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Case Study

Revisiting the dwell effect on friction behavior of molybdenum disulfide

T.F. Babuska [a], B.A. Krick [b], N. Argibay [a], M.T. Dugger [a], M. Chandross [a], J.F. Curry [a,*]

[a] Materials, Physical, and Chemical Sciences Center, Sandia National Laboratories, Albuquerque, NM, 87185, USA
[b] FAMU-FSU College of Engineering, Florida State University, Tallahassee, FL, 32310, United States

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ABSTRACT

Solid lubricants, like molybdenum disulfide (MoS_2) are often used in space applications, and subject to prolonged periods where components sit stationary before use (i.e., during storage, transport or in between duty cycles). When sliding is resumed after dormancy, the friction behavior of MoS_2 can vary due to a variety of factors such as accrued adsorbates and oxidation. This phenomenon, referred to as the dwell or stop-time effect, was first investigated over 50 years ago, and is characterized by an increase in the coefficient of friction and a prolonged run-in back to steady-state friction. After nearly five decades, the fundamental driving mechanism for the dwell effect is still not well understood. In this work, the dwell-effect for MoS_2 coatings is studied through intermittent sliding experiments with dwell times ranging from 30 s to 48 h (172,800 s) at pressures from 7×10^{-9} to 2×10^{-1} torr. Vacuum pressure was varied to investigate the role of surface contaminants (i.e., water). Results suggest that the change in the coefficient of friction is driven by interactions of water with the sliding interface. The role of microstructure on the dwell effect was investigated using nanocrystalline sputter-deposited and highly oriented spray-deposited MoS_2 coatings. Results show that the shear-modified surfaces of sputter-deposited coatings have a $\sim 2 \times$ smaller dwell effect than spray-deposited surfaces due to the reduction of potential edge-sites that can interact with contaminants. Additionally, intermittent sliding experiments after vacuum annealing show that the contribution of latent water can be minimized by driving intercalated water from the coating.

1. Introduction

Tribological materials for applications in harsh space environments must exhibit a variety of characteristics such as low vapor pressures, low coefficients of friction in vacuum, performance over a wide range of operating temperatures, and radiation resistance [1]. Molybdenum disulfide (MoS_2) is a lamellar solid lubricant with a low steady-state coefficient of friction ($\mu < 0.05$) in inert and vacuum environments [2], making it an ideal candidate for low-cycle and single actuation mechanisms (i.e., antennas, solar panels, latches) [1]. A defining characteristic of MoS_2 friction is the initial transition from a higher initial coefficient of friction ($\mu > 0.1$) to a lower steady-state coefficient of friction, a process known as run-in. The run-in process results from the transfer of material across the shearing interface (i.e., formation of a transfer film) and shear-induced reorientation of surface MoS_2 lamellae to a basal orientation (parallel to the surface) to create a low shear-strength sliding interface [3–6].

Though the initial run-in behavior of MoS_2 is well known, a widely overlooked and often forgotten aspect of the friction behavior of MoS_2 is the tendency for the coefficient of friction to increase once sliding resumes following a period where the contact was stationary [7–11]. This is referred to as the stop-time or dwell effect. Specifically, this effect is characterized by an increase in the initial coefficient of friction and a prolonged run-in time to steady state friction. These effects have been observed in other materials such as diamond-like carbon (DLC) [12] and $MoSe_2$ [13] and are found in environments ranging from dry nitrogen [9] to ultra-high vacuum [7,8].

It is also well known that sliding in the presence of water and oxygen increases the coefficient of friction [14–16] and wear rate [17] of MoS_2. In oxygen rich, water deficient environments (i.e., dry air), the coefficient of friction for MoS_2 is low ($\mu < 0.05$) as the removal of surface oxides occurs more quickly than they can form and inhibit sliding [16]. Water rich, low oxygen environments (i.e., humid N_2 or low-vacuum) have been shown to increase the coefficient of friction ($\mu \sim 0.09$) [16, 18], and with the inclusion of oxygen (i.e., humid air) MoS_2 performance continues to degrade ($\mu \sim 0.13$). Though the environmental

* Corresponding author.
E-mail address: jcurry@sandia.gov (J.F. Curry).

