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A Vacuum (10^{-9} Torr) Friction Apparatus for Determining Friction and Endurance Life of MoS_x Films

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A VACUUM (10^{-9} TORR) FRICTION APPARATUS FOR DETERMINING FRICTION AND ENDURANCE LIFE OF MoS$_x$ FILMS

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SUMMARY

The first part of this paper describes an ultrahigh vacuum friction apparatus (tribometer). The tribometer can be used in a ball-on-disk configuration and is specifically designed to measure the friction and endurance life of solid lubricating films such as MoS$_x$ in vacuum at a pressure of 10^{-7} Pa (10^{-9} torr). The sliding mode is typically unidirectional at a constant rotating speed. The second part of this paper presents some representative friction and endurance life data for magnetron sputtered MoS$_x$ films (110 nm thick) deposited on sputter-cleaned 440C stainless-steel disk substrates, which were slid against a 6-mm-diameter 440C stainless-steel bearing ball. All experiments were conducted with loads of 0.49 to 3.6 N (average Hertzian contact pressure, 0.33 to 0.69 GPa), at a constant rotating speed of 120 rpm (sliding velocity ranging from 31 to 107 mm/s due to the range of wear track radii involved in the experiments), in a vacuum of 7×10^{-7} Pa (5×10^{-9} torr), and at room temperature. The results indicate that there are similarities in friction behavior of MoS$_x$ films over their life cycles regardless of load applied. The coefficient of friction μ decreases as load W increases according to $\mu = kW^{-1/3}$. The endurance life E of MoS$_x$ films decreases as the load W increases according to $E = KW^{-1.4}$ for the load range. The load- (or contact-pressure-) dependent endurance life allows us to reduce the time for wear experiments and to accelerate endurance life testing of MoS$_x$ films. For the magnetron-sputtered MoS$_x$ films deposited on 440C stainless-steel disks, the specific wear rate normalized to the load and the number of revolutions was 3×10^{-8} mm^3/N·revolution, the specific wear rate normalized to the load and the total sliding distance was 8×10^{-7} mm^3/N·m, and the nondimensional wear coefficient was approximately 5×10^{-6}. The values are almost independent of load in the range 0.49 to 3.6 N (average Hertzian contact pressures of 0.33 to 0.69 GPa).

INTRODUCTION

The objective of this paper is to describe a high-vacuum tribometer. The motivation for the design and fabrication of this system is to achieve the capability to determine the friction and endurance life of molybdenum disulfide and other solid lubricating films in an atmosphere and in high-vacuum sliding conditions (ref. 1).

Because, in the high vacuum conditions of low Earth orbit, tribocomponents and lubricants might experience vacuum pressures in the range of 10^{-4} to 10^{-7} Pa (10^{-6} to 10^{-9} torr) (ref. 2), the experimental vacuum atmospheres that simulate spacecraft environment should be as low. Sputtered MoS$_x$ films are often nonstoichiometric, with coefficients of friction that are sensitive to oxygen partial pressure in the test environment. The coefficient of friction under ultrahigh vacuum conditions is relatively high but is reduced by a factor of 3 if the partial pressure of oxygen is high enough (ref. 3). Therefore, a vacuum friction apparatus must first

and foremost achieve a pressure in the 10^{-7} Pa (10^{-9} torr) range without a system bakeout, which may thermally stress the MoS_x films.

Solid lubricants designed for spacecraft applications must not only display low coefficients of friction (0.01 to 0.1), but also maintain good durability and environmental stability (refs. 2 to 7). The ability of a lubricant to allow rubbing surfaces to operate under load without scoring, seizing, welding, or other manifestation of material destruction in hostile environments is an important lubricant property. For solid lubricating films to be durable under sliding conditions, they must have low wear rates and high interfacial adhesion strength between the films and substrates. The actual wear rates, wear modes, and interfacial adhesion strength of solid lubricating films, however, are not fully understood.

The primary purpose of this paper is to describe the vacuum friction apparatus and its use in an accelerated test for the endurance life for MoS_x films. The friction, endurance life, and wear rates of some of our magnetron-sputtered MoS_x films were examined in this investigation. Sliding friction and wear experiments were conducted with uncoated 440C stainless-steel bearing balls in contact with MoS_x films deposited on sputter-cleaned 440C stainless-steel disks at loads of 0.49 to 3.6 N (average Hertzian contact pressure, 0.33 to 0.69 GPa), at a constant rotating speed of 120 rpm, and in a vacuum of approximately 7×10^{-7} Pa (5×10^{-9} torr). The sliding velocity ranged from 31 to 107 mm/s due to the range of wear track radii involved in these experiments.

VACUUM FRICTION APPARATUS

The vacuum friction apparatus is shown in figure 1. The apparatus consists of a ball-on-disk assembly mounted in an ultrahigh vacuum chamber, a drive system, and a friction force measuring system. All components within the vacuum chamber are compatible with oxidizing, inert, and reducing gases.

The specimens of the vacuum friction apparatus are a 19-mm-diameter flat disk (5 mm thick) and 6-mm-diameter ball specimen (shown in the insert of fig. 1). The disk specimen is mounted on a shaft which is driven by a gear motor connected to a rotary feedthrough with a ferrofluidic seal. The drive assembly provides rotation at various speeds, which are regulated by a direct current motor speed controller. For this study, all experiments were performed at a constant rotating speed of 120 rpm. During disk rotation, the ball slides on a constant-diameter wear track on the disk. The bellows assembly permits rotation at various track diameters ranging from 5 to 17 mm, producing sliding velocities ranging from 31 to 107 mm/s, respectively.

