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Study of solid lubrication with MoS_2 coating in the presence of additives using reciprocating ball-on-flat scratch tester

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Abstract. Molybdenum disulphide (MoS_2) based solid lubricant mixtures containing zirconia and graphite were prepared in the laboratory and coated on steel specimens. The experiments were done using reciprocating scratch test for various numbers of cycles. The results showed that the addition of zirconia and graphite into the MoS_2 lubricant has improved its properties in terms of both friction and wear. In addition, it was observed that the presence of moisture affects the life of the lubricating film. It was shown that at high temperature the moisture evaporation enhanced the coating performance of the film.

Keywords. Friction, reciprocating scratch tester, wear, molybdenum disulphide.

1. Introduction

A solid lubricant material is used as powder or thin film to provide protection from damage during relative movement and to reduce friction and wear. Other terms commonly used for solid lubrication include dry lubrication, dry-film lubrication, and solid-film lubrication. Although these terms imply that solid lubrication takes place under dry conditions, fluids are frequently used as a medium or as a lubricant with solid additives. The desirable properties of a solid lubricant (Lansdown 1999; Lipp 1975; Lansdown 1992) are to (a) provide low, constant and controlled friction between two bearing surfaces, (b) be chemically stable over the required temperature range, and not to attack or damage the bearing material, (c) preferably adhere strongly to one or both bearing surfaces so that it is not rapidly lost from the bearing, (d) have sufficient resistance to wear to provide a useful life, (e) be non-toxic and economical. These requirements are so demanding that most substances are ruled out for one reason or another and the vast majority of solid lubrication involves only few substances like graphite, MoS_2 and poly tetra fluoro ethylene (PTFE).

Solid lubrication can be implemented where unusual circumstances exist which make oils and greases unsuitable. For e.g. in space applications, where lubricating oils are avoided due

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to their out gassing under vacuum, and high temperature conditions. Other uses include food industries, textile industries, etc., where products are likely to get contaminated with oils and greases. Under such situations the solid lubrication plays an important role. Some of the materials used as solid lubricants are: (i) layer-lattice compounds such as MoS_2, graphite, tungsten disulphide, titanium disulphide, molybdenum diselenide, tungsten diselinide, titanium diselinide, graphite fluoride and calcium fluoride, (ii) polymers such as PTFE, nylon, acetol, polymide and polyphenylene sulphide, (iii) metals such as Lead, Gold, Tin, Silver and Indium, (iv) other inorganics such as Molybdic oxide, Lead monoxide, Boron trioxide and Boron nitride. In addition, many of them are incorporated in composites. These are mixed materials in which, for example a lubricant powder may be dispersed in a strong solid to reduce its friction, or a binder may be used to hold together the particles of a powdered lubricant. Even though some commercial solid lubricants are available for aviation and space applications, they are highly un-economical to use for general engineering purpose. So efforts are being put to develop solid lubricant, which is economical and easy to prepare in-house.

Graphite and MoS_2 are important solid lubricants, and these have a layered structure (Lansdown 1992, Brudnyi & Karmadonov 1975). The layer consists of flat sheets of atom or molecules, and the structure is called a layer-lattice structure. The important effect is that the materials can shear more easily parallel to the layers than across them. They can therefore support relatively heavy loads at right angle to the layers while still being able to slide easily parallel to the layers. This property is being effectively used for lubrication process. The coefficient of friction is more or less equal to the shear stress parallel to the layers divided by the yield stress or hardness perpendicular to the layers. Because the low friction only occurs parallel to the layers, it follows that these solid lubricants will only be effective when their layers are parallel to the direction of sliding. It is also important that the solid lubricant should adhere strongly to the bearing surface; otherwise it would be easily rubbed away and gives very short service life.

In this study, MoS_2 (Lansdown 1999; Lipp 1975; Lansdown 1992; Brudnyi & Karmadonov 1975; Bartz 1971; Holinski & Gansheimer 1972) was used as solid lubricant. It is a dark blue-grey or black solid, which feels slippery or greasy to the touch. It has hexagonal layer-lattice structure (Lansdown 1992; Brudnyi & Karmadonov 1975). It has the advantages such as (i) practically no tendency to flow, creep or migrate, (ii) minimum tendency to contaminate products or environment (most suitable for textile and food industries), (iii) very low volatility enables it to be used in high vacuum, (e.g. space applications), (iv) chemical inertness enables it to be used in reactive chemical environment, (v) generally stable to radioactivity, (useful in nuclear power plants), (vi) good load carrying capacity and (vii) non-toxic. Unlike graphite, MoS_2 does not depend on the presence of adsorbed vapors to act as lubricant. Therefore, it can be used satisfactorily in high vacuum and temperatures. MoS_2 begins to oxidize at 350°C in air, although it can still be used for short periods up to 450°C. The oxidation produces molybdic oxide (MoO_3) which itself is a fair lubricant at higher temperature but wears rapidly. Apart from oxidation, it is stable to most chemicals, but is attacked by strong oxidizing acids and alkalis. On the whole MoS_2 is a versatile and useful material where oils or greases cannot be used or do not have sufficient load-carrying capacity.

