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ADAPTATION OF AN MoS_2 "IN SITU" PROCESS FOR LUBRICATING SPACECRAFT MECHANICAL COMPONENTS

by Charles E. Vest

Goddard Space Flight Center
Greenbelt, Maryland

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SUMMARY

This paper presents one approach to overcome some of the difficulties of using MoS_2 as a space lubricant. These difficulties occur in obtaining a tenacious bond of MoS_2 film to the substrate material, and in devising a method of application which is subject to good quality control so that a known film thickness (within micro-inches) can be consistently placed on specimens. The procedure described in this technical note is known as the *in situ* method of applying MoS_2 lubricating film. It is accomplished by electrodeposition of an MoO_3 complex film onto the substrate material and converting this film to MoS_2 by subjecting the coated specimen to H_2S gas at elevated temperature and pressure. This procedure is an adaptation of a patented IBM process for lubricating mild steel components.

Results from this investigation have shown that film thicknesses from approximately 50 to 250 micro-inches can be successfully applied to within ±50 micro-inches to a number of materials used in spacecraft components. Materials that have been coated successfully are: stainless steels - 303, 304, 316, 416, and 440C; aluminum alloys - 2024, 6061, and 7075; mild steels; and M-10 tool steel. Test specimens were coated and then subjected to standard basic friction and wear tests in air and vacuum. The results show that (1) the *in situ* film has a coefficient of friction between 0.025 and 0.05, (2) film wear life is slightly better than it is for a dry MoS_2 powder film, much better than it is for inorganic bonded MoS_2 powder film, and not quite as good as it is for organic bonded MoS_2 film, and (3) quality control and reproducibility of the film are very good.

In addition, the *in situ* film has been successfully applied to instrument ball bearing races and retainers and to instrument gears. Test results of these components operated in a vacuum environment substantiate the results of the basic friction and wear tests.

CONTENTS

Summary i

INTRODUCTION 1

APPROACH 2

APPARATUS AND PROCEDURE 3

TESTS AND RESULTS 5

DISCUSSION 11

CONCLUSIONS 12

ACKNOWLEDGMENTS 12

References 13

Bibliography 13

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INTRODUCTION

Exposure to space has proven to be a particularly harsh condition for mechanical elements that must operate with a very high degree of reliability. Where relative motion must take place and the replenishment of protective oxide layers happens very slowly or not at all, a vacuum, in particular, poses severe operating problems from the standpoint of friction and wear.

Normal liquid and semi-solid grease lubricants have vapor pressures higher than the ambient pressures in outer space (estimated at 10^{-13} torr interplanetary and 10^{-16} torr interstellar); hence these lubricants evaporate rapidly. In addition to the low pressure problem, space lubricants must not be adversely affected by 10^9 rads of ionization radiation.

A literature survey of the space lubricant field shows that, basically, five approaches have been taken to reduce the problem of friction and wear in space. These involve the use of:

1. Special low-vapor-pressure oils and greases.
2. Metallic films.
3. Ceramics or other extremely hard surfaces.
4. Plastics with low vapor pressures and low coefficients of friction.
5. Laminar solids.

The desirable space lubricant must, in addition to satisfying the vacuum and radiation requirements, meet the following criteria: no outgassing to contaminate optical devices, low coefficient of friction to conserve available power, and low wear rate for extended lifetime.

An analysis of the five basic approaches for these conditions listed above shows that none of them will meet all of the requirements because of (1) high vapor pressure, (2) high coefficient of friction, (3) short wear life, and (4) contamination. The approach that meets the majority of the requirements involves the use of laminar solids, in particular MoS_2. Therefore, this material was chosen as the space lubricant to be investigated.

A literature search revealed that there are a number of methods (References 1 and 2) of bonding MoS_2 onto a substrate. The most widely used methods are inorganic - sodium silicate and graphite, organic - phenolic and epoxies, and MoS_2 powder - burnished or sprayed onto a surface. Each of these has its strong and weak points. In wear life comparison under identical conditions these bonded MoS_2 lubricants rate as follows: an epoxy bond is better than a burnished powder bond which is better than a sodium silicate bond. In vacuum stability, the epoxies outgas more than the other binders. The control of the film thickness in the organic or inorganic bonded methods is ±100 micro-inches, at best. In some of these films, 300 micro-inches is required to cover the surface completely. It is very difficult to measure the film thickness of burnished MoS_2 powder and this was not attempted.

With the undesirable points as prime considerations - short wear life, little control of film thickness, and vacuum instability—an *in situ* MoS_2 technique was considered and tested as the possible solution to elimination of these undesirable features.

The literature states that natural MoS_2 powders adhere tenaciously to a metal because of the attraction of the sulphur atoms to the substrate. This attraction is not sufficient to produce a long lasting film under test conditions. As one of the goals in this investigation is to assure a long wear life, it was felt that this natural bond could be strengthened by the ionic forces produced in electrodeposition and by the chemically clean surface produced by surface activation treatment. Also, as electrodeposition is a definitely controllable process, it was felt that good control of film thickness could be attained. Furthermore, since no binder material is used, the problem of outgassing and contamination would be alleviated.

The particular process investigated is an adaptation of the IBM-patented MoS_2 *in situ* process (References 3 and 4). The process consists of surface activation, electrodeposition of a MoO_3 complex ion onto the substrate surface, and converting this film to MoS_2 in an atmosphere of H_2S gas at 400 psig and 195 °C with an exposure period of 4 to 8 hr.

APPROACH

The approach taken was to examine the processing conditions shown in the IBM patent and, by experimentation, modify these conditions to produce a MoS_2 *in situ* film suitable for spacecraft components.

