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Effect of Humidity on Friction of Molybdenum Disulfide Films Produced by Thermal Evaporation on Titanium Substrates

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Authors' contributions

This work was carried out in collaboration between both authors. Authors BRM and AHJ performed experimental work, data analysis and preparation of the paper. Both authors read and approved the final manuscript.

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ABSTRACT

MoS_2 is one of the most advanced solid lubricants that have been used in many machineries. In this work, the effect of humidity on the friction of MoS_2 solid film lubricant is investigated using a pin-on-disk tribometer. MoS_2 film was coated by a thermal evaporation method on a titanium substrate. Steel and aluminum pins were used for the evaluation of friction coefficient of MoS_2 films using a tribometer. The tests showed that with an increase in humidity, the coefficient of friction also increased. In practical applications at high humidity levels, the MoS_2 films could be covered by condensed water molecules. Therefore, an experiment to understand the effect of water film on the variation of friction coefficient was also conducted. The results showed that the coefficient of friction remained constant for experiments using both aluminum and steel pins, and then the coefficient of friction increased once the water film was evaporated by the heat generated due to friction. An optical and XRD characterization of the MoS_2 film was also conducted.

Keywords: Friction coefficient; tribology; humidity effect; molybdenum disulfide; thin film.

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1. INTRODUCTION

Extensive research on the friction and wear of solid lubricants has been conducted after the emergence of space technology. Astronomical applications require the lubricants to work under extreme conditions of temperature, vacuum, and atmosphere. It is challenging to design and implement liquid lubrication systems for these applications. Fueled by need, a substantial amount of investigations on the mechanisms and experimental validations of tribological properties of different solid lubricants has been reported. Two-dimensional materials such as MoS_2 and graphene gained significant attention and interest due to their symmetric two-dimensional layered structure. This structure allows its layers to slide over each other with lower shear resistance than metals like aluminum and steel. For instance, MoS_2 films have a shear resistance of only 25 MPa [1]. Because of the unique crystal structure of MoS_2, the tribological performance can be substantially modified by using advanced manufacturing processes that help to design and model grain formation and inclusions at a microscopic level.

One of the established processes to improve the tribological properties of film lubricants is alloying [2]. MoS_2 is usually alloyed with chromium and titanium to improve the friction and wear performance under humidity [3]. Carbon is another material added to dichalcogenides to reduce oxidation [4]. Other methods of increasing wear life include additive manufacturing techniques such as powder metallurgy or laser sintering. These methods involve mixing two-dimensional materials in a metal oxide or polymer matrices, which prolongs wear life [5,6]. The wear life of MoS_2 can be increased under high contact pressure, a property highly exploited in space applications [1,7]. The microstructure and hardness of the sputtered films can be altered by changing the deposition rates and the coating atmosphere to enhance the performance of the film [8].

Most of the current research on the friction and wear of MoS_2 films focus on increasing the wear life and corrosion resistance by processes such as multilayer film deposition [8,9], metal matrix composites [10-12] and polymer composites [13,14]. Krantz et al. has conducted some research to quantify the effect of oxidation and humidity on the deterioration of MoS_2 films during storage [15,16]. Also, the effect of humidity on friction and wear of industrial grade titanium plate has been conducted [17]. However, publications on the real-time performance evaluation of MoS_2 film lubrication working under different levels of humidity are rarely reported. Tribological performance of thin films also depends on coating methods and substrate. In this study, the effects of humidity and presence of water film on the tribological performance of MoS_2 film produced by thermal evaporation on titanium substrate were investigated.

2. EXPERIMENTAL PROCEDURE

The manufacturer's specification of the titanium substrate used for these experiments is shown in Table 1. The titanium plate surface was mechanically polished using an automatic grinding machine. Aluminum oxide abrasive grinding tool was used for this operation. The ground samples were again polished using a liquid suspension containing 0.25 µm size silicon carbide, to get a smoother appearance. The roughness of titanium plate after this process was in the range of 0.130-0.150 microns. The polished plates were then cleaned for 5 min. in an ultrasonic cleaner, rinsed with distilled water, sprayed with ethanol, and dried under a heat gun.