There are many ways to produce MoS_2 films (Lansdown 1999): (a) The flotation process involves floating fine MoS_2 powder on the surface of a liquid and lifting it off on to the surface of the flat metal plate. Liquid is removed by draining and drying. A weakly adherent thin uniform film of the powder is left on the metal surface. The film appears to consist of randomly oriented crystals, and has been extensively used for research purpose, but not for use in practical machinery. (b) MoS_2 films can also be produced by the use of dispersion of MoS_2 powder in volatile liquids. The dispersion can be applied to a solid surface by dipping, brushing, or spraying and the liquid is then allowed to evaporate, either at room temperature or with additional heating. Film produced by this process is very soft and randomly oriented. (c) Strong adherent MoS_2 films can be produced by the use of bonding agents. Bonding agents can be of organic or inorganic in nature. MoS_2 powder mixed with the binding material in appropriate proportion can be applied on the component by dipping, brushing, or spraying using the suitable carriers. The component is cured under high temperature to allow for evaporation of carriers and reaction to take place for binding material to get hardened. This type of coating is useful for practical applications since there will be a good adherence between base material and MoS_2 mixture.

Efforts have been made to study the influence of MoS_2 on friction and wear during sliding (Takahashi & Okada 1975; Brudnyi & Karmadonov 1975; Tanaka *et al* 1981; Ravindran & Ramasamy 1984; Roberts 1989; Klenke 1990; Jain & Shukla 1991; Ogilvy 1993; Cunningham *et al* 1994; Semenov 1995; Donnet 1996; Hirvonen *et al* 1996; Morimoto 1997; Wahl *et al* 2000). Takahashi & Okada 1975 examined the frictional properties of MoS_2 on a microscopic scale. Ravindran & Ramasamy (1984) studied the influence of film thickness and normal load on the frictional properties of phenolic-bonded MoS_2 films on steel substrates. It was shown that the friction coefficient varies with the square root of the ratio of the film thickness to the load. Klenke (1990) analysed the effectiveness of boric oxide (B_2O_3), when used as an additive in MoS_2, for substrate temperatures ranging from 21°C to 316°C. Jain & Shukla (1991) studied the interaction of metallic stearates with graphite and MoS_2 combinations on friction, wear and seizure using four-ball wear and EP testers. The interaction of stearates of chromium, copper and aluminium with solid lubricants has been shown to be beneficial in reducing wear and friction, and can reduce seizure, whereas stearates of sodium and tin have shown the reverse effect. Cunningham *et al* (1994) have done a series of experiments to study the friction coefficients and durability of steel substrates coated with thin films of MoS_2 for various values of the substrate roughness and the film thickness. The authors concluded that a kind of surface textures is important to optimize the performance of tribological coatings. Morimoto (1997) studied the effect of MoS_2 on the coefficient of friction and the wear of the steel ring against silicon nitride and cemented carbide in comparison with mineral oil lubricants. It was concluded that MoS_2 was more effective in reducing the coefficient of friction and the wear of the ring than the oil lubricants.

2. Experimental

EN8 steel material of size 20 × 20 × 10 mm thick, were used as test specimen to lubricate MoS_2 and to perform scratch tests. The specimen surface was ground against the specific emery sheet, such as 220 or 600 grades, to get required roughness value. The test specimens were then cleaned with soap solution to remove the dust and oil contents from the surface. These were then ultrasonically cleaned in the acetone bath.

To carry out MoS_2 coating on the steel flat, the binding material used was sodium silicate (Na_2SiO_3) with and without additives. For doing this, two types of test samples were prepared one with bare surface with different surface roughness and the other with phosphated surface with zinc and manganese.

It is found from the literature (Milne 1957; Rajagopal & Vasu, 2000; Kundra 1974) that the phosphate coating provides good base for lubrication on steel surface. Phosphate coatings being microporous, the lubricant is trapped into the interstices between phosphate crystals and improves its bonding strength. Further, a phosphate coating offers a much larger surface area for the lubricant to hold on. A phosphate coating also gives excellent protection against corrosion. Hence, phosphating operation of steel specimens was carried out successfully in the laboratory. MoS_2 solid lubricant with zirconia and graphite as additives were prepared in the laboratory and tested on phosphated specimens.

Phosphate coating is produced on the steel by the action of phosphating solution. The solution is based on phosphoric acid and contains acid metal salts such as Zinc, Manganese, etc. Under normal conditions these metal salts remain dissolved in the solution. However when a cleaned steel surface is introduced in such a solution at a specific concentration and temperature, dynamic conditions set in at the metal/liquid interface. The localized change in the pH conditions at the metal/solution boundary reduce the solubility of the metal phosphates and cause the same to be deposited on the surface. A good phosphated surface should have features like (i) relatively uniform, non-conductive, and corrosion resistant, (ii) an integral chemical coating and is not alkaline in nature, (iii) phosphate coating, due to its microcrystalline structure, provides much more surface area for stronger anchorage to MoS_2, (iv) the coating provides capillaries and micro-cavities which inturn provide good mechanical interlocking with MoS_2, (v) the phosphate coatings are non-metallic.

