AN ASSESSMENT OF SOLID LUBRICANT FILMS FOR USE IN HIGH TEMPERATURE SPACE APPLICATIONS

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ABSTRACT

A number of solid lubricant coatings (MoS_2, Ag, WS_2 and PS212) were applied to Inconel 600 substrates and evaluated under reciprocating sliding motion against unlubricated Inconel 600 pins. Tests were carried out in a nitrogen atmosphere at room temperature, 400 Deg C and 800 Deg C. It was generally found that materials which provided effective room temperature lubrication were far less effective at high temperatures. The exception was a PS212 film (a composite of CaF_2, BaF_2, Ag and Cr_3C_2 in a nickel cobalt matrix) which gave rise to lower friction as the temperature was increased above room temperature.

Keywords: High temperature lubrication, tribology, re-usable spacecraft.

1 INTRODUCTION

High temperature lubricants are required for applications in re-usable spaceplanes, such as elevon hinges on HERMES, where temperatures of 800 Deg C will be reached during re-entry into the Earth's atmosphere. These lubricants should also be capable of providing effective lubrication at lower temperatures as ground testing and "cold start" operation may be necessary. Both conventional thin film lubricants (ie MoS_2, Ag and WS_2) and one NASA composite specifically developed for high temperature lubrication (Refs.1 and 2)) were identified as candidate lubricants and tested in a nitrogen environment. The test environment was chosen to establish baseline data for comparisons with a future test programme which will involve carrying out similar tests in an environment with an oxygen content representative of that of the upper atmosphere.

2 TEST SPECIMENS

The lubricant films tested were molybdenum disulphide and silver (both deposited by magnetron sputtering at ESTL), tungsten disulphide (produced by the Dicronite process by the so-called "impingement process") and PS212 (a CaF_2-BaF_2, Ag and chromium carbide mixture in a nickel-cobalt matrix deposited by plasma spraying). The MoS_2, Ag and WS_2 films had thicknesses in the order of one micron. The thickness of the PS212 coating was measured, following sectioning, using a travelling microscope fitted with a displacement scale. The thickness was of the order of 300 microns.

The substrate and pins were machined from Inconel 600 which is a nickel-based superalloy (76% Ni, 16% Cr and 7% Fe). Superalloys are often chosen for high temperature applications because they maintain their hardness and tensile strength at high temperatures and are also oxidation resistant. The Inconel substrates were polished using Grade 600 silicon carbide paper to achieve a surface finish of 0.05 microns (CLA). The Inconel pins were supplied already polished and were cleaned by ultrasonic cleaning in Arklone-P. Substrates were cleaned in the same manner before coating and also prior to testing in their unlubricated state.

3 TEST APPARATUS AND CONDITIONS

The tests were performed on a reciprocating tribometer which was capable of producing temperatures up to 800 Deg C. Temperatures were measured on thermocouples clamped to the sample surface. A schematic diagram of the test apparatus is shown in Figure 1. Flat test specimens were mounted on a specimen stage and pin samples were clamped in a holder affixed to the reciprocating arm. The test stage was mounted on flexure pivots so that the reaction force on the flat specimen, due to friction, could be measured on a force cell in contact with the specimen stage. The normal load was applied to the test specimen by a lever system located outside the test region.

A stainless steel lid (inner containment) was fixed over the sample mounting stage and the resulting enclosure was purged by nitrogen gas. A positive pressure within the sample enclosure enabled gas to exit via a polarimetric oxygen meter enabling

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continuous monitoring of the oxygen content to take place. The oxygen content was found to be less than 15 ppm and is equivalent to a partial pressure of $15x10^{-3}$ mbar of O_2 in a nitrogen carrier gas at atmospheric pressure. The moisture content of the nitrogen purge was also measured and was found to be less than 10 ppm.