The ball specimen is mounted in a holder attached to one end of a stainless-steel beam. The beam is welded into a bellows assembly, which is gimbal mounted to the vacuum-chamber wall. The gimbal mounting permits deadweight loading of the ball against the disk surface. At right angles to the deadweight loading, the beam containing the ball can move in two directions in the horizontal plane. Movement of the ball (with the disk as it rotates) is restrained by a cable which is attached to a beryllium-copper ring. The ring contains four sets of strain gauges to measure the friction force between the ball and disk specimens. The friction force can be continuously recorded on a strip chart or in a computerized data acquisition system during friction experiments.

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The vacuum system was evacuated in 12 to 15 hr without bakeout to a pressure in the 10^{-7} Pa (10^{-9} torr) range using an oil-sealed mechanical pump, a turbomolecular vacuum pump, an ultraviolet lamp, and a sublimation pump with a titanium ball source. To achieve a pressure in the 10^{-7} Pa (10^{-9} torr) range, the vacuum system is first rough pumped to 27 Pa (200 μm of mercury) with an oil-sealed mechanical pump and to 10^{-3} to 10^{-4} Pa (10^{-5} to 10^{-6} torr) with the turbomolecular vacuum pump. When this vacuum is achieved, the titanium sublimator and the ultraviolet lamp were started at the same time. Sublimation time intervals for the titanium sublimator are set with on periods of 2.5 min, off periods of 48 min, and 10 to 12 cycles of 8.4 to 10.1 hr total duration. The ultraviolet timer was set for 6 hr. After 10 to 12 cycles of sublimation, the pressure was in the 10^{-7} Pa (10^{-9} torr) range. Pressure is measured by a nude ionization gauge. Residual gas analysis is made before, during, and after the friction and wear experiment by using a quadrupole gas analyzer.

DESCRIPTION OF TRIBOEXPERIMENT

MoS_x Specimen Preparation

Films of MoS_x were deposited by magnetron radiofrequency sputtering to a nominal thickness of 110 nm on the 440C stainless-steel disks. The average surface roughness of the 440C stainless-steel disks, as measured by surface profilometer, was 12 ± 2 nm root-mean-square roughness and 9 ± 2 nm centerline-average roughness. Each roughness value is the average of 20 measurements. The average Vickers microhardness measured for the uncoated 440C stainless-steel disks is 695 (6.8 GPa) at loads from 0.49 to 4.9 N (fig. 2).

The 440C stainless-steel disks were scrubbed with ethanol using a wipe and then rinsed sequentially in ethanol and acetone baths. The disks were then placed on the platform in the deposition chamber.

The MoS_x films were deposited by using a commercial magnetron radiofrequency sputtering system. The deposition conditions are presented in table I. The disk substrates were first argon ion sputter-cleaned at 500 W with an argon pressure of 2.7 Pa (20 mtorr) for 5 min. Afterwards, the platform was rotated 180°, and the molybdenum disulfide target was sputter-cleaned at 900 W. Before deposition, the argon pressure and power were adjusted to the deposition conditions. The platform was rotated 180°, the disk substrates were placed under the molybdenum disulfide target, and MoS_x films were deposited at room temperature.

After a deposition procedure is completed, the MoS_x disk specimens were immediately stored in a stainless-steel specimen tray placed in the vacuum chamber (fig. 1), and the system was evacuated to a pressure of 10^{-7} Pa (10^{-9} torr), as described earlier. Absorption of the common contaminants, such as hydrocarbon vapor and water vapor from the laboratory air, and their effects may be minimized by storing MoS_x disk specimens in an ultrahigh vacuum environment.

The average Vickers microhardness values for MoS_x films deposited on 440C stainless steel disks are approximately 10 percent lower than those for uncoated 440C stainless steel disks in the load range 0.1 to 0.25 N (fig. 2). At higher loads (1 to 5 N), however, the microhardness values for MoS_x coated disks are the same as those for the uncoated disks (fig. 2).

Ball Specimen

The 6-mm-diameter ball specimens were 440C stainless steel bearing balls. The average surface roughness of the as-received 440C stainless steel balls, measured by surface profilometer, was 8 ± 2 nm root-mean-square roughness and 7 ± 2 nm centerline-average roughness. Each roughness value was the average of 20 measurements. The average Vickers microhardness for uncoated 440C stainless steel bearing balls is proportional to and approximately 25 percent greater than that for uncoated stainless steel disks over the load range 0.49 to 4.9 N.

Friction and Wear Experiments

Sliding friction experiments were conducted in the chamber shown in figure 1 under the conditions shown in table II. Before each experiment, the as-received ball specimen was ultrasonically rinsed in an ethanol bath. The ball specimen was then ultraviolet ozone cleaned for 1 min at room temperature (ref. 8). The cleaned ball and the as-coated MoS_x disk specimen were positioned in the vacuum chamber (fig. 1), and the system was evacuated to $\approx 7\times10^{-7}$ Pa (5×10^{-9} torr), as described in the previous section. All friction experiments were conducted at this pressure. As the disk rotated, the ball scribed a circular wear track on the flat surface of the disk. In each experiment, a new surface of the ball specimen was used. The loads used were 0.49, 1, 2, and 3.6 N (average Hertzian contact pressures of 0.33 to 0.69 GPa).