The tribology tests were conducted using a custom-made pin-on-disk tribometer [18]. It consisted of a cantilever with a cylindrical pin having a spherical tip of 6mm diameter attached at the free end. The pin had provision to allow the addition of different weights for experiments at different loads. Two strain gauges were attached to the lateral faces of the cantilever to measure the deflections. The test specimen was screwed to a rotating platform and rotated at a constant speed. Once the platform reached steady rotation, the pin, with normal weight, was placed carefully so that the initial interaction between the pin and the titanium plate was measured with the least amount of error. Due to friction, a lateral force was exerted on the cantilever, which deflected the strain gauges. The inner strain gauges underwent compression, whereas the outer one was subjected to tension. The piezoelectric property of these strain gauges gave rise to a differential voltage, which was collected via a data acquisition system and was interfaced with a computer running custom LabVIEW software (Serial No. 3KT54V1). Real-time deflection and frictional force data were collected and saved for further analysis. A normal load of 1.2 N was used for all test runs.

Table 1. Chemical composition of titanium substrate

Weight %	Ti	C	Fe	N	O	H
Titanium TA2 Grade	Balance	0.10	0.30	0.03	0.25	0.015

The maximum contact pressure between the pin and MoS_2 film was calculated using Hertz's equation for contact between a sphere and plate, as shown in Equation (1) [19].

$$P_{max} = \frac{3F}{2\pi w^2}, \quad (1)$$

Where, w and F are the width of the wear track and applied normal load, respectively. For this work, the pin-on-disk tribology test was conducted to plot the variation of coefficient of friction with time on the MoS_2 sample produced by thermal evaporation. An X-Ray Diffraction analysis of the coating was conducted to characterize the coating. Experiments were conducted using steel and aluminum pins under a relative humidity of 15% RH, 35% RH, and 50% RH. In order to control humidity levels, the experiments were conducted in a closed ambient while passing air with different humidity levels. The humidity presented above was measured using a digital humidity meter (calibrated) attached to a side of closed chamber. A water film was produced by dipping the MoS_2 coated sample in distilled water, followed by drying under a stream of air.

The crystallinity of the films was examined with the X-ray diffraction (XRD, Cu, Kα radiation, PANalytical X'Pert Pro MPD). The ultra-violet (UV) and visible double beam spectrometer (UV-1650 PC Shimadzu) was used to measure the optical reflection of sample with respective to an aluminum surface at an incident angle of 5º. The optical images were recorded with a high-resolution optical microscope (Karl Suss MJB3).

3. RESULTS AND DISCUSSION

3.1 MoS_2 Film Characterization

Microscopic photograph of the coated samples on both titanium and glass substrates are shown in Fig. 1. The glass substrate was used for the X-Ray Diffraction analysis to characterize the coating. The MoS_2 film on glass is very smooth and its average surface roughness was evaluated to be 0.009 μm whereas the average surface roughness of MoS_2 film on Ti plate is 0.134 μm. Fig. 2 indicates the X-Ray Diffraction (XRD) analysis of the MoS_2 film on the glass substrate. The XRD pattern indicated that the MoS_2 film has an amorphous structure due to lack of significant peaks.

Fig. 3 shows the optical reflectivity of pure titanium and MoS_2 film coated substrate as a function of wavelength with reference to Al coated glass surface. The incident angle of beam was kept standard 5 degree to the normal of the surface. The reflectivity of titanium sample is smaller than aluminum surface due to the scattering of light beam at the rough Ti plate. The reflectivity of MoS_2 coated titanium was much lower than the pure titanium substrate primarily due to the absorption of light by MoS_2 layer. Optical reflection spectroscopy gives qualitative information about MoS_2 layer and its thickness profile. Also, such information can be used to monitor the in-situ film thickness motoring related to coating technology.

3.2 Relative Humidity - 15%

For the test conducted at 15% RH, the variation of the coefficient of friction values with the number of cycles is shown in Fig. 4. At initial stage, a high friction coefficient was observed, which gradually dropped to a steady value with time for both steel and aluminum interactions. This experiment gave a mean coefficient of friction value of 0.08 for steel and 0.15 for aluminum pins interacting with MoS_2.