As MoS_2 has a natural tendency to adhere to a metallic substrate through the attraction of the sulphur atom to the metal, the first step was to obtain a chemically clean surface to assist in assuring or improving this natural tendency. The second step was to investigate the composition and temperature of the bath and electrodeposition time. The third step was to assure the conversion of the electrodeposited film to MoS_2, which was accomplished by investigating the conversion time, temperature, and pressure. This approach required the investigation of surface preparation, composition and temperature of the electrodeposition bath, time and current employed in electrodeposition, and time and pressure required for conversion.

To determine the merits of the various processing modifications, a fourth step or the test step was established which made maximum use of "calibrated" test apparatus. Experts in the lubricant testing field were enlisted to work on this step. Included were tests on the Alpha LFW-1 equipment*, and vacuum tests on the friction and wear test equipment at the NASA Lewis Research Center, Cleveland, Ohio (Reference 5). In addition, actual space components such as bearings, gears, and sliding devices were processed and tested under simulated space conditions at the Goddard Space Flight Center.

As the H_2S atmosphere is corrosive to most materials used in spacecraft construction, the fifth step included a study of the microstructure of materials subjected to this atmosphere.

In summary, the approach included the investigation of the following:

1. Surface preparation.
2. Electrodeposition.
3. Conversion.
4. Testing.
5. Substrate deterioration.

APPPARATUS AND PROCEDURE

The apparatus used in the experimental work is shown in Figures 1-3. The cleaning, surface activation, and electrodeposition apparatus consist of an ultrasonic cleaner, acid baths (glassware),

*Personal communication with Mr. A. DiSapio, Alpha Molykote Corp., Stamford, Connecticut.

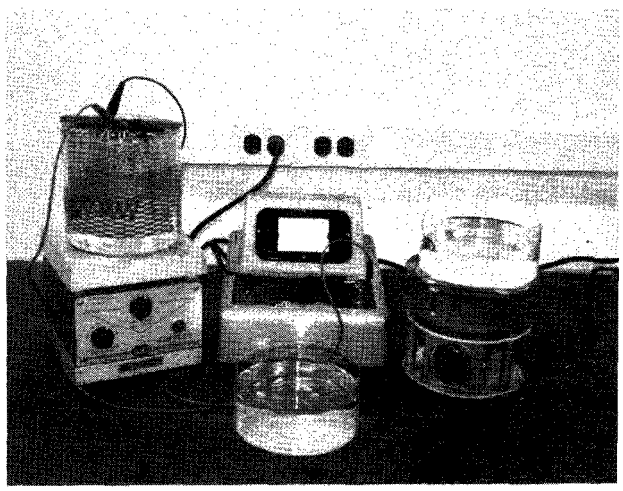


Figure 1—The surface activation and electrodeposition apparatus.

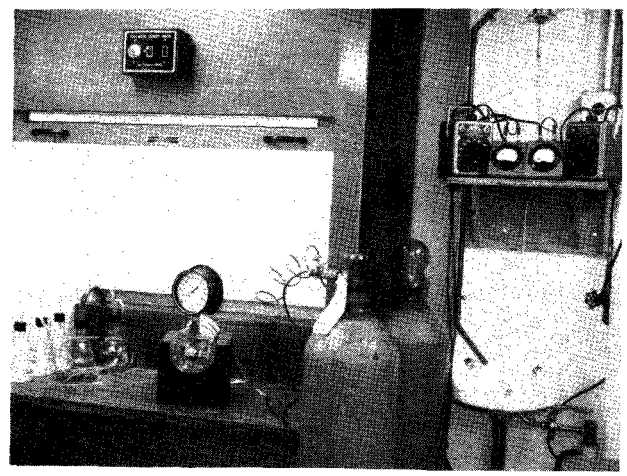


Figure 2—The conversion apparatus - pressure vessel, KOH bottles, H_2S gas cylinder, and heater controls.

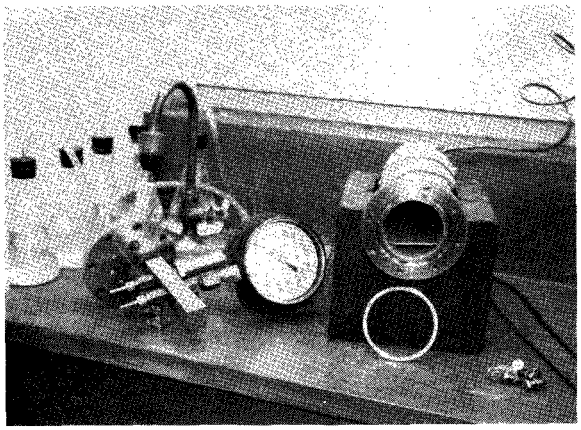


Figure 3—The conversion vessel, 1100 aluminum alloy seal ring, pressure gauge, and pressure relief valve.

a magnetic stirrer and hot plate, an electrodeposition bath with a platinum-plated titanium wire mesh anode, rinse tanks (glassware), and a power supply.

The conversion apparatus consists of a conversion vessel fabricated from 304SS pipe with the back closure welded and the front closure bolted in position. A 304SS manifold is threaded into the front closure which contains (1) a pressure gauge reading 0-600 psi, (2) a pressure relief valve set at 450 psig, (3) two outlet valves (one for H_2S and one for vacuum and purging). As H_2S gas is poisonous, the spent gas is bled through a series of five bottles containing 20v/o KOH; also the conversion vessel and the KOH bottles are operated under a chemical exhaust hood. The vessel is wrapped with heating tapes controlled by variacs. The front closure is sealed with an 1100 aluminum alloy ring. This closure also contains two thermocouple lead-throughs for determining temperatures at two places in the vessel.