2.1 *Preparation of lubricant*

Sodium silicate that is available in powder form has been mixed with double distilled water in different proportions and MoS_2 coatings were tried on the specimen. The coatings used to be either very hard (high friction) or very soft (bad adhesion). An optimum proportion was experimentally arrived at, which is as follows: 4·6 gm of sodium silicate was added to 100 gm of water. The solution was mixed thoroughly until all the salt dissolved in the water. Usage of this solution with MoS_2 gave reasonably good coating for lubrication purpose.

A ratio of one part of MoS_2 and two part of sodium silicate solution, by weight, was found to be an optimum composition for brushing the mixture on the specimen. To increase the wear life of MoS_2 lubricant film it was decided to add zirconia as an additive into the lubricant and mixture was prepared as follows.

(a) Zirconium nitrate $[Zr(NO_3)_4 \cdot H_2O]$, when heated in the furnace at 1000°C for 8 h get converted into zirconia $[ZrO_2]$, which is basically a ceramic material.
(b) Fine zirconia powder (less than 5 microns particle size) separated from the bulk through filtration process.
(c) Lubricant was prepared in the following ratios by weight.
$MoS_2 + ZrO_2 + Na_2SiO_3$ solution in (1) 1:0·05:2·10, (2) 1:0·08:2·16.

To reduce the friction coefficient of the film it was decided to add graphite into the lubricant. Graphite gives very low friction in humid conditions. Lubricant was prepared in the following ratio by weight. MoS_2 + Graphite + $ZrO_2 + Na_2SiO_3$ solution in 1:0·25:0·08:2·66.

The procedure for lubricating the test specimen is as follows:

(i) Heat the prepared specimen to around 80°C so that the moisture present on the specimen will get evaporated.
(ii) Apply (brush) the MoS_2 mixture, 2–3 coats.
(iii) Heat and cure the specimen for one hour at 150°C.

(iv) Remove loose MoS_2 powder from the surface using soft brush.
(v) Burnish the specimen for 15 minutes using ball tumbler.

The coating thickness varies from 15 to 20 microns. This was measured using coating thickness measuring instrument, such as electro-magnetic induction and eddy-current techniques, and can be used for non-magnetic coatings on a ferromagnetic substrate or for non-conducting coatings on a conducting substrate.

Reciprocating scratch test was performed on these steel flats using steel ball as indenter to study the coefficient of friction and ability of coating to withstand the number of cycles during the scratching operation. The details of scratch testing machine are explained elsewhere (Menezes *et al* 2006(a–e)). Scratch testing machine consists of vertical slide and horizontal slide which are driven by stepper motors. The vertical slide is used for indentation purpose whereas the horizontal slide for scratching. Vertical slide can be detached from the screw rod so that it can apply its dead weight on the specimen, which enables to conduct constant load scratch test. The load cell mounted on the vertical slide can detect both normal load and traction forces experienced during sliding. A LVDT mounted on the body can measure the movement of the vertical slide, i.e. the depth of the scratch. The scratching tool consists of 3 mm diameter steel ball, which was bonded using super glue to the holder. The speed and the number of strokes of horizontal slide can be programmed and the normal force, traction force, and depth of scratch can be acquired online on to the computer using data acquisition software. After the tests the plots of 'number of cycles' against 'friction force' are done for all samples. The test conditions are as follows:

- Normal load on the specimen (applied with 3 mm diameter steel ball): 30 N
- Stroke length (length of the scratch): 8 mm
- Slider velocity (forward and reverse stroke): 2 mm/s
- Failure criteria: if coefficient of friction $> 0{\cdot}2$
- Test conditions: ambient (temperature 20–35°C and humidity 50–90%).

Thus, reciprocating scratch tests are conducted in the following sequence. (i) Scratch test under dry sliding conditions between steel specimen and steel ball. (ii) Scratch test between MoS_2 coated steel specimen and steel ball. (iii) Scratch test between the phosphated steel specimens and steel ball. (iv) Scratch test between the phosphated steel specimens coated with MoS_2 and steel ball. (v) Scratch test between MoS_2 coated phosphated steel specimen with zirconia as an additive and steel ball. (vi) Scratch test between MoS_2 coated phosphated steel specimen with graphite and zirconia as an additives and steel ball. (vii) High temperature scratch tests.

3. Results

3.1 *Scratch test under dry sliding conditions between steel specimen and steel ball*

Experiments were carried out to study the dry friction characteristics of the rubbing surfaces of steel specimen having roughness, $R_a = 0{\cdot}19\,\mu$m. The initial coefficient of friction was around 0·13 and attained a value of 0·5 within 20 cycles. Similar tests were conducted on the specimen having $R_a = 0{\cdot}39\,\mu$m. The initial coefficient of friction was around 0·15 and attained a value of 0·5 within 20 cycles. From these results it is clear that the coefficient of friction between two steel surfaces is greater than 0·5 under non-lubricated conditions.