The friction coefficient for molybdenum disulfide film decreases with an increase in contact pressure [1]. The contact pressure is less during the run-in stage, with time, the contact pressure between the pin and the film gradually increases, thus decreasing the coefficient of friction to a constant value.

The optical micrographs of wear tracks for both aluminum and steel pins at 75x magnification are shown in Fig. 5. The tracks were thin, and a higher contact pressure value was observed in comparison to other experiments. The wear track thickness was less for steel than for aluminum though the experiment was conducted for a greater number of cycles. At lower humidity levels, the friction coefficient of MoS_2 films is lower for steel and aluminum.

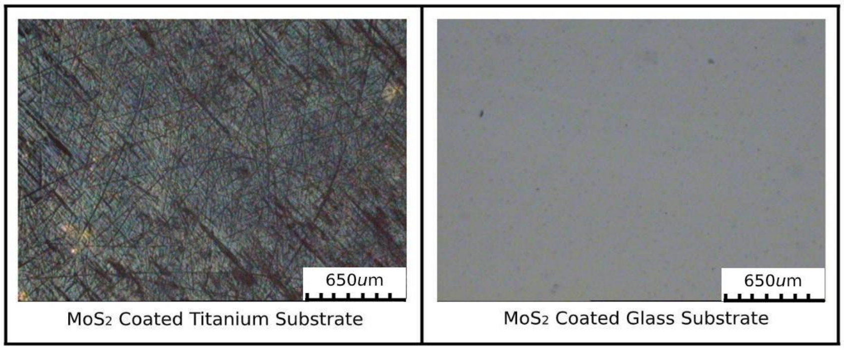

Fig. 1. The optical microscopic photograph of the film produced by thermal evaporation at 75x magnification on titanium and glass substrates