Before experimentation began, a discussion was held with the IBM Corporation on the background of the process and their present production line. From this meeting, processing conditions in the patent were narrowed down to more specific values, i. e., a current density of 12 ma/in.^{2}, a conversion time of 8 hr, and a range of deposition times. A limited number of experimental specimens (1/4" × 1" × 1-1/2" mild steel) were examined metallographically, and the lifetime vs. current density data were evaluated. The results showed that the 12 ma/in.^{2} current density is suitable for their process and a film thickness of approximately 45 micro-inches is produced in the operation. IBM also processed for evaluation a number of 440C and M-10 tool steel specimens (1/4" × 1" × 1-1/2") through their production line with varying time in the electrodeposition step and with their standard 8 hour conversion time. A metallographic examination of these specimens showed that for film thicknesses of 45, 90, and 170 micro-inches, the electrodeposition times were 6, 12, and 18 min., respectively. The film appeared converted and adherent for all specimens (Figure 4).

The developmental work at the Goddard Space Flight Center for adaptation of this process to space components started with the design and set-up of the laboratory *in situ* processing apparatus. After the minimum amount of apparatus was set up, work began on surface preparation and electrodeposition for a study of their effects of film thickness and adherence. Various modifications were made to these two steps in the process before a suitable film was produced. Table 1 is a summary of the solutions used and results obtained.

After the conversion vessel and its ancillary apparatus were set up and tested for leaks, specimens with electrodeposited films were processed under various temperatures, pressures, and

times. The results are also shown in Table 1. After the developmental work was completed, a number of specimens were processed for the various tests and substrate reaction examination.

A summary of the major differences between the IBM-patented process and the adapted process are shown in Table 2.

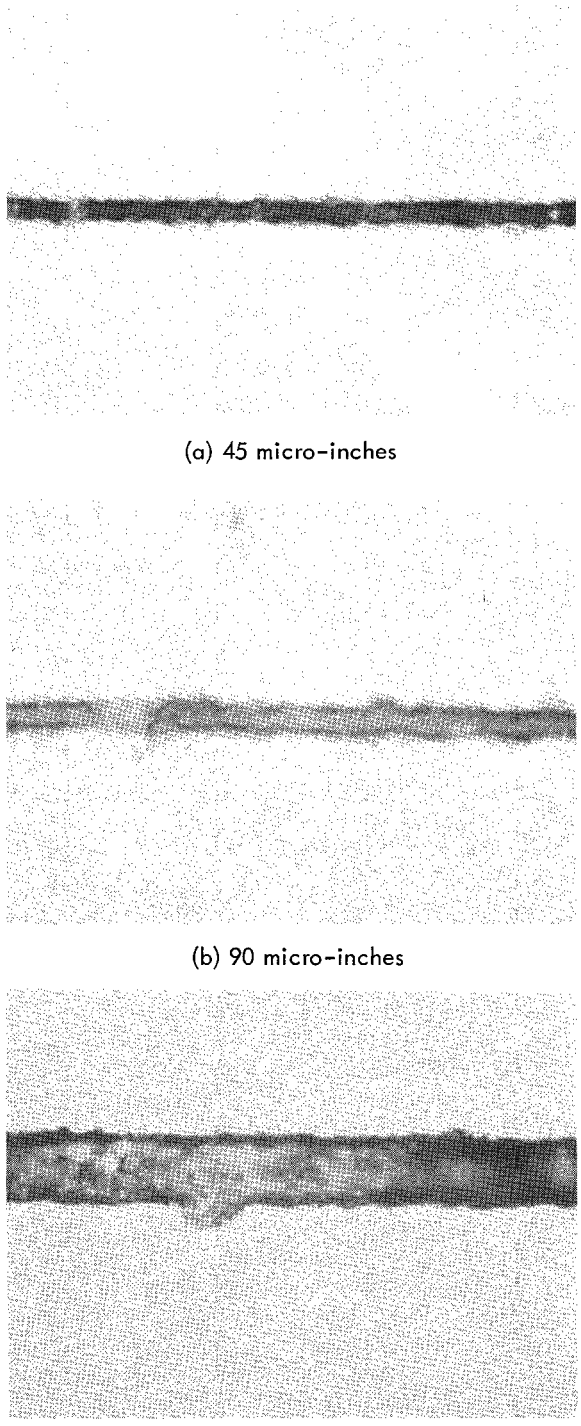
TESTS AND RESULTS

To evaluate the merits of the *in situ* method of depositing MoS_2 onto a space component, a laboratory test program was established. This program covered the following areas:

1. Determination of the coefficient of friction.
2. Wear life in air and vacuum.
3. Life test of instrument gears coated *in situ*.
4. Life test of R-2 size instrument bearings coated *in situ*.
5. Effect of H_2S on substrate materials.
6. Film thickness vs. time in plating bath—film adherence.
7. X-ray diffraction analysis of *in situ* film.

Where possible, tests were conducted on calibrated equipment under the control of experts in the various fields of lubrication. In this manner the results obtained could be correlated directly to tests conducted on other materials having MoS_2 as the lubricant. Also, when possible, actual space components were used as the test specimens; for example, instrument bearings.

The facilities of the Naval Research Laboratory were utilized to determine the



(a) 45 micro-inches

(b) 90 micro-inches

(c) 170 micro-inches

Figure 4—Photomicrographs showing the IBM in situ film with thicknesses of 45, 90, and 170 micro-inches. Because of pullout of the MoS_2 layer during specimen preparation, the focus is on the MoS_2 and the plane of the steel is out of focus. Mag. 1000X, as polished. All photomicrographs have been reduced for printing.

Table 1
Summary of Trials.