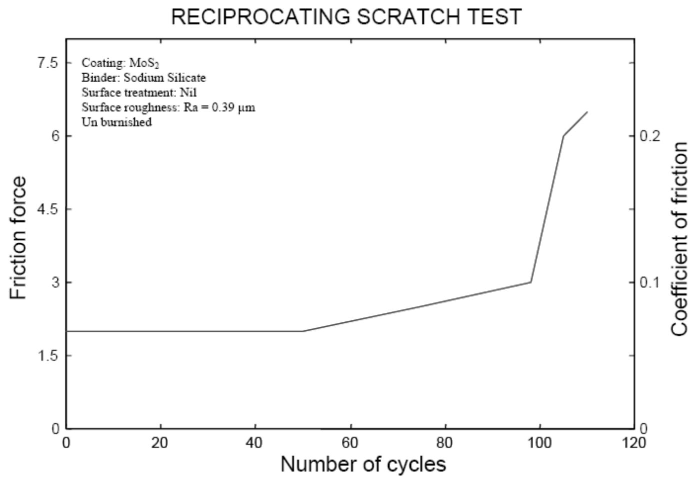

Figure 1. Variation of friction force and coefficient of friction with number of cycles when steel ball slid against MoS_2 coated steel specimen under un-burnished condition.

3.2 *Scratch test between MoS_2 coated steel specimen and steel ball*

Figure 1 shows the test results of the specimen which is coated with MoS_2. The binding material is sodium silicate. The roughness of the specimen before coating was maintained to $R_a = 0{\cdot}39\,\mu$m. The specimen was left un-burnished. The test result showed that the specimen failed at around 105 cycles. When the specimen was burnished for 30 minutes, as shown in figure 2, the specimen failed at 45 cycles. This gives a clear indication that the adhesion between the specimen and coating is getting weakened due to the impact of balls during burnishing process. When the roughness of the specimen before coating was maintained to $R_a = 0{\cdot}19\,\mu$m, under un-burnished conditions, the specimen failed at 115 cycles and under burnished (for 30 minutes) conditions, it failed at 55 cycles. Again, indicating that the adhesion between the specimen and coating is getting weakened due to the impact of balls during burnishing process and also the film thickness is getting reduced during burnishing operation.

From the above results it is clear that the adhesion between specimen and lubricant is poor and should be improved. It is found that the phosphating process (Milne 1957; Rajagopal & Vasu 2000; Kundra 1974) for steel specimen would improve the adhesion properties. As there is hardly any difference between the performance of the specimens which are having surface finish of $R_a = 0{\cdot}19\,\mu$m and $R_a = 0{\cdot}39\,\mu$m, for all future tests, specimens are finished with $R_a = 0{\cdot}19\,\mu$m value. The specimens are phosphated either with zinc or manganese phosphate. Over the phosphated surface, MoS_2 coatings are done and scratch tests are performed. The burnishing duration was fixed for 15 minutes, which is enough for orienting the layers of MoS_2. It is found that more duration of burnishing operation would start removing MoS_2 layer, which will reduce coating thickness.

3.3 *Scratch test between the phosphated steel specimens and steel ball*

The scratch test conducted on the zinc phosphated and manganese phosphated surface showed that coefficient of friction crossed 0·12 after 10 to 15 cycles. Figures 3a and b show the optical photographs of zinc phosphated and manganese phosphated surfaces, respectively.

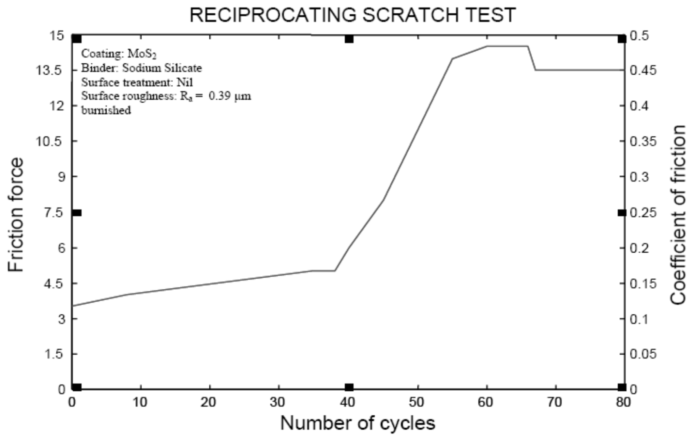

Figure 2. Variation of friction force and coefficient of friction with number of cycles when steel ball slid against MoS_2 coated steel specimen under burnished condition.

The porous structure of phosphated surface is clearly visible in the photographs. This type of porous structure provides more surface area and better mechanical interlocking for lubricant to adhere on it.

