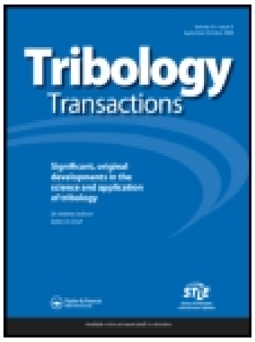

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In Situ Lubrication with Boric Acid: Powder Delivery of an Environmentally Benign Solid Lubricant

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In situ deposition of boric acid in dry powder form is investigated as a potential environmentally benign solid lubricant for sliding metal contacts. Boric acid is widely used in industrial processes and agriculture, is not classified as a pollutant by EPA, and produces no serious illnesses or carcinogenic effects from exposure to solutions or aerosols. In this study, boric acid powder is aerosolized and entrained in a low-velocity jet of nitrogen gas, which is directed at a self-mated 302 SS sliding contact in a rotating pin-on-disc tribometer. The effects of powder flow rate, sliding speed, normal load, and track diameter on coefficient of friction and wear rate are investigated. Friction coefficients below $\mu = 0.1$ can be consistently reached and maintained as long as the powder flow continues. Wear rates are reduced over 2 orders of magnitude.

KEY WORDS

Solid Lubrication; Powder Delivery; Environmentally Friendly; Green Manufacturing

INTRODUCTION

Petroleum-based lubricants are widely used in the manufacturing and industrial sectors, as well as in automotive and many other mass market products. It is well recognized that use of these lubricants introduces significant quantities of used petroleum-based substances into the waste stream (Sutherland, et al. *(1)*). These lubricants impose significant negative impacts to environment and health both during their primary use and after disposal (Allen, et al. *(2)*). This article introduces a lubrication concept aimed at reducing the need for petroleum-based fluids in a wide range of industrial processes and consumer applications by delivery of boric acid, an environmentally benign solid lubricant, in powder form. The proposed lubricant and delivery method will avoid the waste stream and environmental and health impacts associated with other lubricants used in many industrial processes and products. This technique is not tied to any particular process or product, but rather it has broad applicability, although customized delivery strategies will need to be developed for the various applications.

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Boric Acid

Boric acid is the common term for orthoboric (or boracic) acid H_3BO_3, which is a hydrate of boric oxide B_2O_3. When in contact with water, boric oxide will readily hydrate, converting to boric acid. Boric acid is a weakly acidic white powder that is soluble in water (about 27% by weight in boiling water and about 6% at room temperature), soft, ductile, stable, free flowing, and easily handled. Finely ground technical grade boric acid powder (>99% pure) is readily available for under US$2 per pound. The Environmental Protection Agency has established that boric acid is benign and it is not classified as a pollutant under the Clean Water Act. Material safety data sheets for boric acid show no serious illnesses or carcinogenic effects from exposure to solutions or aerosols. The United States is the world's largest producer of boron compounds (U.S. Geological Survey *(3)*). Boric acid is recovered from brines at Searles Lake in California, with large domestic reserves of boron materials residing in other lake sediments and brines. Large quantities of boron ore are also collected from an open pit mine in California.

The consumption of boric acid and boric oxide in the United States is distributed among glass making (78%), fire retardant (9%), agricultural fertilizer (4%), and industrial applications such as metal plating and finishing, paints and pigments, electroplating, and cosmetics (9%) (U.S. Geological Survey *(3)*). A dilute water solution of boric acid is also commonly used as a mild antiseptic and eyewash. The use of boric acid as a food preservative apparently dates back to the ancient Greeks. The earliest known scientific study of boric acid of which the authors are aware was conducted in 1902 and reported in *Science* in 1904 *(4)*. In this study, boric acid (0.5 g) was introduced into food and ingested by a group of participants with each meal. This produced occasional occurrences of "fullness in the head," nausea, and loss of appetite in a few subjects.