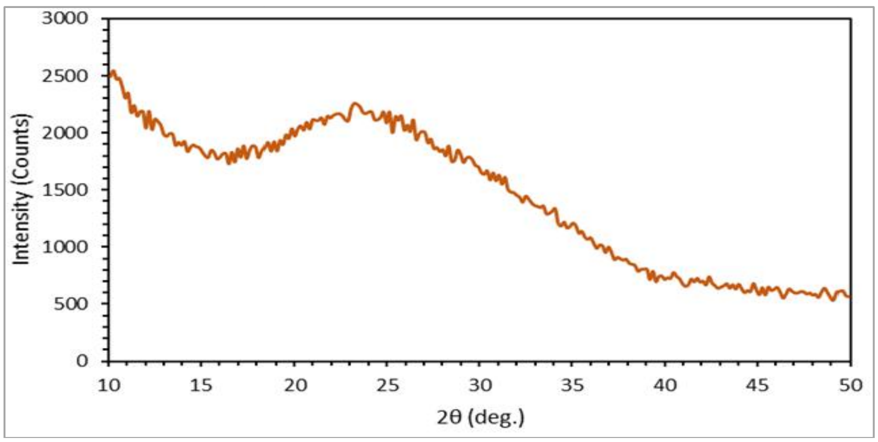

Fig. 2. X-Ray diffraction pattern of MoS_2 film on glass substrate

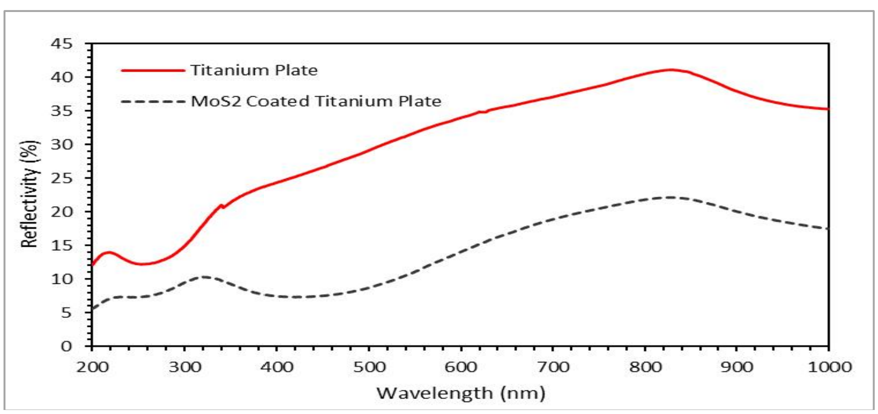

Fig. 3. Reflectivity analysis of pure titanium and MoS_2 coated sample

3.3 Relative Humidity - 35%

As the atmospheric humidity was increased to 35% RH, the coefficient of friction values also increased. The coefficient of friction appeared to be more for aluminum than for steel. Since aluminum is softer than steel, the tip surface could be worn off quickly, thus increasing the interfacial friction. Fig. 6 shows the variation of friction coefficient with the number of cycles for both steel and aluminum pairs.

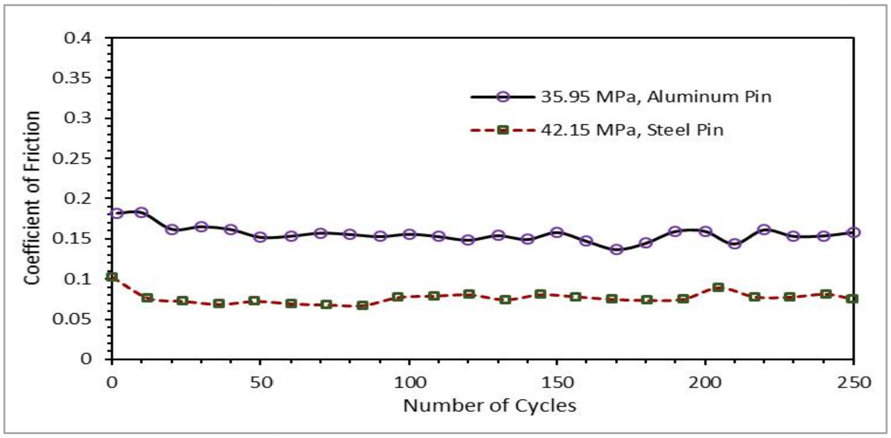

Fig. 4. Coefficient of friction values for steel /MoS_2 and aluminum/ MoS_2 interactions at 15% RH: steel/42.15 MPa; aluminum/35.92 MPa; 120 rpm

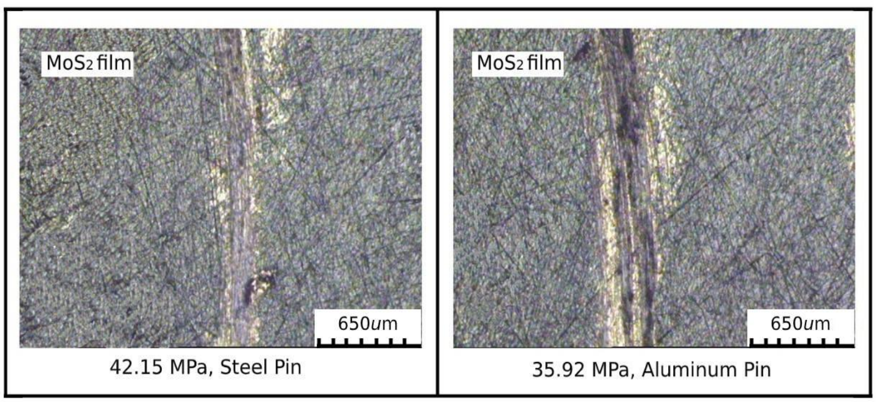

Fig. 5. Optical microscope photograph of wear track of steel/MoS_2 and aluminum/MoS_2 interaction at 15% RH at 75x magnification: steel /42.15 MPa; and aluminum/35.92 MPa

