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THE THERMAL STABILITY AND FRICTION
OF THE DISULFIDES, DISELENIDES, AND
DITELLURIDES OF MOLYBDENUM AND
TUNGSTEN IN VACUUM (10-9 TO 10-6 TORR)

by William A. Brainard
Lewis Research Center
Cleveland, Ohio



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By William A. Brainard

Lewis Research Center Cleveland, Ohio

ABSTRACT

Thermal stability and friction experiments were conducted with the disulfides, diselenides, and ditellurides of molybdenum and tungsten in vacuum (10^{-9} to 10^{-6} torr). Weight loss rates were plotted as a function of temperature; thermal dissociation temperatures were observed by mass spectrometry. Friction experiments showed the materials were limited as high temperature lubricants by evaporation, dissociation, and wear. Generally, as lubricants, the diselenides provided the best performance ($f_k = 0.04$ to 0.08) up to 1400° F (760° C), whereas the ditellurides provided the poorest because of a low thermal dissociation temperature.

THE THERMAL STABILITY AND FRICTION OF THE DISULFIDES, DISELENIDES, AND DITELLURIDES OF MOLYBDENUM AND TUNGSTEN IN VACUUM (10^{-9} TO 10^{-6} TORR)

by William A. Brainard Lewis Research Center

SUMMARY

Thermal stability and friction experiments were conducted in vacuum (10^{-9} to 10^{-6} torr) with the disulfides, diselenides, and ditellurides of molybdenum and tungsten in order that useful operating temperature ranges could be established. Thermal stability experiments were conducted in vacuum by heating small pressed compacts of each of the six inorganic compounds and monitoring weight loss rates and observing dissociation products on a mass spectrometer. The results show the disulfides are the most thermally stable and the ditellurides the least stable. The temperatures at which dissociation was observed by mass spectrometry were as follows: MoS_2 , 2000^{0} F (1090^{0} C); WS_2 , 1900^{0} F (1040^{0} C); $MoSe_2$, 1800^{0} F (980^{0} C); WSe_2 , 1700^{0} F (930^{0} C); $MoTe_2$ and WTe_2 , 1300^{0} F (700^{0} C). It was observed that the weight loss rate of WS_2 increased more slowly with increasing temperature above 1700^{0} F (930^{0} C) than MoS_2 .

Friction tests were conducted using thin rubbed films of each of the compounds on a 6.35-centimeter-diameter cobalt-base alloy disk which rotated in vacuum against a 0.475-centimeter-radius hemispherical rider. Tests were conducted at temperatures to 1650° F (900° C) in a pressure range of 10^{-8} to 10^{-6} torr. The results showed that thin, unreplenished films of the inorganic compounds were limited as effective lubricants to temperature below those expected from the thermal stability limits because of evaporation of the compounds. Thin films of MoS_2 and WS_2 provided effective lubrication to 1200° F (650° C) and 1350° F (730° C), respectively. Above these temperatures the materials were lost from the disks presumably by evaporation and wear. Both diselenides provided lubrication up to 1400° F (760° C), beyond which temperature, they were lost from the lubricated surface by evaporation, dissociation, and wear. The ditellurides generally were poor as high temperature lubricants because of their relatively low (1300° F or 700° C) thermal dissociation temperature. It was shown that friction increases with time at temperatures where lubricant loss occurred.

INTRODUCTION

 ${
m MoS}_2$, ${
m MoS}_2$, ${
m WS}_2$, ${
m WT}_2$, and ${
m WS}_2$ have found wide usage as solid lubricant materials. They possess intrinsic lubricating ability which is associated with their layer-lattice structure. These solid lubricants have been used very successfully in areas where conventional lubricants could not be used because of extremes of temperature, pressure, or atmosphere (refs. 1 to 6). Recently, data were presented (ref. 7) on the oxidation characteristics of several of the solid lubricants derived from heavy metals. There is, however, a lack of information concerning the stability of these materials in a vacuum environment at elevated temperatures. What data are available are often not consistent. For example, the thermal dissociation temperature of ${
m MoS}_2$, the most widely used inorganic solid lubricant, is variously given as 1832° F (1000° C) (ref. 8), 2012° F (1100° C) (ref. 5), 2498° F (1370° C) (ref. 9), and 3002° to 3092° F (1650° to 1700° C) (ref. 10).

Further, there is a lack of complete information on the lubricating ability of these solid lubricant materials in a vacuum environment at elevated temperatures.

The objectives of this investigation were to examine the following:

- (1) The thermal stability of the molybdenum and tungsten disulfides, diselenides, and ditellurides in vacuum
- (2) The lubricating ability of these compounds at elevated temperatures in vacuum The six compounds were pressed into compacts, and heated in vacuum to determine their thermal stability.

Friction experiments were also conducted in vacuum using thin, rubbed lubricant films of each of the six compounds at temperatures to 1650° F (900° C).

