FRETTING WEAR BEHAVIOUR OF MOS_2 DRY FILM LUBRICANT

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ABSTRACT

Dry film lubricants (DFL) are used as palliative coatings to prevent fretting wear. In this work fretting tests are carried out on coated Ti6Al4V cylinders on coated flat samples under dry sliding conditions, using an amplitude of 300 µm, 2.5 Hz frequency and 575 N normal loads. During the tests the coefficient of friction (CoF) was monitored, with tests being terminated when the coefficient of friction reached 0.7. Wear scars were analysed by profilometry and SEM to elucidate wear mechanisms. Results show that CoF initially increases rapidly to 0.4, this is then followed by a plateau region that finishes in a sudden step decrease in CoF following which CoF rises steadily. This behaviour is shown to be characteristic and interrupted tests are presented to allow elucidation of the wear scar at different stages in the lifetime and thus aid an understanding of the mechanisms of degradation which control the tribological behaviour.

Keywords: Dry film lubricant, Coefficient of friction, Wear, Fretting, MoS_2.

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INTRODUCTION

Dry film lubricant (DFL) coatings were originally developed for aerospace applications, and are currently employed in many fields [1]. They are used to reduce coefficient of friction (with its influence on wear rate) in applications where conventional fluid lubricants are ineffective and undesirable such as in environments at high vacuum or high temperature, or with highly-loaded contacts with velocities too low to generate fluid-film separation [1, 2]. Molybdenum disulphide is a well-known dry film solid lubricant [3] which has a lamellar hexagonal structure [4, 5] and is commercially available as a polymer bonded dry film lubricant. Various investigations into the fretting behavior of MoS_2 have been reported [6-11]. Xu et al. carried out fretting wear tests on MoS_2 solid lubricant using various normal loads and amplitudes and saw a rapid increase of CoF with higher loads [6]. Hu et al. investigated the tribological properties of hot pressed MoS_2 coating on laser textured surface and found a significant increase in wear life compared to a burnished coating on textured steel surface [7], they also noted that the coefficient of friction could be low as 0.05. Ye et al. saw that MoS_2 based solid lubricant on steel had excellent anti-friction and wear-resistant performance [8] within a wide load range of 20 N to 800 N [9]. Holmberg et al. found that the use of MoS_2 coatings on steel and silicon substrates improved the fracture performance of the coated surfaces [10].

In service DFL coatings frequently use a compliant underlayer such as CuNiIn [12]. This work concentrates on the behavior of the DFL in isolation and does not use an underlayer, potentially resulting in the test conditions being harsher than reality.

In this paper, the behavior of a DFL coating in a highly-loaded fretting contact is examined. In particular, the evolution of the coefficient of friction is investigated with the aid of interrupted tests and examination of the wear scars.

EXPERIMENTAL METHODS

Fretting wear tests were conducted with a crossed cylinder-on-flat arrangement, as shown in Figure 1, with samples being cut by electro-discharge machining (EDM) from Ti6Al4V plate followed by surface grinding. The length of the contact was 10 mm (determined by the width of the flat sample) and the cylindrical sample had a 15 mm radius of curvature. Both parts of the fretting couple were grit blasted with aluminum oxide grit (grade NK36), grit size 0.50-0.59 mm yielding a surface with a roughness (Ra) of around 2 µm. Immediately following grit blasting, a commercially available DFL paint was applied using a spray deposition technique; the primary lubricant phase in the DFL was MoS_2. To achieve a final coating thickness typical of industrial use, ~ 50 µm, eleven separate coats were needed. A time of approximately ten minutes was allowed between each paint coat for solvent evaporation.

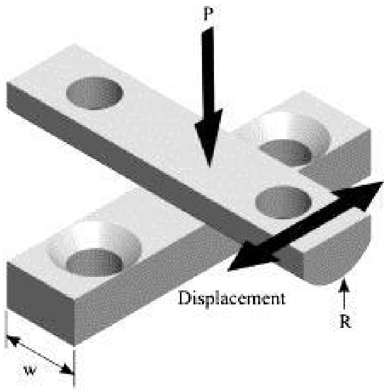

Figure 1. Cylinder-on-flat specimen setup for the fretting test [13], where w= 10 mm, R= 15 mm and P = 575 N.

