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Quantification of a Lubricant Transfer Process that Enhances the Sliding Life of a MoS_2 Coating

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A lubricant transfer process that enhanced the wear life of a MoS_2 coating has been identified and quantified. A steel ball sliding against a coated steel flat in reciprocating motion produced reservoirs at the turnaround part of the track ends, then emptied them, to provide replenishment similar to what is expected of liquid lubricants. The dynamics of the process were inferred from measurements of material loss and/or buildup in the track and on the ball; measurements were performed with Michelson interferometry and energy dispersive x-ray spectroscopy.

Keywords: Solid Lubrication, Molybdenum Disulfide, Lubricant Transfer, Coatings, Transfer Film

MoS_2 is a remarkable solid lubricant. It produces some of the lowest friction coefficients ever measured [1], and it can be used to lubricate sliding contacts at high contact stresses (on the order of GPa) even though its shear strength is only ~25 MPa. In addition, thin coatings of MoS_2 can withstand hundreds of thousands of sliding cycles, having overall wear rates <<1 nm/cycle. Although some of its behavior can be explained in terms of the bulk mechanical properties of MoS_2 (e.g. low friction coefficient in terms of the plasticity/shear strength), the high endurance has never been accounted for. Furthermore, while MoS_2 provides endurant sliding, it is well known that most of the coating is lost early in life, both in sliding [2,3,4,5] and rolling [6]. How, then, does the remaining lubricant sustain sliding?

We began this study to learn how a coating, worn heavily early in sliding, was able to endure the remaining 90% of sliding life [7]. But our investigation led to the recognition that the endurance was influenced not only by the coating wear rate but also by a lubricant replenishment process. This

paper identifies the transfer processes that contribute to the endurance of MoS_2 coatings in reciprocating sliding contact.

MoS_2 coatings were deposited by ion-beam assisted deposition (IBAD) to thicknesses from 285 to 1020 nm on a hardened steel substrate [7] precoated with a thin (30-40 nm) TiN interlayer [8]. Reciprocating sliding tests were performed at 4 mm/s sliding speed in a dry air environment (RH < 2%) using 6.4 mm diameter 52100 steel balls. The load was 9.8 N, providing an initial mean Hertzian pressure of 0.92 GPa. This paper reports results of one of the coatings, ~320 nm thick.

Friction and wear behavior were studied using a reciprocating sliding test methodology described in more detail elsewhere [7]. Briefly, each track was run a length of 5 mm for the first n = {1,3,10,30,100,...} cycles, and then the stroke length was shortened to 3 mm for an additional 2n cycles; a new ball was used for each track. Each track contained 3 turnaround points, and adjacent tracks had segments worn to duplicate sliding cycles. Friction coefficients were monitored throughout the tests, and failure (if reached) was defined as the number of sliding cycles the coating survived before the average friction coefficient reached 0.2. After the sliding tests, wear tracks, debris patches at the turnaround points and ball transfer films were examined by optical (Nomarski) microscopy. Michelson interferometry (MI) was used to measure wear track depths; maximum track depths are reported here. Energy dispersive x-ray spectroscopy (EDS) was used to estimate thickness of wear tracks, debris patches, and transfer films; beam conditions of 10 and 20 keV gave linear signal intensity with thickness (see e.g. Ehni and Singer[9]). Auger spectroscopy (AES) was used to identify film compositions.

Three distinct stages of coating wear can be seen in the depth vs. log cycle curve (Figure 1). Initially, there was no measurable wear (first 100 cycles, Stage I). This was followed by a period of rapid wear to nearly the full coating

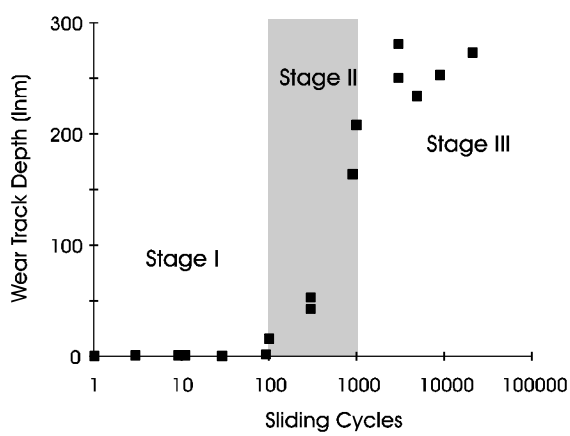

Figure 1. Wear track depths of IBAD MoS_2 coating measured by interference microscopy.

thickness during the next 1000+ cycles (Stage II), and finally a long period of low average wear for the remaining ~20000 cycles (Stage III). Friction coefficients remained low (0.02 to 0.06) throughout testing until late in sliding life near failure at ~21000 cycles.

