THE TRIBOLOGY OF SOME MoS_2-LUBRICATED ENGINEERING CERAMICS

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ABSTRACT

This paper reports on a study of the tribological properties of ceramics involving friction and wear slide tests in both air and vacuum. Tests were undertaken with and without lubrication (the lubricant employed was sputtered MoS_2). The ceramics tested (in either bulk or thin-film form) were silicon nitride, silicon carbide, titanium carbide. titanium nitride and zirconia. The study has demonstrated that, in general, unlubricated ceramics yield high friction in both air and vacuum. MoS_2-lubricated ceramics give low friction in vacuum, their friction and wear properties comparing favourably with that of MoS_2-coated bearing steels. The results indicate that MoS_2-lubricated ceramic bearings should prove superior to similarly coated steel bearings in terms of reduced torque and prolonged low-friction lifetime. This is particularly attractive to the space industry where increasingly the demands are for components having lower torque and torque noise together with longer lifetimes.

Keywords: ceramics, molybdenum disulphide, vacuum, tribology.

1. INTRODUCTION

Some ceramic materials are potentially superior to hardened steels as structural elements of tribological components because of their:

- higher hardness
- corrosion resistance
- higher elastic modulus
- ability to withstand higher temperatures
- lower density (hence reduced weight).

However, the tribological properties of ceramics, particularly under vacuum conditions, are not well characterised and, in general, effective methods of lubrication, ones which are suited to space application, have yet to be established.

The objective of the work reported here was to investigate the tribological properties of various engineering ceramics in both an unlubricated and solid-lubricated condition. The tests were undertaken in both air and vacuum environments. Where possible, the ceramic materials were procured in bulk form. Where bulk specimens were not available, ceramic coatings were deposited onto bearing steel substrates. The ceramics tested were silicon nitride (Si_3N_4), silicon carbide (SiC), titanium carbide (TiC), titanium nitride (TiN) and zirconia (ZrO_2). Lubrication was provided by sputter deposition of molybdenum disulphide.

2. BACKGROUND

Despite their hardness, clean unlubricated ceramics do not have intrinsically good tribological properties. Friction coefficients are usually in excess of 0.3 and wear can be high (Ref.1). However, with lubrication, as for example with sputtered MoS_2 on Si_3N_4, lower friction coefficients and higher endurance are obtained than those of equivalent films deposited on bearing steels (Ref.2).

This reduction in film friction has been explained, for elastically deformed contacts, in terms of the higher elastic modulus of the substrate (Ref.2). For a smooth elastic sphere sliding against a smooth, flat substrate coated with a thin solid lubricant film the friction coefficient is given by (Ref.3),

$$\text{Friction coefficient} = \frac{s}{W^{1/3}} \left[\frac{3R}{4E^*}\right]^{2/3} \qquad (1)$$

where R is the radius of the sphere, s is the shear strength of the film, W is the applied load. E^* is the effective (reduced) elastic modulus, defined by:

$$1/E^* = (1-v_1^2)/E_1 + (1-v_2^2)/E_2 \qquad (2)$$

where v is Poisson's ratio.

It follows from equ. (1) that if the contact geometry and load are held constant, then, for a given lubricant film, the friction coefficient will be proportional to $E^{*-2/3}$. Thus the higher the

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effective elastic modulus of the contact materials then the lower the film friction. Certain ceramics have elastic moduli which are appreciably higher than that of bearing steel and thus offer the prospect of very low friction when used in conjunction with thin solid lubricant films.

An additional advantage is that film durability may be increased when MoS_2 is applied to ceramic, as opposed to steel, substrates. Thus it has been demonstrated that the in-vacuo endurance of sputtered MoS_2 applied to silicon nitride and silicon carbide exceeds by an order of magnitude that of similar films of MoS_2 applied to bearing steel (Refs. 2,4).