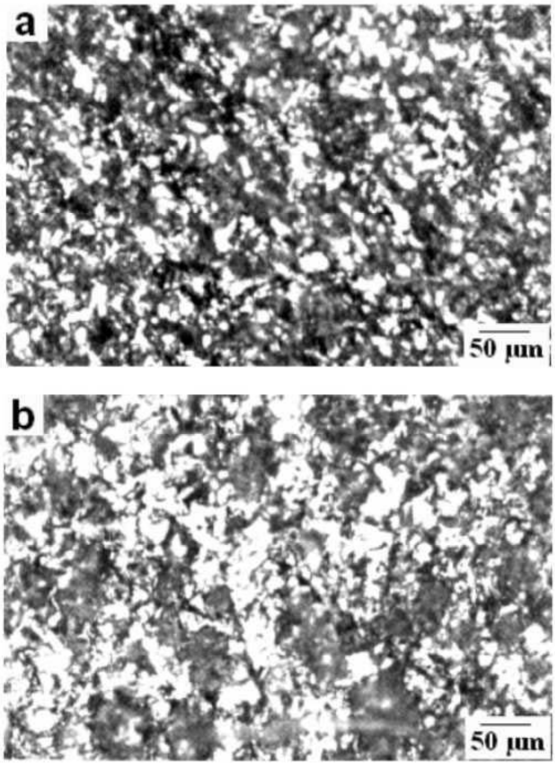

Figure 3. Optical photographs of **(a)** zinc and **(b)** manganese phosphated steel specimens.

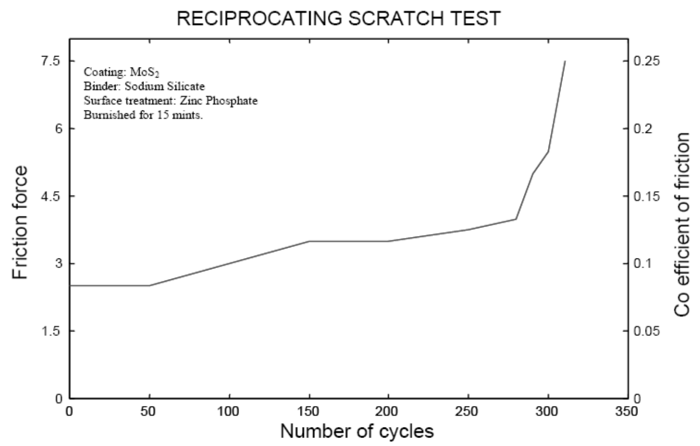

Figure 4. Variation of friction force and coefficient of friction with number of cycles when steel ball slid against zinc phosphated steel specimens coated with MoS_2 under burnished condition.

3.4 *Scratch test between the phosphated steel specimens coated with MoS_2 and steel ball*

Figure 4 shows the test results of specimen, which was zinc phosphated and then, coated with MoS_2. The specimen was burnished for 15 minutes. The scratch test results indicate that it has withstood around 300 cycles before failure and initial friction value is around 0·08. Wear rate was around 0·02 μm/cycles. When the specimen is un-burnished, the specimen withstood around 250 cycles before the coating failed. The initial friction value was around 0·1 and wear rate was around 0·023 μm/cycles. The burnishing orients the molecules of MoS_2 and compacts the film. Thus, it reduces the friction and improves wear properties.

For the case of manganese phosphated specimen coated with MoS_2, and then burnished for 15 minutes, the test underwent for 260 cycles before failure. The wear rate was around 0·02 μm/cycles. However, when the specimen is un-burnished it has taken around 190 cycles before failure. The wear rate is around 0·023 μm/cycles.

From the above results, it is evident that both zinc and manganese phosphated surface will provide a good base for lubricant. The porous structure of phosphated surface provides better mechanical interlocking for MoS_2 and binder.

Figures 5(a–d) show optical photographs of the scratched portion of the specimen at different stages of the test. Figure 5(d) indicates the coating failure. Here the bare surface started coming in contact with the steel ball.

3.5 *Scratch test between MoS_2 coated phosphated steel specimen with zirconia as an additive and steel ball*

Figure 6 shows the test results of the specimen coated with MoS_2 on zinc phosphated surface. Here 5% (by wt.) zirconia was added as an additive material. Specimen has taken around 590 cycles. The average wear rate was around 0·02 μm/cycles. Zirconia is basically a ceramic material, which offers high resistance to wear. Its crystal structure exists both in tetragonal and monoclinic. During scratching operation the strain induced structural transformation takes place. The metastable tetragonal structure converts into monoclinic thereby increasing its volume by 4%. If these zirconia particles are properly distributed in the matrix, then better results can be expected in terms of wear. X-ray diffraction test was carried out and found that