Wear volumes of the flat, disk specimens were obtained from stylus tracings across the wear tracks of at least four locations. The average cross sectional area of the wear track was then multiplied by the wear track length computed from the diameter of the track at its center to determine the wear volume.

MAGNETRON SPUTTERED MoS_x FILMS

Auger electron spectroscopy (AES) analysis provided elemental depth profiles for the MoS_x films deposited on the 440C stainless-steel substrates. A typical example of an AES depth profile, with concentration shown as a function of the sputtering distance from the surface of MoS_x film, is presented in figure 3. The concentrations of sulfur and molybdenum at first rapidly increase with an increase in sputtering distance, while the concentrations of carbon and oxygen contaminant decrease. They all remain constant thereafter. The deposited MoS_x films contain small amounts of carbon and oxygen at the surface and in the bulk, and they have a sulphur to molybdenum ratio of approximately 1.7.

SLIDING FRICTION AND WEAR DATA

Friction

Typical coefficient of friction as a function of number of disk revolutions is presented in figures 4 and 5. In figure 4 the coefficients of friction for MoS_x films are plotted as a function of the number of disk revolutions to 400 (the initial run-in period). In figure 5, where the plots are extended to the endurance life, the coefficient of friction rapidly rises to a fixed value around 0.15. The values of coefficient of friction given in figures 4 and 5 are typical, and the trends

with number of disk revolutions are quite reproducible. For comparison, some data points in the range to 400 revolutions shown in figure 5 are repeated from figure 4.

Qualitatively, the coefficient of friction usually starts relatively high (point A) but rapidly decreases and reaches its minimum value of approximately 0.01 (point B) after 40 to 150 disk revolutions. Afterwards, the coefficient of friction gradually increases with increasing number of disk revolutions (fig. 4). It reaches its equilibrium value at point C. From point C to point D it remains constant for a long period. At point D the coefficient of friction becomes lower and remains low from point E to point F. Finally, the sliding action causes the film to break down whereupon the coefficient of friction rapidly increases (line F–G). The plots of figure 5 reveal the similarities in the friction behavior of MoS$_x$ films regardless of load applied. This evidence suggests that increasing the load may not affect the wear mode of MoS$_x$ films.

The friction data presented in figures 4 and 5 clearly indicate that the coefficient of friction for steel balls in contact with MoS$_x$ films varies with load. In general, the higher the load, the lower the coefficient of friction. Therefore, the coefficients of friction as a function of load for the regions designated in figures 4 and 5 were replotted in figure 6 on logarithmic coordinates. The logarithmic plots reveal a generally strong correlation between the coefficient of friction in the steady-state condition (C–D region) and load. The relation between coefficient of friction μ and load W is given by $\mu = kW^{-1/3}$, which expression agrees with the Hertzian contact model (refs. 9 to 13). Similar elastic contact and friction characteristics (load-dependent friction behavior) for polymers (refs. 9 and 14, p. 214-241), diamond (ref. 14, p. 159-185), ceramics (ref. 15), and thin, solid lubricating films like molybdenum disulphide and boron nitride (refs. 16 to 18) can also be found. The load- (or contact pressure-) dependent friction behavior allows us to deduce the coefficient of friction from design concept (e.g., component design parameters). Further, a better understanding of the mechanical factors controlling friction such as load (or contact pressure) would improve the design of advanced bearings and performance of solid lubricants.

Endurance Life

Endurance lives of the magnetron-sputtered MoS$_x$ films (110 nm thick) deposited on sputter-cleaned 440C stainless-steel disks are determined to be the number of revolutions or sliding distance at which the coefficient of friction rapidly rises to approximately 0.15 (see line F–G in fig. 5). The endurance lives as a function of load are presented in figure 7. Even in very carefully controlled conditions, repeat determinations of wear life can show considerable scatter. Although the endurance life determined by the sliding distance showed larger variation than that determined by the number of disk revolutions, the trends are similar; that is, the endurance life of MoS$_x$ films decreases as the load increases.

To express the relation between endurance life and load by an empirical relation, the endurance life data of figure 7 were replotted on logarithmic coordinates (fig. 8). A straight line was easily placed through the data in both plots of figure 8, which, once again, reveal the strong correlation. To a first approximation for the load range investigated, the relation between endurance life E and load W on logarithmic coordinates was expressed by $E = KW^n$, where K and n are constants for the MoS$_x$ films under examination and where the value of n was approximately -1.4.

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Specific Wear Rates and Wear Coefficient

An attempt to estimate average wear rates for MoS_x films was made with the primary aim of generating specific wear rates and wear coefficient data that could be compared with those of other materials in the literature.

It is recognized that the contact by the ball tip is approximately continuous and that the contact by any point on the disk track is intermittent. A fundamental parameter affecting film endurance life is the number of compression and flexure cycles to which each element of the film is subjected. Therefore, normalizing the disk wear volume to the total sliding distance experienced by the ball is fundamentally incorrect. To account for the intermittent and fatigue aspects of this type of experiment, the volume worn away should be given by

$$\text{Wear volume} = c_1 \times \text{normal load} \times \text{number of revolutions} \tag{1}$$

where the dimensional constant c_1 is an averaged specific wear rate expressed in $mm^3/N \cdot$revolution. However, a great quantity of historical ball-on-disk or pin-on-disk results have been reported using an expression of the form

$$\text{Wear volume} = c_2 \times \text{normal load} \times \text{sliding distance} \tag{2}$$

where the dimensional constant c_2 is the averaged specific wear rate expressed in $mm/N \cdot m$. Also, a Holm-Archard relationship of the type

$$\text{Wear volume} = \frac{c_3 \times \text{normal load} \times \text{sliding distance}}{\text{hardness}} \tag{3}$$

where the nondimensional constant c_3 is the nondimensional averaged wear coefficient reported in references 19 to 22.