Boric Acid as a Solid Lubricant

In the early 1990s, the lubricity of boric acid, an overlooked but extremely available and environmentally benign lamellar solid, was demonstrated by Erdemir, et al. *(5)-(8)*. Figure 1 shows the lamellar molecular structure of boric acid. The shear strength of boric acid has been experimentally determined to be 23 MPa by Gearing, et al. *(9)*, who performed high-pressure (above 500 MPa) thrust washer experiments on a 6111 aluminum alloy plate coated

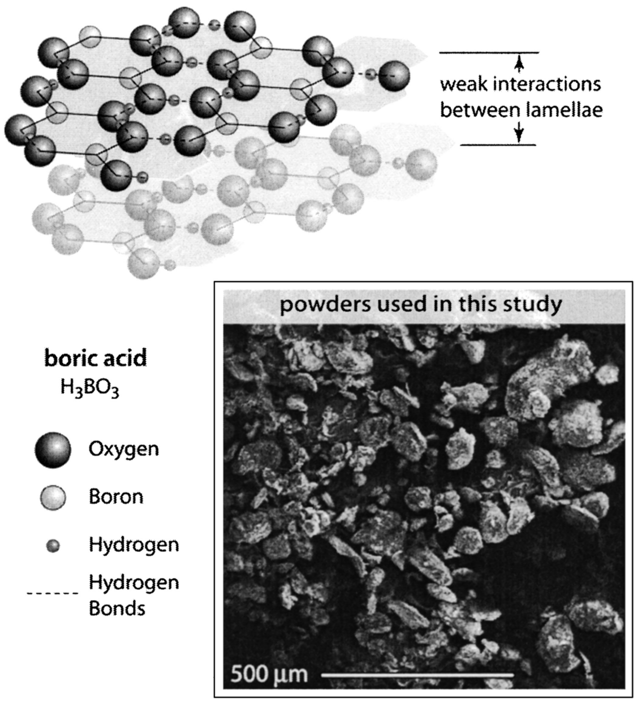

Fig. 1—A schematic showing the lamellar structure of boric acid and a scanning electron micrograph of the boric acid powders used in this study. The particle sizes ranged from 10 μm to over 100 μm.

with boric-acid. This is almost the same as the experimentally determined shear stress of molybdenum disulphide, which was found to be 24 MPa by Singer, et al. *(10)*. In Gearing, et al. *(9)*, the friction coefficient lowers with increased contact pressure and is well below 0.1 for average pressures above 100 MPa. At subatmospheric pressures, boric acid dehydrates (i.e., reverts back to boric oxide) above 170°C. Although the contact temperatures may reach this level in some potential applications, it is unclear whether or not the boric acid will transform to boric oxide at the higher contact pressures.

Some of the original boric acid solid lubricant films were created by dissolving the boric acid in water or alcohol, spraying the solution onto the samples, and then allowing the solution to evaporate, leaving the boric acid behind as a thin coating. This film formation method creates an extra step that is not compatible with many current processes or products and may be one reason that boric acid lubrication has not been adopted by industry. The work presented here indicates that delivery of dry powder in an air jet can provide sufficient lubricant to cling to the workpiece and achieve good lubrication in situ. The feasibility of boric acid powders to sustain low friction when delivered as a powder was shown in Lovell, et al. *(11)*. In this study, a concentrated sliding contact between an alumina pin and AISI-M50 bearing steel was loaded to an initial maximum central Hertzian contact pressure of 1.9 GPa. The sliding speed was approximately 1 m/s. Boric acid powder was delivered by manually sprinkling it onto the disk surface; the corresponding lubricous surface film lowered the friction coefficient from $\mu = 0.4$ to approximately $\mu = 0.15$. Further, the reduced friction was sustained until the powder delivery was halted.

EXPERIMENTAL SETUP

The experiments reported here were carried out on a high-speed rotating pin-on-disc tribometer described in more detail in McCook, et al. *(12)*. Figure 2 shows a schematic of the apparatus. The pin samples were 302 stainless steel balls with a diameter of 4.76 mm; the disk samples were 302 stainless steel and were 50.8 mm in diameter. The initial surface roughnesses of the specimens were measured using scanning white light interferometry, with the pins having a mean average roughness value of Ra = 150 nm and the discs a mean Ra = 172 nm. All samples were washed and sonicated in methyl alcohol prior to testing. During the tests, frictional forces were continuously monitored via a computerized data acquisition system, allowing real-time computation and display of friction coefficient. Wear rates were obtained by computing the volume of material lost on the pin and the disk at the conclusion of the test using optical microscopy and scanning white light interferometry, respectively.