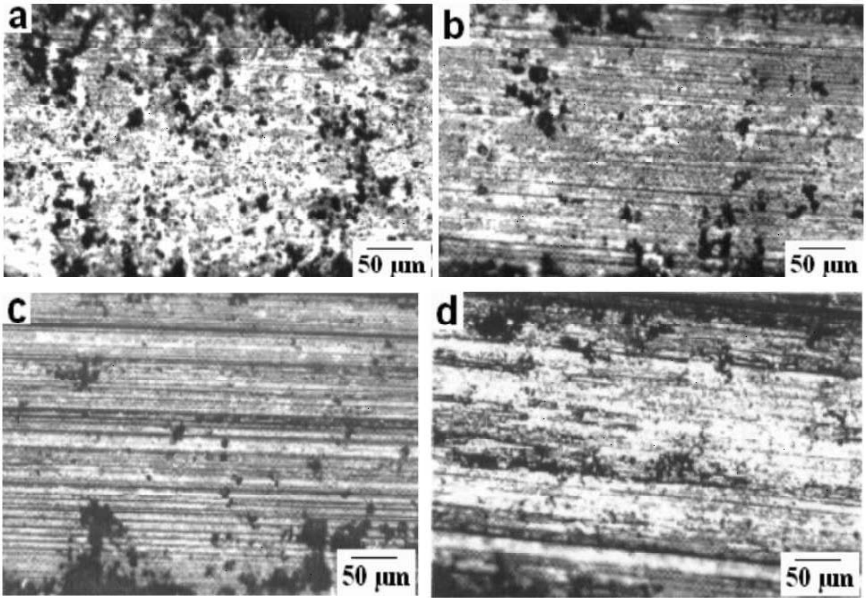

Figure 5. Optical photographs of the scratched portion of the specimen at the end of **(a)** 25, **(b)** 50, **(c)** 100, **(d)** 275 cycles.

more than 90% of its crystal structure was of monoclinic. If zirconia can be prepared such that more percentage of tetragonal structure exists in it and then it would give better result when used in the lubricant.

When the specimen was coated with MoS_2 on manganese phosphate base and 5% of zirconia added as additive failed at 340 cycles. Wear rate was around 0·016 μm/cycles. When the coating thickness was increased failure occurred at around 500 cycles. The initial friction

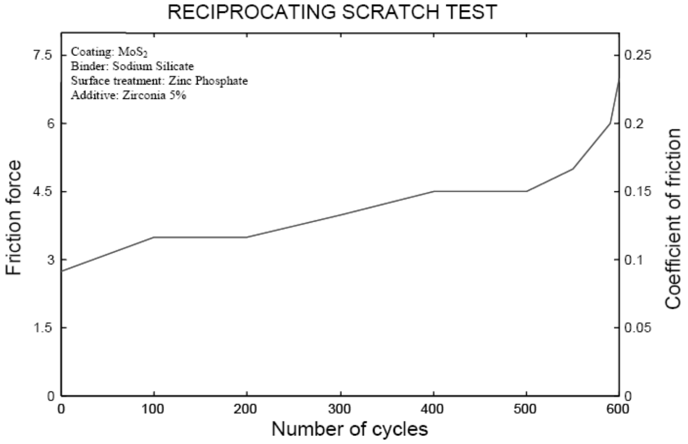

Figure 6. Variation of friction force and coefficient of friction with number of cycles when steel ball slid against zinc phosphated steel specimens coated with MoS_2 and 5% zirconia additive.

value was greater than 0·1. The wear rate was around 0·018 μm/cycles. From the above results it can be understood that the wear resistance has improved with the addition of zirconia.

When the tests were conducted with specimen coated with MoS_2 on zinc phosphated surface and 8% (by wt.) of zirconia is added as an additive material, the specimen has withstood around 1400 cycles before failure. Initial friction value was greater than 0·1. The average wear rate was around 0·014 μm/cycles.

When the tests were conducted with specimen coated with MoS_2 on manganese phosphate base and 8% of zirconia additive, it has failed at around 730 cycles. Wear rate was around 0·014 μm/cycles. When the coating thickness is increased then it has taken around 600 cycles. The initial friction value was greater than 0·1. The wear rate was around 0·010 μm/cycles.

From these results, it can be inferred that the wear resistance has further improved with the increase in the quantity of zirconia from 5 to 8%. From the above results it is certain that zirconia will improve the wear resistance property of the lubricant film. The only disadvantage was with initial friction coefficient, which was more than 0·1 in most of the cases.

Figure 7 shows the optical photographs of the scratched portion of the specimen at different stages of the tests. The random distribution of ZrO_2 can be seen clearly in all the photographs.

3.6 *Scratch test between MoS_2 coated phosphated steel specimen with graphite and zirconia as an additives and steel ball*

Graphite is one of the good solid lubricants to use in ambient conditions. It is a grey-black crystalline form of carbon in which the carbon atoms are arranged hexagonally in regular layers. In this hexagonal structure the bonds between the carbon atoms in a layer are strong