Figure 9 presents the two specific wear rates and wear coefficient as a function of load. They are almost independent of load for the loads investigated. This evidence suggests that increasing the load in the range of 0.49 to 3.6 N may not affect the wear mode of MoS_x films.

The worn surfaces of MoS_x films took on a burnished appearance and a low wear form of adhesive wear, namely, burnishing wear, was encountered. The two average specific wear rates and nondimensional wear coefficient for the MoS_x films studied herein are approximately 3×10^{-8} $mm^3/N \cdot$revolution (c_1 in eq. (1) and fig. 9(a)), 8×10^{-7} $mm^3/N \cdot m$ (c_2 in eq. (2) and fig. 9(b)), and 5×10^{-6} (c_3 in eq. (3) and fig. 9(b)).

Note that the very concept of specific wear rates and a wear coefficient implicitly assumes a linear relation between the volume of material removed and either the number of revolutions (passes) for the disk (flat) or the distance slid for a ball (rider). If it were true that roughly the same amount of material were removed from a disk specimen by each revolution of the disk, then the relation between wear volume and number of revolutions would be roughly linear, and the specific wear rates and wear coefficient would be meaningful. A consequence for solid lubricating films would be that a film twice the thickness of another similar film should last

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twice as long. However, if material is not removed from the disk (flat) at a constant rate, a measurement of the wear volume after a number of revolutions only gives the average amount of material removed per revolution. The calculated value of wear rate in this case, being an average, would change with the number of revolutions completed, and doubling a solid lubricating film initial thickness would not double the film endurance life. The specific wear rates and wear coefficient for a material such as MoS_x film, therefore, should be viewed with caution.

SUMMARY OF RESULTS

This paper has described a tribometer designed primarily to measure the friction and endurance life of solid lubricating films in ultrahigh vacuum and has presented the friction and wear properties of magnetron sputtered MoS_x films (110 nm thick) deposited on sputter-cleaned 440C stainless steel disks when slid against a 6-mm-diameter 440C stainless steel ball. The following remarks can be made:

1. The tribometer has performed satisfactorily in unidirectional rotation in vacuum at a pressure of 10^{-7} Pa (10^{-9} torr).

2. Similarities are observed in the life cycle friction behavior (coefficient of friction as a function of number of disk revolutions) for MoS_x films at average Hertzian contact from 0.33 to 0.69 GPa.

3. The coefficient of friction μ decreases as load W increases according to $\mu = kW^{-1/3}$. This load- (or contact-pressure-) dependent friction behavior allows us to deduce the coefficient of friction of MoS_x films from design concept (e.g., component design parameters).

4. The endurance life E of MoS_x films decreases as the load W increases according to $E = KW^{-1.4}$. Increasing the load does not change the wear mode of MoS_x films for the load range. The load- (or contact-pressure-) dependent endurance life allows us to reduce the time for wear experiments and to accelerate life testing of MoS_x films.

5. For the MoS_x films investigated the average specific wear rate normalized to the load and the number of revolutions was 3×10^{-8} mm^3/N·revolution, the specific rate normalized to the load and the total sliding distance was 8×10^{-7} mm^3/N·m, and the nondimensional wear coefficient was 5×10^{-6}. These values are almost independent of load for the range 0.49 to 3.6 N (average Hertzian contact pressures of 0.33 to 0.69 GPa).

The correlation between endurance life and load would allow us to reduce the time required for wear experiments. Further, the load- (contact-pressure-) dependent endurance life would enable rapid screening of advanced solid lubricants such as sputtered MoS_x films at higher loads and result in faster technology transfer.

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TABLE I. – MAGNETRON RADIOFREQUENCY SPUTTERING CONDITIONS

Substrate	440C Stainless steel
Ion—etching of substrate before deposition	500 W, 20 mtorr argon for 5 min
Target cleaning	900 W, 20 mtorr argon for 5 min
Deposition conditions	900 W, 20 mtorr argon
Target-to-substrate distance	90 mm
Deposition rate	1100 A min^{-1}
Power density	4.9×10^4 Wm^{-2}
Film density	4.4 g·cm^{-3}
Value of x in MoS$_x$	1.7