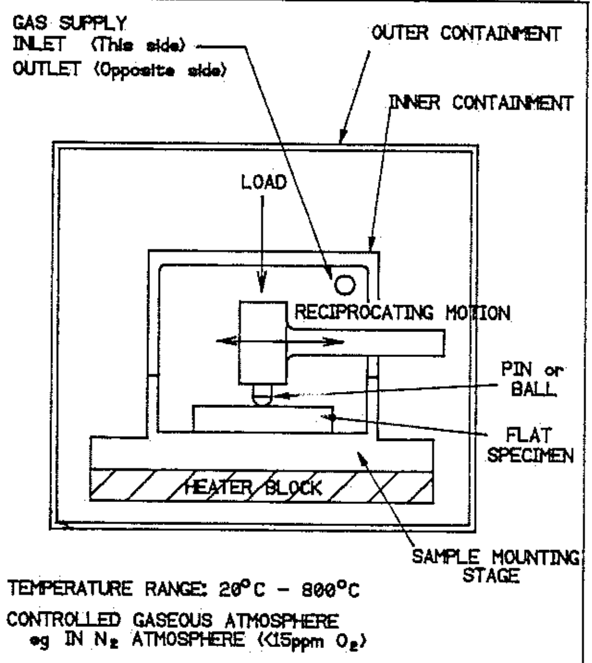


Figure 1
Schematic View of
Reciprocating High Temperature Rig

A ceramic housing (not shown in Fig.1) surrounded this inner containment for thermal insulation purposes and also helped prevent the back diffusion of contaminant gas species into the specimen chamber. An outer gas containment stage was provided by an aluminium enclosure and was independently purged by nitrogen.

The following parameters were used in the tests: 50±5 N load, 1 sample traverse of 15 mm every second, pin radius of curvature 3.57 mm.

The mean and peak frictional forces were calculated from the load cell signal, the output of which approximates to a square wave in reciprocating tests (Fig.2). The definitions of mean and peak frictional forces, from which friction coefficients were calculated, are shown in Figure 2. It was noted that the maximum friction (peak friction) always occurred when the direction of motion changed, ie when the sliding velocity was zero. These friction peaks are therefore taken to be a measure of the static friction.

Film failure was taken to be the point at which the film no longer provided effective lubrication. This occurred when the friction coefficient increased to a value representative of that of unlubricated Inconel 600.

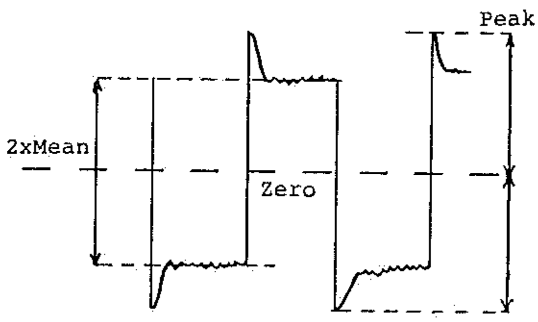


Figure 2

Typical Square Wave Output
From Load cell Showing
Definitions of Mean And
Peak Frictional Force

4 RESULTS

4.1 Unlubricated Substrates (Figure 3)

Room temperature. The initial value of friction coefficient of 0.19 increased to 2.17 within 3 traverses. A maximum value of 2.49 was recorded at 7 traverses. The friction coefficient was erratic throughout the test.

400 Deg C. A range of values from 0.8 to 0.9 was recorded up to 1,794 traverses. A minimum of 0.64 was measured at 2130 traverses.

800 deg C. The initial value of friction coefficient was 1.38 and increased to a maximum of 1.99 at 10 traverses. A decrease then occurred and a minimum value of 0.7 was reached at 700 traverses. The friction remained at this level for a further 410 traverses, after which the test was terminated.

4.2 Criteria for film failure

The above measurements of friction coefficient can now be used as a guide to assess the point at which coating (lubricant) failure occurs. The measurements indicate that, in nitrogen, Inconel rubbing against itself yields a friction coefficient in the range 0.64 - 2.5 (ignoring any running-in effects). The lowest value of friction coefficient (0.64) which was achieved in the unlubricated condition was therefore taken as the film failure point.

4.3 MoS_2-Lubricated Substrates (Figure 4)

Room Temperature. Steady state friction coefficients of 0.02 were measured, with a minimum value of 0.016 being obtained. The endurance was approximately 3,000 traverses.

400 Deg C. The steady state friction coefficient was generally 0.08. A minimum level of 0.06 was achieved at 4 traverses. The endurance was 440 traverses.

