ADVANCES IN MOLYBDENUM DISULPHIDE FILM TECHNOLOGY FOR SPACE APPLICATIONS

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ABSTRACT

This paper reviews recent advances made by the European Space Tribology Laboratory in the area of thin-film MoS_2 lubrication for space. The focus is on two developments, these being: (a) the modification of sputtered molybdenum disulphide films to improve their life in moist air, thereby extending their ground test capability and (b) the setting-up of a new coating facility which produces dense, high-durability molybdenum disulphide films and which has the capability of uniformly coating large tribo-components and those of complex geometry.

In the first development, the moisture-resistance and oxidation-resistance of sputter deposited molybdenum disulphide films have been improved by modifying the films through the introduction of gold inclusions. Two types of film were produced which contained known quantities of gold. In the first type the gold was uniformly distributed throughout the film whilst the second type of film was a multi-layered coating comprising alternate layers of MoS_2 and gold. The best overall performance was shown by the first type of coating (uniform distribution of gold) which, when compared to the unmodified MoS_2 films, brought about a substantial improvement in film durability, whilst the friction coefficient was essentially unchanged. Thus, the mean durability in dry nitrogen gas was more than doubled, and the durability in air improved by a factor of three to four.

1. INTRODUCTION

Whilst sputter-deposited MoS_2 provides excellent lubrication in vacuum it has a number of drawbacks which sometimes mitigate against its use in space applications. Foremost in this respect is its inability to reproduce its in-vacuo lubricating properties in air, particularly moist air, where friction and wear rate are substantially higher than in vacuum. Often therefore, ground testing of MoS_2-lubricated mechanisms in air is precluded. In such cases the test engineer has to arrange for testing to take place in an evacuated chamber or in an inert gas environment. Where this is impracticable a replacement lubricant might be chosen, one having similar properties in air and vacuum, - even though MoS_2 might be the most appropriate lubricant to employ in the vacuum of space. To try and circumvent this problem ESTL has undertaken studies on films of MoS_2 modified so that they are better able to withstand operation in moist air. These modifications, which involve the introduction of small quantities of gold into the MoS_2 coating, have proved successful inasmuch as the in-air durability of the coatings has been appreciably extended.

Secondly we report on a further advance in our capabilities for the deposition of solid lubricant films. In this second development, films of molybdenum disulphide have been produced on a new coating rig known as an unbalanced magnetron sputtering system. In conventional magnetron sputtering the plasma is confined by magnetic fields to a region near the sputter target. In unbalanced magnetron sputtering the magnetron sources are designed to allow part of the plasma to extend beyond the target and interact with the substrate thus allowing ion bombardment of the growing film. Results are presented which show that molybdenum disulphide coatings produced by this method are superior in durability to those produced by conventional techniques. An additional advantage of this system is that its design allows much larger components (up to 330mm in diameter and 300mm in length) to be coated with sputtered MoS_2 than was hitherto possible.

2. MODIFICATIONS TO MoS_2 FILMS FOR LONGER LIFE IN AIR

2.1 Background

Initially a literature survey was undertaken of published works relating to the degradation of MoS_2 when operated in humid environments. It was concluded that the process of degradation is initiated by the adsorption of water molecules at the most chemically active sites on the MoS_2 lattice (these being the "edge" sites). This action increases the shear strength of the lubricant film and the attendant rise in friction promotes oxidation of the MoS_2 to MoO_3.

It became clear from this survey that there was no easily definable route by which to develop moisture-and oxidation-resistant MoS_2 films. However, it was also apparent that if such films were to be developed then this would most likely be achieved through some or all of the following:

- film densification to reduce porosity
- minimisation of active sites to reduce H_20 adsorption
- neutralisation of active surface sites to prevent oxidation
- incorporation of getter materials to inhibit oxidation

It emerged from this study that one approach to obtain, potentially, both film densification and site neutralisation was through the inclusion of gold - either by sequential deposition to produce gold layers or by co-deposition to give a uniform distribution of gold within the MoS_2 film. Attempts were made to produce both types of MoS_2/Au films as described below.