A perceived disadvantage of ceramics is their brittleness. To a degree this may be overcome by the use of thin ceramic interlayers. Thus by applying thin (<5 microns) ceramic coatings to steel components the ductility of the steel is retained whilst the surface, being ceramic, may have an improved wear resistance. In space applications, this approach has been taken in ball bearings in which 440C balls are coated with titanium carbide and the races lubricated with sputtered MoS_2 (Ref.5). It is claimed that this combination extends the low-torque life of the bearings since on penetration of the MoS_2 film by ball asperities, steel-to-steel contact is avoided thus preventing microwelding and adhesive wear.

3. TEST MATERIALS AND SAMPLE PREPARATION

Specimens comprised discs and balls for testing on ESTL's pin-on-disc apparatus. Wherever practicable, the ceramic materials were procured in bulk form. Otherwise, ceramic coatings were deposited onto 440C stainless steel or EN31 steel substrates using well established deposition techniques. Details of the ceramic samples are given below.

Silicon nitride: silicon nitride discs were produced by hot pressing (by Morgan Matroc). Silicon nitride balls were manufactured by hot isostatic pressing (from Spheric Engng.).

Silicon Carbide: SiC discs were obtained from Morgan Matroc and were produced by hot isostatic pressing (HIP). SiC balls were supplied by Spheric Engineering.

Titanium Carbide: neither the ball or disc were manufactured from bulk TiC. Instead, 440C steel balls and discs were coated with a 3 micron coating of TiC. TiC coatings were produced by Vilab, Switzerland by a CVD process.

Titanium nitride: as with the titanium carbide, titanium nitride was applied in the form of a thin coating (in this case to a EN31 steel substrate). The TiN was applied by the technique of arc-evaporation to a thickness of approximately 4 microns. The arc-evaporated coating is characterised by the presence of titanium-rich nodules which protrude above the film surface. Previous tests had demonstrated that the presence of these nodules was detrimental to the durability of sputtered MoS_2. Thus, for this study, the nodules were removed, prior to deposition of MoS_2, by polishing with 400 SiC grit.

Zirconia: yttria-stabilised zirconia balls were obtained from the Swiss company Jean Sandez et Fils. Disc samples were prepared by coating 440C steel discs with ZrO_2 by plasma spraying to a thickness of nominally 100 microns.

For consistency with previous work (Ref.2), the preferred disc surface finish was R_a=0.05 microns (centre-line-average). The surface finish of the test discs was measured using a stylus instrument (Talysurf). For each disc, measurements were made in the radial and circumferential directions at three locations. The best finish achievable with the ZrO_2 discs was limited, as a result of the granular structure of the coating, to an R_a value of 0.2 micron. Likewise with the TiN coatings, the surface roughness was in excess of that desired, being of the order 0.1 micron, the roughness being determined by the recesses remaining following removal of nodules. Surface finishes of R_a= 0.05 ± 0.02 micron were achieved on TiC, SiC and Si_3N_4 surfaces.

Following polishing, discs to be tested in the lubricated condition were coated with MoS_2. Nominal 1.0 micron-thick films were deposited by magnetron sputtering according to ESTL procedure ESTL/TP/073. Discs to be tested unlubricated were degreased by ultrasonic cleaning in a freon-based solvent for ten minutes and the process repeated. All rider materials were run unlubricated and were degreased by the above process.

4. TEST APPARATUS

Sliding friction coefficients were measured using a pin-on-disc apparatus in which three spherically tipped riders (pins or balls) were loaded against a rotating disc. The total load was 50N and the sliding speed approximately 1.3ms^{-1}. The frictional torque was measured by means of a torque transducer connected to the pin holder. It was possible to accommodate two test tracks on each side of the discs. The test parameters are summarized in Table 1.