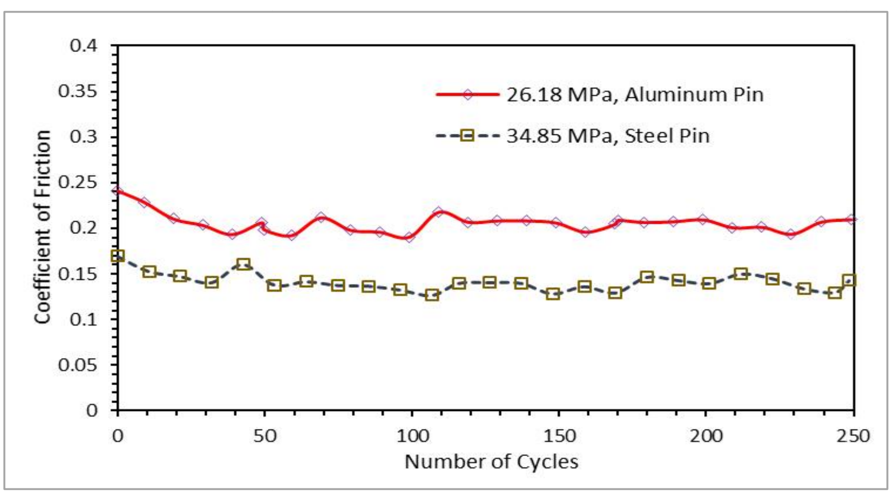

Fig. 6. Coefficient of friction values for steel /MoS_2 and aluminum/ MoS_2 interactions at 35% RH: steel/34.85 MPa; aluminum/26.18 MPa; 120 rpm

The thickness of the wear track increased for both steel and aluminum experiments when the humidity level was increased to 35%. For steel/MoS_2 interaction, a small amount of wear was present outside the significant wear track. For MoS_2 film lubrication, the friction and wears decrease with an increase in the contact pressure [1]. Since spherical pins were used for the tribology test, the contact pressure decreases away from the pin-tip substrate contact point. Small wear observed outside the wear track was due to the sliding of the pin substrate surfaces at reduced contact pressure. Since aluminum was more easily wearable than steel, the pin contact surface becomes rougher easily compared with steel, hence significant wear on the film surface was caused even away from the contact point; thus, generating a broader wear track, as shown in Fig. 7. An experiment has been conducted by Krantz et al. [16] to understand the shelf life of MoS_2 coated gears up to 60% RH; they concluded the wear and friction of molybdenum disulfide films are affected by humidity. With high humidity levels, the coefficient of friction and wear increased, giving more substantial proof for this study.

3.4 Relative Humidity-50%

At a relative humidity of 50%, the friction coefficient increased substantially. The average coefficient of friction is about 0.3 for aluminum and about 0.2 for steel. The friction coefficient value was close to the pure titanium/aluminum interaction, which was around 0.5. However, at this humidity level, steel/MoS_2 tribopair showed better performance compared with pure titanium/steel interaction [13,20]. These experiments showed that the coefficient of friction value is dependent on atmospheric humidity. As humidity levels were increased, the friction coefficients also increase. The variation of coefficient of friction with the number of cycles is shown in Fig. 8.

The optical micrograph of the wear tracks is shown in Fig. 9. The contact pressure value was slightly larger for steel MoS_2 interaction than for aluminum MoS_2 interaction. The wear tracks were more deeply grooved for aluminum than steel. Liu et al. has conducted a molecular dynamics simulation at the interfacial surface of the water and silicon carbide [21]. The results showed an increase in surface energy due to the formation of hydrogen bonds by dissociation of the water molecules. Since friction is determined by the surface energy interactions between the sliding surfaces, this increased the friction coefficient. Also, with an increase in humidity, the oxidation of MoS_2 increases, thus deteriorating the quality of the film formed.

3.5 Presence of Interfacial Water Film

The increase in friction is due to the adsorption of water molecules on the MoS_2 film surface, which increases the surface energy [21]. In practical application, there is a significant chance of condensation of atmospheric water vapor on the interfacial surface at high atmospheric humidity. An experiment was conducted to check whether the frictional behavior of the tribo-system was affected if a layer of water film was present on the surface of the MoS_2 film. In order to mimic this condition, the MoS_2 coated titanium sample was sprayed with distilled water and excess water was removed by a stream of air using an air gun. The readings were collected at 50% relative humidity and 26ºC.