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2.2 MoS_2 film modification thorough gold inclusions

Multi-layered MoS_2/Au films

Multi-layer MoS_2/Au films (Fig.1) were produced by sequential sputtering from separate gold and MoS_2 targets. A low energy ion beam source was used to sputter-etch the surface of the test specimens prior to sputter-deposition, in order to remove any surface contaminants which would hinder coating adhesion. Total film thicknesses were in the order 1 μm, the thickness of the individual gold layers within the "sandwich" structure being varied between 5 to 50 nm. Examination of cross-sections of these coatings by SEM indicated that regular, periodic layered structures had been achieved and that the films were dense in comparison to conventional sputtered MoS_2.

Fig.1 SCHEMATIC DIAGRAM OF MoS_2 /Au MULTI-LAYER FILMS

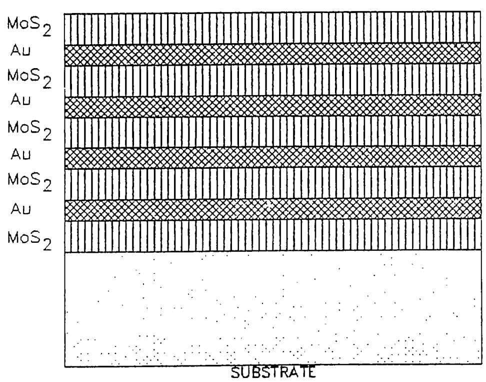
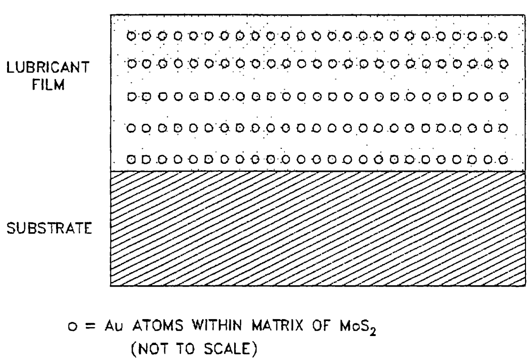


Fig.2 SCHEMATIC DIAGRAM OF MoS_2 /Au Co-SPUTTERED FILMS



Co-sputtered MoS_2 Au coatings

The co-sputtered MoS_2/Au films (Fig.2) were produced by magnetron-sputtering. The surface of the MoS_2 sputter-target was partially covered with thin strips of gold foil so as to effectively create a binary (Au/MoS_2) target. The magnetron arrangement results in enhanced sputtering of target material in an annular shaped area and the gold strips were arranged to lie within this annulus. The gold content of the sputter-deposited film was adjusted by varying the number of the gold strips. For comparison, some films containing only MoS_2 were also produced.

2.3 Test methods

Films were deposited onto Type En31 steel discs of external diameter 19 mm, supplied by INA Bearing Company Ltd. The discs selected had a surface finish (R_a value) not exceeding 0.12 μm, and were cleaned ultrasonically in propan-2-ol (iso-propanol) prior to sputter deposition. The coated discs were tested in dry nitrogen and in laboratory air (ESTL standard conditions, temperature 20-22°C, relative humidity 40-50%), using a CSEM tribometer (single pin/disc test machine), with an applied load of 20 N (mean Hertzian contact stress 1.04 GPa), and a rotation speed of 500 rpm. The film durability was defined as the number of disc revolutions performed until the friction coefficient (μ) exceeded 0.15, which in these tests corresponded to the onset of film failure.

The same coating procedures were used to apply selected films to the running surfaces of SNFA Type ED20 ball bearing inner & outer races. Two pairs of bearing races were coated in each deposition run. Two bearing tests were performed using the MoS_2/Au coated bearings, and, for comparison, two tests were performed with bearings coated only with MoS_2. The bearings each contained ten steel balls and a Duroid (PTFE/MoS_2/glass fibre) cage - these were not coated with MoS_2 or MoS_2/Au films. After coating, a pair of ED20 bearings was assembled in a back-to-back configuration, and, utilising spring washers, a preload of 40 Newtons was applied. The bearings were then loaded into the test chamber, which was evacuated until the pressure was 1×10^{-6} torr or less.

The bearings were rotated for an initial 100,000 revolutions in vacuum followed by alternate periods of rotation in air (Standard ESTL laboratory environment, 20±1 °C, Relative humidity 45±5 %.) and in vacuum, typically as follows:

1,000 revolutions in air,
99,000 revolutions in vacuum,
2,000 revolutions in air
98,000 revolutions in vacuum, etc.