TABLE II. – CONDITIONS OF TRIBOEXPERIMENTS

Load, N	0.49 to 3.6
Disk rotating speed, rpm	120
Track diameter, mm	5 to 17
Sliding velocity, mm/s	31 to 107
Vacuum pressure, Pa (torr)	10^{-7} (10^{-9})
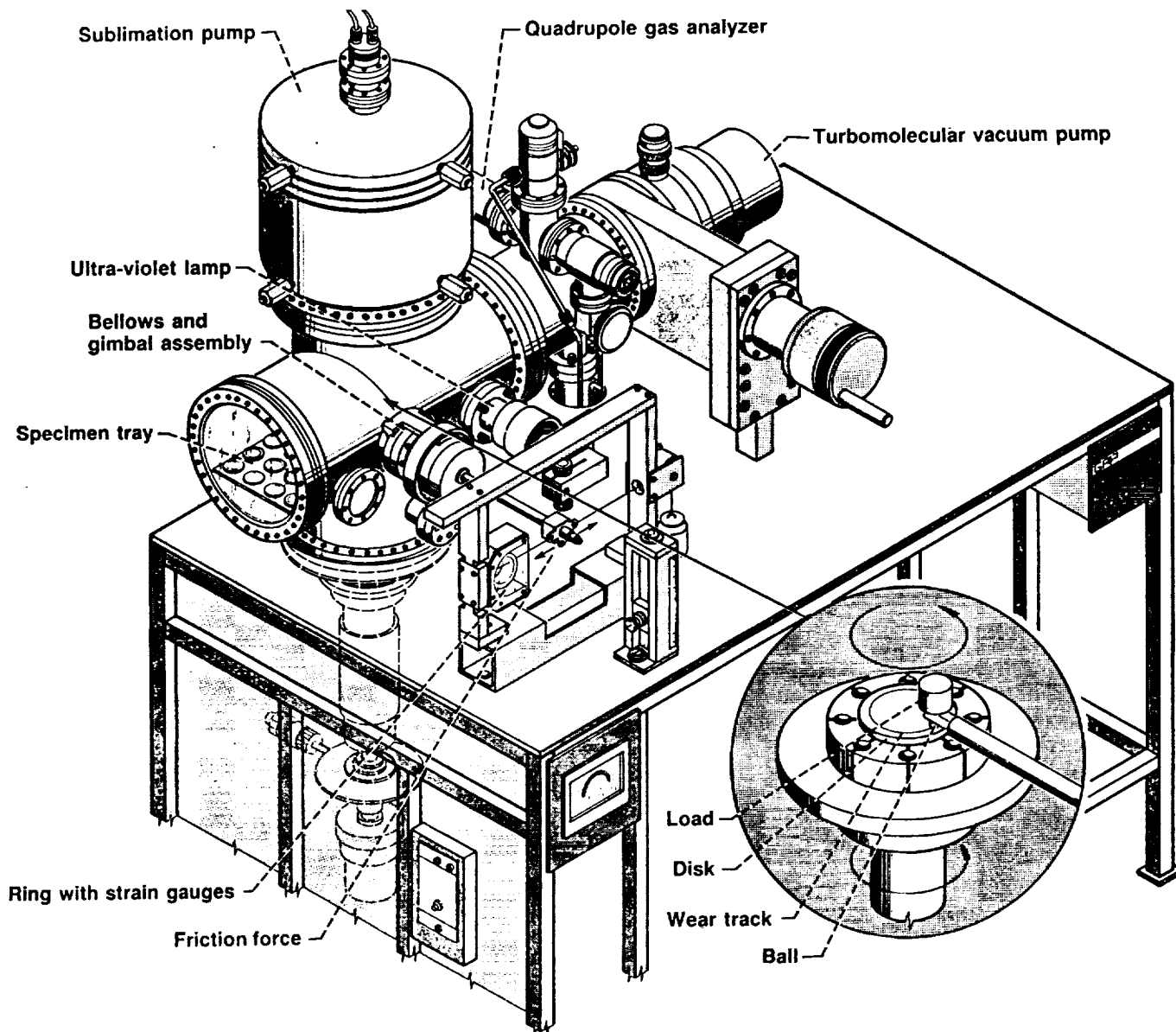
Figure 1.—Vacuum friction apparatus.
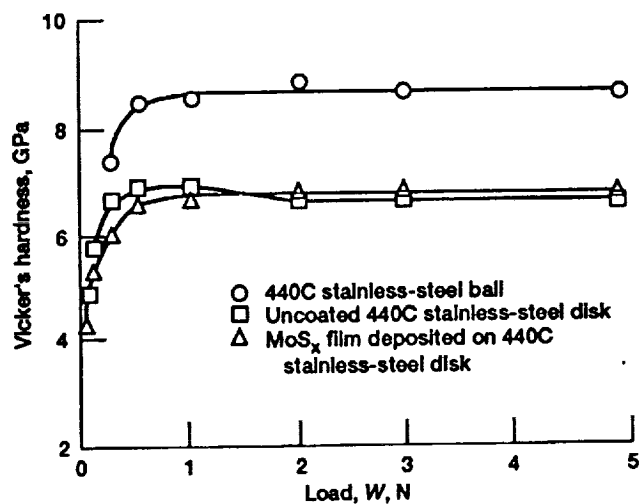
Figure 2.—Vicker's microhardness as function of load.
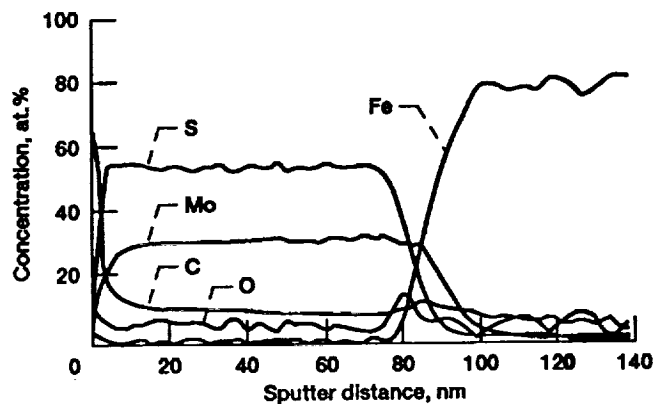
Figure 3.—Auger electron spectroscopy (AES) depth profile of MoS$_x$ film on 440C stainless-steel.
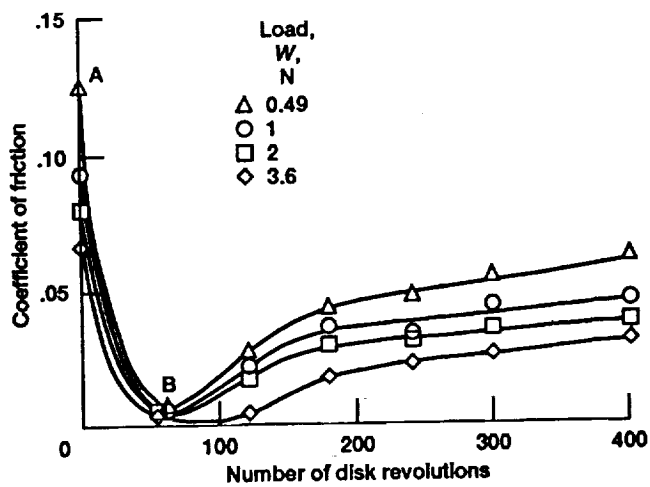
Figure 4.—Run-in average coefficient of friction of MoS$_x$ film as function of number of disk revolutions.
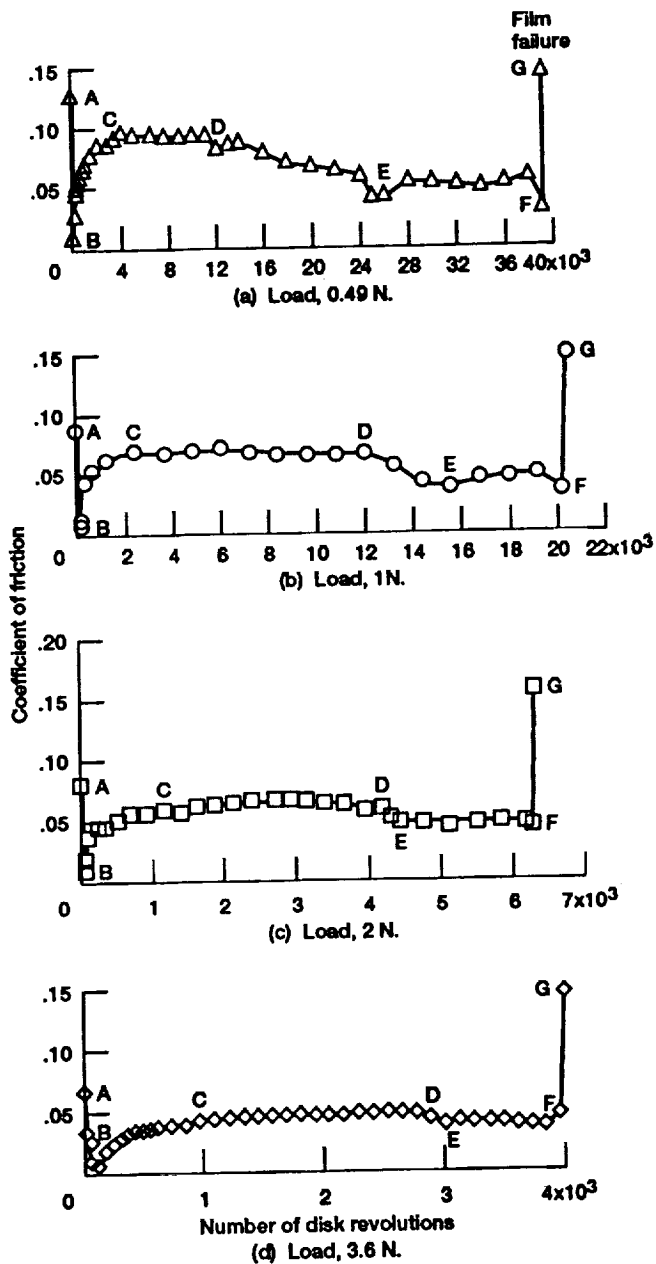