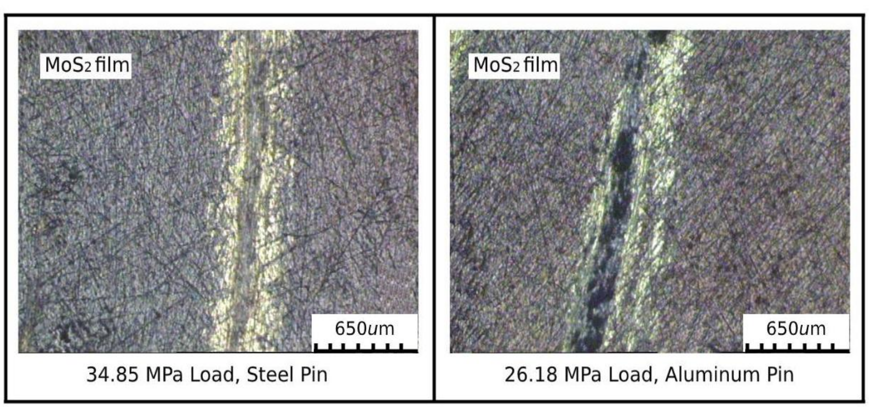

Fig. 7. Optical microscope photograph of wear track of steel/MoS_2 and aluminum/MoS_2 interaction at 35% RH at 75x magnification: steel /34.85 MPa; and aluminum/26.18 MPa

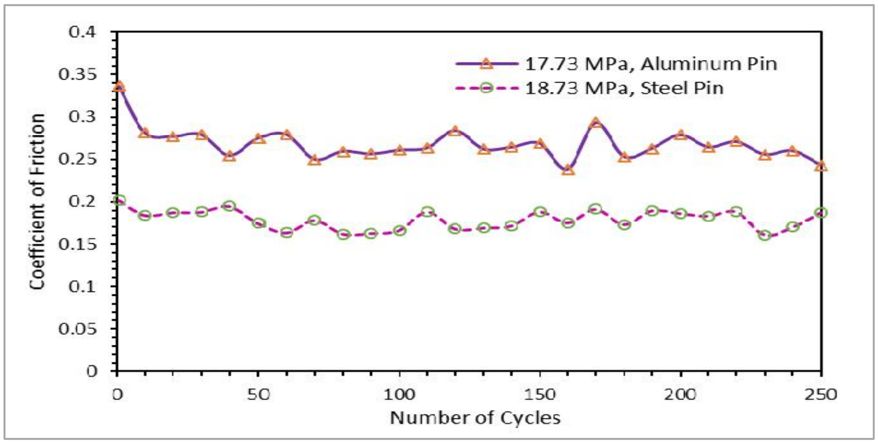

Fig. 8. Coefficient of friction values for steel /MoS_2 and aluminum/ MoS_2 interactions at 50% RH: steel/18.73 MPa; aluminum/17.73 MPa; 120 rpm disk speed

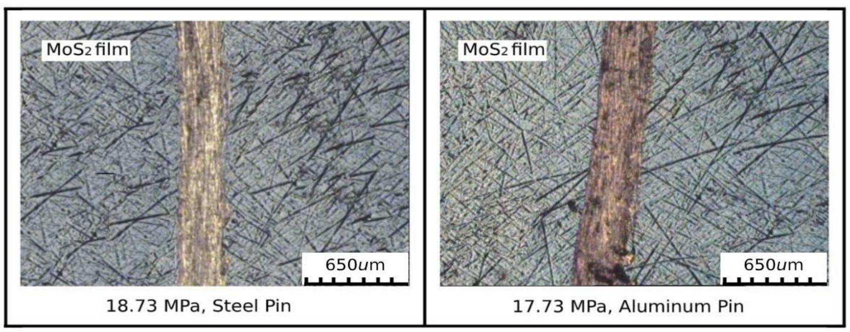

Fig. 9. Optical microscope photograph of wear track of steel/MoS_2 and aluminum/MoS_2 interaction at 50% RH at 75x magnification: steel /18.73 MPa; and aluminum/17.73MPa

