° intervals as shown in Enclosure 43.

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A computer program for linear regression analysis was used to compute least squares estimates of the quantities  $\mathcal{M}_{20}$ ,  $\mathcal{M}_{11}$ and  $\mathcal{M}_{02}$  using the measured value of  $\mathcal{M}_{20}$  as the dependent variable and  $x_1 = \cos^2\theta$ ,  $x_2 = \sin^2\theta$  and  $x_3 = \sin^2\theta$  as independent variables according to Eq. (4) of Appendix I.

The result was found to be

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The principal direction is calculated in Eq. (5) of Appendix I to be

$$\bar{\theta} = 90^{\circ}27'$$

i.e. consistent with the obvious directionality seen in Enclosure 42.

From the above equation one has,

$$(m_2)_{max} = 18.56$$
  
 $(m_2)_{min} = 9.15^{-1}$ 

Thus the RMS slope angle varies with tracing direction between a maximum of 4.3° and a minimum of 3.0°, a rather modest degree of anisotropy.

4.4.3 Experimental Study of Friction and Wear Using "Pin on Disk" Apparatus and Lapmaster.

Attached in their entirety as Appendix II are Professor Rabinowicz's four quarterly reports on friction and wear tests conducted at MIT with hot pressed silicon nitride and other ceramic specimens. In the friction tests a simple "pin on disk" apparatus was used. The top specimen, a 1/4" rod was pressed against the bottom specimen, a plate rotated at a speed of 0.3 cm/sec in the friction tests and 30 cm/sec in wear tests. Wear tests were conducted in a "Lapmaster" machine. Enclosure 44 shows a scanning electron micrograph (SEM) taken at EDST of the wear debris produced in a typical

Lapmaster test with silicon nitride. This photograph shows the presence of faceted particles typical of brittle fracture. The most important findings in this study, including the SEM work at ED is T. are:

1. The results indicate that the friction coefficient on ground silicon nitride surfaces seems to be lower when the pin traverses across the grinding grooves than when it runs parallel to the grooves.

2. Some of the six lubricants tested give a higher friction coefficient than the unlubricated condition. The silicone gives a much higher friction coefficient than all the other lubricants. The hydrocarbon Teresso V78 gave the lowest friction coefficient, by a slight margin. Turbojet 2380 oil was later added to the list and found to have almost the same friction coefficient as Teresso V78.

3. These studies suggest that silicon nitride behaves in a brittle fashion on a microscale at least under surface finishing conditions.

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### APPENDIX 1

A DESCRIPTION OF A PORTION APPLICABLE TO THE PRESENT STUDY OF THE PAPER "STATISTICAL PROPERTIES OF ANISOTROPIC RANDOM SURFACE" BY M. S. LONGUET-HIGGINS

The definition of spectral moments is as follows: If the power spectrum of the two-dimensional height process is  $\pi(\omega_x \omega_y)$ and that of a profile traced in the direction  $\Theta$  relative to x is  $\overline{\pi}_{\mathbf{a}}(\omega)$ , then the two-dimensional spectral moment of order  $[\mathbf{p},\mathbf{q}]$  is

$$m_{\beta q} = \iint_{-\infty} \pi(\omega_x \omega_y) \ \omega_x^{\beta} \ \omega_y^{\gamma} \ d\omega_x \ d\omega_y + \infty$$

and the profile spectral moment of order n is mne. [ To ( w) w dw

The first two non-zero spectral moments of a profile are designated  $m_0$  and  $m_2$ . (In a symmetrical (Gaussian) process, odd order moments vanish).  $m_0$  is identical to the variance  $\sigma^2$ of the roughness amplitude process, and  $m_2$  is the variance of the slope of the roughness process in the  $\Theta$  direction.

Of the two-dimensional spectral moments of the surface (with two subscripts), the lowest order non-zero values are  $m_{00}$ ,  $m_{10}$  and  $m_{01}$ .

If a cartesian coordinate system is arbitrarily established on a rough surface and a profile trace is made zt an angle  $\theta$ to the x axis of this coordinate system, the profile moments  $m_{o\theta}$ and  $m_{\lambda\theta}$  are, according to Longuet-Higgins, related to the twodimensional spectral moments as follows:

$$m_{00} = m_{00} = G^2$$
 (3)

$$m_{20} = m_{20} \cos^2 \theta + 2 m_{11} \sin \theta \cos \theta + m_{02} \sin^2 \theta$$
 (4)

Eq. (3) shows that the RMS roughness height is independent of  $\boldsymbol{\Theta}$ .

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Eq. (4) shows that  $\mathfrak{M}_{20}$  is equal to  $\mathfrak{M}_2$  for a profile in the direction  $\Theta = 0$ , i.e. a profile taken along the x axis. Similarly,  $\mathfrak{M}_{02}$  is the value of  $\mathfrak{M}_2$  from a profile trace along  $\Theta = \pi/2$ . Since  $\mathfrak{M}_2$  is a variance, it cannot be negative in any direction, and hence it follows that  $\mathfrak{M}_{02}$  and  $\mathfrak{M}_{20}$  are positive quantities.

For an isotropic surface  $\mathfrak{M}_{10}$  is by definition independent of  $\Theta$  and hence for an isotropic surface one must have, regardless of the orientation of the coordinates:

# m,, =0

### m20 = m02 = m2

If <u>either</u>  $m_n$  differs from zero, <u>or</u>  $m_{20}$  and  $m_{21}$  from each other, then the surface is anisotropic.