Table 1: Test parameters

Rotation rate:	400 rpm
Disc dimensions:	approx. 78mm o.d, 50mm i.d, 6mm thick.
Running track diameters:	61mm and 67mm
Sliding speeds:	1.28ms^{-1} and 1.40ms^{-1}
Rider diameters:	7.14mm (TiC, ZrO_2, Si_3N_4 and 52100) and 8.0mm (SiC)
Load:	50N on 3 riders
Environment:	
air	class 10,000 clean-room, RH=45% ± 5%
vacuum	pressure $<10^{-6}$mbar
Measurement accuracy:	± 10%
Failure criterion:	Friction coefficient greater than 0.3
No.tests per combination:	
Vacuum/lubricated	3-5
In air or unlubricated	2

TABLE 2 MATERIAL COMBINATIONS TESTED & UNLUBRICATED TEST DATA

SUBSTRATE MATERIAL	RIDER MATERIAL	INITIAL FRICTION		DURABILITY	
		AIR	VACUUM	AIR	VACUUM
SILICON NITRIDE	SILICON NITRIDE	0.2-0.45	0.05 - 0.4	350	>500
TIN ON EN31	EN31 STEEL	0.29	0.27	20	20
TIC ON 440C	a) EN31 STEEL b) TIC-COATED 440C	0.25 0.22	0.15 0.15	1045 856	386 187
SILICON CARBIDE	a) EN31 STEEL b) SILICON CARBIDE	>0.3 >0.3	>0.3 >0.3	- -	- -
ZIRCONIA ON EN31 STEEL	a) EN31 STEEL b) ZIRCONIA	0.26 0.23	0.25 >0.3	500 66	165 -

During all tests, the frictional torque was recorded continuously. Film failure was defined to have occurred when the friction coefficient exceeded a value of 0.3.

The material combinations tested are listed in Table 2.

5. RESULTS

5.1 Unlubricated ceramics

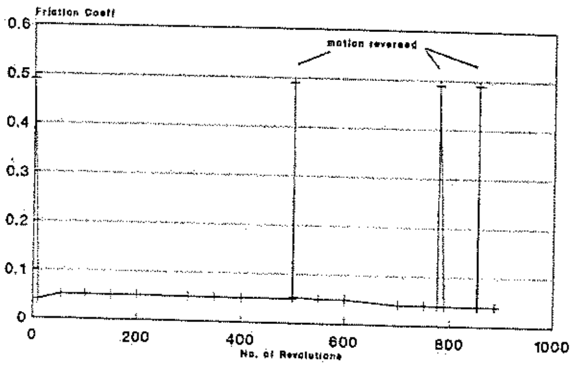
Unlubricated ceramics gave high and noisy friction coefficients, and the tests either failed to start (initial friction coefficients exceeded 0.3) or were short lived. The initial friction coefficients and endurances are summarized in Table 2.

An exception to the above behaviour occurred with silicon nitride sliding against itself under vacuum. Here, low values of friction coefficient (0.04) were observed following running-in. However, on each reversal of disc direction high transitional values of friction coefficient (0.5) were seen (Fig.1).

5.2 MoS_2-Lubricated Ceramics

The frictional behaviour of MoS_2 films generally shows three distinct phases (Fig.2). Initially there is a brief "running-in" period in which the friction coefficient decreases to a low stable value. This is followed by a prolonged period of running, in which low friction is maintained (the "steady-state"). In the steady-state, the friction coefficient ranges between minimum and maximum values as defined in Figure 2. Finally, on wear-through of the film (film failure) the friction coefficient increases. Often, this failure is rapid, as for MoS_2 films on steels, but can be more gradual - as, for example, with MoS_2 on silicon nitride (Ref.2). Fig.2 also defines "friction bands" for a set of data obtained from repeat tests. These bands represent the upper and lower limits of friction coefficient and film endurance.

FIG. 1 SLIDING FRICTION COEFFICIENT
SI3N4 BALL vs SI3N4 DISC
In vacuum, unlubricated



In general, the friction behaviour of the lubricated ceramics tested here followed the above pattern in both air and vacuum. An exception to this trend occurred with zirconia-on-zirconia contacts where, under vacuum, the film failed in two stages. In the first stage the friction coefficient increased to values in the range 0.1 to 0.2 and remained at this level until a second sharp increase signalled complete failure. Thus whilst the tests ran for tens of thousands of disc revs. the actual low-friction film life was low (a few thousand revs. only).

FIG. 2 Schematic diagram to show the analysis of a set of friction data.