Coated specimens were cured by being placed in an air-circulatory oven, initially at 100°C. The oven temperature was then increased by 2°C min^{-1} to 195±1°C and then held for 2 h; the oven was then switched off and the cured samples remained in the oven whilst they cooled to room temperature. A Positector 6000 eddy current gauge was used to confirm that the cured coating thickness was 50 µm ± 5. Figure 2 shows an image of the as-cured DFL coating, where it can be seen that the film is a bonded agglomerate of individual particles. The cured DFL had a Knoop hardness of 20 ± 0.5 kgf mm^{-2} and a Ra value of 1.4 ± 0.5 µm.

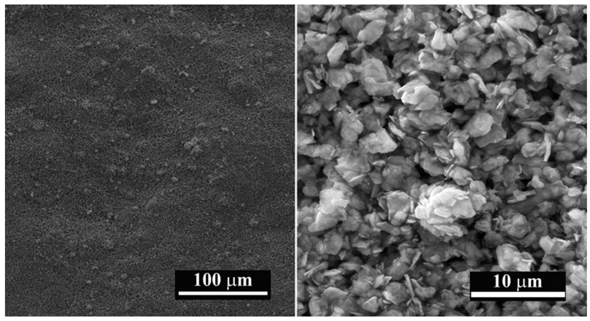

Figure 2. Low magnification (left) and high magnification (right) SEM micrograph of the surface of the as-cured DFL paint coating MoS_2.

For the fretting tests, specimens were mounted in a cylinder on flat configuration as shown in Figure. 1 with fretting motion being applied perpendicular to the axis of the cylindrical specimen; a schematic diagram of the fretting rig used for the experiments is shown in Figure 3. A normal load is applied, and relative displacement between the cylindrical and flat specimens is achieved through control of an electromagnetic vibrator (EMV). The tangential force, Q, at the contact is measured as a reaction force by a piezoelectric load cell. The applied displacement amplitude is controlled via position measurement of the upper specimen mounting block using a linear variable differential transformer (LVDT). The applied displacement and tangential force were measured throughout the test. The relationship between measured friction force and displacement amplitude during a fretting cycle is plotted, generating the fretting loops seen in Figure 5.

A normal load, P, of 575 N was applied; if the presence of the DFL is ignored, such a load would result in a mean Hertzian contact pressure of 222 MPa (assuming a Young's modulus of 115 GPa and a Poisson's ratio of 0.342 for Ti6Al4V [14]). The flat specimen was fixed and the cylinder specimen was fretted against it, moving with a displacement amplitude of 300 µm at a frequency of 2.5 Hz. The temperature and relative humidity of the environment were 20±3°C and 30±5% respectively.

The maximum tangential force, Q, in each fretting cycle was recorded; from this, the coefficient of friction (CoF), µ, was calculated by dividing by the applied normal load.

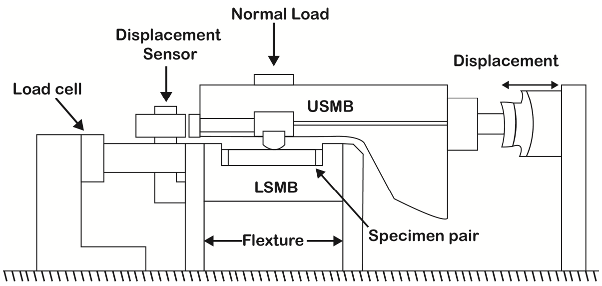

Figure 3. A schematic diagram of the fretting rig.

During the rig tests, the values for coefficient of friction (CoF) were continuously recorded. Initially, tests were continued until the friction of coefficient reached a value of 0.7 (initial experiments proved this value to be the coefficient of friction between uncoated Ti6Al4V samples in this configuration); this was reached after 68000 cycles. Subsequently, tests were run to lower numbers of cycles (5000 cycles and 36000 cycles) to allow the surfaces at different stages throughout the damage process to be characterized. Throughout this paper, these samples are referred to as 5k, 36k and 68k (indicating the number of cycles that the test ran for).