For given values of  $m_{20}$ ,  $m_{02}$  and  $m_{11}$  Eq. (4) above yields the profile slope variance  $m_{20}$  as a function of  $\Theta$ , i.e. it represents  $m_{20}$  in a polar coordinate system with coordinate angle  $\Theta$ .

It can be shown that another polar system can always be obtained by an axis rotation through the angle  $\bar{\Theta}$ , in which the term  $m_{ij}$  vanishes. The required angle of rotation is given by

$$\overline{\Theta} = \frac{1}{2} \tan^{-1} \left( \frac{2 m_{11}}{m_{20} - m_{02}} \right)$$
(5)

The form of the equation in the new system is

$$m_{20} = \overline{m}_{20} \cos^2 \phi + m_{02} \sin^2 \phi \qquad (6)$$

where

$$\Phi = \Theta - \overline{\Theta}$$
 (7)

$$\overline{m}_{20} = m_{20} \cos^2 \overline{\Theta} + m_{11} \sin 2\overline{\Theta} + m_{02} \sin^2 \overline{\Theta}$$
 (8)

and

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$$\overline{m}_{02} = m_{10} \sin^2 \overline{\Theta} - m_{11} \sin 2\overline{\Theta} + m_{02} \cos^2 \overline{\Theta}$$
 (9)  
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Note that  $\overline{m}_{20} + \overline{m}_{02} = m_{20} + m_{02}$  irrespective of  $\overline{\bullet}$ , i.e. the sum of the coefficients of the sin<sup>2</sup> and cos<sup>2</sup> terms is invariant with respect to an axis rotation.

The maximum value of  $m_2$  is readily shown to occur at  $\phi = 0$  and the minimum at  $\phi = \overline{u}/2$ , thus

 $(m_2)_{max} = \overline{m}_{20}$  $(m_2)_{min} = \overline{m}_{02}$ 

and

The directions  $\overline{\Theta}$  and  $\overline{\Theta} + \overline{\mathbb{1}/2}$  with respect to the original axis are the principal directions for  $\mathfrak{M}_2$ , i.e.  $\mathfrak{M}_2$  is maximum in the direction  $\overline{\Theta}$  and minimum in the direction perpendicular to  $\overline{\Theta}$ .

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### APPENDIX II

## FRICTION AND WEAR TESTS WITH HP SILICON NITRIDE

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Professor Ernest Rabinowicz Massachusetts Institute of Technology

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### APPENDIX II

#### FIRST QUARTERLY REPORT

### Introduction

The starting material for these tests were a silicon nitride rod of length 1" and diameter 1/4", with a trunkated conical end, also a silicon nitride plate of dimensions 2" x 2" x 1/4". These were kindly supplied to me on a no-charge basis by Norton. Tests have been carried out using these materials as well as similarly shaped specimens of M 50 steel.

As received, the silicon nitride flat had a ground finish with surface roughness approximately 24 microinches RMS. In carrying out friction tests using a rider sliding circumferentially on the disk, there was a sharp rise in friction whenever the rider moved parallel to the grinding grooves. In later tests, the surface finish was improved to about 4 microinches RMS by lapping the flat on a Lapmaster with 600-grit abrasive. Friction and wear results have also been obtained on this smoother surface.

It was noticed that the rate of removal of silicon nitride on the Lapmaster was excessively high. Tests on other ceramic materials have been conducted to check out this impression, and these tests are also described below.

#### Apparatus

The simple pin on disk apparatus used in the friction and wear tests is shown in Figure 1. The top specimen, the 1/4" rod, was pressed against the bottom specimen under a load which was generally 2 kg. The bottom specimen, the plate, was rotated at a speed of

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0.3 cm/sec in the friction tests and a higher speed of 30 cm/sec in the wear tests. In order to obtain measurable amounts of wear, the wear tests generally lasted many hours. For lubricated tests, the bottom plate was covered by the lubricant in question.

### Results

A. Friction coefficient f

	Si <sub>3</sub> N <sub>4</sub> on Si <sub>3</sub> N <sub>4</sub> (ground)		Si <sub>3</sub> N <sub>4</sub> on M50	M50 on <u>(gro</u>	Si <sub>3</sub> N <sub>4</sub> und)	M50 on Si <sub>3</sub> N <sub>4</sub> (lapped)	
	along groove	across groove		along groove	across groove		
Unlubricated	.17	.17	.16	.16	.15	.16	
Cetane	.20	.15		.16	.12		
Palmitic acid in cetane	.17	.15		.17	.11		
Silicone DC 200-50	.24	.24		. 36	.36	.36	
Fluorocarbon Halocarbon 11-14	.17	.15	.16	.17	.16	.16	
Ucon fluid DLB 180 BX	.17	.15	.17	.15	.12	.13	
Hydrocarbon Teresso V78	.16	.13	.13	.16	.11	.14	

It will be seen that combination involving rough surfaces (i.e.  $\text{Si}_3\text{N}_4$  flats, sliding along the groove) generally gave friction coefficients of about .16-.17, more or less the same as for unlubricated surfaces. In contrast, smooth  $\text{Si}_3\text{N}_4$  surfaces frequently gave friction

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coefficients in the range .11-.14. The silicone was a terrible lubricant, especially in combinations involving M 50 steel.