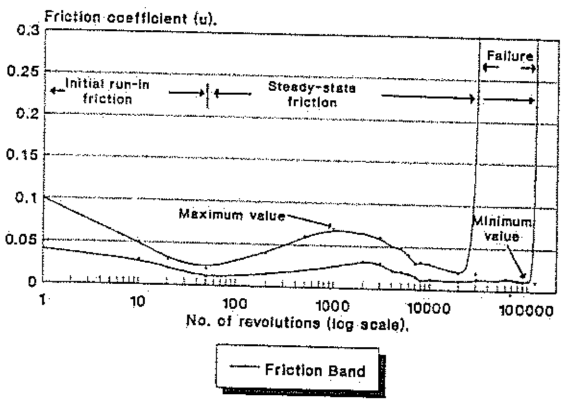
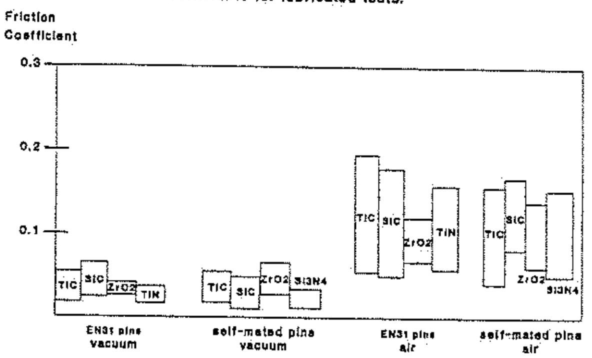
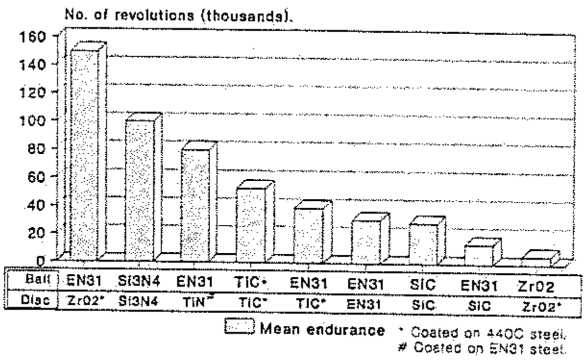


Fig. 3 Summary of range of steady-state friction coefficients for lubricated tests.



Mean endurances of various MoS2 lubricated ceramic/steelball on disc tests. Load 50N: Sliding speed 1.35 m/s +/-10%. Vacuum pressure < 10E-6 mbar.



Disc Ra 0.05 +/- 0.02 microns. (ZrO, Ra 0.2 microns)
MoS2 nominally 1.0 microns.
Fig. 4

Fig.3 summarises the measured ranges of steady-state friction coefficients. A summary of MoS_2 mean film durability in vacuum for different material combinations is shown in Fig.4 (for comparison we also show MoS_2 film durability on EN31 steel). For each material combination, MoS_2 film durability in air was always less than in vacuum (typically a factor five less). The following observations are made:

- tests conducted in vacuum gave lower friction coefficients and higher endurances than tests conducted in air. In vacuum, steady-state friction coefficients were typically in the range 0.01 to 0.06. This compares with a much wider range of steady-state friction coefficients (0.04 to 0.20) for the air tests.

- in vacuum, MoS_2 film durability is greatly dependent on the substrate and rider material combination: the highest mean film durability being obtained for a combination of EN31 steel rubbing against MoS_2-coated zirconia.

- the use of a zirconia rider (ball) gave rise to the shortest film lifetimes in both the air and vacuum tests.