APPARATUS

Thermal Stability Apparatus

The apparatus shown schematically in figure 1 was used to determine the vacuum thermal stability of the inorganic solid lubricants. The apparatus is similar to that described in reference 4. A small (200-mg) pressed compact of the solid lubricant material is placed in a tantalum pan and suspended from a highly sensitive recording electronic balance. In these experiments, weight losses as small as 1.0×10^{-5} gram could be detected. The suspended sample is enclosed by a 1.91-centimeter-diameter tantalum tube, which was used as a thermal susceptor. Directly under the specimen in the tube was a large bead Chromel-Alumel thermocouple which was used to monitor temperatures inside the tube. This thermocouple was calibrated against a 0.0025-centimeter-diameter thermocouple embedded in the test sample. At equilibrium tem-

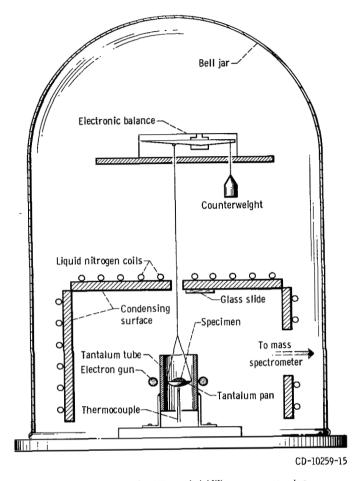


Figure 1. - Schematic of thermal stability vacuum apparatus.

perature, no discernible temperature difference between the two thermocouples was observed. To further insure temperature measurement accuracy, the temperature measuring equipment was calibrated with the standard millivolt reference, with the maximum error being $\pm 10^{\circ}$ R (5.5° C) at the maximum operating temperature.

Approximately 0.5 centimeter from the outside wall of the tube were two electron gun filaments which were used to heat the tube to a maximum inside temperature of 2300° F (1260° C). The gun filament voltage was controlled by a proportional controller using the thermocouple for a signal. The tube temperature could be controlled to within $\pm 10^{\circ}$ F ($\pm 5.5^{\circ}$ C).

Directly above the furnace and on both sides are liquid nitrogen cooled surfaces to trap any evaporated or desorbed material. Glass slides were mounted on these surfaces so condensate could later be analyzed by X-ray techniques.

A mass spectrometer was mounted adjacent to the test area so that gaseous species could be monitored and dissociation products observed.

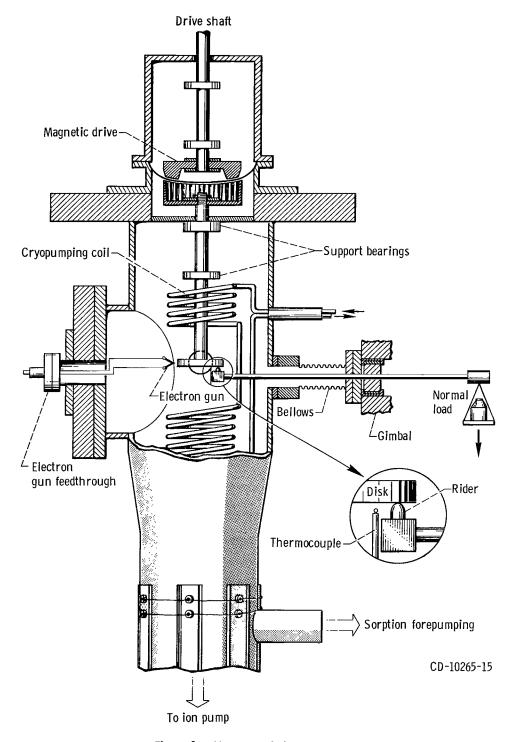


Figure 2. - Vacuum-friction apparatus.

The system was evacuated by two 400-liter-per-second ion pumps and a titanium getter pump. Ultimate pressure is 10^{-10} torr but during heating, the pressure rose into the range, 10^{-8} to 10^{-6} torr.

Friction Apparatus

Friction experiments with the inorganic solid film lubricants were conducted in the vacuum-friction apparatus shown in figure 2. A set of test specimens consisted of a 6.35-centimeter-diameter flat disk with an inorganic rubbed solid lubricant film applied to it and a 0.475-centimeter-radius bullet specimen. The disk specimen is mounted horizon-tally on a magnetically driven shaft. The rider is mounted in a holder which bolts to a bellows-sealed, gimbal-mounted load arm. The hemispherical tip of the rider specimen is loaded against the flat side of the rotating disk by dead weight loading. The friction force is transmitted by the arm to a strain gage assembly mounted outside the chamber. The output of the strain gage is read on a multirange potentiometer.

Attached to the lower end of the chamber are two cryosorption pumps which are used to rough pump the system to 1 micron. A 500-liter-per-second ion pump is used for pumping in the UHV range. A stainless steel coil of 0.955 centimeter diameter, 6 meters long can be used for cryopumping of the system. No cryopumping was used in these experiments. The pumping system is free from any organic contamination that might result from mechanical or diffusion pumping.

A cold cathode ionization gage located adjacent to the test specimens was used to monitor the chamber pressure. Pressure varied during the experiment, depending upon test temperature, but never exceeded 10^{-6} torr.

The disk specimen was heated by a 1750-watt electron gun. The electron gun filament was located approximately 0.63 centimeter from the edge of the disk specimen. The disk temperature was monitored by a Chromel-Alumel thermocouple located 0.158 centimeter from the disk surface near the rider-disk contact zone. The thermocouple temperature was calibrated against a contacting thermocouple while the disk was stationary. The temperature measured by the proximity thermocouple was accurate to within approximately 25° F (14° C).