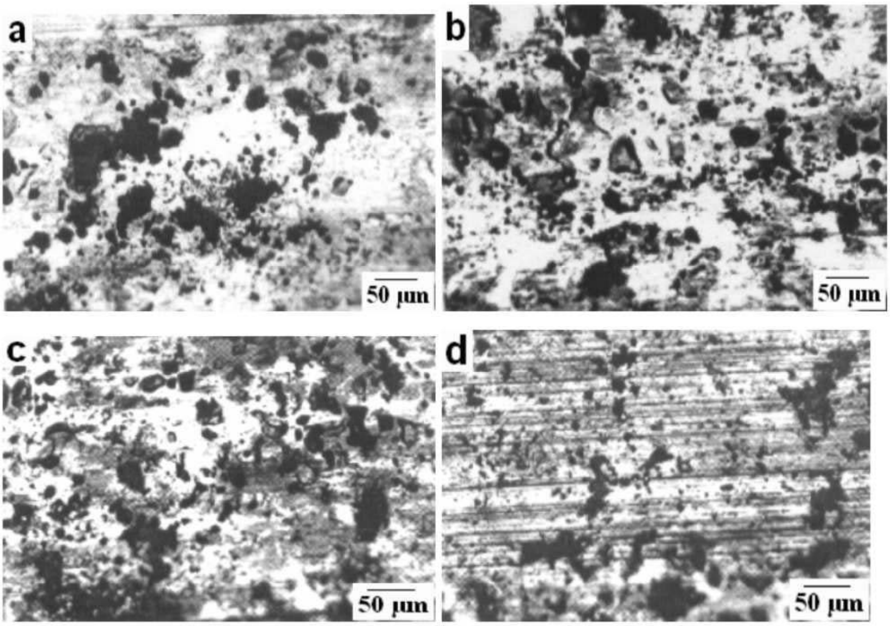

Figure 7. Optical photographs of the scratched portion of the specimen at the end of **(a)** 25, **(b)** 100, **(c)** 300, **(d)** 650 cycles.

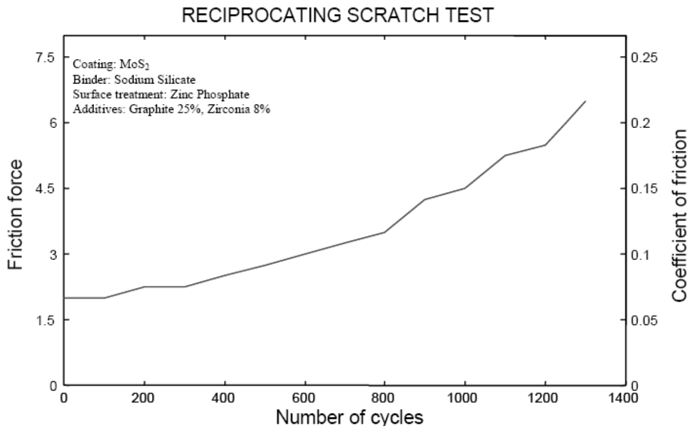

Figure 8. Variation of friction force and coefficient of friction with number of cycles when steel ball slid against zinc phosphated steel specimens coated with MoS_2 and 8% zirconia and 25% graphite as additive.

chemical covalent bonds where as the bond between the layers are weak Van der Walls forces. The layers will slide readily one over the other with the application of shear stress. The property of graphite is such that it shows good natural low-friction behaviour when it is contaminated by water vapor or other condensable vapors.

From the previous experiments it is observed that even though the addition of zirconia has improved the wear properties but this also increased the initial friction value. To possibly reduce the friction it was decided to add graphite into the lubricant.

Figure 8 shows the test results of the specimen coated with MoS_2 on zinc phosphated surface. Here 8% of zirconia and 25% of graphite (by wt.) are added as additive materials. Specimen has withstood around 1250 cycles before failure. Initial friction coefficient is around 0·07 and crossed 0·1 after 600 cycles. The average wear rate was around 0·01 μm/cycles.

When the specimen was coated with MoS_2 on Manganese phosphated surface and when 8% of zirconia and 25% of graphite (by wt.) are added as additive materials, the specimen has withstood around 1525 cycles before failure. Initial friction coefficient was around 0·053 and crossed 0·1 after 1300 cycles. The average wear rate was around 0·018 μm/cycles.

3.7 *High temperature scratch tests*

To carry out high temperature tests, the specimen was coated with MoS_2 on manganese phosphated surface, 8% of zirconia and 25% of graphite (by wt.) were added as additive materials. Three types of high temperature experiments were conducted. In the first type, the specimen was subjected to reciprocating scratch test at 200°C. The result is shown in figure 9. The test was carried out for 5500 cycles. The specimen has not shown any sign of failure even at 5500 cycles. It has maintained the friction coefficient as low as 0·05. However, same specimen when tested at ambient condition had failed at 600 cycles.

In the second set of experiment, the specimen was heated up to 200°C and then allowed to cool down to room temperature. The scratch test was conducted after 90 minutes of cooling. The specimen has taken around 3000 cycles.

In the third set of experiments, the specimen was heated up to 200°C and then allowed to cool down to room temperature. The scratch test was conducted after two days. The specimen failed at 600 cycles.