6. DISCUSSION

6.1 Lubricated ceramics

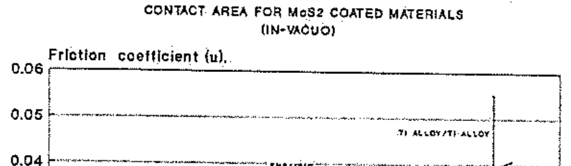
6.1.1 In-vacuo behaviour compared to MoS_2-lubricated steel. Polished EN31 steel specimens (R_a < 0.05 micron) lubricated with sputtered MoS_2 yield, in vacuum, minimum friction coefficients of 0.02 and film durabilities of typically 30,000 revs (Fig.4). Our present results show that ceramic contacts give lower minimum friction coefficients (Fig. 3) and have mean MoS_2 film durabilities which in general compare favourably with that of MoS_2-coated steel. The lower friction of MoS_2-coated Si_3N_4 and SiC is attributed to a higher effective elastic modulus and is in line with prediction based on equ.1. It is probable that zirconia, which has an elastic modulus similar to that of EN31 steel, yields a lower friction coefficient than that of MoS_2-coated EN31 because of its rougher surface (Ref. 8 shows that the friction coefficient of MoS_2 films varies with the surface roughness of the substrate).

From Fig.4 it can be concluded that the most consistently effective performances (durabilities in the order 10^5 disc revs.) are obtained with combinations of EN31 steel on zirconia and silicon nitride versus itself. Fig.4 also demonstrates that the use of thin ceramic coatings (TiN, TiC) as interlayers between a steel substrate and an MoS_2 lubricant film, can effect increases in film durability over that of MoS_2 on bearing steel.

It can be shown that the values of friction coefficient made here for MoS_2 films applied to bulk ceramics are consistent with the model of film friction discussed in section 2 and described mathematically by equ.1. This model relates film friction to true contact area which, in turn, is governed by the elastic moduli of the contact materials (for elastic contacts). In Fig. 5 we plot measured friction coefficient as a function of the Hertzian contact area using data obtained in this work and elsewhere (Ref.2). We see that the best fit curve is linear such that the friction coefficient is directly proportional to contact area in accordance with equ.1.

6.1.2 Effect of air environment. The results show that MoS_2 film lubricity (defined as the inverse of friction coefficient) and durability in vacuum are consistently higher than those observed in air. This observation is in agreement with

RANGE OF STEADY-STATE FRICTION COEFFICIENTS VS HERTZIAN CONTACT AREA FOR MoS2 COATED MATERIALS (IN-VACUO)



Surface Ra nominally 0.05 microns.
MoS_2 coating nominally 1.0 microns.
Fig. 5

the behaviour of sputtered MoS_2 films on steel (Ref.6). Film friction increases in air because of adsorption of water molecules by the MoS_2 film which results in an increase in the shear strength of molybdenum disulphide and hence friction coefficient (equ.(1)). In air, the higher friction forces generate higher surface temperatures and film oxidation occurs. This, in turn, reduces film durability.

6.1.3 The behaviour of MoS_2-lubricated zirconia contacts. The use of zirconia either as a substrate material or rider has produced results which deviate considerably from those obtained with the other ceramics tested and as such call for some comment. Specifically, it is observed that:

a) the use of zirconia riders in conjunction with zirconia substrates leads to very low MoS_2 film lifetimes.

b) the use of zirconia substrates in conjunction with EN31 steel riders leads to consistently high film durabilities.

Low film durabilities often arise as a result of poor adhesion between film and substrate. Therefore, in examining the reasons for (a) above we must first examine the role of the substrate and the adhesion of MoS_2 to zirconia. However, two pieces of evidence lead to the conclusion that poor adhesion is not a factor. First, it has been shown in a parallel investigation (Ref.7) that chemical bonds are generated between MoS_2 and ZrO_2 during sputter deposition of MoS_2. The presence of chemically bonded entities is indicative of strong interfacial adhesion. Secondly, MoS_2 films applied to zirconia substrates, but tested in conjunction with steel riders, show high mean durabilities (Fig.4). This latter observation confirms that adhesion of sputtered MoS_2 to zirconia is strong, thus precluding poor adhesion as the reason for short film durability. This being so, the reason for poor film durability must be to do with the zirconia riders. SEM examination reveals the surface of ZrO_2 balls to be relatively rough and having large and distinct asperities. Such hard protruding asperities will penetrate the lubricant film and enhance its wear by abrasion. It seems likely therefore that the observed premature failure of MoS_2 arises as a result of the poor finish of the zirconia riders. The use of smoother zirconia riders might well lead to much longer film lives.