The geometry of the wear scars on the flat sample in each wear couple was characterized using a Talysurf CLI 1000 contact profilometer. The central 8 mm region of the wear scar was scanned with 54 lines, all parallel to the fretting direction and spaced 150 µm apart. The surfaces of the wear scars were characterized via using scanning electron microscopy (Philips XL30), using both secondary and backscattered electron imaging.

RESULTS

<u>Evolution of coefficient of friction in the contact</u>

Figure 4 shows the evolution of coefficient of friction (CoF) with number of cycles during fretting of the DFL-coated titanium alloy fretting pairs. Where comparable, the evolution of friction over the three tests examined can be seen to be very reproducible, with similar changes in the coefficient of friction occurring at similar numbers of cycles. Initially, the CoF rose quickly to a value just below 0.6 and then fell to a value around 0.5 (the 5k sample represents a point in the damage evolution associated with this phase of behavior). The coefficient of friction continued to fall slowly over the next 15000 cycles; at this point, a change in behavior was observed, with the coefficient of friction more rapidly falling to a value below 0.35 over the next 5000 cycles. From this point, the coefficient of friction began to slowly rise again; (the 68k sample represents a point in the damage evolution associated with this phase of behavior). At a point after 45000 cycles, the coefficient of friction was observed to begin to oscillate over small numbers of cycles, and also to increase rapidly, until a CoF of 0.7 had been reached after about 68000 cycles.

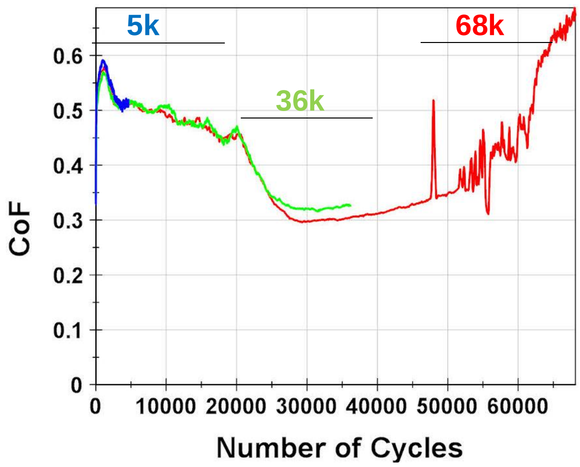

Figure 4. Evolution of coefficient of friction with fretting cycles. Three tests conducted under identical conditions are included, with tests being terminated at different points.

Fretting loop results associated with the three selected stages in the evolution of behavior are shown in Figure 5. At 5k cycles, the fretting loop has regular rectangular shape and the coefficient of friction was around 0.6. At 36k cycles, the fretting loop shows much lower lateral force for sliding (CoF around 0.3); however, it has an unusual shape in that during the sliding stage, the lateral force decreases towards the mid-point of the slide and then increases again (such behavior is difficult to rationalize in terms of the geometry of the wear scar [15]). At 68k cycles, the fretting loop shows a return to higher coefficients of friction, with a significant variation in the lateral force in the sliding region, probably associated with the disruption of the sample surface due to wear.

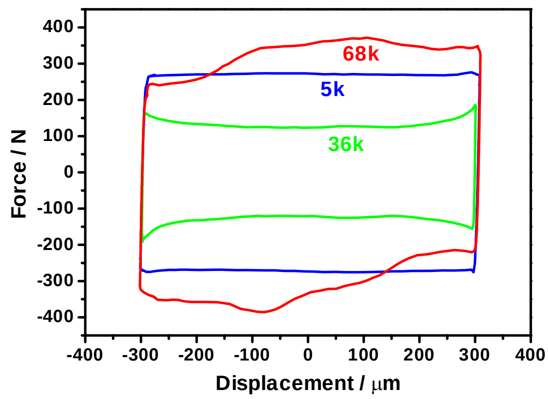

Figure 5. Fretting loops obtained from EMV rig for 5k, 36k and 68k.