The powder used in this study was technical grade (99% pure) with particle sizes ranging from a few micrometers up to 100+ micrometers. The continuous powder delivery was achieved by entraining a cloud of particles above a fluidized bed of boric acid into a directed gas stream of dry nitrogen. The apparatus is shown in Fig. 3. Pressurized nitrogen was introduced into the container through a tube that entered the top and extended nearly to the bottom of the container and into the boric acid powder. The exit tube also penetrated the top of the container but did not extend into the powder bed. The container was placed on a mechanical shaker and the combination of the mechanical agitation and air flow into the powder created a fluidized bed. Some of the powder was aerosolized and left with the air flow in the exit tube. Powder flow rate into the tribological contact was coarsely controlled by regulating the nitrogen flow through the boric acid container. The nitrogen stream with entrained boric acid powder was mixed downstream with another nitrogen line such that the total gas flow rate into the chamber was maintained at a constant value regardless of particle loading. The flow of nitrogen and boric acid particles were directed at the tribological contact via a nozzle within the plastic enclosure. The actual mass flow rate of the powder was determined by weighing the container before and after each test and dividing by the test duration (powder flow and motion were initiated simultaneously).

A matrix of experiments was conducted to investigate the effectiveness of a continuous delivery of dry boric acid powder into the vicinity of a self-mated stainless steel sliding contact. The experimental parameters studied were normal force, sliding speed, wear-track diameter, and powder delivery rate. A 3 × 3 × 3 × 3 matrix of test conditions was developed as shown in Table 1. This

TABLE 1—THE EXPERIMENTAL MATRIX (3 × 3 × 3 × 3) PLUS NINE REPEATS AT THE MIDPOINT AND ONE UNLUBRICATED CONTROL

Fn (N)	Ω (RPM)	Track Diameter, D (mm)	Flow Rate, f (grams/min)
0.65	400	3.8	10
3.30	2,000	25	20
6.30	4,000	38	30