B. Wear coefficient k

This is obtained via Archard's formula

In this case the wear volume was always the total volume lost from both surfaces, while the hardness was that of the softer member of the pair, either M 50 at 800 kg/mm<sup>2</sup> or  $\text{Si}_3\text{N}_4$  at 1500 kg/mm<sup>2</sup>.

	Si <sub>3</sub> N <sub>4</sub> on Si <sub>3</sub> N <sub>4</sub> (ground)	Si <sub>3</sub> N <sub>4</sub> on M50	M50 on S1 <sub>3</sub> N <sub>4</sub> (ground)	M50 on Si <sub>3</sub> N <sub>4</sub> (lapped)
Unlubricated	$5600 \times 10^{-6}$	$220 \times 10^{-6}$	$50 \times 10^{-6}$	$20 \times 10^{-6}$
Palmitic acid in cetane		30 ·	85	

Teresso V78

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For a non-metallic sliding system (either non-metal against non-metal or non-metal against metal), we would generally expect wear coefficients of about 5 x  $10^{-6}$  unlubricated and 2 x  $10^{-6}$  lubricated. E. Rabinowicz, Friction and Wear of Materails, John Wiley and Sons, New York, 1965, p. 164. Indeed, the system M 50 on lapped Si<sub>3</sub>N<sub>4</sub> lubricated by Terresso V78, did give a wear coefficient in this range. The other wear values were higher, often much higher, suggesting that brittle fracture wear was occurring.

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### C. Wear in the Lapmaster apparatus

The wear of various ceramic materails was determined, and an abrasive wear coefficient  $\tan \theta$  was computed, according to the formula

$$\tan \theta = \frac{3 \times \text{wear volume x hardness}}{\text{Load x distance slid}}$$

### The values obtained were as follows

<u>Material</u>	Estimated Hardness	Measured tan $\theta$ value
GE 330		
carbide	1250 kg/mm <sup>2</sup>	$3 \times 10^{-3}$
HP SI 3 <sup>N</sup> 4	1500	$30 \times 10^{-3}$
Silicon carbide	2000	$1 \times 10^{-3}$
Boron carbide	2500	$5 \times 10^{-3}$

For three-body abrasion processes, as in a Lapmaster,  $\tan \theta$ values of 2 to 6 x  $10^{-3}$  might be considered normal. The silicon nitride falls way outside this range, suggesting again that brittle fracture has occurred.

### Discussion

It is too early to make any definite statements about the friction and wear properties of  $Si_3N_4$ , but our samples appear to be brittle, and there seems to be considerable sensitivity to surface roughness.

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Future tests will, hopefullv, explore these and other variables more fully. We plan to run a few tests involving other ceramic materials, to see to what extent silicon nitride is characteristic, and also to run tests involving lubricated silicon nitride at elevated temperatures.

As further samples of silicon nitride become available, perhaps manufactured in slightly different wavs, it may be of interest to evaluate them in the Lapmaster. I have a hunch that samples which show a low wear rate under abrasive conditions will also show low wear rates under ordinary sliding conditions.

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## APPENDIX II (CONTINUED)

## SECOND QUARTERLY REPORT

#### Introduction

During the past three months, the main focus of our investigations has been to generate further data on the friction of lubricated pairs of materials, one of which is silicon nitride or some other ceramic. The aim here is to see to what extent the frictional properties of silicon nitride are exceptional or anomalous.

At the same time, we have had the opportunity to carry out further wear tests of other silicon nitride samples on the Lap-master, our aim here being to see if this technique is able to measure the structural integrity of various silicon nitride samples. These results, too, are presented below.

#### Results

#### A. Friction tests

The data were obtained under the identical conditions as those described in the first quarterly report. A pin on disk tester was used, and the top specimen, a 1/4" rod, was pressed into the bottom specimen under a load of 2 kg. The bottom specimen, the plate, was rotated at a speed of 0.3 cm/sec.

The specimens used in these tests consisted of a tungstentitanium carbide (GE 330), silicon carbide (Crystallon N), boron carbide, as well as the Norton-derived hot pressed silicon nitride specimens used in previous tests.

#### -II/6-

•	ladie 1.	Friction	Coerricien	t values		
	WC on WC	M50 on	S1 <sub>3</sub> N <sub>4</sub> on	SiC on SIC	M50 on S1C	S13 <sup>N</sup> 4 on SiC
	<u></u>				010	
Unlubricated	.34	.19	.22	.52	.29	.31
<u>Silicone</u> <u>DC 200-50</u>	.40	. 39	.33	.23	.42	.35
Fluorocarbon Halocarbon 11-14	4.20	.18	.18	.14	.14	.14
Ucon Fluid DLB 180 BX	.21	.16	.14	.20	.16	.17
Hydrocarbon Teresso V78	.18	.13	.18	.14	.13	.15
	B <sub>4</sub> C	M50	S1 3N4	M50		
	B4C	B <sub>4</sub> C	<u>B<sub>4</sub>C</u>	<u>M50</u>		
Unlubricated	. 53	.29	.43			
Silicone	.28	.30	.34			
Fluorocarbon	.27	.25	.31	.15		
Ucon Fluid	.23	.20	.31	.20		
Hydrocarbon	.25	.25	.29	.12		

### B. Wear tests on the Lapmaster machine

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These tests were carried out under the same conditions as those described in the last report, using a load of 1 kg, a sliding speed of 30 cm/sec, and 600 grit silicon carbide as abrasive. The duration of the tests was 1-2 hours. The results are shown below.