Optical micrographs revealed wear morphologies and material transfer

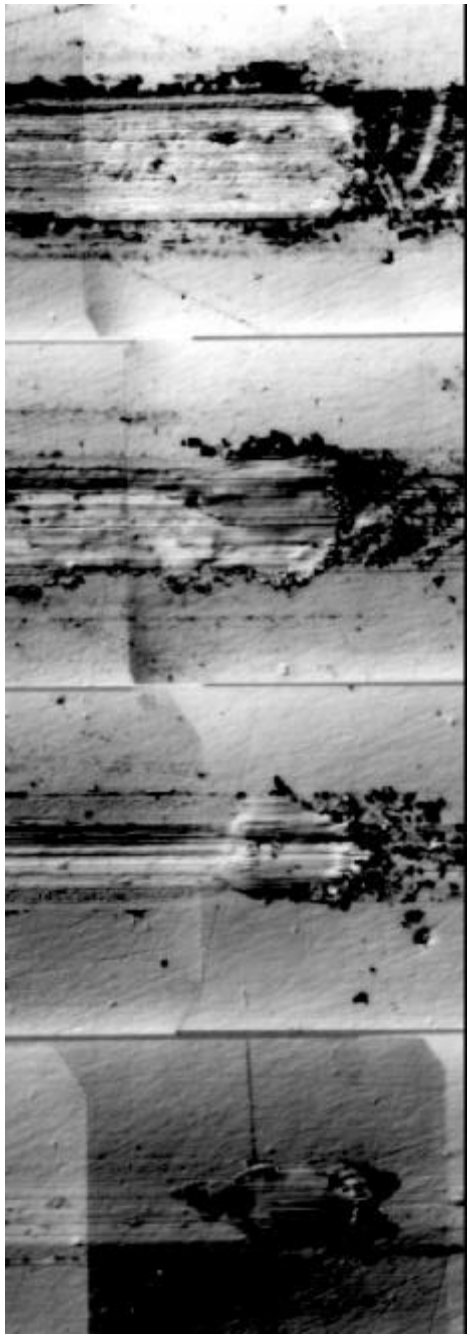

Figure 2. Micrographs of MoS_2 wear tracks and end patches, taken near the turnaround point in the middle of each track. (9000, 3000, 900, and 300 cycles from upper to lower.)

associated with each stage of wear. During Stage I, tracks appeared burnished, with only a small amount of debris at the turnaround points. The tracks at the turnaround points (Figure 2, 300 and 900 cycles); these debris patches appeared thinner or absent by Stage III (Figure 2, 3000 and 9000 cycles). In addition, during Stages II and III, material was ejected from the contact and deposited as loose debris along the edges and beyond the ends of the tracks.

On the ball surfaces, transfer films began forming during the first few sliding cycles. The transfer films were distributed in and around three distinct regions of the contact [10]. During Stage I, a very thin film of MoS_2 was transferred to the center of the contact (as determined by AES) and compacted debris was collected around the border of the contact zone (Figure 3a). During Stage II, more material was observed in and around the contact zone, and loose debris accumulated outside the contact zone (Figure 3b). The transfer process continued in Stage III, with increasing amounts of debris in and around the contact.

The buildup and depletion of material on track and ball surfaces was quantified using EDS and is shown in Figure 4. The material loss in the tracks agrees with the optical interferometry result (Figure 1), as expected for dense coatings. Most dramatic is the buildup (Stage II) then depletion (Stage III) of material in patches at turnaround points. The buildup occurred during the period of rapid coating loss from tracks in Stage II. Depletion of these patches to nearly zero thickness occurred during more gradual loss from tracks in Stage III. On balls, a continuous buildup of a relatively thin transfer film was observed. (The ball transfer film at failure contained significant amounts of oxygen, making it difficult to determine the thickness by EDS.)