MATERIALS

Some physical properties of the molybdenum and tungsten disulfides, diselenides, and ditellurides are given in table I(a). With the exception of the orthorhombic WTe_2 , the materials all have the hexagonal crystal structure. The materials used in this investigation were obtained from a commercial supplier; the purity levels were 99.5 percent with the exception of MoS_2 which was 99.99 percent and WS_2 which was 99 percent.

TABLE I. - PROPERTIES AND ANALYSES OF MATERIALS

(a) Properties of the molybdenum and tungsten disulfides, diselenides, and ditellurides (ref. 2)

| Compound | Crystal structure | Color | Molecular weight | Density, g/cm ³ | Lattice parameter, Å | | Resistivity, Ω , | Air stability, |
|------------------|----------------------|----------|---------------------|-------------------------------|-------------------------|-------------|-------------------------|-------------------|
| | | | | | a-direction | c-direction | cm | °C |
| MoS ₂ | Hexagonal | Gray | 160.07 | 4.80 | 3.16 | 12.295 | 8.5×10 ² | 350 |
| MoTe, | Hexagonal | | 351.14 | 7.7 | 3.52 | 13.97 | 8.7×10 ² | 400 |
| MoSe, | Hexagonal | | 253.86 | 6.9 | 3.2 9 | 12.80 | 1.8×10 ⁻² | 400 |
| ws, 1 | Hexagonal | | 2 47.98 | 7.50 | 3.29 | 12.97 | 1.4×10 ¹ | 440 |
| WTe2 | Orthorhombic | | 439.05 | 9.4 | | | 3.1×10 ⁻³ | |
| WSe ₂ | Hexagonal | \ | 341.78 | 9.0 | 3.29 | 12.95 | 1.1×10 ² | 350 |

(b) Analyses of materials

| Compound | Supplier's analysis | Stoichometric percentage | | |
|-------------------|-------------------------|--------------------------|--|--|
| MoS ₂ | | 40.06 percent sulfur | | |
| MoTe ₂ | 72.00 percent tellurium | 72.65 percent tellurium | | |
| MoSe ₂ | 61.88 percent selenium | 62.20 percent tellurium | | |
| ws_2 | 26.15 percent sulfur | 25.77 percent sulfur | | |
| WTe ₂ | 57.46 percent tellurium | 58.12 percent tellurium | | |
| WSe ₂ | 45.50 percent selenium | 46.20 percent selenium | | |

Analysis of the materials are given in table I(b) along with the composition for the stoichometric compounds. No attempts were made to further purify the materials.

PROCEDURE

Thermal Stability Experiments

Thermal stability experiments were conducted with compacts pressed from each of the six solid lubricant materials. Each compact was suspended from the electronic balance inside the tantalum tube susceptor. After system pumpdown and bakeout (to a maximum specimen temperature of 212° F (100° C), the condensing surfaces were cooled with liquid nitrogen and specimen heating started.

The specimens were heated in steps of 100° F (59° C) from 400° to 2300° F (204° to 1260° C) or until excessive dissociation rates occurred. At each temperature, weight loss readings and mass spectrometer readings were made. Large weight loss readings

coupled with the observation of sulphur, tellurium, or selenium peaks on the mass spectrometer were indicative of thermal dissociation. After heating to maximum temperature and subsequent cooling, the chamber was opened and the condensate on the glass slides analyzed with X-ray techniques.

Friction Experiments

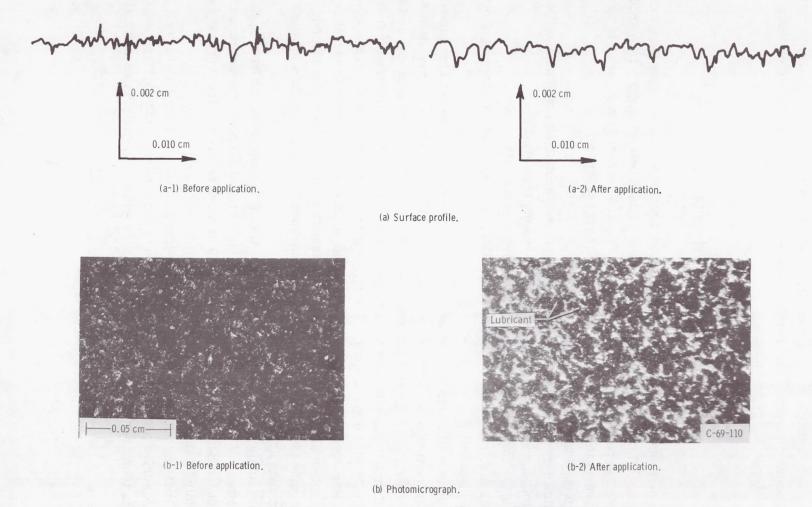
Friction testing of the solid lubricant materials was carried out using very thin rubbed lubricant films on a cobalt alloy substrate. The cobalt-base alloy (32 percent Cr, 17 percent W, 2.5 percent C, 3.0 percent max Fe, 2.5 percent max Ni, and 41 percent Co) was chosen as the disk material for the following reasons:

- (1) It has good hot hardness characteristics.
- (2) Preliminary friction tests with bare metal combinations showed no significant change in friction upon heating to the maximum testing temperatures.
- (3) The alloy has been used to make high temperature ball bearings. The rider was also machined from the cobalt alloy. Hardness of the riders and disks was 55 to 57 Rockwell C.