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# Table II. Abrasive Wear Coefficient Values Obtained on the Lapmaster

Specimen	Wear Coefficient Va	lue (tan θ)
H.P. Si <sub>3</sub> N <sub>4</sub> flat, 2" x 2" x 1/4" Used in previous tests Obtained from Norton.	10 x 10	-3
H.P. Si <sub>3</sub> N <sub>4</sub> ring, 1 3/8" OD, 13/16" ID, 5/8" long	8 x 10	)-3
Kindly lent by Dr. H. Mahnke, SKF Obtained from Norton.		2
H.P. Si <sub>3</sub> N <sub>4</sub> flat, 2 1/4" x 2 1/4" x 1/4" Purchased for this program from Norton.	80 x 1	n <sup>-3</sup>

The range in wear coefficient values is quite marked. Tt is of interest to note that the most recently obtained specimen may indeed be the worst, in terms of structural integrity.

### Discussion

Looking at the friction results, and c mparing them with those presented in the last report, there seems little unusual in the frictional properties of  $Si_3N_4$  as compared to those of other hard nonmetallics. Silicon nitride gives relatively high friction with the silicone fluid, but so do the other materials. The general range of lubricated friction values is also typical. Perhaps the only unique thing about silicon nitride is the exceptionally low friction coefficient of .17 for the unlubricated material sliding on itself.

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The wear values obtained on the Lapmaster show a lot of scatter. In future tests, we plan to see if these wear values correlate with other frictional properties of these samples.

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### APPENDIX II (CONTINUED)

### THIRD QUARTERLY REPORT

### Introduction

During the period covered by this report, a number of friction tests were run using a sample of the lubricant Turhojet 2380 (MIL L 23699 Exxon) obtained from SKF, to see how this practically important lubricant would perform. At the same time, adhesive wear tests were carried out using the new samples of eilicon nitride recently obtained from Norton. The results of this testing are shown below.

### Regults

### A. Friction tests using Turbojet 2380

The data was obtained under the same conditions as those described in earlier reports using a pin on disk tester in which a 0.25" diameter rod is pressed against a flat specimen. The normal force was 2 kg and the speed of rotation was 0.3 cm/sec.

The results obtained are shown in Table I. For comparison, results obtained with Teresso V 78 (a mineral oil used as a general purpose lubricant), and shown in earlier reports, are also given.

### -11/10-

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## Table I. Values of the friction coefficient f

Material combination	f with Turbojet 2580	F with Teresso V78
Silicon nitride on silicon nitride	.12	.13
N 50 steel on silicon nitride	.11	.11
Boron carbide on silicon nitride	.24	.29
Boron carbide on boron carbide	. 31	.25
M 50 steel on boron carbide	.24	.25
M 50 steel on M 50 steel	.13	.12

It will to seen that the Turbojet and the Teresso produced almost the same friction values each time. Also significant is the fact that both silicon nitride on silicon nitride and H 50 steel on silicon nitride are combinations which give quite low friction values. Thus, these are combinations which can be readily lubricated.

### B. Wear tests using Turbojet 2380

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The tests were carried out using specimen of HP silicon nitride recently obtained from Norton. The results obtained are shown in Table II. For comparison, a result reported earlier using another sample of HP silicon mitride is also shown.

### Table II. Values of the wear coefficient k

Combination	k with Turbojet 2380	k with Teresso V78
Silicon nitride on silicon nitride	$2.2 \times 10^{-6}$	
M 50 steel on silicon nitride	$1.0 \times 10^{-6}$	$3.2 \times 10^{-6}$

### -11/11-

These k values are quite reasonable for well lubricated sliding systems involving non-metallic materials. (For such combinations, the book "Friction and Wear of Materials," E. Rabinowicz, Wiley, 1955, suggests on p. 164 a wear coefficient of  $2 \times 10^{-6}$ ).

### Future tests

Currently we have under way an exploratory series of tests to compare the abrasive wear properties of silicon nitride samples with other mechanical properties of these same samples, in an attempt to see if abrasive wear testing can form the basis of a simple materials test for selecting good samples of silicon nitride.

Following this work, we have scheduled a meries of tests to measure the friction and wear of lubricated silicon nitride at elevated temperatures. It is important to see whether the good friction and wear behavior of lubricated silicon nitride at room temperature extends to elevated temperatures.

### -11/12-

## APPENDIX II (CONTINUED)

## FOURTH QUARTERLY REPORT

The 3-pin high temperature friction test apparatus was repaired. Friction-temperature tests were conducted using four combinations of  $Si_3N_4$  and M-50 riders and flats.

The test conditions and test results are listed in Table 1. Plates 1 and 2 present the same information in graphical form.

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### TABLE 1

TEST CONDITION	TFMP. (°C)	FRICTION COEPF.
M-50 riders on	36	0.122
Si <sub>2</sub> N, flat, lubricated	85	0.126
with4 MIL-L-23699	150	0.128
	200	0.130
SiaN, riders on SiaN,	30	0.126-0.180
flat, unlubricated	105	0.122-0.173
Run 1	150	0.155-0.180
	180	0.158-0.183
	200	0.155-0.180
Come condition as	85	0.151-0.173
above, Run 2	50	0.158-0.180
	110	0.158-0.184
	150	0.169-0.187
	200	0.173-0.194
Si.N. riders on M-50	32	0 152
flat lubricated with	90	0.157
MIL-L-23699	155	0,161
	200	0.165
SieW. mideme on Si N.	22	0.124
flat lubricated with	90	0.126
MTL-L-23699	11.5	0 120
	180	0 117
	200	0.111

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TABLE I, cont.