Trial	Procedure	Deposition Bath	Material	Remarks	
				Deposited Film	Conversion
I	Ultrasonically cleaned in trichlorethylene for 5 min., air dried	12.6 gm MoO_2 + 54.6 gm NH_4 COOH + 1000 ml distilled H_2O at 60°C, bath agitated	6061A1	Film nonuniform and flaky	Not converted
II	Hot detergent wash + I, polished through 400 grit paper	Same as above	6061A1	Film nonuniform and flaky	Not converted
III	II + I + 10V/o NaOH at boiling temperature for 5 min.	Same as above	6061A1	Film nonuniform and flaky	Not converted
IV	I + 2 min. immersion in 2V/o HF + 20V/o HCl	Same as above	440C SS	Film nonuniform and flaky	Not converted
V	I + IV + a rinse of 0.1V/o HCl + 1.0V/o H_2SO_4	Same as above	M-10 tool steel	Film nonuniform and flaky	Not converted
VI	I + 2 min. immersion in 2V/o HF + 20V/o HCl	12.6 gm (85V/o) MoO_3 + 54.6 gm NH_4 COOH + 1000 ml distilled H_2O at 60°C, bath agitated	M-10 tool steel	Film black, uniform, and adherent	Converted in 6 hr at 397 psig, 140°C, film dull gray.
VII	I + hot detergent wash + 20V/o H_2SO_4 at 82°C (anodic) + a rinse in 0.1V/o HCl and 1.0V/o H_2SO_4	Same as above	M-10 tool steel	Film black, uniform, and adherent	Not converted
VIII	Same as above	12.6 gm (85V/o) MoO_3 + 54.6 gm NH_4 COOH + 1000 ml distilled H_2O, bath at room temperature	Four 416 SS gears	Film gray, grainy, and uniform	The two thin gears were shiny and uniform. The two thick gears were dull. It was converted in 6 hr at 400 psig
IX	Same as above	Same as above	Three R-2 440C bearing components	Film non-adherent in spots	Film dull and adherent, converted in 6 hr at 400 psig
X	I + hot detergent + wash + a rinse in 0.1V/o HCl and 1.0V/o H_2SO_4	Same as above	440C SS	Film adherent and black	Film shiny and adherent, converted in 10 hr at 400 psig
XI	I + hot detergent wash + 20V/o H_2SO_4 at 82°C (anodic) + a rinse in 0.1V/o H_2SO_4 and 50V/o HCl (anodic)	Same as above	M-10	Film glossy and black	Film dull, satin, and adherent, converted in 18 hr at 400 psig
XII	Same above	Same as above	416 SS gear	Film flaky, dull and black	Excessive peeling of film, converted in 12 hr at 400 psig
XIII	Hot detergent wash + trichlorethylene (both ultrasonically for 3 min.) + immersion in hot 20V/o H_2SO_4 for 1/2 min.	Same as above	416 SS gear	Film uniform and adherent	Film not completely uniform, flaky in spots, converted in 8 hr at 400 psig
XIV	Same as above	Same as above	440C SS (test specimen for Alpha LFW-1 test.)	Film black and adherent	Film uniform, black and adherent, converted in 4 hr at 400 psig

coefficient of friction. The equipment used was a standard Bowden-Leben friction and wear machine. The test conditions were a 1/2 in. diameter hardened steel ball, a 2 cm/sec stroke, and an 800 gm load on the ball. The three test specimens were M-10 steel, one with no coating other than atmospheric contaminants and a 32 rms surface finish, one with 150 micro-inches of MoO_3 complex film, and one with 150 micro-inches *in situ* MoS_2 film. The results are: for the coefficient of friction for bare metal, 0.32 average, for MoO_3 complex, 0.12 average, and for MoS_2 *in situ*, 0.05 average. The latter coefficient of friction is approximately the same as that for natural MoS_2 powders. The same coefficient of friction was obtained in the wear life tests.

Table 2
Comparison of the Processes.

Condition	IBM Patent	Adapted
Surface cleaning	Not specified	Chemical reactants
Plating time	Not specified	specific time—3 to 10 min.—determined by thickness desired
Conversion time	30 min. to 110 hr.	4 to 8 hr.
Conversion temperature	60° to 125°C	195°C
Conversion pressure	Atmosphere to 400 psi	395 psig

Two different tests were performed to determine the wear life of the *in situ* film, one performed in air on an Alpha LFW-1 machine and one performed in vacuum using a disc and rider set-up. The first test specimens were Timken rings coated with 200 micro-inches of *in situ* MoS_2 and the friction and wear test was performed per military specification MIL-L-25504A Lubricant, Solid Film. This test includes controlled humidity (50 percent), 72 rpm, test block at R_c60, and a step-wise loading of the block from 30 to 630 lb. The average Hertz stress was of the order of 80,000 psi. The wear life data shows an average cycles-to-failure for the MoS_2 *in situ* film of 120,000, compared with 100,000 for burnished MoS_2 powder film, 60,000 for sodium silicate bonded film, and 300,000 for epoxy-bonded MoS_2 film.

The second wear life test was conducted by Lewis Research Center on their vacuum friction and wear test equipment. The conditions of the test are 10^{-9} torr pressure, a 396 ft/min. disc speed, and a rider load of 200 to 1000 gm applied in increments. The rider is hemispherical in shape with a 3/16 in. radius. Both disc and rider are of 440C SS. The disc had an *in situ* film thickness of 200 micro-inches. The results* of this test showed the wear life of the film to agree with the results found on the Alpha LFW-1 machine in air.