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sensitivity of MoS_2 is partly a result of surface oxidation or water adsorption causing increases in shear strength [19,20], Curry et al., [21] proposed that the friction behavior of MoS_2 is a structurally-driven process dependent on the defect-density of the sliding interface. Interactions between MoS_2 edge sites with water inhibit the coalesce of MoS_2 lamella during sliding, resulting in smaller (i.e., more defective) MoS_2 crystallites with a higher shear-strength.

Developing a fundamental understanding of the dwell effect is important for aerospace applications where critical systems that require only a single or limited number of actuations stay dormant for days to decades. This requires coatings to solely operate in the transient friction regime as steady-state friction can never be achieved. Haltner [9] hypothesized that surface contamination from adsorbates (i.e., water) was the cause for increased coefficients of friction once sliding resumes but noted that degassed samples had no observable increase in friction after an 800-h dwell. Matsunaga et al., [7,8] expanded on the Haltner's hypothesis by including the contribution of diffused bulk contaminants, such as water, using Fick's law of diffusion and attributing changes in friction to the contaminant concentration gradient at the sliding interface. Khare et al., [16] observed that diffused water in the bulk increases the coefficient of friction by 2 × compared to a coating that has been annealed. Additionally, the bulk water content was found to increase with humidity and exposure time and decrease with increased interface temperature [16].

It is also important to consider the role of surface oxidation on friction behavior after prolonged periods of dormancy. While the dwell effect is observed on the timescale of minutes to hours, exposure to water and oxygen for days to months without sliding, known as aging, has been shown to increase wear [22–24], initial coefficients of friction [25], and to prolong run-in times to low friction [26]. Oxidation of MoS_2 has been shown occur in oxygen environments devoid of moisture [27], but oxidation is more severe in environments containing both oxygen and water [27–29]. The formation of oxidative species on MoS_2 has been observed with even brief exposures to air, as little as 10 min, [28]. The adverse effects of aging can be mitigated by creating basally-oriented coating microstructures through deposition [23,30] or shear-induced reorientation [21,31,32]. Basally-oriented microstructures have been shown to limit oxidation by limiting pathways for oxygen to penetrate the coating and minimizing the reactive edge sites that promote oxidation [26,31].

In this work, we revisit the dwell effect using controlled experiments in dry nitrogen and low to ultra-high vacuum environments to understand the effects of adsorbates and diffused water on the increases in friction. Additionally, the relationship between microstructure and dwell time is investigated using pure highly-oriented nitrogen spray-deposited MoS_2 and randomly-oriented sputter-deposited MoS_2 coatings.

2. Methods

2.1. Material synthesis

Pure molybdenum disulfide (MoS_2) coatings were nitrogen spray deposited on 1″ diameter x 1/8″ thick 440C steel disks (average roughness (R_a) ~ 20 nm) with a thickness of ~100–250 nm resulting in basally oriented MoS_2 films, similar to those described by Curry et al., [30]. Sputter-deposited pure MoS_2 coatings were manufactured by Tribologix Inc. using DC magnetron sputtering. A 3" MoS_2 target at 150 W with 1.5 mTorr Ar and 30 V bias was used to deposit 1 μm thick coatings on 440C steel flats (R_a ~20 nm).