The torque of the bearing pair was measured using a Teldix DG1.3 torque transducer, connected to a Teldix torque meter. The output from the meter was fed to a chart recorder to give a continuous record of the torque during the tests. The tests were stopped when it became clear from increases in torque that there was insufficient residual sputtered MoS_2 to lubricate the contacting surfaces of the ball bearings.

The MoS_2 film thickness on discs was measured by X-Ray fluorescence (XRF). All the film thickness results are "estimates", assuming that MoS_2 was the sole component of the film whilst making the measurements, since calibrated thickness standards for the various co-sputtered MoS_2/Au films were unavailable. The film thickness of MoS_2 was measured at accessible locations on the lands on either side of the raceway of the ED20 inner races, the actual raceway being inaccessible by our equipment.

2.4 Tribological properties of modified films

Multi-layered MoS_2/Au films

Although the multi-layered MoS_2/Au films were dense their tribological properties were on average no better than MoS_2 films containing no gold, i.e. their friction and wear properties in air were similar. Likewise, in dry nitrogen, the introduction of gold layers did not bring about any significant advantage over the standard MoS_2 film.

Co-sputtered MoS_2/Au films

In contrast, co-sputtered MoS_2/Au films did exhibit an improvement over the standard film. Tribometer test results from the co-sputtered films are summarised in Figure 3. This shows the film durability in air as a function of the gold concentration in the coating and indicates a substantial enhancement in the in-air durability at an optimum level of gold. Figure 4 shows typical friction plots for tribometer tests in air of MoS_2 films with and without gold.

The optimum level of gold that maximises the MoS_2 film durability in air corresponds to between 12 and 16 atoms % . In air, the durability of films containing the

Fig. 3 SUMMARY OF MoS_2/Au FILM DURABILITY RESULTS IN AIR TRIBOMETER TESTS, LOAD 20 N, 500 RPM

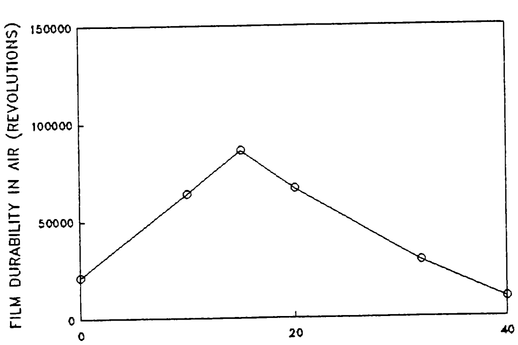
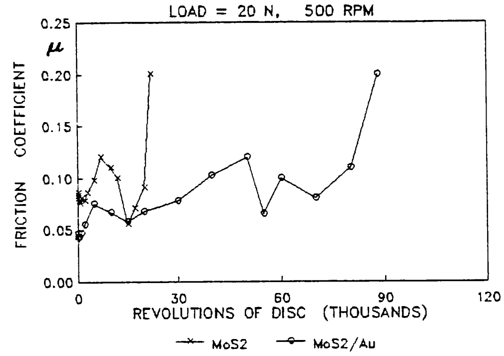


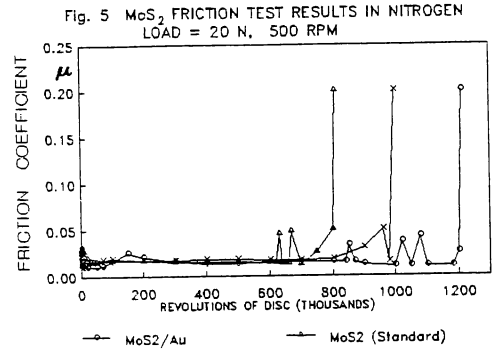
Fig. 4 MoS_2 FRICTION TEST RESULTS IN AIR LOAD = 20 N, 500 RPM



optimum gold content was typically 3 to 4 times greater than that of films which contained only MoS_2.

Although not the main objective of our tests, it was noted that the presence of gold also increased the durability of MoS_2 films in nitrogen, even in those cases when there was no improvement in air. Almost all the films produced using between 9 and 30 atoms % Au had durabilities exceeding 1 million disc revolutions in nitrogen in ESTL's standard tribometer test, typically 1.5 to 2 times the durability of "pure" MoS_2 films produced on the same coating facility. However, films containing 30 - 40atoms % Au were no better than "pure" MoS_2 films. Figure 5 shows typical friction plots from tribometer tests in nitrogen of MoS_2 films with and without gold.