Characterization of wear scars

The shape of the wear scars on the flat samples for the three cycle durations were characterized by profilometry. In each case, the individual profiles across the scar were very similar, indicating relatively uniform wear across the width of the width of the flat. As such, it was appropriate to combine these individual line profiles into an average profile. Figure 6 shows the average profiles on the flats for the three fretting durations. A dotted line on the graph also indicates the depth at which the coating-substrate interface occurs (the DFL thickness being ~ 47 μm). The maximum depth of the wear scar after fretting for 5k, 36k and 68k cycles was 14 μm, 70 μm and 127 μm respectively. After 5k cycles, it can be seen that the material removal is well within the coating thickness, but that following 36k cycles, material has been removed to a thickness greater than that of the coating, although it is clear that this tests has been terminated very soon after the coating has been penetrated. In contrast, at 68k cycles, the wear scar is much deeper than the coating thickness, indicating that there has been substantial removal of the titanium alloy substrate.

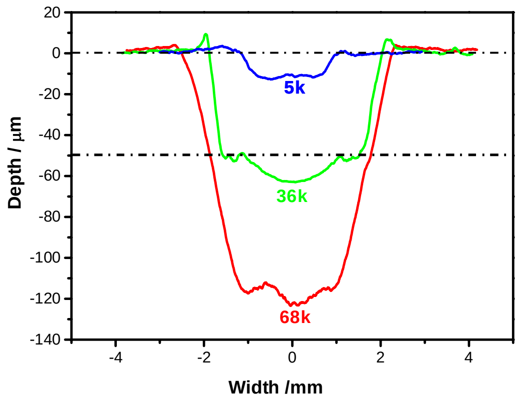

Figure 6. Average profiles of the wear scar after 5k, 36k and 68k cycles. The dotted lines indicate the location of the DFL coating.

Table 1 shows the EDX analysis results from a 100 µm x 100 µm area from the centres of the wear scars following testing for the three durations. It is recognized that there will be significant errors in the absolute values quoted (due to the measurement errors associated with carbon and oxygen); however, some clear conclusions can be drawn despite this uncertainty. After 5k cycles, there is no indication of the titanium alloy on the surface, but at 36k, there is evidence of substrate exposure, which then increases in magnitude by 68k cycles. In contrast, there is evidence of MoS_2 after 5k and 36k cycles, but after 68k cycles, all the MoS_2 lubricating phase has been removed. Although the carbon and oxygen measurements are associated with significant error, the trends observed are consistent with the other data, namely that the carbon level falls as the DFL layer is removed (the carbon being associated primarily with the polymeric bonder phase) and the oxygen level increases (associated with the formation of an oxide debris layer as the titanium alloy substrate begins to fret [16]). It is also observed, the contact size is much smaller than the width of the wear scars in Figure 6, i.e. there is notable growth of the contact area during the wear process. The effect of the resulting progressive reduction in contact pressure will be the subject of our future work. The positive values in the wear scar at the contact edges could be due to wear debris or material transfer from the counter surface, this has not yet been investigated in detail.

Figure 7 shows images of the surfaces of the centre of wear scars following testing for the three durations (SE images are presented on the left with BSE images being presented on the right). Following 5k cycles, the surface appears to be basically flat. It is clear that material is being removed from the surface by cracking and delamination of flakes, indicating attritive wear. No surface cracking was observed. Following wear for 36k cycles, the surface still exhibits a generally smooth appearance, with some evidence of delamination of flakes. However, in contrast with the 5k case, the BSE image following wear for 36k cycles indicates the presence of a different (lower contrast) phase; it is proposed that this material is titanium oxide associated with the exposure (and fretting of) of the titanium alloy substrate. Following 68k cycles, the wear surface has a very different morphology, with the surface now being roughened and covered with particulate debris. Within the surface, there are some regions which appear to be more smooth (with an absence of particulate debris) and these are associated with regions of higher contrast in the BSE images. These may be regions where the titanium alloy substrate has had any debris layer removed, exposing the alloy itself.