Each lubricant film was applied in the same manner. A new set of specimens was used for each friction run. The disk was sandblasted to an average roughness of 40 to 45 microinches rms. Following blasting, each disk was rinsed with acetone, and then scrubbed with moist polishing grade aluminum oxide. It was then rinsed with distilled water and finally with ethyl alcohol. The rider specimens were cleaned in the same manner. Following cleaning, a coarse cloth was used to rub the solid lubricant on the disk surface in the direction of sliding until a smooth uniform film was achieved. No attempt was made to control or measure film thickness. Typical surface profiles and photomicrographs are shown in figure 3.

Following application of the lubricant film, the specimens were mounted in the vacuum chamber. After subsequent system pumpdown and bakeout, the specimens were heated to test temperature and held there for 30 minutes before the friction test was started. Sliding was then initiated and the friction recorded.

Sliding continued for 30 minutes, and the representative friction for that test was recorded. A series of runs, each with a new set of specimens and newly applied films, were made to temperatures of 1650° F (900° C) for each of the solid lubricant materials.



 $Figure \ 3. \ \ \hbox{-Typical disk surface profile and photomic rograph before and after application of lubricant film.}$

RESULTS AND DISCUSSION

Thermal Stability Results

Pressed compacts of each of the solid lubricant compounds were heated in vacuum and the rate of weight loss measured. The weight loss rate curves for the disulfides of molybdenum and tungsten are given in figure 4. No weight loss was observed for MoS_2 up to 1700° F $(930^{\circ}$ C). Beyond 1700° F $(930^{\circ}$ C), the weight loss rate increased with temperature. At 2000° F $(1090^{\circ}$ C), sulfur peaks were observed on the mass spectrometer, indicating the liberation of sulfur because of thermal dissociation. Dissociation undoubtly begins at a lower temperature but the amount of elemental sulfur liberated was not sufficient for detection. Reference 11 gives 842° F $(450^{\circ}$ C) as the sublimation temperature of MoS_2 , while reference 12 gives 1922° F $(1050^{\circ}$ C). Reference 13 heated MoS_2 powder in vacuum to 1832° F $(1000^{\circ}$ C) and reported a constant weight loss rate at 1742° F $(950^{\circ}$ C) which is in good agreement with the data of figure 4.

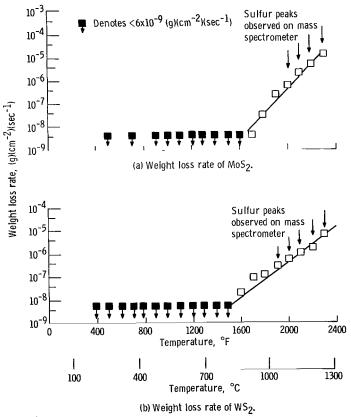


Figure 4. – Weight loss rate curves for ${\rm MoS_2}$ and ${\rm WS_2}$ when heated in vacuum (${\rm 10^{-9}}$ to ${\rm 10^{-8}}$ torr).

The WS $_2$ compact began to have a detectable loss rate at 1600 $^{\rm O}$ F (870 $^{\rm O}$ C), and sulfur peaks were observed at 1900 $^{\rm O}$ F (1040 $^{\rm O}$ C). The rate-temperature slope for WS $_2$ is less than that of MoS $_2$. It is of interest to note that, during oxidation studies of MoS $_2$ and WS $_2$ (ref. 14), it was shown that MoS $_2$ oxidizes slower than WS $_2$ at lower temperatures, but at the higher temperatures, the rate of oxidation of MoS $_2$ is more rapid than for WS $_2$.

The weight loss rate curves for $MoSe_2$ and WSe_2 are presented in figure 5. Both these compounds exhibit weight loss at temperatures lower than the equivalent disulfides. Selenium peaks were observed at 1800° F (980° C) for $MoSe_2$ and 1700° F (930° C) for WSe_2 . Again dissociation was probably occurring at lower temperatures but could not be detected. Both diselenides have similar rate-temperature slopes. At the lower temperatures some weight losses were detected; these losses were likely impurities.

The results for the ditellurides are given in figure 6. Both of these compounds exhibited high weight loss rates up to 800° F (427° C) with the liberation of tellurium. It is believed that these peaks at 800° F (427° C) represent the liberation of uncombined

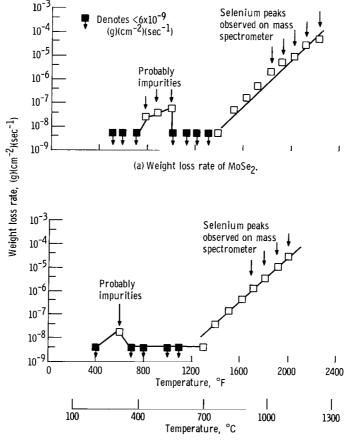


Figure 5. - Weight loss rate curves for MoSe $_2$ and WSe $_2$ when heated in vacuum (10^{-9} to 10^{-8} torr).