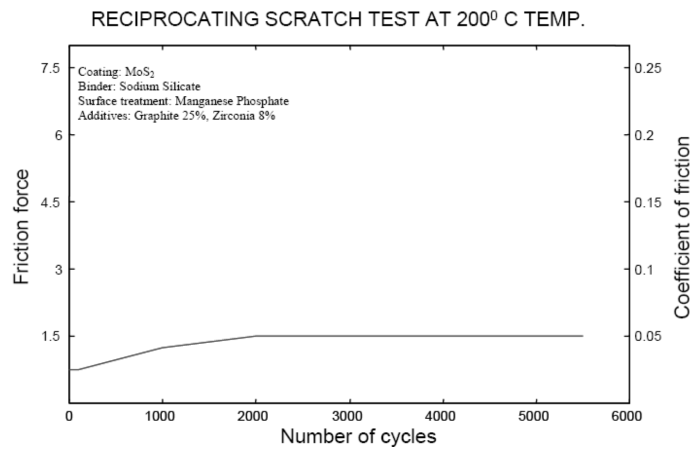

Figure 9. Variation of friction force and coefficient of friction with number of cycles when steel ball slid against manganese phosphated steel specimens coated with MoS_2 and 8% zirconia and 25% graphite as additive at 200°C.

From the above three tests it is clearly understood that moisture present in the air played an important role in terms of wear rate and friction coefficient. The behaviour of MoS_2 film in the presence of moisture was not found to be good. As seen in the first high temperature test, the moisture evaporated and then film performed well. The second test clearly indicated that sufficient time was not given for the specimen to absorb moisture completely. That was the reason it could sustain more number of scratches. The third test was one where specimen had fully absorbed moisture to its saturation level and the result was comparable with those of ambient conditions.

Similar tests were also conducted on the specimen that was coated with MoS_2 (without additives) on zinc phosphated surface. Effect of moisture was observed in all the tests.

In the literature, the effect of humidity on the friction for the case of MoS_2 films has also been reported. Pritchard & Midgley (1969) reported that the friction of molybdenum disulphide was governed essentially by the amount of water physically adsorbed. The authors concluded that humidity affects the adhesion component of friction and therefore the intercrystallite forces; the wear rate of the lubricant layer also increases as more water is adsorbed.

4. Discussion

MoS_2 lubricant with sodium silicate as a binder has been developed and tested on the steel specimens. From the literature it was found that phosphated surface can provide better base for lubrication, and hence the process of phosphating was developed and tested in the laboratory. The MoS_2 coatings were done on the phosphated specimens and tested. The test results revealed that the adhesion between phosphated surface and the lubricant film is much better than the adhesion between bare surface and the lubricating film.

MoS_2 coating was done on phosphated steel specimens. Scratch test was performed on these specimens. The average wear rate was found to be 0·215 μm/cycles and initial coefficient of friction values was around 0·08.

MoS_2-based lubricant with zirconia as an additive material was prepared in the laboratory. Zirconia was developed in the laboratory from zirconium nitrate. Zirconia is a ceramic material, which offers high resistance to wear. Initially, coating was done with 5% of zirconia in the lubricant. Scratch test results showed that the average wear rate was 0·018 μm/cycles and initial coefficient of friction value was around 0·1. Coating was also carried out with 8% of zirconia in the lubricant. The tests were performed and the average wear rate was found to be 0·0125 μm/cycles. The coefficient of friction at the beginning of the test was around 0·11.

From the previous experiments, it was observed, even though the addition of zirconia has improved the wear properties but this also increased the initial friction value. The problem of increase in friction coefficient has been overcome by the addition of graphite into the lubricant. The graphite is a solid lubricant to use in humid conditions. The property of graphite is such that it shows good natural low-friction behaviour when it is contaminated by water vapor or other condensable vapors. The lubricant was prepared with 25% graphite and 8% zirconia as additive material. The lubrication was carried out on phosphated steel specimens and scratch tests were preformed. Scratch tests showed the average wear rate of 0·003 μm/cycles and initial coefficient of friction value was around 0·06. The addition of 8% of zirconia and 25% of graphite to the lubricant increased the wear resistance of the film by about 72% and reduced the friction coefficient to 0·06 from 0·11.

The tests conducted under high temperature (200°C) clearly showed that the moisture present in air played an important role in terms of wear rate and friction coefficient. The behaviour of MoS_2 film in the presence of moisture was not found to be good. At high temperature the moisture evaporated and thus enhancing the film performance. The results of scratch tests conducted for 5500 cycles at 200°C had not shown any sign of failure and maintained the friction coefficient at 0·05. The specimen that was heated up to 200°C then allowed to cool down to room temperature. The scratch test was then conducted after 90 minutest of cooling. The specimen failed at around 3000 cycles. Another series of tests were conducted after two days under similar conditions. The specimen failed at 600 cycles. These tests clearly demonstrated the effect of moisture on MoS_2 film.

5. Conclusions

- The addition of zirconia and graphite into the MoS_2 lubricant improves the MoS_2-based lubricant properties in terms of both friction and wear.
- The moisture present in air also plays an important role in terms of reducing friction coefficient and wear rate. At high temperature the moisture evaporates and enhances the coating performance of the film.

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