TEST CONDITION	TEMP. ( <sup>o</sup> C)	FRICTION COEFF.
Si N, riders on Si N, flet; lubricated with Teresso V-140 and V-65 (volume ratio 1:4.8)	29 80 150 200 250	0.104 0.115 0.132 0.15 0.156
M-50 riders on M-50 flat, lubricated with MIL-L- 23699	28 85 160 213 250	0.110 0.115 0.123 0.128 0.134
SigNy riders on SigNy flat lutricated with palmitic acid in cetane	, 33 85 150 200 250	0.115 0.116 0.116 0.116 0.116 0.116
Si <sub>3</sub> N <sub>1</sub> riders on Si <sub>3</sub> N <sub>4</sub> flat lubricated with Halocarbon 11-14	28 50 150 200 210 (stick-slip) 230 250	0.126 0.140 0.160 0.182 0.185 0.193 0.200

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Figure 1. Schematic Illustration of the Single Rider Friction Apparatus.

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#### ENCLOSURE 2

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SPECIAL 201-SIZE BALL BEARING WITH CEMENTED CARBIDE RINGS, CERAMIC BALLS AND MOLYBDENUM CAGE AFTER 12 HOURS OPERATION AT 1500°F, 8000 RPM, THRUST LOADED TO 300,000 PSI CONTACT STRESS, AND LUBRICATED WITH MoS2 POWDER IN ARGON CARRIER.



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## ENCLOSURE 3

## COMPARISON OF PROPERTIES\* OF SILICON NITRIDE AND BEARING STEELS

	Silicon Nitride	<u>52100 Steel</u>	M50 Steel
Property	500,000	400.000	340,000
Compressive Strength (psi)	500,000		$30 \times 10^{6}$
Elastic Modulus (psi)	$45 \times 10^{\circ}$	$30 \times 10^{-1}$	30 x 10
Linear Thermal Expansion (°F) <sup>-1</sup>	$1.5 \times 10^{-6}$	$8 \times 10^{-6}$	$8 \times 10^{-5}$
Density (g/cc)	3.18	7.89	7.89
Hardness (Rc)	80-85	60	62
Maximum Use lemp. (°F)	3,000	400	700

\* Room Temperature Unless Otherwise Stated.

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## ENCLOSURE 4

# TYPICAL PROPERTIES OF NORTON HOT PRESSED SILICON NITRIDE

1)	Bulk density (g/cc) 45	$3.2 \times 10^{6}$	
2)	Modulus of elasticity (psi) 1000°C 45	$\mathbf{x} 10^{0}$	
- >	n Terrenture compressive strength (ps	<b>i)</b> 5	00,000
3)	Room lemperature compressive services	1	30.000
4)	Modulus of rupture (psi) 25°C 900°C	1	20,000
	1300°C		75,000
5)	Kncop Hardness with 100 g. load (kg/mm2)		2200
6)	Linear thermal expansion in the range 25-1500°C (per °C)		2.75 X 10 <sup>-6</sup>
7)	Thermal conductivity (BTU-in/hrft2-or)	25 °C 1000 °C	116 70
8)	Specific heat (BTU/lb-°F)	25 °C 1 000 °C	0.17 0.39

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### ENCLOSURE 5

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Spall morphology on a Silicon Nitride ball run in a five-ball tester. 100x

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### ENCLOSURE 6

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Surface of an unrun Silicon Nitride Ball as Received 1000X

BEAF		ENCLOSURE 8	THEIR PROPER'	LES		
RC	LIST -	JF TEST LUBRICANTS AND			lemp.	
		Kinematic Viscosity c.s. (100/210°F)	Surface Tension (dyne/cm)	Sp. Wt. (	Viscosity Coefficjent (	kerracture Index (25°C)
BORA	Ester Uil With Additive (Mobil Jet II)	28/5.1	30.5 (100°F	) 0.987	0.0154	<b>1.</b> 4570
TORY	Synthetic Paraffinic With Additive (XKM-177F)	412/42	31.0 (25°C)	0.81	0.0216	1.4690
BKP	. Mineral Oil (DTE Med. Hvy.)	63.6/8.1	33.13 (100°F 27.53 (210°F	) 0.882(60 %	i) 0.0187	
IND.	Synthetic Paraffinic (XKM-109F)	447/42.6	30.8 (23°C)	19.0	0.0214	1.4689
ы USTI	Polyphenyl ether	346/13	47.4 (100°F)	1.195	0.297	1.6310
JIES.	,Ester Base Oil (XRM-234)	28/5.1	30.5 (100 %	196.0 (1	0.0154	AL 74P00
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### ENCLOSURE 9

TYPICAL SILHOUETTE OF OIL DROPS ON THE SILICON NITRIDE SUBSTRATE



a) MIL-L-23699 (No.1 - Table 1)



b) Polyphenyl ether
 (No.5 - Table 1)