800 Deg C. The initial value of friction coefficient of 0.02 increased to 0.59 within the first few traverses. A steady increase to 1.31 was noted within 320 traverses. The film was deemed to have

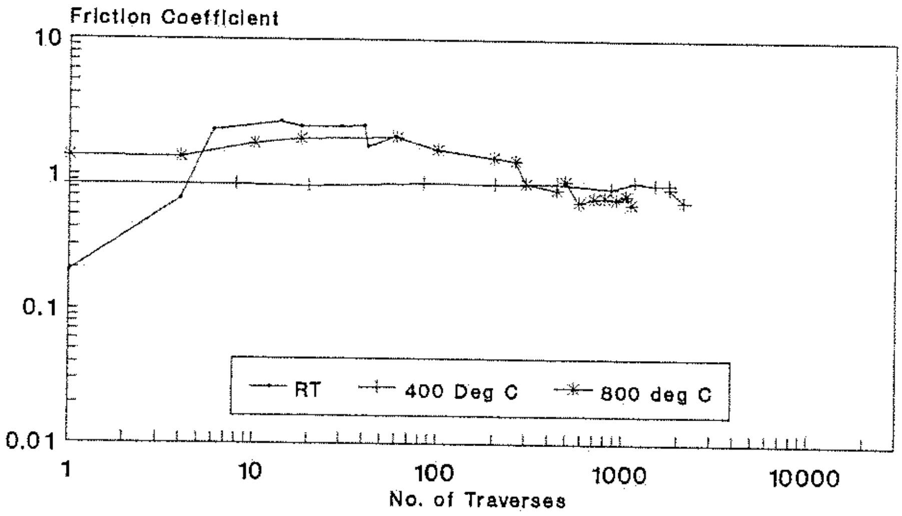


Figure 3

Unlubricated Inconel Substrate And Pin

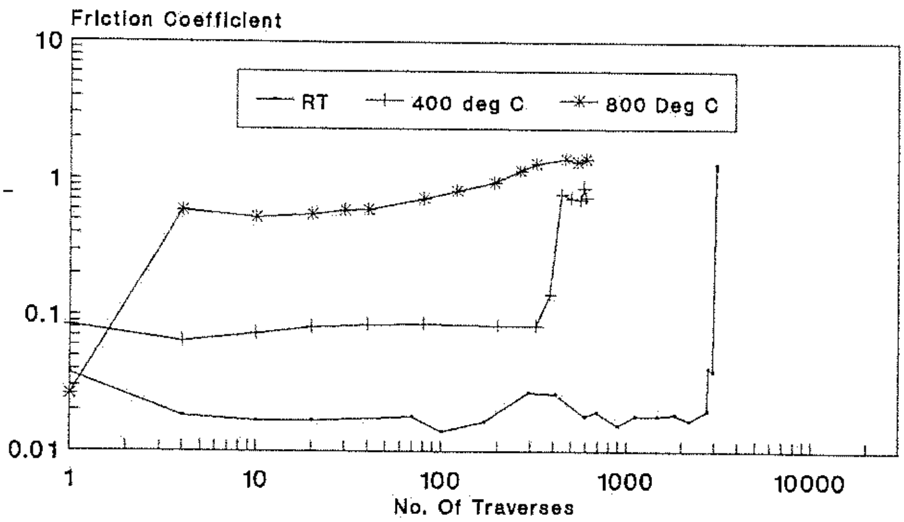


Figure 4

Magnetron-Sputtered MoS_2-Lubricated Substrate

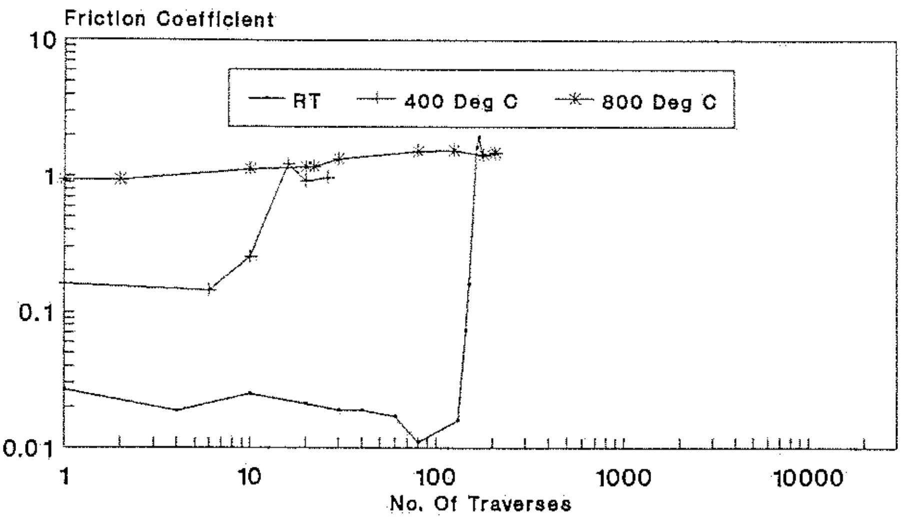


Figure 5

WS_2-Lubricated Substrate

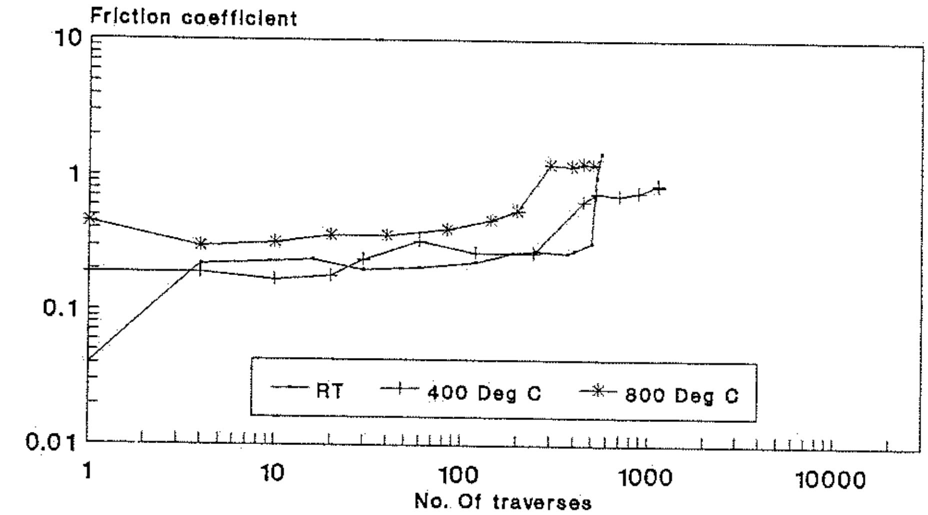


Figure 6

Magnetron-Sputtered Ag Lubricated Substrate

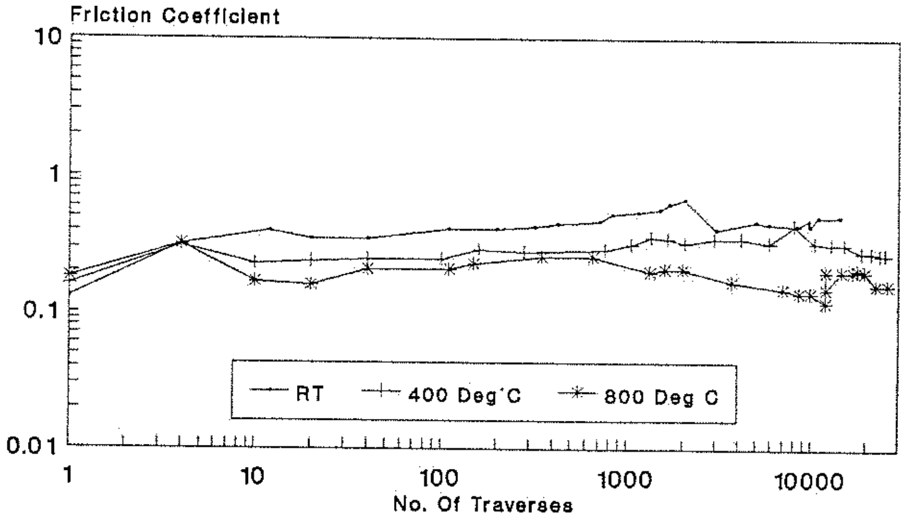


Figure 7

Plasma-Sprayed PS212-Lubricated Substrate

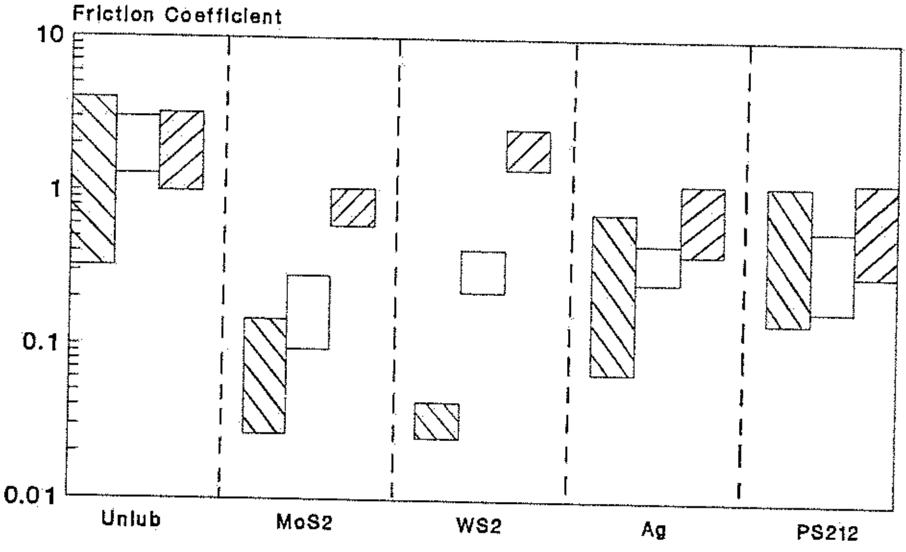
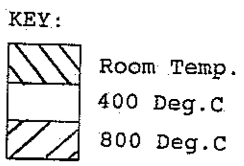


Figure 8

Range Of Values Of Peak Friction Coefficients



failed at 80 traverses.

4.3 WS_2-Coated Substrates (Figure 5)

Room Temperature. The steady state friction coefficient of 0.02 suddenly increased to greater than 1 at 150 traverses. A minimum value of 0.011 was measured at 80 traverses.

400 Deg C. The initial friction coefficient was 0.15±0.01. This increased to 0.26 at 10 traverses and failure occurred at 12 traverses.

800 Deg C. The friction coefficient lay within the range 0.93 to 1.48 during a test duration of 208 traverses. Thus the film is deemed to have failed immediately on commencement of testing.

4.4 Ag-Lubricated Substrates (Figure 6)

Room Temperature. Steady state friction coefficients were within a band ranging from 0.20 to 0.32. The onset of film failure was noted between 500 and 530 traverses.