To determine how this film would function in actual application, space components (instrument gears and bearings) were coated with the *in situ* film and subjected to life tests. The instrument gears were Pic precision class 3, of 48 diametrical pitch and 416SS, heat treated to R_c30. Four gears were coated with 250 micro-inches and loaded with 20 oz.-in. of torque in a four square gear test fixture (Figures 5 and 6). The gears operated for 3.4×10^6 cycles with a load of 8.7 lb./in. of tooth width before failure. Tests are now underway to compare this film with sodium silicate bonded burnished MoS_2 powder and epoxy bonded films.

*Personal communication with Mr. D. H. Buckley, Lewis Research Center, NASA, Cleveland, Ohio.

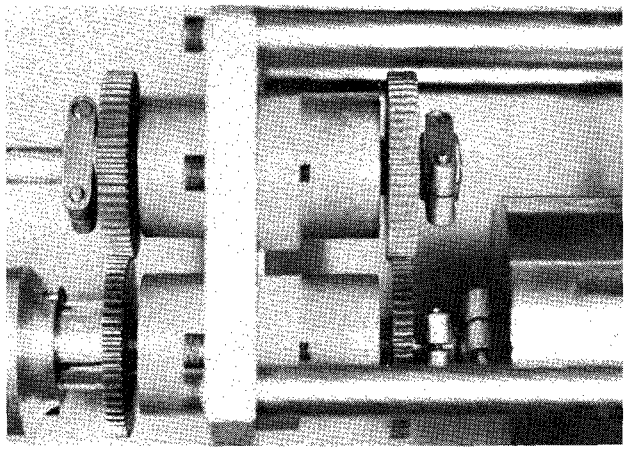


Figure 5—The in situ coated gears assembled in the four square gear tester, before the test.

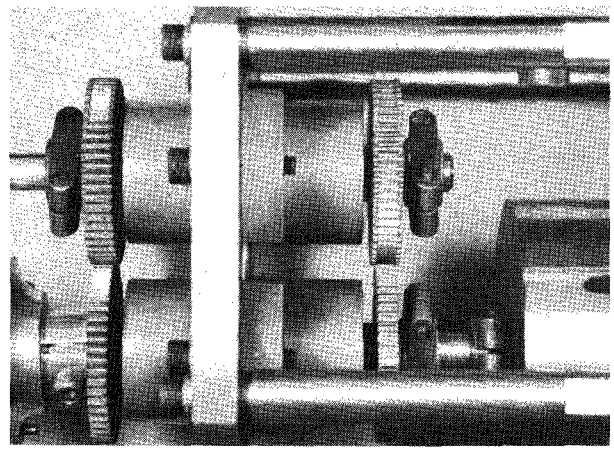


Figure 6—The in situ coated gears after the test. Note the wear path of the narrow gear in the larger gear.

The second space component to be coated with *in situ* MoS_2 and tested were R-2 size instrument ball bearings. The outer and inner races and the machined retainers were processed with 150 micro-inches of *in situ* MoS_2. Two bearings were set up in a bearing tester with a 5 oz. radial load, at 600 rpm and in vacuum. They have operated over 3200 hr in a vacuum of 10^{-9} torr with no indication of failure. A comparison of this film with the other MoS_2 films will be made when the test fails. Figure 7 shows the coated bearings and Figure 8 shows typical coated specimens.

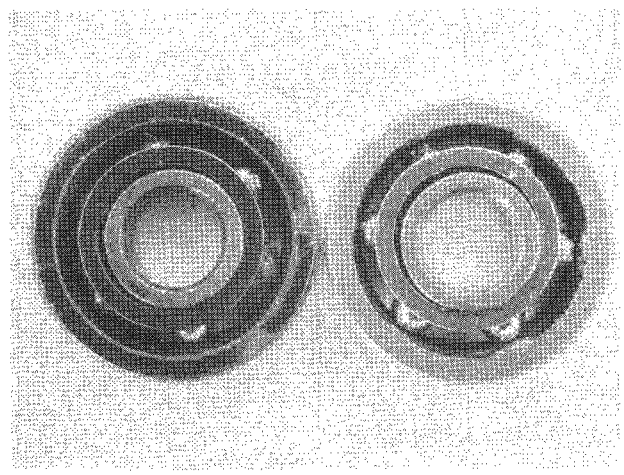


Figure 7—The R-2 size instrument bearing coated with the in situ film. The bearing at left has the coating. Note the difference in color. Mag. 4X; reduced for printing.

As H_2S gas is very reactive to most materials, and since the materials are subjected to this gas at 195°C and 400 psig for 4 to 8 hours during the conversion step, a program was set up and completed to determine the effect, if any, of the processing conditions on the substrate materials. A number of specimens - 2024, 6061, and 7075 aluminum alloys, 303, 316, 416, and 440C stainless steels, mild steel, and M-10 tool steel - were processed with an excessively thick film at 250° to 300°C for 8 hr., under H_2S gas to 400 psig. The high temperature was necessary to preserve pressure because of a leak in the gas system, and the longer conversion time was due to a desire to accelerate the possible

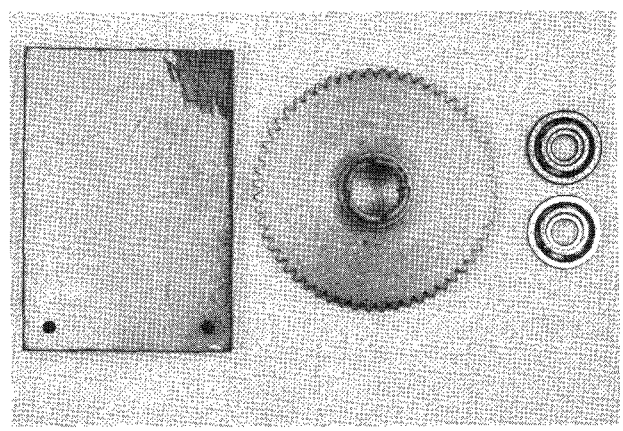


Figure 8—Typical specimens coated with the in situ film. Mag IX; reduced for printing.