2.2. Tribological testing

Intermittent sliding experiments (Fig. 1) were performed at varying vacuum pressures (Figs. 2 and 3) in a custom pin-on-disk bi-directional reciprocating ultra-high vacuum tribometer consisting of a double leaf cantilever and capacitance probes (Lion precision), similar to that described by Krick et al., [33]. A 1/8" dia. 440C ball with a normal load of 200 mN (584 MPa) and a sliding speed of 2 mm/s was used for all tests in vacuum. Chamber pressure was varied between 1×10^{-1} to 1×10^{-9} torr by controlling turbo pumping speed and leaking dry N_2 gas (99.999%) through a crystal seat leak valve. Samples were initially characterized by running 500–2000 sliding cycles, or until the steady-state coefficient of friction was below 0.05. Re-run in was studied by pausing sliding after running the sample in to steady-state and leaving the contact loaded for between 30 and 172,800 s (Fig. 1). Dwell times were randomly varied to minimize history effects on re-run-in. After the dwell time elapsed, sliding resumed for 500 cycles or until a steady-state friction coefficient <0.03 was attained. Where noted, samples were annealed before testing at 150 °C under 1×10^{-9} torr for 24 h to reduce adsorbed and latent water and contaminants. The change in friction coefficient (Δμ) was determined by taking the difference between the coefficient of friction for the first sliding cycle after resuming sliding (μ_i) and the friction coefficient averaged from the last 50 sliding cycles (μ_{ss}) of the previous run in the same location (i.e., μ_i - μ_{ss}) (Fig. 1).

Additional tribological experiments were run in dry N_2 (O_2 < 0.5 ppm, H_2O < 0.5 ppm) (Figs. 1 and 4) using a pin-on-disk bi-directional reciprocating tribometer with a ¼" diameter 440C ball, a normal load of 450 mN (470 MPa), and a sliding speed of 2 mm/s. Re-run in experiments were performed similarly to the vacuum experiments mentioned above with randomly varied dwell times between 10 and 36,000 s and 100 sliding cycles between dwells.

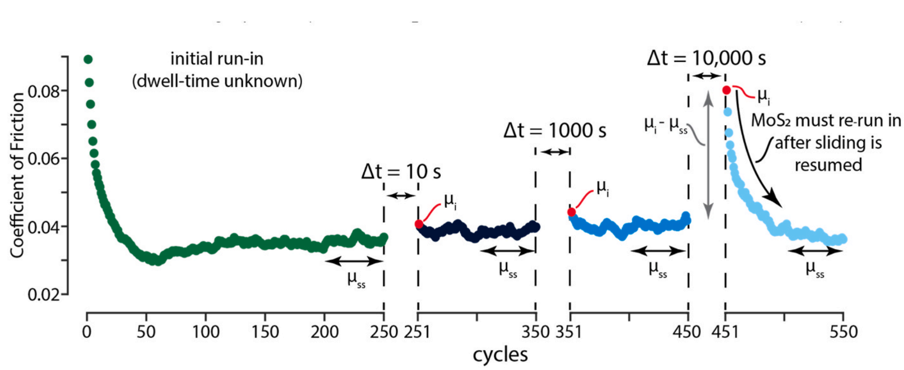

Fig. 1. Friction traces after dwell times of 10, 1000 and 10,000 s for N_2 spray-deposited MoS_2 in dry N_2 showing the dwell effect and defined friction values, steady-state friction (μ_{ss}), initial coefficient of friction once sliding is resumed (μ_i), and the change in friction after stopping (μ_i - μ_{ss}).

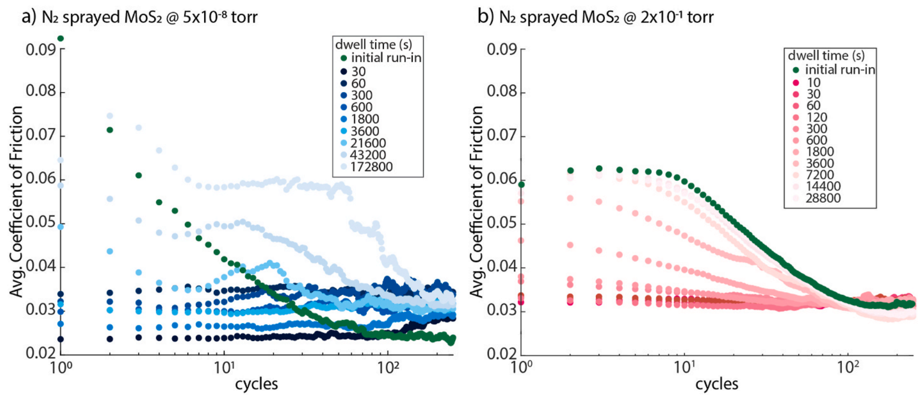

Fig. 2. Friction traces for N_2 spray MoS_2 coupons after increasing dwell times in a) 5×10^{-8} torr and b) 2×10^{-1} torr.