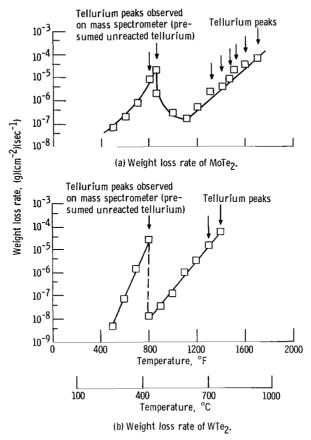


Figure 6. - Weight loss rate curves for MoTe $_2$ and WTe $_2$ when heated in vacuum (10 $^{-9}$ to 10 $^{-8}$ torr).

tellurium rather than the reduction to some lower order telluride. X-ray diffraction and fluorescence patterns were taken of $MoTe_2$ powder before and after heating to 800° F (427° C) in vacuum. The fluorescence patterns indicated a definite loss of tellurium, but the diffraction patterns showed no change in structure.

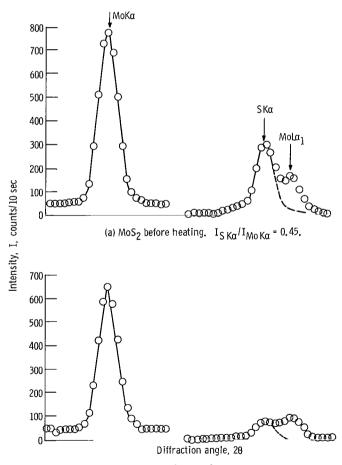
Upon further heating, weight loss rates again increased and tellurium peaks were monitored on the mass spectrometer at 1300° F (700° C).

Following each run, the chamber was opened and the compact and condensate were analyzed by X-ray techniques. The results of the analyses on the condensate are given in table II. Only the condensate from the ditellurides and WS₂ were thick enough for detection by X-ray analysis. The pressed compacts were analyzed by X-ray fluorescence and the ratios of its constituent elements peaks were compared with prerun ratios. Figure 7 shows the change in the sulfur to molybdenum ratio in MoS₂ after heating in vacuum. There was still sulfur remaining in the compact but the ratio of intensities changed from 0.45 to 0.18 indicating the sample consisted of Mo and MoS₂. In the case

TABLE II. - ANALYSES OF DISSOCIATION PRODUCTS CONDENSATE BY

X-RAY DIFFRACTION OR FLUORESCENCE

| Compound | Deposit color | Amount deposited | Results of X-ray analysis |
|------------------|---------------|------------------|---|
| MoS ₂ | Yellow-brown | Very light | Not sufficient material for diffraction or fluorescence |
| ${\tt MoTe}_2$ | Black | Heavy | Diffraction indicates elemen- tal tellurium |
| $MoSe_2$ | Reddish brown | Very light | Not sufficient material for diffraction or fluorescence |
| WS_2 | Yellow-brown | Light | Fluorescence detects sulfur |
| WTe ₂ | Black | Heavy | Diffraction indicates elemental tellurium |
| WSe_2 | Reddish brown | Very light | Not sufficient material for diffraction or fluorescence |



(b) MoS $_2$ after heating to 2300° F (1260° C) in thermal stability apparatus. I $_{S~K\alpha}/I_{Mo~K\alpha}$ = 0.18.

Figure 7. - Ratio of intensities of constituent element peaks in X-ray fluorescence patterns of ${\rm MoS}_2$ before and after heating in vacuum.

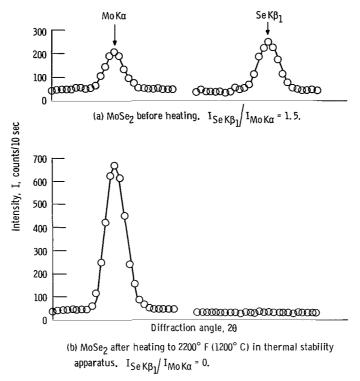


Figure 8. - Ratio of intensities of constituent element peaks in X-ray fluorescence patterns of MoSe₂ before and after heating in vacuum.

of $MoSe_2$ (fig. 8), no selenium was detected by fluorescence after heating to 2200° F (1200°C) indicating that the decomposition reaction was complete.

Friction Results

The molybdenum and tungsten compounds were examined as thin lubricant films at elevated temperatures in vacuum in order that limiting temperatures for these materials could be established under these conditions. Figures 9 and 10 present friction data for cobalt alloy disks lubricated with the six inorganic compounds at temperatures to 1650° F (900° C). At each temperature, a newly coated disk was used. The disk was brought to test temperature and held there for 60 minutes. During the final 30 minutes, sliding was initiated and the representative friction recorded.

For all the compounds except WTe_2 , the friction below 1200° F (650°C) was low (0.04 to 0.08). The higher friction for the WTe_2 (0.20 to 0.25) lubricated disks is probably related to the orthorhombic crystal structure of WTe_2 as compared with the hexagonal structure for the other compounds (ref. 8).