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### ENCLOSURE 10

# RESULTS OF CONTACT ANGLE MEASUREMENTS

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No	011	Surface Rou	ghness, <b>P</b> in,	
	222	15	15	1.5
		Contact (Perpendicular to lay)	Angle (Parallel to lay)	(Isotropic)
1	Mobil Jet II (MIL-L-23699)	10°	6°	5 <b>°</b>
2	XRM 177 F	18°	6°	16°
3	DTE Medium Heavy	5°	4°	15°
4	XRM 109 F	17°	12°	13°
5	05 124	37°	29°	290
6	XRM 234	26°	11°	6°

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BASIC TEST CONFIGURATION



ENCLOSURE 12 EHD TEST RIG ASSEMBLY

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### ENCLOSURE 13

# EHD TEST RIG OPTICAL SYSTEM

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### ENCLOSURE 15

### FRINGE PHOTOGRAPHS OF EHD CONTACTS WITH STARVATION

Lubricant: Ester Oil (MIL-L-23699) Speed: 40 in/sec 52100 steel ball

Inlet



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Sapphire - Steel Contact Pò = 305 ksi



Glass - Steel Contact PO = 126 ksi

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### ENCLOSURE 16

FILM THICKNESS AS A FUNCTION OF INTERFERENCE COLOR FOR XENON AND INCANDESCENT LIGHT SOURCES



Xenon Incandescent

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### ENCLOSURE 18

### SURFACE OF AN UNRUN SILICON NITRIDE BALL AFTER FERRIC OXIDE POLISHING



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a) 1000X

b) 3000X



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### ENCLOSURE 19

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STATIC FRINGE PHOTOGRAPH OF POLISHED SILICON NITRIDE BALL IN OPTICAL EHD RIG



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Optical Path of Light Beam Reflected From Coated Glass Plate and the Ball Surface.

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# ENCLOSUBE 21

# PROPERTIES OF COATINGS

<u>Coating Material</u>	Reflectivity (%)	Absorption (%)
CeO	25	
Ce 0	20	0.5
SiO	20	10

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CALCULATED FRINGE VISIBILITIES FOR VARIOUS COMBINATIONS OF BALL AND COATING MATERIALS

Mate	<u>rials</u>	Refle	<u>ectivity</u>	<u>Absorption</u>	Relat Beam <u>Inten</u>	ive <u>sity</u>	Interference Fringe <u>Visibilit</u> .
<u>Ball</u>	Layer	R <sub>2</sub> (Ball)	R <sub>1</sub> (Layer)	A <sub>l</sub> (Layer)	<u></u>	<sup>1</sup> 2/10	<u>~</u>
Steel	CeO	60%	30%	0.5%	0.30	0.27	0 <b>.99</b> 86
Si N 34	CeO (1)	40%	30%	0.5%	0.30	0.19	5 0.9772
<sup>Si</sup> 3 <sup>N</sup> 4	CeO (2)	40%	25%	0.5%	0.25	0.22	4 0.9984
Si 3 <sup>N</sup> 4	CeO (3)	40%	20%	0.5%	0.20	0.250	<b>0.99</b> 24
si <sub>3</sub> n <sub>4</sub>	Si0	40%	20%	10%	0.20	0.196	0.9999



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### Enclosure 24. Dynamic Fringegram of a Silicon Nitride-Glass Contact.

Speed = 40 in/sec. Max. Hertz Stress = 113 ksi. Lubricant: Mobil Jet II



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Variation with Kolling Speed of the Katio of

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Color Fringegrams of  $Si_3N_4$ -Glass Contacts at Loads = 5 and 15 lbs, and Three Speeds for Each Load.



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Q = 5 lbs. V = 20 in/sec. D A Q = 15 lbs. V = 5 in/sec.



Q = 5 lbs. V = 100 in/sec. E B Q = 15 lbs. V = 00 in/sec.



Q = 5 lbs. V = 200 in/sec. F C Q = 15 lbs. V = 100 in/sec.





Distance of Contact Center of the Meniscus (r\*) Scaled by the Hertz Contact Radius (a) as a Function of Rolling Speed (V). Lubricant: Mobil Jet II

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Traction Curves for Oil No. 6 (Ester Base Oil)



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# MAXIMUM TRACTION COEFFICIENTS OF SIX TESI OILS

# Material: Silicon Nitride-Glass, Axis Hatio 1:1

	Moximum	Roll	ing Speed	
0 i 1	Stress	40 in/sec (90°F)	100_in/sec (100°F)	(113°F)
Synthetic paraffinic	$   \begin{array}{c}     113 \\     141 \\     163   \end{array} $	0.0161 0.0168 0.026	0.010 0.0093 0.0172	0,0085 0,0077
Synthetic Paraffinic with addit	113 2 141 tive 163	0.0140 0.0181 0.0275	$0.0126 \\ 0.0094 \\ 0.0225$	0,007 0,0154
Ester oil with addi	113 tive 141 163	0.0186 0.03 <b>58</b> 0.0362	0.0143 0.0240 0.0292	0.0150 0.0203
Ester bas oil	e 113 141 163	$0.0256 \\ 0.0294 \\ 0.0346$	$0.0197 \\ 0.0222 \\ 0.0251$	0.0127 0.0145 0.0224
Mineral O (DTE Med.	il 113 Hvy) 141 163	0.0434 0.0445 0.0467	0.032 0.033 0.0388	0.0134 0.0217 0.0243
Polypheny ether	y1 113 141	0.0376 0.038	0.034 0.0306	$0.0187 \\ 0.0290$