Fig. 5 MoS_2 FRICTION TEST RESULTS IN NITROGEN LOAD = 20 N, 500 RPM



"STANDARD" = CONVENTIONAL SPUTTER-DEPOSITION FACILITY
"PLASMAG" = UNBALANCED MAGNETRON FACILITY

Two ball bearing tests were performed with the bearing races lubricated with MoS_2/Au containing the optimum level of gold, and two tests with the races lubricated only with MoS_2. The mean film thicknesses of MoS_2 on the inner race lands, close to the race track, were all in the range 0.7 - 0.8 µm. The mean torque was monitored continuously during the ball bearing tests. The results from the first test are summarised in Figures 6-7, which show the torque behaviour in vacuum and in air during the initial stages of that test, and the overall trend of the torques in vacuum & air throughout the test. In the first vacuum stage of this test, after an initial running-in period, the torque fell to about 5 g.-cm. In air, the torque rose to 28 g-cm. This behaviour was repeated throughout the alternate test stages in vacuum and air, although the torque levels gradually fell to 2-3 g.-cm in vacuum and about 10 - 15 g.-cm. in air. Film failure occurred after 171,000 revolutions of the ball bearings in air (total revolutions 3,540,000 in vacuum & air)

Fig. 6 BALL BEARING TEST TORQUE RESULTS

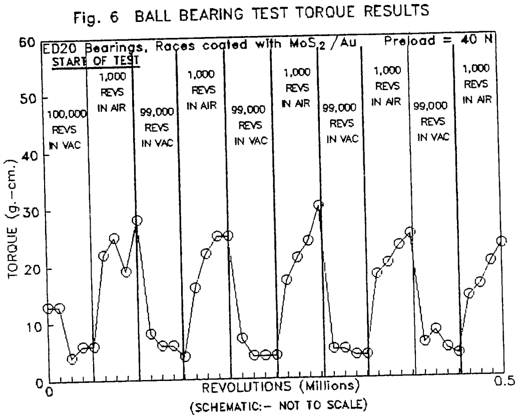
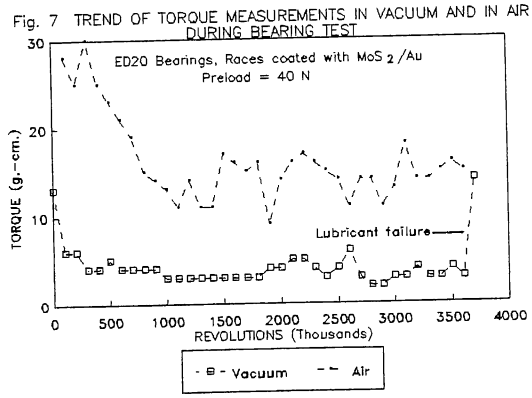


Fig. 7 TREND OF TORQUE MEASUREMENTS IN VACUUM AND IN AIR DURING BEARING TEST



Qualitatively, ball bearing tests nos. 2-4 showed similar torque behaviour to test no.1, the film durabilities being:

Test 2 (MoS_2/Au)	276,000 revs. in air (Total 2.4 million revs)
Test 3 (MoS_2)	105,000 revs. in air (Total 1.6 million revs)
Test 4 (MoS_2)	120,000 revs. in air (Total 1.5 million revs)

The most significant factor was that tests 1 and 2 (with MoS_2/Au films) lasted much longer than tests 3 and 4 (with MoS_2 alone). It is very encouraging that both the MoS_2/Au lubricant films had much greater durabilities than either of the films with MoS_2 alone. The greatest damage to MoS_2-based lubricant films occurs in air, due to their susceptibility to the effects of water vapour and oxidation. Considering only the number of revolutions in air, the mean durability for tests 1 & 2 (MoS_2/Au) was about 220,000 revolutions, compared with a mean of about 113,000 revolutions for tests 3 & 4 (MoS2 alone). Thus, these tests have clearly demonstrated the improvement in performance that is obtained by using co-sputtered MoS_2/Au films rather than "pure" MoS2 films.