We now turn to observation (b). We believe that the high film durability of MoS_2 on ZrO_2 substrates is, in part, a result of the surface roughness of the zirconia substrate. Whereas the surface roughness of the TiC, Si_3N_4 and SiC substrates was in the order 0.05 micron (Ra), the surface of the zirconia could be polished to a finish no better than 0.2 micron (Ra). It is known that the endurance of MoS_2 on 52100 steel is very much dependent on the surface roughness of the substrate (Ref.2). Indeed, recently it has been shown that there exists an optimum surface roughness for steel substrates at which the endurance of sputtered MoS_2 films of thickness 1 micron is maximised (Ref.8). This optimum surface roughness corresponds to an Ra value in the order 0.2 microns. For Ra values of 0.05 micron MoS_2 film durability was an order of magnitude less. Although the sensitivity of MoS_2 film lifetime to the surface roughness of steel has yet to be fully explained it seems reasonable to assume that a similar effect would occur with ceramic substrates. We therefore propose that the higher film durability of MoS_2 on zirconia substrates arises because of the greater surface roughness of the zirconia coatings.

6.2 Unlubricated ceramics

In general, unlubricated ceramic substrates run against both EN31 steel and self-mated riders gave high friction coefficients and endurances of less than 1,000 revolutions. In vacuum the lowest friction measured was that of Si_3N_4 rubbing against itself. That combination gave a friction coefficient of 0.05 during unidirectional sliding. However, on reversing the direction of sliding the friction momentarily became much higher. The next best performer was 52100 steel rubbing against TiC-coated steel, this yielding a friction coefficient of 0.12 but this relatively low value was only maintained for some 250 disc revolutions. All the unlubricated SiC tests failed to start. The results demonstrate convincingly that ceramics do require lubrication if they are to be fully exploited as tribological components.

6.3 The way forward

The low friction and long durability of MoS_2 films applied to sliding contacts of silicon nitride suggest that sputtered MoS_2 would provide highly effective in-vacuo lubrication for silicon nitride ball bearings. In principle, the low friction observed under sliding would give rise to low bearing torque and low torque noise (in comparison with steel bearings similarly lubricated). Additionally we might expect, from our pin-on-disc results, that the low-torque lifetime of MoS_2-coated silicon nitride bearings to be longer than that of their steel counterparts. Since precision ball bearings manufactured from silicon nitride are now available commercially these propositions could readily be tested (and it is our intention to do so in the near future). Low torque bearings have potential uses in high-precision pointing mechanisms and in spacecraft mechanisms required to operate under conditions of microgravity. Dry-lubricated ceramic components are also of interest for high temperature applications.

The use of zirconia interlayers also led to impressive enhancements in MoS_2 film-life under vacuum when tested in conjunction with bearing steel balls. However, the use of zirconia riders (balls) led to very poor film life. It would be instructive to repeat this work as and when high quality zirconia balls become available. Such a study would confirm, or otherwise, that the observed poor film durability was due to abrasion by poor quality (ie. rough) zirconia balls.

7. CONCLUSIONS

1. In general, unlubricated ceramics yield high friction coefficients in both air and vacuum. Of the unlubricated tests performed, the combination of Si_3N_4 vs. Si_3N_4 in vacuum yielded the lowest friction.

2. Ceramics lubricated with sputtered MoS_2 give low friction in vacuum and their friction and wear properties compare favourably with those of MoS_2-coated steel tested under similar conditions. MoS_2 film durability was highest on zirconia and silicon nitride substrates.

3. In air, MoS_2-lubricated ceramics exhibit higher friction and reduced durability than in vacuum. This is consistent with the in-air behaviour of sputtered MoS_2 on bearing steel.

4. Tests involving zirconia riders (balls) led to rapid wear-out of sputtered MoS_2 films. Failure is ascribed to abrasion of the MoS_2 films by the relatively rough-surfaced zirconia riders.

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