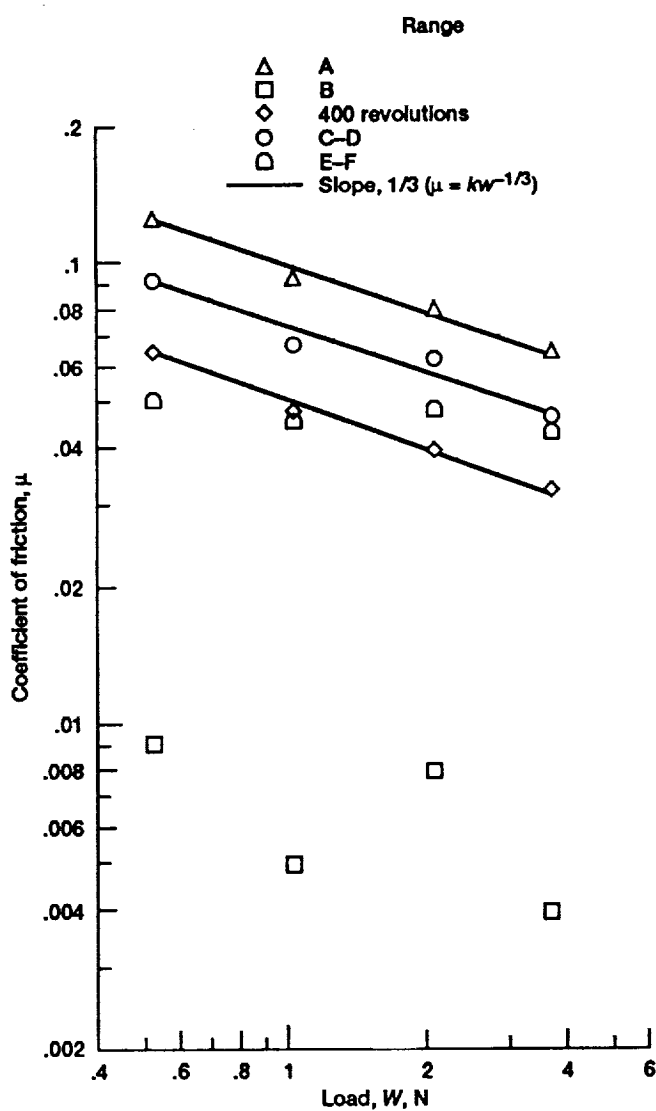**Figure 5.—Coefficient of friction of MoS$_x$ films as function of number of disk revolutions.**