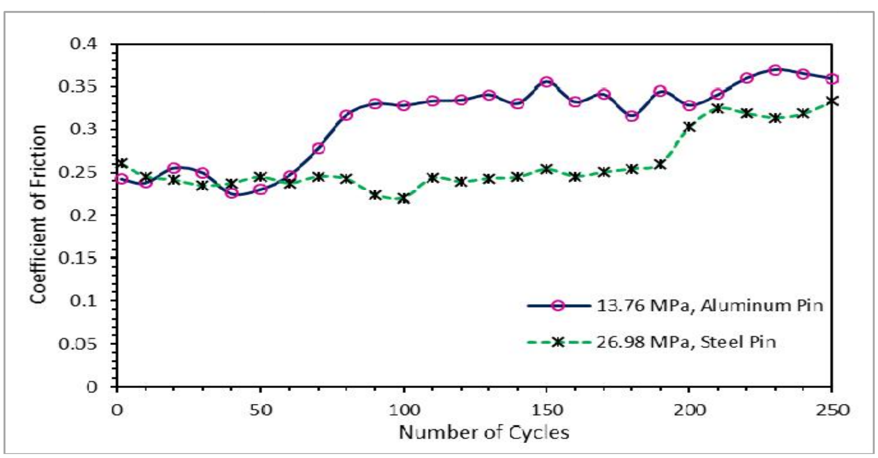

Fig. 10. Coefficient of friction values for steel /MoS_2 and aluminum/ MoS_2 interactions at 50% RH and presence of water film: steel/26.98 MPa; aluminum/13.76 MPa; 120 rpm

Initially, the variation of the coefficient of friction was almost the same for aluminum and steel pin. However, the coefficient of friction increased with time. For steel pin, this increase happened around 240 cycles, but for the aluminum pin, the initial increase happened much earlier, at about 60 cycles.

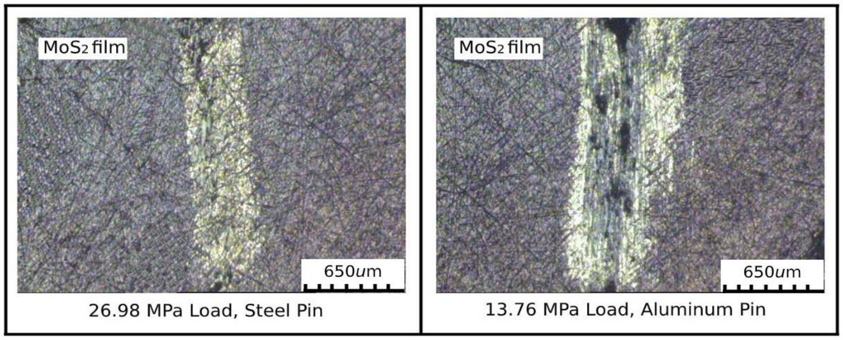

Fig. 11. Optical microscope photograph of wear track of steel/MoS_2 and aluminum/MoS_2 interaction at 50% RH and the presence of water film at 75x magnification: steel /26.98 MPa; and aluminum/13.76 MPa

The wear tracks formed with water film on the surface were different from the ones in the previous experiments. For steel, the wear tracks were thinner in comparison to aluminum. The significant wear was observed for aluminum but for steel, the life of the film was high.

In this experiment, the water film masked the contacting surfaces and prevented the formation of metal-metal contact tribofilm. Hence during the initial stages of the experiment, the variation of coefficient of friction appeared to be similar for both aluminum and steel pins as the contact happened between the water layers of pin and film. Only after the interfacial water film was evaporated by friction heat, the surfaces come in contact with each other. Since the coefficient of friction value was higher for aluminum than steel, the interfacial surfaces were exposed earlier for aluminum.

4. CONCLUSION

The friction coefficient for MoS_2 interacting with steel and aluminum pins was verified as a function of humidity. As the humidity was increased, the coefficient of friction also increased. The friction coefficient for steel/ MoS_2 interaction was lower than aluminum/MoS_2 tribopair. Since aluminum is softer than steel, wear on the aluminum pin was more significant, which changes the surface morphology of the aluminum /MoS_2 tribosystem leading to a higher friction coefficient. The experiment to capture the effect of condensed water molecules at high humidity levels showed that water masked the interacting surface of MoS_2 film and metal pin. Pin-film interaction happened only after the water film was evaporated by heat generated by friction. Also, a higher friction coefficient was observed during the surface interaction in the presence of a water film.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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