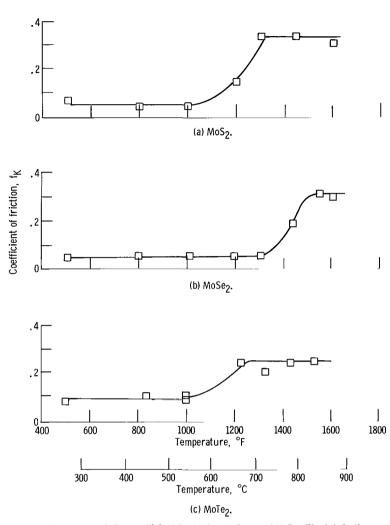


Figure 9. - Friction coefficient for MoS $_2$, MoS $_2$, and MoT $_2$ film lubrication at various temperatures. Exposure time, 60 minutes in vacuum (10^{-8} to 10^{-6} torr); load, 100 grams; speed, 2.0 centimeters per second.

At the higher temperatures, the measured friction of all the lubricant films increased. These increases are associated with loss of the lubricant films from the disk surface by evaporation, dissociation, and wear.

For ${\rm MoS}_2$ and ${\rm WS}_2$, the mass spectrometer data shown in figure 4 indicate that dissociation is probably not the cause for lubricant loss up to $1650^{\rm O}$ F ($900^{\rm O}$ C). The lubricant loss is due primarily to thermal evaporation and wear. Even though the measured weight loss rate of both disulfides at $1400^{\rm O}$ F ($760^{\rm O}$ C) is $<6\times10^{-9}$ (gram) (centimeter $^{-2}$)(second $^{-1}$), the actual area of lubricant exposure is much larger than the apparent area so the amount of lubricant lost was greater than calculation based on apparent area would predict. The ${\rm WS}_2$ films provide lubrication to a slightly higher temperature than the ${\rm MoS}_2$ films and this may be due to the higher molecular weight

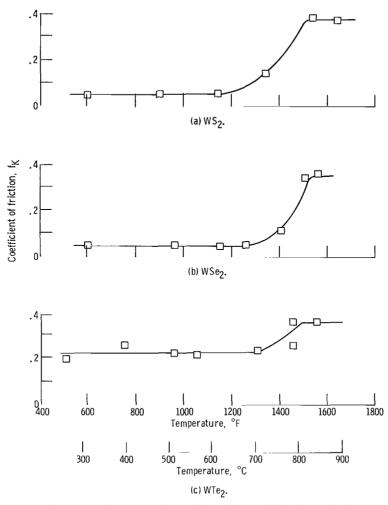


Figure 10. - Friction coefficient for WS $_2$, WSe $_2$, and WTe $_2$ film lubrication at various temperatures. Exposure time, 60 minutes in vacuum (10^{-8} to 10^{-6} torr); load, 100 grams; speed, 2.0 centimeters per second.

(247.98) of WS $_2$ than MoS $_2$ (160.07). Confirming these results, reference 15 reported failure of MoS $_2$ films in vacuum above 1470 $^{\rm O}$ F (800 $^{\rm O}$ C) which the author attributes to decomposition of the MoS $_2$.

The disclenides both provide lubrication over the same temperature range. Above 1400° F (760°C), the increase in friction is due to evaporation and wear with the possibility of some dissociation at the maximum temperatures (fig. 5). MoSe₂ and WSe₂ provided the best lubricant performance of the compounds tested.

From the data of figure 6, it was seen that thermal dissociation of the ditellurides occurred at 1300° F (700° C) and very likely below; thus the increase in friction for lubricant films of the ditellurides is due to thermal dissociation. Some slight lubricant action is probably afforded by the mixture of the metal and the remaining metal telluride.

Figures 11, 12, and 13 present photomicrographs of the disk surface with lubricant

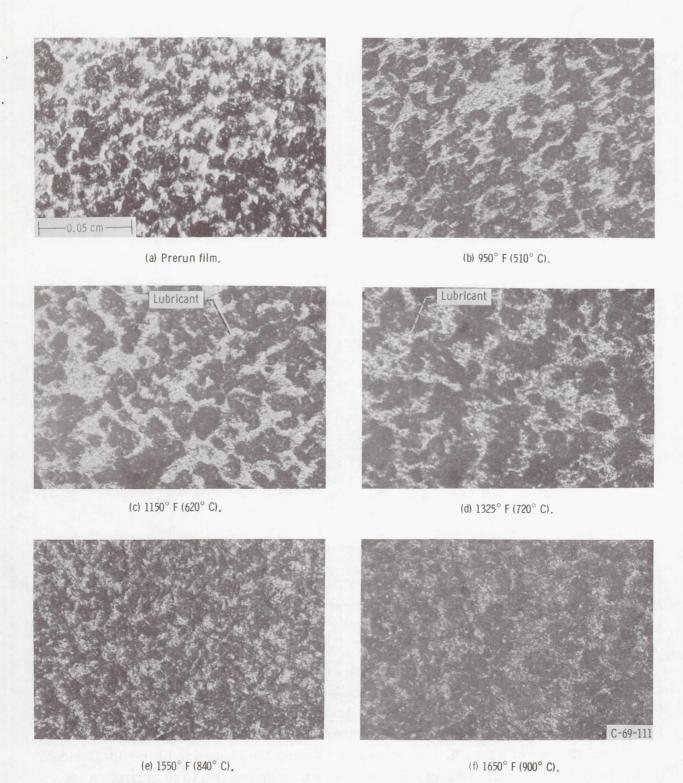


Figure 11. - Photomicrographs of disk surface after runs at various temperatures with rubbed WS $_2$ film lubrication. Pressure, 10^{-8} to 10^{-6} torr.