H_2S attack. The specimens were prepared for metallographic examination. For examination at up to 1250 magnification, under bright light, polarized light, and sensitive tint, no indication of attack on the microstructure could be detected (Figures 9-12).

A study of the heat treating practices for the above materials shows that at 195 °C the temper of the steels will not be affected and that slight over-aging of the aluminum alloys will take place. As this temperature is not excessively over the normal aging temperature, the physical properties will not be adversely affected.

Adherence of the film was a very important factor in deciding the preconversion conditions, such as surface activation and electrodeposition time. The adapted procedure produced a good

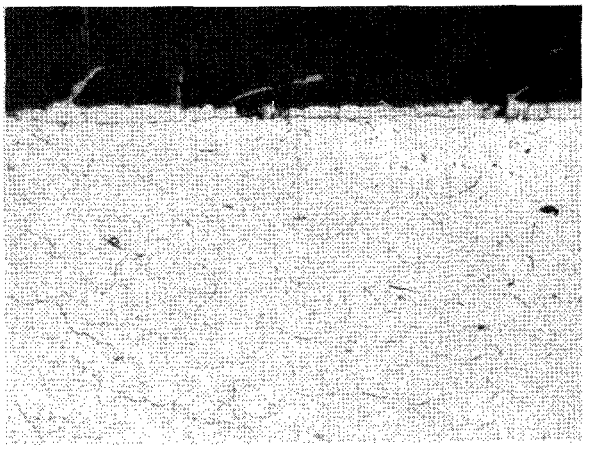


Figure 9—Photomicrograph showing the absence of any reaction of the in situ processing to 1020 mild steel. Mag. 360X, 4 percent Nital etch.

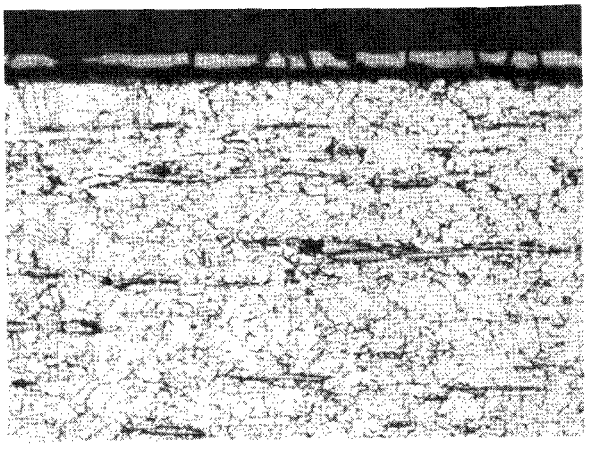


Figure 10—Photomicrograph showing the absence of any reaction of the in situ processing to 416SS. Mag. 360X, hydrofloric picral etch.

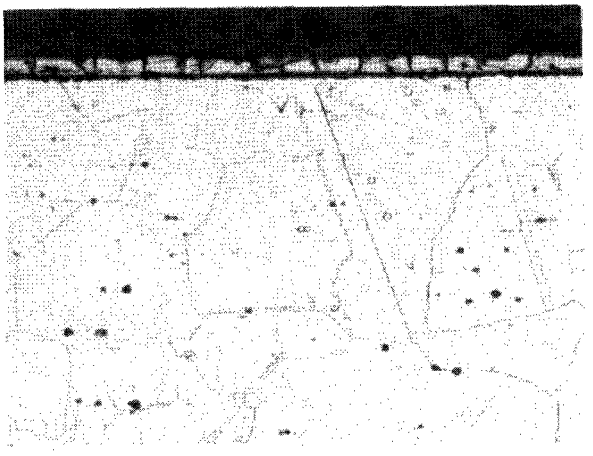


Figure 11—Photomicrograph showing the absence of any reaction of the in situ process to 316SS. Mag. 360X, Marbles etch.

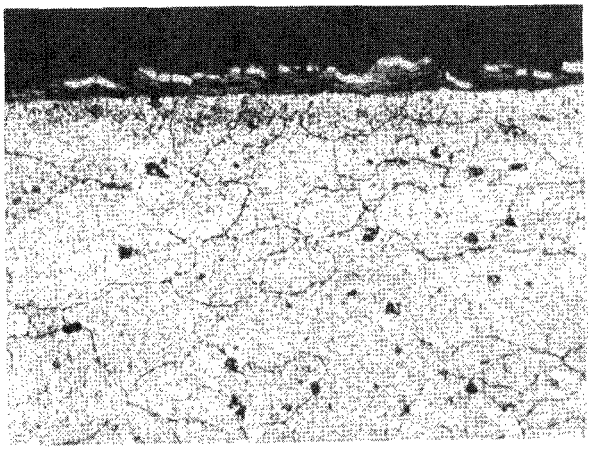


Figure 12—Photomicrograph showing the absence of any reaction of the in situ process to 2024 aluminum alloy. Mag. 360X, Kellers etch.

adherent film. The adherency of the film was determined by shaking or dropping the specimen onto a hard surface and placing a 1" × 1/2" piece of cellophane tape onto the specimen under medium thumb pressure and swiftly peeling off the tape. A good adherent film did not flake or appear powdery (Figure 13). Also, a good film did not peel off with the tape, although a trace of the MoS_2 did show up on the tape.

A number of specimens were processed to determine film thickness vs. time in the electrodeposition bath. The film was measured with a Magne Gage, a Dermitron, a Federal Precision Master Comparator, and a microscope. Correlation was good between the Magne Gage and the microscope. Figures 14 and 15 show thickness vs. deposition time. Figure 16 shows film thicknesses.