The observed progression of wear morphology (Figures 2 and 3) along with quantification of thickness changes (Figure 4) can be interpreted as a lubricant transfer process diagrammed in Figure 5. In Stage I, a film transfers from the coating to the ball surface, and a small amount of the lubricant is deposited in patches at the turnaround points directly from the coating or indirectly from the ball transfer film (Figure 5a). During Stage II (Figure 5b), some of the lubricant lost from the track is transferred to patches at turnaround points and onto the ball; these deposits can act as reservoirs to replenish lubricant lost from the sliding contact. At the same time, some of the lubricant is ejected (as debris) beyond the track and ball contact zone and excluded from participation in the replenishment process. During the long period of sliding, Stage III (Figure 5c), the reservoir provided by the end patches becomes depleted as it replenishes lubricant to the track. Meanwhile, some material continues to be ejected and lost during the sliding process.

The individual processes illustrated in Figure 5 have been documented in earlier studies. Fleischauer et al. [4] as well as Ehni and Singer [11] have shown

that the transfer can occur in the first pass. Sliney, using real-time optical analysis, has shown how loose MoS_2 debris can be extruded through the sliding contact and incorporated as both transfer film and track material [12]. Fusaro has also documented similar retransfer processes for MoS_2 [13] and other solid lubricants [14]. Fleischauer et al. [4] and Singer et al. [5] have shown that early loss of lubricant from tracks correlates with buildup of transfer film on the counterface, and Lancaster [15] has argued that transfer films play a crucial role in the endurance of solid lubricants.

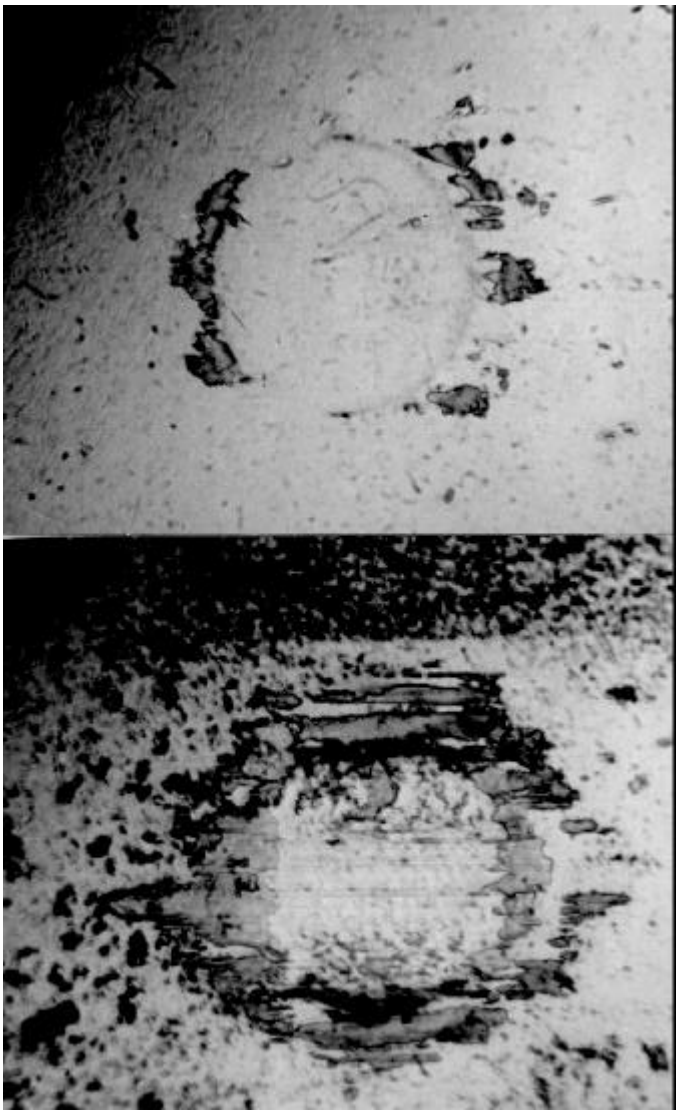

Figure 3. Transfer films formed on steel balls during sliding against IBAD MoS_2 coating after (a) 9 and (b) 900 cycles.