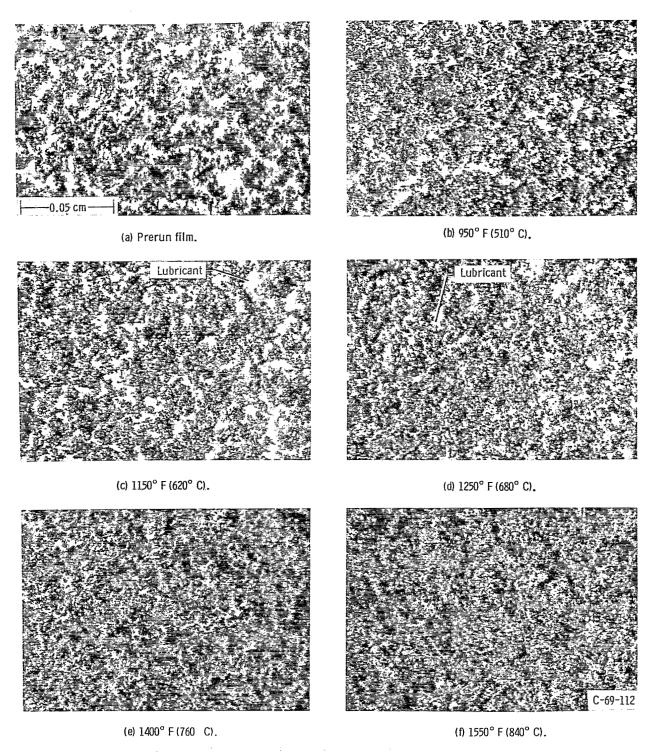


Figure 12. – Photomicrographs of disk surface after runs at various temperatures with rubbed WSe $_2$ film lubrication. Pressure, 10^{-8} to 10^{-6} torr.

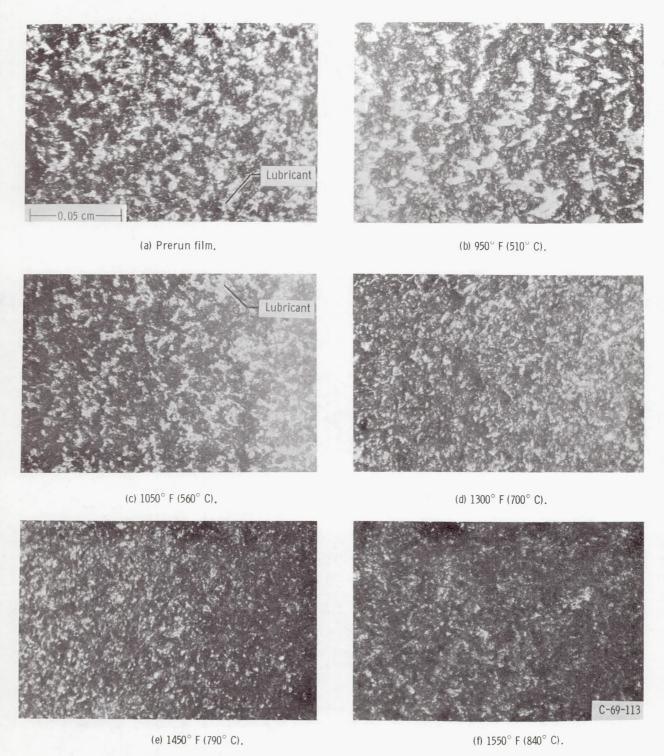


Figure 13. – Photomicrographs of disk surface after runs at various temperatures with rubbed WTe $_2$ film lubrication. Pressure, 10^{-8} to 10^{-6} torr.

films of the tungsten compounds before running and after various thermal-vacuum exposures. The lubricant appears white because of the illumination. The photomicrographs show very definite loss of the lubricant material from the disk surface, and there is good agreement between the observed lubricant loss and the friction-temperature curve of figure 10.

In order to observe the effect of exposure time on friction, runs were made with the disulfides and the diselenides as lubricants with exposure times to 5 hours. The ditellurides were not examined because of their low dissociation temperatures. The results are given in figure 14. The runs were made at 1325° F (720° C) and the friction measured during a 5-minute interval each hour. As expected, there is a general increase in friction for all the compounds. The longer a lubricant film was exposed, the greater the lubricant loss and consequently the higher the friction.

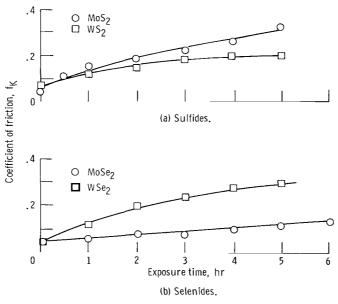


Figure 14. - Coefficient of friction at 1325° F (720° C) of cobalt-base alloy with four lubricants. Pressure, 10^{-8} to 10^{-7} torr; load, 100 grams; sliding speed, 2.0 centimeters per second.