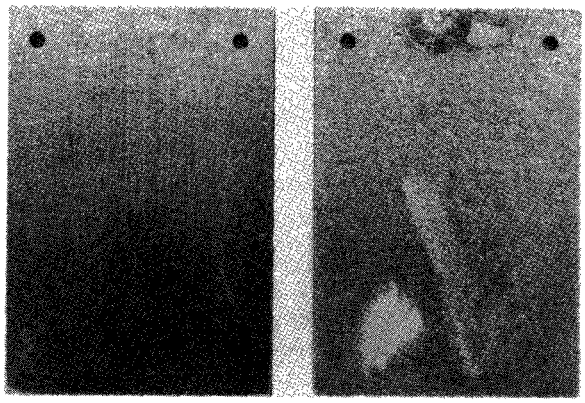


Figure 13—An adherent in situ film (150 micro-inches, left-hand specimen) and a nonadherent film (300 micro-inches, right-hand specimen).

During the phase wherein conversion time was being determined, samples were submitted for x-ray diffraction analysis to determine if all of the MoO_3 complex had been converted to MoS_2. Preliminary results showed an amorphorous pattern which indicated that the MoS_2 crystallites

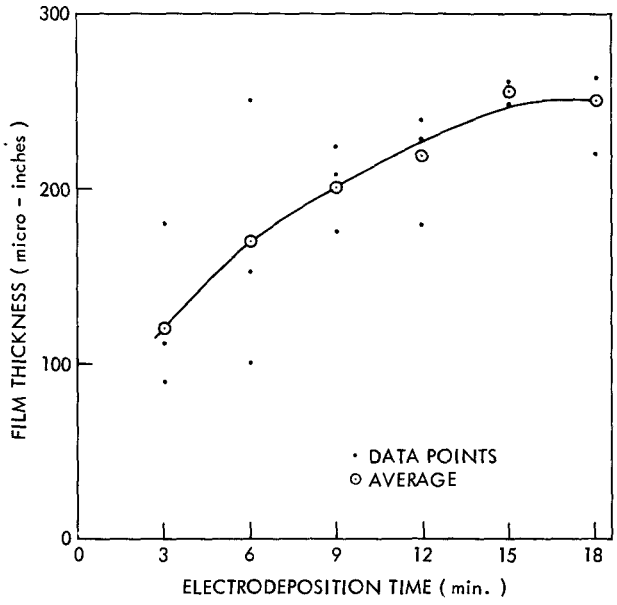


Figure 14—Electrodeposited in situ film thickness vs. electrodeposition time.

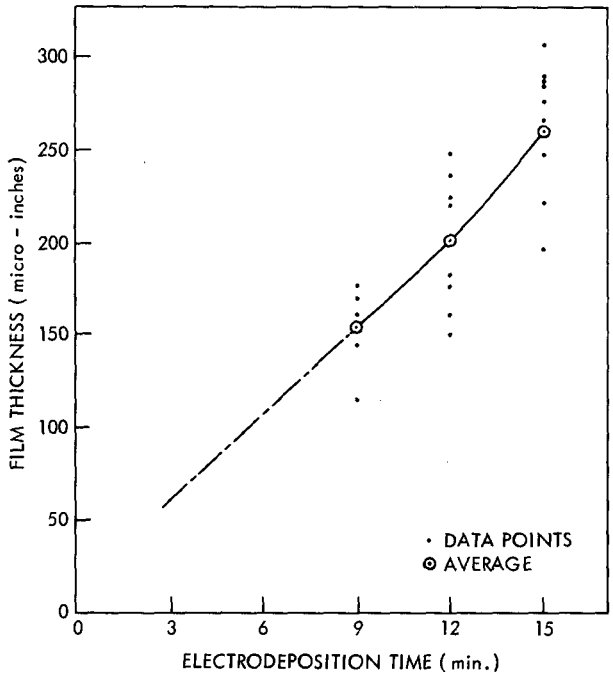


Figure 15—Converted in situ film thickness vs. electrodeposition time.

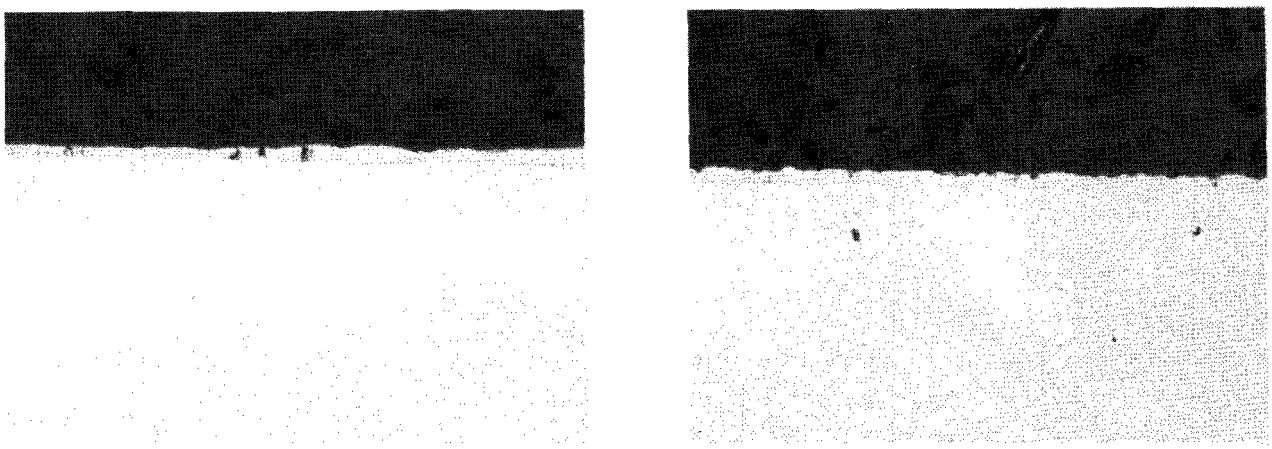


Figure 16—Photomicrographs showing steel specimens coated with in situ MoS_2 prepared for microscopic measurement. The thickness is 250 micro-inches. Mag. 300X, as polished.