Here we carry that argument one step further: we contend that the endurance of a MoS_2 coating (in dry air environment) is not determined simply by the wear rate of MoS_2 but rather by the dynamics of the replenishment process illustrated in Figure 5. The track depths (Figure 1) and coating losses (Figure 4) determine the *net loss of material* from the track rather than a *coating "wear rate."* The amount of lubricant in the track is established by both

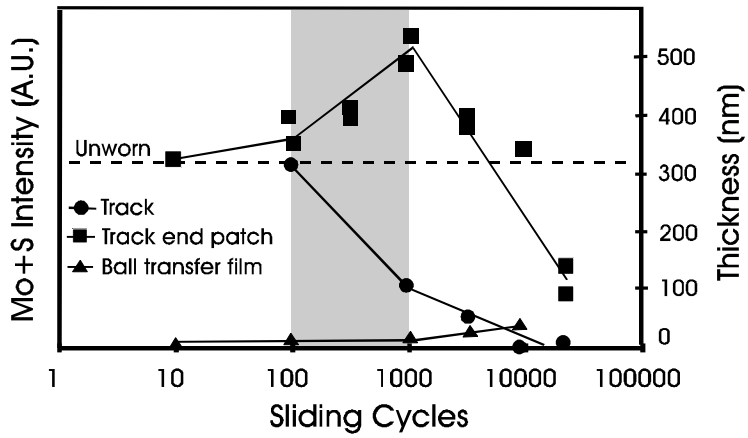

Figure 4. Track, patches at turnaround points (track end patch), and ball transfer film thicknesses measured by EDS.

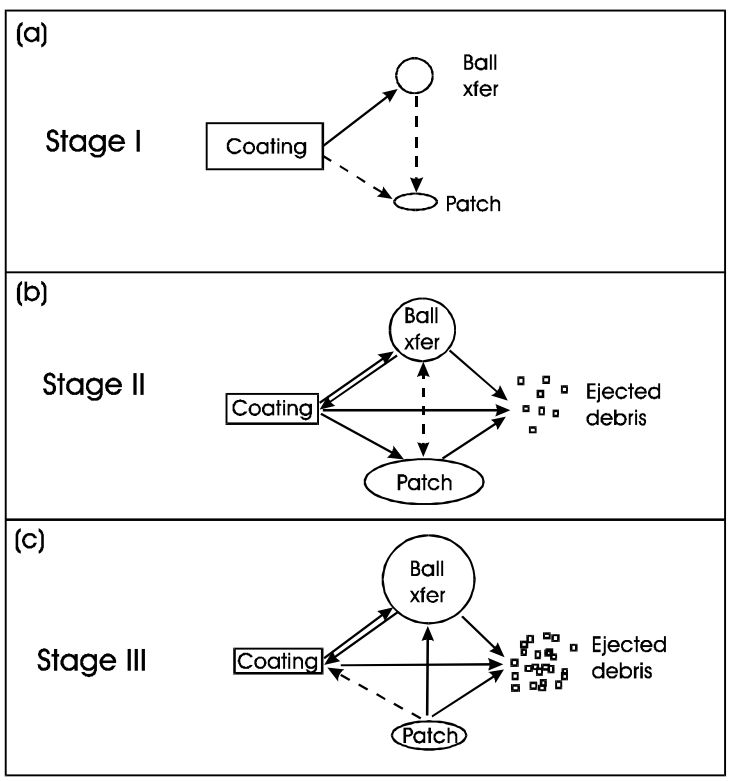

Figure 5. Schematic representation of the transfer processes between the coating wear track (coating), ball transfer film (ball xfer), and patch material at turnaround points (patch) for (a) Stage I, (b) Stage II, and (c) Stage III sliding. Solid lines indicate observed material transfer directions, while dashed lines indicate other possible transfer routes.

In summary, IBAD MoS_2 coatings wore rapidly during the first 5-10% of sliding life. Nonetheless, solid lubrication persisted for the remaining 90% of sliding life by redistribution of worn lubricant to the track and ball surfaces. A dynamic transfer process that produced reservoirs of lubricant, and then emptied them, was inferred from quantitative analysis of worn coatings and transferred materials.

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