3. NEW UNBALANCED MAGNETRON SPUTTER-DEPOSITION FACILITY.

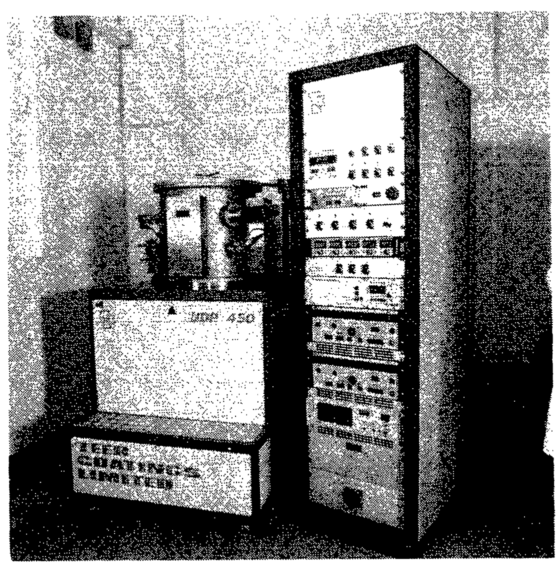


Fig. 8 The unbalanced magnetron sputter-deposition facility

3.1 Description of facility

In conventional magnetron sputtering, such as used for the work described above, the plasma is confined by magnetic fields to a region near a single sputter-target. In unbalanced magnetron sputtering, part of the plasma interacts with the substrate allowing ion bombardment of the coating as it grows. In ESTL's new system this is achieved by having two vertical MoS_2 targets, the plasma being confined to the area between these two targets. Components to be coated are located centrally. Since this area is relatively spacious, several small components or a small number of relatively large components may be coated in a single deposition run. Fig. 8 shows the new system.

3.2 MoS_2 films produced on new facility

The first objective with this facility has been to establish the deposition conditions leading to MoS_2 films with the optimum tribological performance. This was achieved by varying the parameters used during sputter-deposition, namely:

- the sputter-gas (Ar) pressure

- the current supplied to the magnetron sources

- the bias voltage applied to the specimen holders during sputter-deposition.

- the location of specimens relative to the sputter-targets

- the background pressure in the sputter-deposition chamber (prior to commencement of deposition)

For the purposes of optimisation, MoS_2 films, approximately 1µm thick, were applied to steel discs, and tested on a pin-on-disc tribometer, as described above. With the optimum parameters, the deposition rate was typically 40 - 50 minutes per 1µm of MoS_2 film. Figure 5 shows a friction plot from tribometer tests of a typical optimised MoS_2 film produced on the unbalanced magnetron facility, together with the plots described in Section 3.1. The film durability is at least as good as that obtained in ESTL's conventional magnetron sputter-deposition facilities. On completion of further optimisation tests with discs, it is planned to test ball bearings coated with MoS_2 in the unbalanced magnetron facility.

In order to apply uniform coatings to components with complex geometric shapes, the components are affixed to a carousel during sputter-deposition. The rotating motion of the carousel ensures that, during sputter-deposition, all the accessible surfaces of the components are coated uniformly with MoS_2 lubricant film. The ability to coat large components uniformly is illustrated in Figure 9, which shows the outer race of a roller bearing (O.D. 165 mm), after coating with MoS_2 in the new facility. If required, up to 8 races of the same size could be coated in a single run.

Fig. 9 Bearing outer race (O.D. 165 mm), coated with MoS2 in unbalanced magnetron sputter-deposition facility.

4. CONCLUSIONS.

Co-sputtered films containing both MoS_2 and gold were compared with films containing only MoS_2 in tribological tests in air and in inert environments. In simple tribometer tests with sliding motion and in tests with Type ED20 ball bearings, films containing 12 - 16 % gold yield much higher durabilities in air (x2 on bearings, x3 to x4 in tribometer tests) than films containing only MoS_2. In sliding motion, the presence of gold at the optimum level also doubled the typical durability of MoS_2 films in inert environments (nitrogen).

ESTL's new unbalanced magnetron sputter-deposition coating facility has been commissioned and tests show that, in sliding motion, the films are at least as effective as lubricants as those produced on the pre-existing facilities at ESTL. The main advantage of the new facility, however, is its ability to coat large numbers of small-to-medium sized components (or alternatively a small number of relatively large components) in a single coating run.