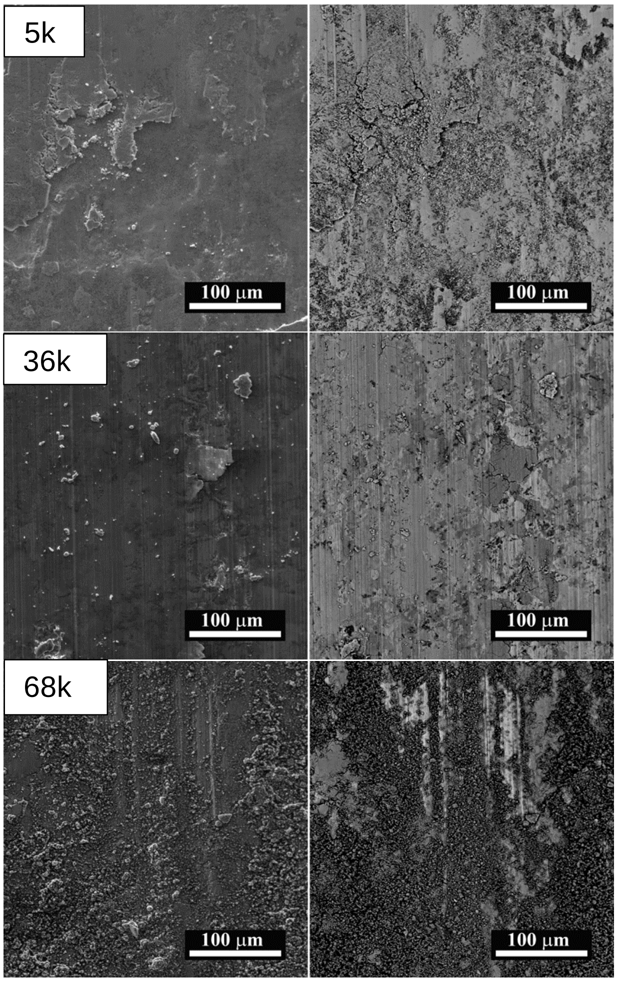

Figure 7. SE (left) and BSE (right) plan view micrographs of representative portions of the wear scars at 5k, 36k and 68k cycles.

Table 1. Area analysis EDX results for 5k, 36k and 68k cycle wear scars.

Element	5k area analysis / wt%	36k area analysis / wt%	68k area analysis / wt%
Ti	0	9	49
Al	0	2	3
V	0	0	2
Mo	38	28	0
S	25	16	0
C	27	21	11
O	10	23	35

DISCUSSION

The significant changes in friction behavior during the fretting of self-mated DFL coated titanium substrates can be understood in terms of microstructural development. After 5k cycles, the coefficient of friction is high, and this is associated with the fretting of the DFL against itself. The DFL wears attritively by delamination, until the titanium alloy substrate begins to be exposed. As the metallic substrate is exposed in the fretting scar, the coefficient of friction is significantly reduced (with this being a very rapid change in behavior). This low coefficient of friction is maintained, but wear continues with more of the DFL being lost from the surface; as the proportion of the surface being titanium alloy (and debris associated with fretting of the alloy – namely oxides) increases, then the coefficient of friction begins to rise. At this point, there is significant fluctuation of the coefficient of friction superposed on a steady rise in the coefficient of friction which increases until that observed during fretting of bare titanium alloy itself, ~0.7, is reached by 68k cycles.

It is proposed that the DFL therefore operates best (in terms of reducing the coefficient of friction) as a component of a composite DFL-metallic surface. The benefits of the composite surface over that of the pure DFL surface are not fully understood at this stage, but it is clear that the metallic nature of the composite surface will serve to provide lateral (shear) stiffness to the material which will limit energy dissipation during fretting cycling, thereby limiting damage of the surface.