3. Results and discussion

3.1. Role of environmental contaminants on the dwell-effect of highly-oriented MoS_2

The re-run-in behavior of spray-deposited MoS_2 coatings for dwell times ranging from 10 to 172,800 s was determined at vacuum pressures ranging from 5×10^{-8} to 2×10^{-1} torr (Fig. 2) to investigate the role of contaminants (i.e., H_2O, O_2). The initial run-in behavior (shown in green in Fig. 2) shows a transition from a higher initial coefficient of friction (μ ~ 0.06–0.09) to a lower steady-state coefficient of friction (μ ~0.02–0.03) after 100 sliding cycles. This steady state value is maintained until the end of the experiment (250 cycles). There are minor differences in the initial run-in behavior between the experiments tested at 5×10^{-8} torr (Fig. 2a) and $\times 10^{-1}$ torr (Fig. 2b), such as number of cycles to run-in (~10 cycles to reach μ < 0.04 at 5×10^{-8} torr vs ~ 50 cycles at 2×10^{-1} torr) and initial coefficients of friction ($\mu_i \sim 0.09$ at 5×10^{-8} torr vs $\mu_i \sim 0.06$ at 2×10^{-1} torr). While differences in initial run-in could be due to the non-uniform coverage of MoS_2 inhibiting transfer film formation [30], we observe similar re-run-in behavior after longer dwell-times (>7200 s) at 2×10^{-1} torr, suggesting that prolonged run-in is due to the presence of adsorbates (i.e., water, oxygen) introduced while the contact was held stationary at higher vacuum pressures.

Fig. 2 demonstrates the severity by which the dwell-effect can change the coefficient of friction and, in cases of long dwell-times, revert the coating to the initial run-in state. To better highlight the dwell effect and the impact of time and contaminants, we plot the change in friction coefficient (Δμ) as a function of dwell time for pressures ranging from 7.7×10^{-9} to 2.2×10^{-1} torr (Fig. 3). In vacuum applications, water is the major constituent remaining in the chamber and the time required to form a monolayer of water is dependent on vacuum pressure. Here, we vary the overall vacuum pressure of the chamber to understand the role of water contamination and its concentration on the dwell effect. Haltner [9] and Matsunaga et al. [7,8] hypothesized that the dwell-effect was due to a combination of surface contaminants (i.e., water) and diffused water in the bulk. We observe that the change in the coefficient of friction as a function of dwell-time exhibits two distinct trends depending on the vacuum pressure (Fig. 3). At low vacuum (1×10^{-3} to 2.2×10^{-1} torr), we observe an increase in Δμ at 300 s while in high vacuum (1×10^{-6} to 7.7×10^{-9} torr) the time to increase is an order of magnitude greater at with Δμ increasing at ~3600 s.

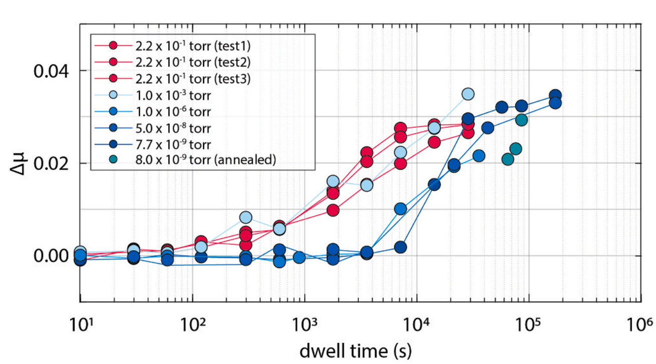

Fig. 3. Plot of the change in friction (Δμ) between the initial friction of a test following a defined dwell period (μ_i) and the steady-state friction of the experiment run before it (μ_{ss}) at various pressures.