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TRACTION FORCE DATA FOR STEEL/SILICON NITRIDE AND STEEL/STEEL CONTACTS

	Dmax :	216	<b>k</b> si	247	(si	271	ks i
kolling Speed (in/sec)	Material: Load (lbs):	5t5t.	6.95	stst. 15	st5i3 <sup>A4</sup> 10.4	StSt. 20	Si 3N4 -5 13.9
40		0.040	0.0430	0.0455	0.0515	0.035	0.0533
100		0.031	0.0418	0.0371	0.0419	F1-0.0	0.0474
200		,	0,0301	0.030	0.035	0.0375	0.0377

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ENCLOSUKE 35

# DIAGRAM OF CROWNED ROLLER



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SILICON NITRIDE FLAT WASHER SPECIMEN DESIGN



NOTES:

FACES TO BE BLANCHARD GROUND PARALLEL WITHIN .001 TIR TO SMOOTHEST POSSIBLE FINISH

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### ENCLOSURE 37

### PHOTOGRAPH OF SEVERAL FLAT WASHER ROLLING CONTACT FATIGUE TESTERS

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# TEST CONDITIONS AND RESULTS OF FLAT WASHER TESTS

TestFlatWasherRollers(oF)(drops/min.)(loss)1.0.004.90.901 Roller15210052100M50170ML-L-2369930100012.42.502 spalled25210052100M50170ML-L-2369930100012.42.502 spalled3513Nd +52100M50205ML-L-236993010002.30.50Rollers4 $$i_3N_d + 52100$ M50230ML-L-236993010002.30.50spalled5 $$i_3N_d + 52100$ M50230ML-L-236993010001.250.50spalled6 $$i_3N_d + 52100$ M50250ML-L-236993010001.250.25spalled6 $$i_3N_d + 52100$ 850250ML-L-236993010001.250.55spalled7 $$i_3N_d + 52100$ 85086x 49295ML-L-236993010001.250.55spalled6 $$i_3N_d + 52100$ 86x 49295ML-L-236993010007.251.45spalled7 $$i_3N_d + 52100$ 86x 49275ML-L-236993010007.251.45spalled7 $$i_3N_d + 52100$ 86x 49275ML-L-236993010007.251.45spalled					0	Derating	Lubricant	Lubricant Feed Rate	Load	Lif.	e II. revs.	Remarks
No.         Bottom         Let           1         52100         52100         M50         235         ML-L-23699         30         1000         12.4         2.50         Spalled           2         52100         52100         M50         170         ML-L-23699         30         1000         12.4         2.50         Spalled           3         513N <sub>4</sub> +         52100         M50         205         ML-L-23699         30         1000         1.3         2.20         Rollers           3         513N <sub>4</sub> +         52100         M50         230         ML-L-23699         30         1000         2.3         0.50         IRoller           4 $$ $13N_4^+ $52100         M50         230         ML-L-23699         30         1000         1.25         0.50         I Roller           5         $13N_4^+ $52100         M50         250         ML-L-23699         30         1000         1.25         0.25         spalled           6         $$3N_4^+ $52100         Kex         49         295         ML-L-23699         30         1000         1.45         0.60         8011er           7         $$$3N_4^+ $52100         Rex         49     $	Test	Flat Was	Top	Roller	s	(oF)		(drops/min.)	1.03.1		00 0	1 Roller
I       J2100       52100       52100       M50       I70       MIL-L-23699       30       1000       12.4       2.50       2 Bollers spalled spalled spalled         3 $533N_4^+$ 52100       M50       205       MIL-L-23699       30       500       11.3       2.20       Rollers spalled         4 $513N_4^+$ 52100       M50       230       MIL-L-23699       30       1000       2.3       0.50       Isollers         4 $513N_4^+$ 52100       M50       230       MIL-L-23699       30       1000       2.3       0.50       Isollers         5 $5i3N_4^+$ 52100       M50       250       MIL-L-23699       30       1000       1.25       0.25       spalled         6 $5i3N_4^+$ 52100       M50       295       MIL-L-23699       30       1000       1.25       0.25       spalled         7 $5i3N_4^+$ 52100       Rex 49       275       MIL-L-23699       30       1000       7.25       1.45       spalled         7 $5i3N_4^+$ 52100       Rex 49       275       MIL-L-23699       30       1000       7.25       1.45       Isoller </td <td>No.</td> <td>Bottom</td> <td>52100</td> <td>M50</td> <td></td> <td>235</td> <td>MIL-L-23699</td> <td>30</td> <td>1000</td> <td>4.9</td> <td>06.0</td> <td>spalled</td>	No.	Bottom	52100	M50		235	MIL-L-23699	30	1000	4.9	06.0	spalled