produced in this process were too small to be determined with the x-ray diffraction equipment as it was set up. After contacting IBM and discussing this point with them, it was decided that additional analysis would not be performed since IBM had found the same amorphorous pattern in their study. Also, IBM had carried the analysis further and by methods of crystal growth had concluded that the MoO_3 had been converted to MoS_2. The tests that were made to determine the coefficient of friction and the metallographic examination of the specimen at high magnification also showed that the MoO_3 had been converted to MoS_2.

DISCUSSION

After discussions with IBM personnel and metallographic examination of various coated specimens, it was determined that 120 ma/in.^{2} current density was suitable for the developmental work.

During the metallographic examination of a number of specimens, it was noticed that the film was deposited into all laps, seams, cracks, and micro-fissures (Figure 17). This indicated that this process may lend itself to lubricating hard-to-reach places, such as bearing outer races.

As shown earlier, the wear life of this film is not quite as good as space conditions dictate. Therefore, additional work will be performed on the process in an attempt to improve wear life. Present work will be carried out to investigate incorporating the *in situ* process with a reservior technique. Also, further adjustment to the process, such as changing Mo valence, current density, etc., may produce a longer wear life.

The effects of the temperature of the deposition bath on the film adherence proved undetectable and the elevated temperature caused evaporation of the bath. Hence, the bath was used at room temperature.

Optimum conversion time was determined by using the coefficient of friction and the adherence test. Times of 18, 12, 10, 8, 6, and 4 hr were used and the results show that 6 hr is sufficient to convert a 250 micro-inch film.

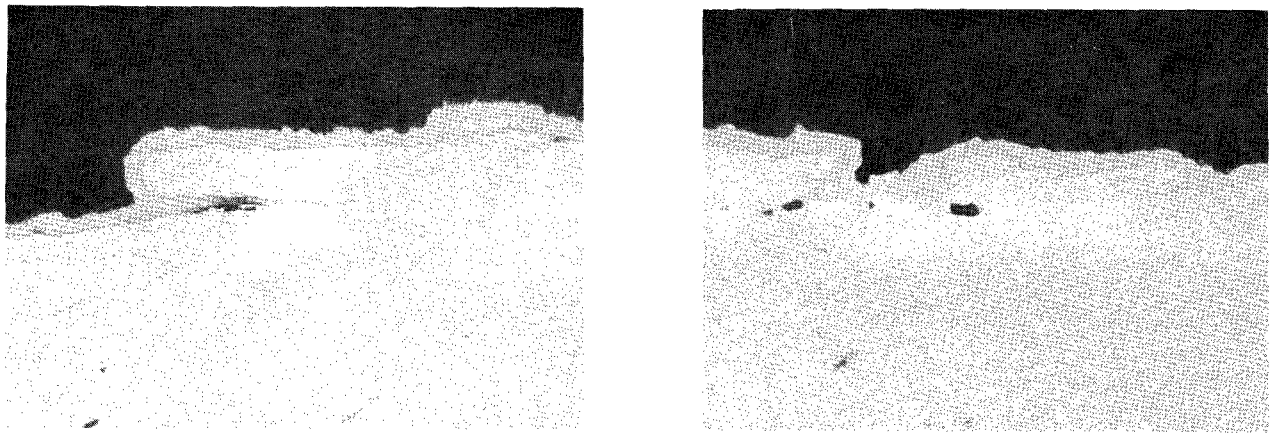


Figure 17—Photomicrographs showing the ability of the in situ film to fill cracks, seams, and laps. Mag. 360X, as polished.

CONCLUSIONS

An evaluation of the test data shows that the following conclusions can be drawn:

1. The *in situ* process for depositing MoS_2 onto space components as a suitable lubricant is feasible and practical.

2. The procedure for depositing MoS_2 *in situ* has been worked out, an adaptation of an IBM-patented process.

3. Adherence tests show that 250 micro-inches is the maximum film thickness advisable under the processing conditions outlined.

4. The film thickness can be controlled within ±50 micro-inches.

5. The average coefficient of friction of this film is 0.05 or less and is comparable with or the same as that of MoS_2 powder and lower than those of bonded MoS_2 films.

6. The film can be easily and safely deposited onto a number of common spacecraft materials, including 2024 Al, 6061 Al, 7075 Al, 303SS, 316SS, 416SS, 440C SS, mild steel, and M-10 tool steel.

7. The film has a better wear life than sodium silicate bonded MoS_2, slightly better wear life than a burnished MoS_2 powder, and a somewhat poorer wear life than epoxy bonded MoS_2.

8. The film follows the surface contour and fills up the smallest crack, lap, seam, or indentation and therefore makes it possible to place a controlled amount of MoS_2 on hard-to-reach surfaces, such as the outer races of ball bearings.

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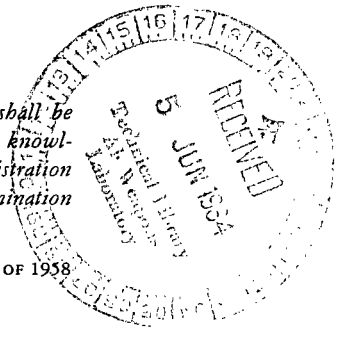

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