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Figure 6.—Relationship of coefficient of friction and number of revolutions (from regions denoted in figs. 4 and 5) to load.
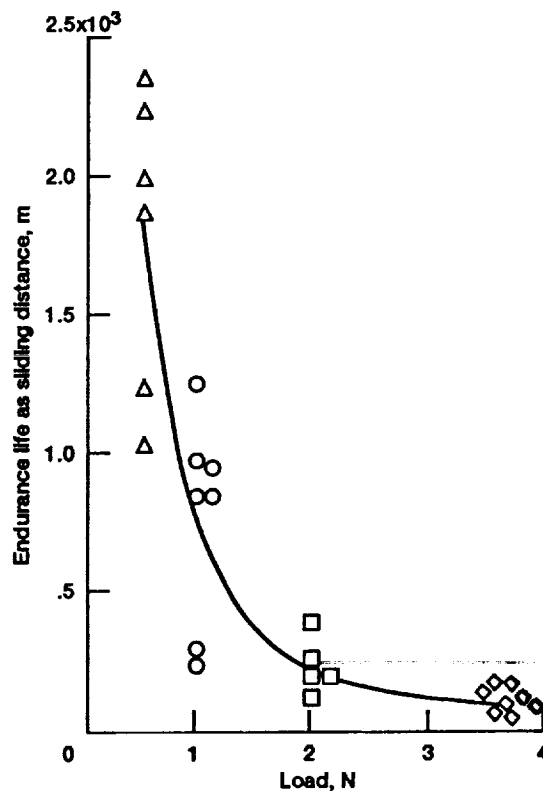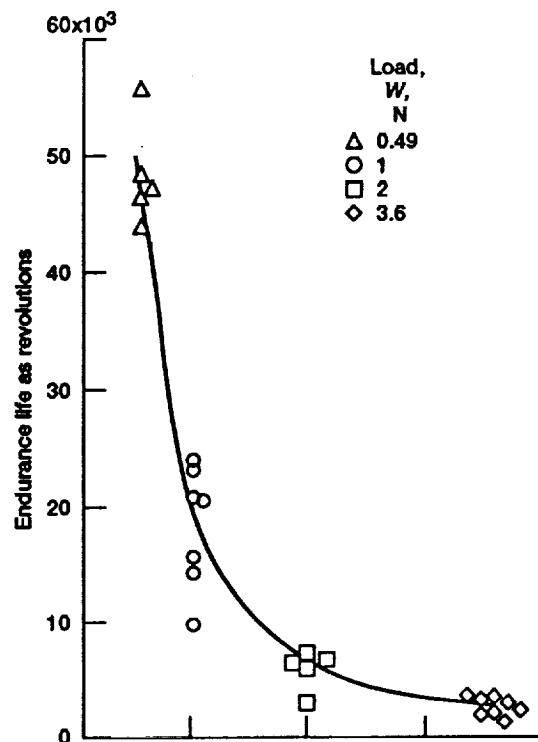
Figure 7.—Endurance life of MoS_x films as function of load; Cartesian plot.
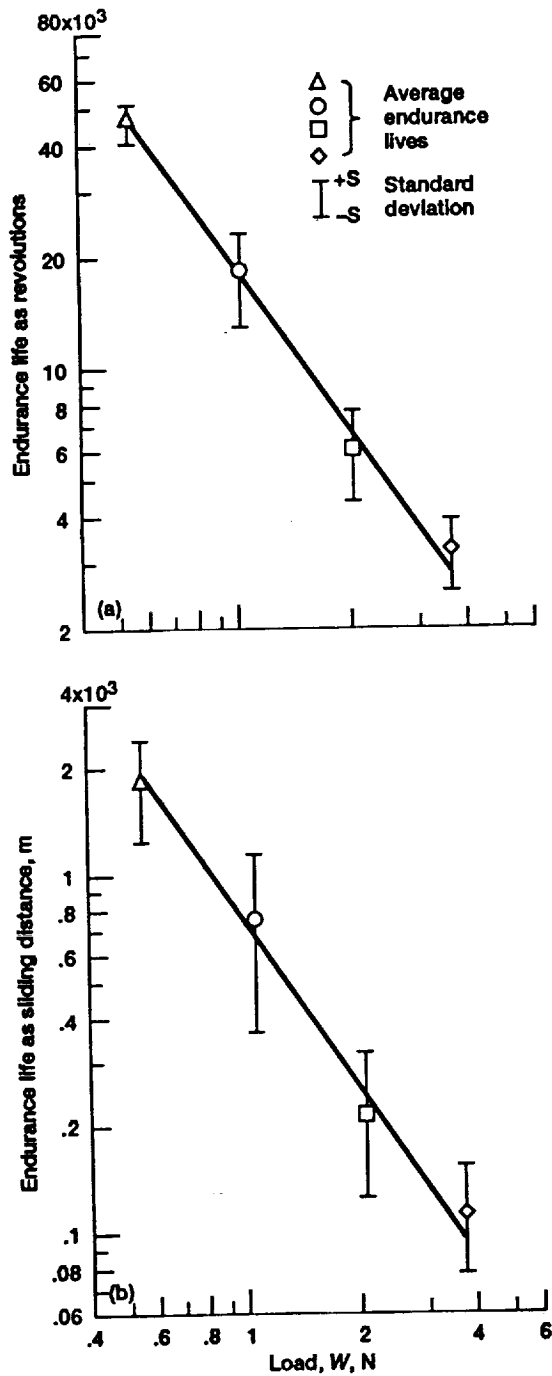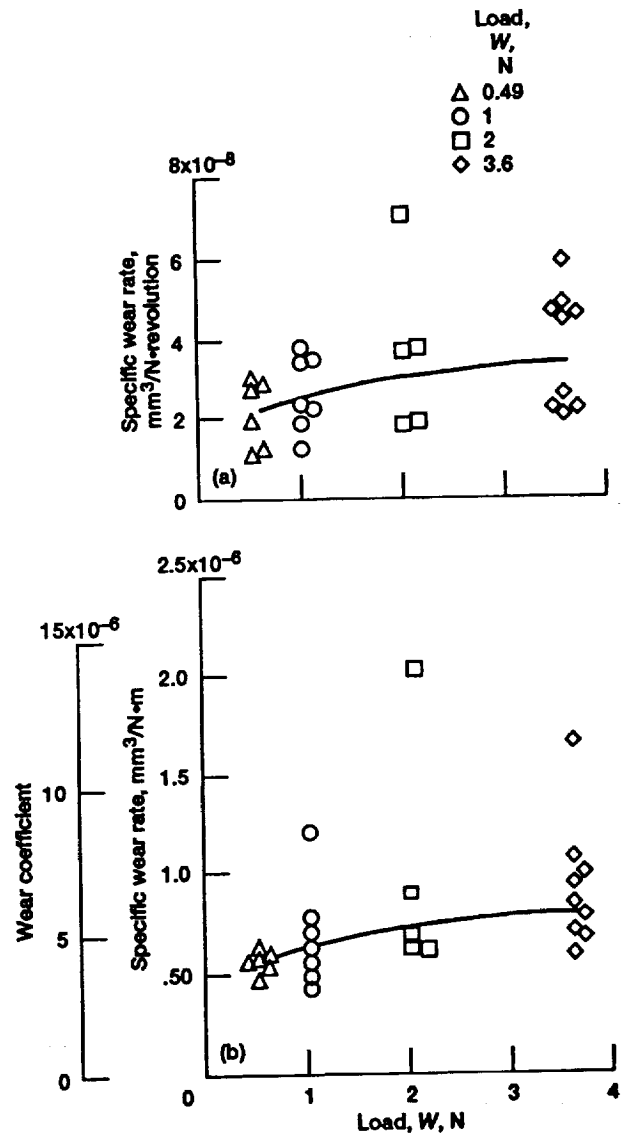
Figure 8.—Endurance life of MoS$_x$ films as function of load in vacuum; Logarithmic plot.