400 deg C. Steady state values of friction coefficient were from 0.17 to 0.27. A gradual rise, from 0.65 to 0.87, began at 450 traverses and ended at 1140 traverses. The film was judged to have failed after 450 traverses.

800 Deg C. Friction coefficients increased from 0.30 and 0.55 (a starting value of 0.45 was measured) during the first 200 traverses. A value greater than 0.65 was attained at 220 traverses and reached a value of 1.22 at 300 traverses.

4.5 PS212-Lubricated Substrates (Figure 7)

These coatings survived at least 10,000 traverses and tests were stopped (due to time constraints) prior to film failure.

Room Temperature. Friction coefficients ranged from 0.3 to 0.6, being typically 0.05 during steady state running.

400 Deg C. The friction coefficient rose from an initial value of 0.15 to its steady state value of between 0.2 and 0.4 within 10 traverses.

800 Deg C. Equilibrium values of the friction coefficient ranged from 0.2 to 0.25.

4.6 Peak Friction

Figure 8 shows the range of values of peak friction coefficient. The general trend, for MoS_2, WS_2 and Ag coated substrates, follows that of the mean friction coefficient ie the lowest values were measured at room temperature. MoS_2 and WS_2 showed more rapid increases with temperature than Ag. In all cases, the minimum values were greater at 400 deg.C than at room temperature and, at 800 deg.C, the values approached that of the unlubricated substrates. A similar trend is followed by PS212 lubricated Inconel, albeit to a lesser extent.

5. DISCUSSION