The ~10 × increase in time for Δμ to increase between low and high vacuum is possibly due to changes in the contaminant source that drives the frictional changes. At low vacuum and ambient temperatures the time for a monolayer of water to form is 10 μs – 1 ms, assuming a sticking coefficient of 1. This is six orders of magnitude faster than at high vacuum, where a monolayer will form in ~2–300 s at 1×10^{-6} to 7.7×10^{-9} torr, suggesting that surface contaminants are driving the observed dwell-effect in the low vacuum regime [34]. As more environmental water is removed in the high to ultra-high vacuum regime, the dwell effect persists. This is likely caused by diffused water in the coating migrating to the sub-surface or sliding interface and inducing structural and chemical changes over time. This hypothesis, first proposed by Matsunaga et al., [8], is supported by experiments in which we vacuum annealed the coating and repeated the dwell experiments at 8×10^{-9} torr (Fig. 3). We observe that vacuum annealing decreases the magnitude of Δμ for similar dwell times compared to the pre-annealed coating. Latent water introduced during deposition can be driven from the bulk by the annealing process [35] and has been shown to reduce the steady-state coefficient of friction of MoS_2 by ~2 × in humid air [16]. During steady-state sliding in dry/inert environments where the concentration of water is low, the influence of latent water is unlikely to be observed because the surface is constantly evolving and wear events dominate changes in friction. Diffusion from the bulk or a sticking coefficient less than one, could also explain why the amount of time required to increase friction is longer than that required for the formation of monolayers of containments. Furthermore, it is possible that competitive adsorption with other containments (i.e., carbon) is driving changes in Δμ as pressure is reduced; this will be a topic of future work.

3.2. Role of coating microstructure on the dwell-effect of MoS_2

The microstructure of MoS_2 coatings can vary from basally-oriented, micron-sized crystallites [30] with nitrogen spraying to amorphous/nanocrystalline and randomly-oriented crystallites with sputter deposition [23,36–38]. Previous works studying the dwell effect by Haltner [9] and Matsunaga et al. [7,8] used MoS_2 pins compressed from MoS_2 powder, trapping moisture during processing that likely contributed to the observed dwell time behavior [35]. In this work, the dwell effect was compared for spray and sputter deposited coatings (Fig. 4).

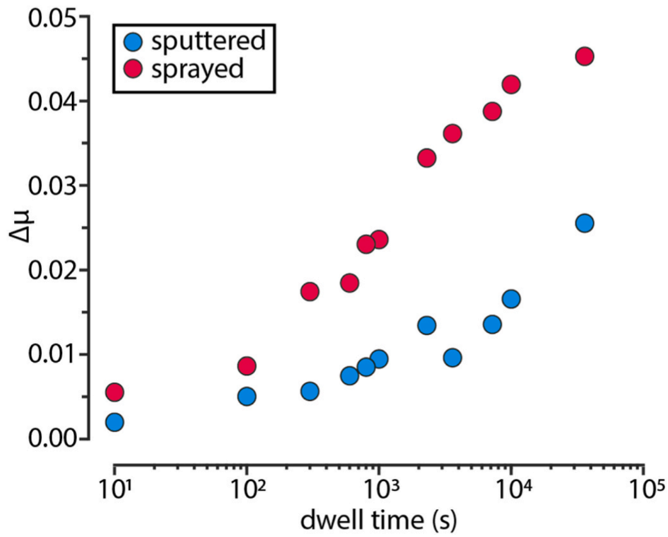

Fig. 4. The change in coefficient of friction for varying dwell times in dry N_2 for sputter and N_2 spray deposited pure MoS_2 coatings.