Figure 9.—Wear rates for MoS$_x$ films as function of load in vacuum.

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12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The first part of this paper describes an ultrahigh vacuum friction apparatus (tribometer). The tribometer can be used in a ball-on-disk configuration and is specifically designed to measure the friction and endurance life of solid lubricating films such as MoS$_x$ in vacuum at a pressure of 10^{-7} Pa (10^{-9} torr). The sliding mode is typically unidirectional at a constant rotating speed. The second part of this paper presents some representative friction and endurance life data for magnetron sputtered MoS$_x$ films (110 nm thick) deposited on sputter-cleaned 440C stainless-steel disk substrates, which were slid against a 6-mm-diameter 440C stainless-steel bearing ball. All experiments were conducted with loads of 0.49 to 3.6 N (average Hertzian contact pressure, 0.33 to 0.69 GPa), at a constant rotating speed of 120 rpm (sliding velocity ranging from 31 to 107 mm/s due to the range of wear track radii involved in the experiments), in a vacuum of 7×10^{-7} Pa (5×10^{-9} torr) and at room temperature. The results indicate that there are similarities in friction behavior of MoS$_x$ films over their life cycles regardless of load applied. The coefficient of friction (μ) decreases as load W increases according to $\mu = kW^{-1/3}$. The endurance life E of MoS$_x$ films decreases as the load W increases according to $E = KW^{-1.4}$ for the load range. The load- (or contact-pressure-) dependent endurance life allows us to reduce the time for wear experiments and to accelerate endurance life testing of MoS$_x$ films. For the magnetron-sputtered MoS$_x$ films deposited on 440C stainless-steel disks, the specific wear rate normalized to the load and the number of revolutions was 3×10^{-8} mm^3/N·revolution, the specific wear rate normalized to the load and the total sliding distance was 8×10^{-7} mm^3/N·m, and the nondimensional wear coefficient of was approximately 5×10^{-6}. The values are almost independent of load in the range 0.49 to 3.6 N (average Hertzian contact pressures of 0.33 to 0.69 GPa).

14. SUBJECT TERMS

Vacuum apparatus; Endurance; Friction; Wear

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