The sliding of unlubricated Inconel 600 against itself led to high friction coefficients. Indeed, dynamic friction coefficients as high as 2.5 were recorded. Such high values are not unexpected for unlubricated metals operating in an essentially inert environment and arise principally as a result of adhesion at the contact interface. Thus, for example, it has been found elsewhere that vacuum outgassed nickel on nickel gives rise to friction coefficients as high as 5 when sliding in an H_2 or N_2 environment (Ref.3). Such measurements and those made in the present study confirm the need for effective lubrication at high temperature.

The performances of the solid lubricant coatings tested within the temperature range 20 deg.C to 800 deg.C are summarised in Table 1 (the Table shows coating life in number of traverses; steady state values of friction coefficient are shown in brackets).

MoS_2 and WS_2: at room temperature the lowest friction is exhibited by the molybdenum disulphide and tungsten disulphide films and, of these two lubricants, the best endurance is given by the films of MoS_2 produced by sputtering. Indeed the durability of WS_2 is a factor 20 less than that of MoS_2. This is ascribed to the different methods of film application. Lubricant film lifetime is determined by factors such as adhesion and cohesion (Ref.4). Magnetron sputtering of MoS_2 results in strongly adherent and coherent films giving a comparatively long film lifetime. The WS_2 was deposited by a method described by the manufacturer as "an impingement process resulting in a molecularly bonded film". The degree of film-to-substrate adhesion afforded by this process is not known to us.