We observe that spray deposited films have a ~2 × higher Δμ than sputtered films after a 28,800 s dwell time in dry N_2 (Fig. 4).

The mechanism driving the dwell effect is dependent on the coating microstructure (Fig. 4). Despite the higher degree of environmental resilience found in sprayed films as a result of their higher degree of basal orientation and ordering [26,30], our results indicate that they are more susceptible to the dwell effect. Generally, it is thought that the size of surface lamellae in MoS_2 contacts will evolve to an equilibrium size based on the initial microstructure, contact conditions and environment [39]. Curry et al., [21] showed that surface microstructures of sputter deposited coatings formed by sliding in dry N_2 were less defective, with larger crystallites than the as-deposited coating due to shear-induced reorientation and coalescence of the initially nanometer-size lamellae. Additionally, the introduction of water inhibited structural-modifications and caused higher coefficients of friction. While previous studies focused on nanocrystalline sputter-deposited coatings, findings from Curry et al., [30] indicate that sliding on spray deposited coatings will also result in shear-induced refinement of the larger, micron-sized initial microstructure (i.e., sliding interface is more defective than as-deposited).

As the dwell effect is caused by environmental contaminants and bulk water interacting with sheared surfaces, the observed differences between sputter and spray deposited coatings (Fig. 4) could be a result of the less defective surface of sheared sputtered coatings compared to sheared spray deposited coatings. Even with similar contact conditions, the resulting shear-modified surfaces of coatings with different initial microstructures are different. Shear-modified sputtered surfaces have less potential edge sites for interactions with water than shear-modified spray-deposited surfaces, and this minimizes the changes in the coefficient of friction. The dwell effect, which can be thought of as short-term aging, is analogous to aging and oxidation that are known to cause oxidative etching and create smaller, more defective lamella [27].

The importance of bulk water on the dwell effect is highlighted by comparing coatings of different microstructures. Spray deposited coatings inherently have less trapped water than sputter deposited coatings because they are thin (~100–200 nm) and the high degree of basal orientation creates a tortuous pathway for water to diffuse to the surface. Sputtered coatings that are 5–10 × thicker (~1 μm) are usually low density and suffer from voiding [40,41] that can trap water and provide less resistance to diffusion, making them more susceptible to oxidation [26]. The observation that sputter deposited coatings, which are prone to latent water, have a smaller Δμ than spray-deposited coatings suggests that the dwell effect is dominated by surface contaminants and that latent water has a minor contribution to changing friction over time.

4. Conclusions

This work revisits the dwell effect for MoS_2 solid lubricant coatings and investigates the role of coating microstructure, surface contamination from water, and latent water on frictional changes with time during intermittent sliding experiments. Intermittent sliding at pressures ranging from 2.2×10^{-1} to 5×10^{-8} torr and dry N_2 was performed on sputter deposited and spray deposited MoS_2 coatings with dwell times ranging from 30 to 172,800 s. Results show that the increase in friction after stopping (Δμ) is a function of the base pressure, dwell time and coating microstructure. Exposure to contaminants (i.e. water) was controlled by varying vacuum pressure. In low vacuum (2.2×10^{-1} to 1×10^{-3} torr), the friction coefficient was observed to increase by 0.005 after 300 s and 0.01 after 1800 s between experiments. In high to ultra-high vacuum (1×10^{-6} to 5×10^{-8} torr), the friction coefficient takes longer to increase, with close to 10,000 s elapsing before a noticeable increase was observed. Dwell time experiments on sputter and spray deposited coatings in dry N_2 show that sputter deposited coatings are more resistant to changes in friction than spray deposited. Differences in friction behavior between coating microstructures is believed to be a result of shear-induced structural modifications of the surface, with sputtered coatings creating less defective surfaces that are more resistant to water-induced changes. Additionally, surface contaminants are shown to be the dominant cause of the dwell effect and any minor contribution of latent water can be minimized with vacuum annealing of the coating prior to testing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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