SUMMARY OF RESULTS

Thermal Stability Results

Thermal stability experiments conducted in vacuum with the disulfides, diselenides, and ditellurides of molybdenum and tungsten yielded the following results:

- 1. The disulfides are the most thermally stable of the inorganic compounds at elevated temperatures in vacuum. The ditellurides are the least thermally stable.
- 2. The temperatures at which thermal dissociation in vacuum was definitely observed by mass spectrometry were: MoS_2 , 2000^O F (1090^O C); WS_2 , 1900^O F (1040^O C); $MoSe_2$, 1800^O F (980^O C); WSe_2 , 1700^O F (930^O C); $MoTe_2$ and WTe_2 , 1300^O F (700^O C).
- 3. The weight loss rate of WS_2 increased more slowly with increasing temperature above 1700° F (930° C) than the rate of MoS_2 .

Friction Results

Friction experiments conducted in vacuum with thin unreplenished films of each of the inorganic lubricant materials on a cobalt base alloy yielded the following results:

- 1. Thin, unreplenished films of the inorganic material were limited as effective lubricants not only by thermal dissociation but also by thermal evaporation and wear. The effects of evaporation lowered the temperature range where these thin inorganic films provided good lubrication.
- 2. ${\rm MoS_2}$ and ${\rm WS_2}$ did not provide effective lubrication above 1200° F (650° C) for ${\rm MoS_2}$ and 1350° F (730° C) for ${\rm WS_2}$ because of evaporation of the materials.
- 3. The diselenides provided the best high temperature performance to 1400° F (760°C). The diselenides were lost from the disk by evaporation, by wear, and at the maximum temperature, by dissociation.
- 4. The ditellurides failed as high temperature vacuum lubricants because of their relatively low $(1300^{\circ}\ \text{F or }700^{\circ}\ \text{C})$ thermal dissociation temperatures.
- 5. For the sulfides and selenides of molybdenum and tungsten, prolonged thermal-vacuum exposure at a temperature where lubricant losses occurred resulted in an increase in friction with time because of a decrease in the amount of lubricant affording protection.

Lewis Research Center,

National Aeronautics and Space Administration, Cleveland, Ohio, January 3, 1969, 129-03-13-09-22.

REFERENCES

1. Winer, W. O.: Molybdenum Disulfide as a Lubricant: A Review of the Fundamental Knowledge. Wear, vol. 10, 1967, pp. 422-252.

- 2. Magie, Peter M.: A Review of the Properties and Potentials of the New Heavy Metal Derivative Solid Lubricants. Lubr. Eng., vol. 22, no. 7, July 1966, pp. 262-269.
- 3. Boes, D. J.: Unique Solid Lubricating Materials for High Temperature-Air Applications. ASLE Trans., vol. 10, no. 1, Jan. 1967, pp. 19-27.
- 4. Buckley, Donald H.; Swikert, Max; and Johnson, Robert L.: Friction, Wear, and Evaporation Rates of Various Materials in Vacuum to 10⁻⁷ mm Hg. ASLE Trans., vol. 5, no. 1, Apr. 1962, pp. 8-23.
- 5. Amateau, Maurice F.; Krause, Horatio H.; Orcutt, F. K.; Glasser, W. A.; and Allen, C. M.: Development of Lubricants for Positive-Displacement Power Sources from 1200° F to 1500° F. Battelle Memorial Inst. (ASD-TR-61-478, DDC No. AD-265 587), July 14, 1961.
- 6. Bisson, Edmond E.; and Anderson, William J.: Advanced Bearing Technology. NASA SP-38, 1965.
- Lavik, Melvin T.; Medved, Thomas M.; and Moore, G. David: Oxidation Characteristics of MoS₂ and Other Solid Lubricants. ASLE Trans., vol. 11, no. 1, Jan. 1968, pp. 44-55.
- 8. Boes, David J.: New Solid Lubricants: Their Preparations, Properties and Potential For Aerospace Applications. IEEE Trans. on Aerospace, vol. 2, no. 2, Apr. 1964, pp. 457-466.
- 9. Scholz, W. G.; Doane, D. V.; and Timmons, G. A.: Molybdenum by Direct Thermal Dissociation of Molybdenum Disulfide. Trans. AIME, vol. 221, no. 2. Apr. 1961, pp. 356-364.
- Touloukian, Y. S., ed.: Thermophysical Properties of High Temperature Solid Materials. Vol. 5. Macmillan Co., 1967, p. 691.
- 11. Killeffer, D. H.; and Linz, Arthur: Molybdenum Compounds, Their Chemistry and Technology. Interscience Publ., 1952, pp. 54-56.
- 12. Cannon, Peter: Melting Point and Sublimation of Molybdenum Disulfide. Nature, vol. 183, 1959, pp. 1612-1613.
- 13. Johnston, R. R. M.; and Moore, A. J. W.: Water Adsorption on Molybdenum Disulfide Containing Surface Contaminants. J. Phys. Chem., vol. 68, no. 11, Nov. 1964, pp. 3399-3405.
- 14. Sliney, Harold E.: Decomposition Kinetics of Some Solid Lubricants Determined by Elevated-Temperature X-ray Diffraction Techniques. Presented at the USAF Aerospace Fluids and Lubricants Conference, San Antonio, Texas, Apr. 16-19, 1963.

15. Rowe, G. W.: Vapour Lubrication and the Friction of Clean Surfaces. Inst. Mech. Eng. Conference on Lubrication and Wear, London, Oct. 1-3, 1957, pp. 333-338.

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