At higher temperatures (400 deg.C and above) film friction increases and film durability are much reduced. Indeed, at 800 deg.C, the life of the WS_2 film is essentially zero. The rise in friction and decrease in coating life at high temperatures is probably accounted for by oxidation of the coating surfaces. This occurs as a result of the presence of small quantities of oxygen (<15ppm) in the test environment. The rates of chemical reactions which result in oxidation of MoS_2 and WS_2 are dependent on the amount of oxygen present and the temperature at which the reaction takes place. The reaction rate is exponentially dependent on temperature such that at room temperature it is negligibly low. However, at the higher temperatures of testing the reaction rate is significant - despite the very low concentrations of oxygen present. Since the oxides of MoS_2 and WS_2 yield higher friction, and since, also, their wear rates are higher than the unoxidised sulphides, the tribological performance is degraded. It appears therefore that, in spite of the precautions taken to reduce

TABLE 1

Summary Of Friction And Wear Lives Of Solid Lubricant Coatings

LUBRICANT	TEMPERATURE 20 deg.C	400 deg.C	800 deg.C
Sputtered MoS_2	3000 (0.02)	440 (0.08)	80 (0.6)
WS_2 (Dichronite)	150 (0.02)	12 (0.2)	0 (>0.65)
Silver	500 (0.25)	450 (0.22)	220 (0.3)
PS212	> 15,000 (0.5)	> 25,000 (0.3)	> 25,000 (0.2)

the contaminant level of the nitrogen environment, trace quantities of oxygen are sufficient to degrade thin metal dichalcogenide films at elevated temperatures.

Silver: at room temperature the tribological performance of sputtered silver is inferior to that of the sputtered MoS_2 in that film friction is higher and film durability lower. However, silver films have the advantage over MoS_2 in that their friction and durability remain essentially unchanged as the temperature increases.

PS212: these coatings gave by far the longest wear lives (in excess of 15,000 traverses) of all the lubricants examined in this study. In general, the friction yielded by these films is higher than that given by the other lubricants tested, particularly at room temperature. However, the frictional properties of the film improved as the temperature was increased, the steady state friction coefficient decreasing from 0.5 at room temperature to 0.2 at 800 deg.C. This behaviour relates to the fact that PS212 contains a number of constituents, selected for their lubricating properties, within a wear resistant matrix. The composition is chosen such that each of the lubricant components lubricates at a particular temperature (Ref.5). Thus the presence of silver is expected to assist lubrication at temperatures below 500 Deg C and the BaF_2-CaF_2 combination provides lubrication at higher temperatures.

It should be noted that PS212 is a thick (about 300 microns) coating in comparison with the other thin film coatings (1 micron thick) tested in this study. Thus, the film endurance of PS212 cannot be directly compared with those of silver and the metal dichalcogenides.

Peak Friction Coefficients: the arguments presented in the previous sections to explain the increasing values of mean friction coefficient with increasing temperature can also be applied to the peak friction coefficients. It is important to consider the value of peak friction coefficient when choosing lubricants for applications where intermittent motion is required since in this study the peak friction corresponded to the point at which the sliding velocity was zero.

The greatest values of peak friction, at room temperature, were observed for PS212 and Ag films. At 400 deg.C and at 800 deg.C the differences in the peak friction coefficients among the lubricant films were less significant.

6 CONCLUSIONS

The following conclusions are drawn and relate to measurements made in nitrogen gas containing <15 ppm of oxygen.

1. Unlubricated Inconel vs unlubricated Inconel gave rise to high friction (friction coefficients in the range (0.64 to 2.5)

2. Thin films of MoS_2 and WS_2 applied to Inconel substrates reduced the friction coefficient to 0.02 at room temperature. At elevated temperature, film friction was increased and film life much reduced.

3. The friction coefficient yielded by silver films was essentially invariant with temperature. Film durability also did not change appreciably with temperature but was relatively short.

4. Thick PS212 films provided the longest endurance, survived the highest test temperature and exhibited their lowest film friction coefficients at 800 Deg C.

5. In general, the overall trend in the values of the peak friction coefficients is towards increased values with increasing temperature.

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