CONCLUSIONS

Analysis of the fretting wear of cylinder and flat Ti6Al4V fretting couples coated with a commercial MoS_2 containing DFL has revealed the following:

- The coefficient of friction varies notably, and reproducibly, as fretting wear progresses.
- The minimum value of the coefficient of friction is reached at an intermediate point in the fretting test.

- The minimum coefficient of friction value corresponds to the generation of a mixed metal/DFL surface.
- The precise mechanisms behind the observed effect are not yet fully understood.

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REFERENCES

1. P.Z. Zhen Yang, Yue Lan Di, Zhi Hai Cai, Qi Li, Investigation on Microstructure of CrN-Based Solid Self-Lubricant Composite Coating, Advanced Materials Research. 538 - 541 (2012) 406-409.
2. C. Donnet, A. Erdemir, Historical developments and new trends in tribological and solid lubricant coatings, Surface and Coatings Technology. 180–181(0) (2004) 76-84.
3. A.M. Korsunsky, A.R. Torosyan, K. Kim, Development and characterization of low friction coatings for protection against fretting wear in aerospace components. Thin Solid Films 516(16) (2008) 5690-5699.
4. M.S. Dallavalle, S. Nadja F. Zerbetto, Stability, dynamics and lubrication of MoS2 platelets and nanotubes. Langmuir 28(19) (2012) 7393-7400.
5. J.M. Martin, C. Donnet, T. Le Mogne, T. Epicier, Superlubricity of molybdenum disulphide. Physical Review B 48(14) (1993) 10583-10586.
6. J. Xu, M.H. Zhu, Z.R. Zhou, P. Kapsa, L. Vincent, An investigation on fretting wear life of bonded MoS2 solid lubricant coatings in complex conditions. Wear 255(1-6) (2003) 253-258.
7. T. Hu, Y. Zhang, L. Hu, Tribological investigation of MoS2 coatings deposited on the laser textured surface. Wear 278–279 (2012) 77-82.
8. X. Li, Y. Gao, J. Xing, Y. Wang, L. Fang, Wear reduction mechanism of graphite and MoS2 in epoxy composites. Wear 257(3-4) (2004) 279-283.
9. Y. Ye, J. Chen, H. Zhou, An investigation of friction and wear performances of bonded molybdenum disulfide solid film lubricants in fretting conditions. Wear 266(7-8) (2009) 859-864.
10. K. Holmberg, H. Ronkainen, A. Laukkanen, K. Wallin, S. Hogmark, S. Jacobson, U. Wiklund, R.M. Souza, P. Ståhle, Residual stresses in TiN, DLC and MoS2 coated surfaces with regard to their tribological fracture behaviour. Wear 267(12) (2009) 2142-2156.
11. X. Shi, W. Zhai, Z. Xu, M. Wang, J. Yao, S. Song, Y. Wang, Synergetic lubricating effect of MoS2 and Ti3SiC2 on tribological properties of NiAl matrix self-lubricating composites over a wide temperature range. Materials & Design 55 (2014) 93-103.
12. K. Kim, A. M. Korsunsky, Dissipated energy and fretting damage in CoCrAlY-MoS_2 coatings. Tribology International 43(5-6) (2010) 861-867.
13. A.R. Warmuth, S.R. Pearson, P.H. Shipway, W. Sun, The effect of contact geometry on fretting wear rates and mechanisms for a high strength steel. Wear 301(1-2) (2013) 491-500.
14. A.L.M. Tobi, J. Ding, G. Bandak, S.B. Leen, P.H. Shipway, A study on the interaction between fretting wear and cyclic plasticity for Ti-6Al-4V. Wear 267(1-4) (2009) 270-282.
15. D.M. Mulvihill, M.E. Kartal, A.V. Olver, D. Nowell, D.A. Hills, Inverstigation of non-coulomb friction behavious in reciprocating sliding. Wear 271(5-6) (2011) 802-816.
16. N.M. Everitt, J. Ding, G. Bandak, P.H. Shipway, S.B. Leen, E.J. Williums, Characterisation of fretting-induced wear debris for Ti-6Al-4V. Wear 267(1-4) (2009) 283-291.

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