2 32.00 0.00 11.3 2.20 Rollers 3 $5i_3N_4^+$ 52100 M50 205 MIL-L-23699 30 500 11.3 2.20 Rollers 4 $5i_3N_4^+$ 52100 M50 230 MIL-L-23699 30 1000 2.3 0.50 isoliter 5 $5i_3N_4^+$ 52100 M50 250 MIL-L-23699 30 1000 1.25 0.25 isolited 6 $5i_3N_4^+$ 52100 Rex 49 295 MIL-L-23699 30 1000 4.0 0.00 Rollers 7 $5i_3N_4^+$ 52100 Rex 49 275 MIL-L-23699 30 1000 7.25 1.45 isoliters	-	00170	52100	M50		170	MIL-L-23699	30	1000	12.4	2.50	2 Rollers spalled
3 $Si_3N_4$ $52100$ M50 230 M1L-L-23699 30 1000 2.3 0.50 1 Roller 4 $Si_3N_4^{+} 52100$ M50 230 M1L-L-23699 30 1000 1.25 0.25 spalled 5 $Si_3N_4^{+} 52100$ M50 250 M1L-L-23699 30 1000 4.0 0.80 distressed 6 $Si_3N_4^{+} 52100$ Rex 49 295 M1L-L-23699 30 1000 4.0 0.80 distressed 7 $Si_3N_4^{+} 52100$ Rex 49 275 M1L-L-23699 30 1000 7.25 1.45 spalled	2	+	00102	05		205	MIL-L-23699	) 30	500	11.3	2.20	Rollers distressed
4 $\mathbf{5i}_{3}\mathbf{N}_{4}$ $52100$ $\mathbf{m50}$ $1000$ $1.25$ $0.25$ $\mathbf{10011er}$ 5 $\mathbf{5i}_{3}\mathbf{N}_{4}^{\dagger}$ $52100$ $\mathbf{M50}$ $250$ $\mathbf{ML-L-23699}$ $30$ $1000$ $1.25$ $0.26$ $\mathbf{8011ers}$ 6 $\mathbf{5i}_{3}\mathbf{N}_{4}^{\dagger}$ $52100$ $\mathbf{Rex}$ $49$ $295$ $\mathbf{ML-L-23699}$ $30$ $1000$ $4.0$ $0.80$ $\mathbf{Ro11ers}_{4}$ 6 $\mathbf{5i}_{3}\mathbf{N}_{4}^{\dagger}$ $52100$ $\mathbf{Rex}$ $49$ $275$ $\mathbf{ML-L-23699$ $30$ $1000$ $7.25$ $1.45$ $\mathbf{18011er}_{5}$ 7 $\mathbf{5i}_{3}\mathbf{N}_{4}^{\dagger}$ $52100$ $\mathbf{Rex}$ $49$ $275$ $\mathbf{ML-L-23699$ $30$ $1000$ $7.25$ $1.45$ $\mathbf{18011er}_{5}$ 7 $\mathbf{5i}_{3}\mathbf{N}_{4}^{\dagger}$ $52100$ $\mathbf{Rex}$ $49$ $275$ $\mathbf{ML-L-23699$ $30$ $1000$ $7.25$ $1.45$ $\mathbf{18011er}_{5}$	3	Si 3N4	00126			230	WIL-L-2369	9 30	1000	2.3	0.50	1 Roller spalled
5 $s_{13}N_4^{+}$ 52100 M50 230 M1L-L-23699 30 1000 4.0 0.80 Rallers 6 $s_{13}N_4^{+}$ 52100 Rex 49 295 M1L-L-23699 30 1000 7.25 1.45 1 Roller 7 $s_{13}N_4^{+}$ 52100 Rex 49 275 M1L-L-23699 30 1000 7.25 1.45 spalled	4	Si 3N4	52100				WTI -I2369	9 30	1000	1.25	0.25	l Roller spalled
6 $si_3N_4^+$ 52100 Rex 49 295 MIL-L-20077 0 1 Roller 7 $si_3N_4^+$ 52100 Rex 49 275 MIL-L-23699 30 1000 7.25 1.45 spalled	ŝ	si3N4	5210	0 M50		250		30	1000	4.0	0.80	Rollers distressed
7 Si <sub>3</sub> N <sub>4</sub> <sup>+</sup> 52100 Rex 49 275 MIL-L-23699 30 1000 spalled	9	si3N4	5210	0 Rex	49	295	11-1-2007			: :	1 45	1 Roller
	7	Si3N4	+ 5210	0 Rex	49	275	MIL-L-236'	99 30	1000		1	spalled

<sup>+</sup>As ground surface finish = 5  $\mu \cdot in$ . AA.

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Appearance of Ground Si<sub>3</sub>N<sub>4</sub> Plate. 1000X

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SURFACE MEASUREMENTS ON GROUND SILICON NITRIDE PLATE

Slope Ang RMS Angle Measur	le Meter Rdgs. cements in Deg. (MLD)	Rdg.	<u>C.L</u> Microin	.A. Met	ter Rdgs. Repeated	4 Times
Angular Location		-	N	က၊	ন	Average
15°	3.1	10	10.5	9.5	9.5	6.9
30°	3.45	11.5	13	11	12.5	12.0
45°	3.75	11	11	11	11	11.0
°0°	4.05	13	13	11.5	11.5	12.3
750	4.15	13	14.5	15.5	13.5	14.1
٥06	4.3	15	14	15	13.5	14.4
105°	4.25	13	11.5	12	12.5	12.3
120°	4.0	13.5	12.5	13	11.5	12.6
135°	3.8	13.5	12.5	13.5	12	12.9
150°	3.55	14.5	14	13	13.5	13.8
165°	2.9	13	13	12.5	13	12.9

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## ENCLOSURE 44

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SEM Photograph of Wear Debris Obtained from Lap-master Wear Tests 3000X

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