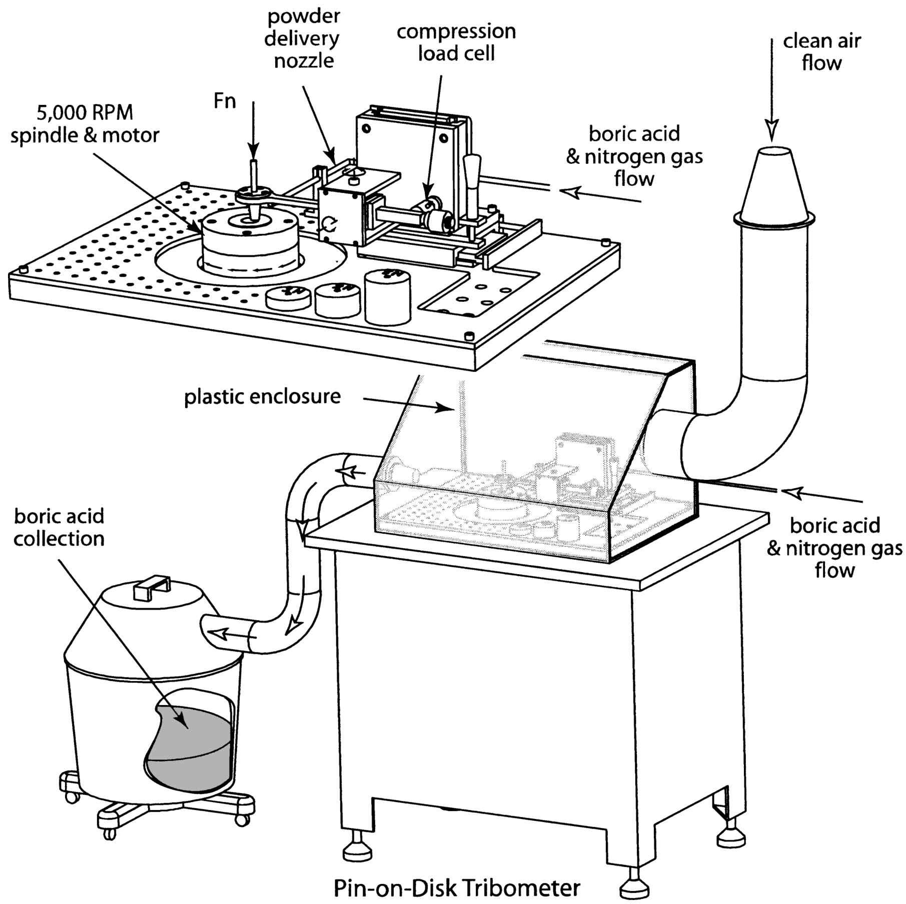

Fig. 2—Schematic of the rotating pin-on-disc tribometer used in this study. The vacuum cleaner and filter stack are attached to the clear plastic enclosure.

resulted in 81 individual tests, one test with no boric acid delivery and nine additional repeat experiments at the midpoint. The sequence of experiments was randomized to prevent any time related bias. All tests were run for 24,000 rotations of the disc in

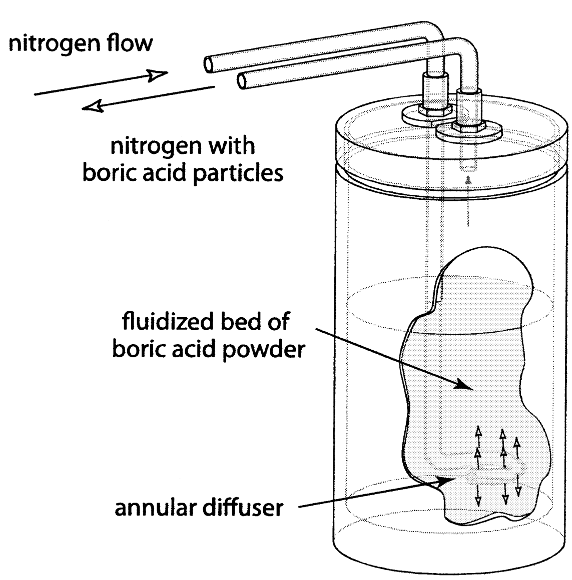

Fig. 3—Schematic of the particle delivery scheme. The container was mounted on a mechanical shaker during operation. Dry nitrogen gas flow rates were controlled through a series of flowmeters.

filtered laboratory ambient air with a relative humidity varying from approximately 20% to 55% and temperatures ranging from 25°C to 28°C. The combination of three rotational speeds for the discs and three track diameters allowed a 100 times speed range variation for the tests. The choice of test conditions allowed significant flexibility in grouping data for analysis. For example, 27 tests were performed at constant diameter while varying sliding speed, contact force, Fn, and flow rate, f; and 18 tests were performed at constant sliding speed while varying track diameter, D, and rotational speed, Ω, Fn, and f.

RESULTS AND DISCUSSION

Table 2 provides the entire set of friction coefficient and wear-rate, K, data collected during completion of the Table 1 experimental matrix. Figure 4 shows the friction coefficient during the nine repeat tests and the control. In each of the midpoint repeats, friction coefficient values less than $\mu = 0.1$ were quickly achieved and maintained throughout the duration of the test, except for instances where malfunctions and clogging in the powder delivery system momentarily shut off the flow of lubricant to the contact. During execution of the experimental matrix two instances occurred where clogging was severe enough that powder delivery was lost completely, resulting in a transition from low to high friction and wear for the remainder of that test; this is shown in Fig. 5. The transitions from low to high friction were very rapid,

TABLE 2—RESULTS FROM THE EXPERIMENTAL MATRIX. D IS THE WEAR TRACK DIAMETER IN MILLIMETERS, F IS THE COMMANDED BORIC ACID FLOW RATE IN GRAMS/MINUTE, AND K IS THE WEAR RATE × 10^{-6} MM^3/(NM)

Ω = 400 RPM				Ω = 2,000 RPM				Ω = 4000 RPM			
D	f	μ	K	D	f	μ	K	D	f	μ	K
Fn = 0.65 N				Fn = 0.65 N				Fn = 0.65 N			
3.8	10	0.196	2.92	3.8	10	0.157	—	3.8	10	0.090	—
3.8	20	0.080	—	3.8	20	0.094	14.93	3.8	20	0.085	—
3.8	30	0.138	—	3.8	30	0.085	1.98	3.8	30	0.092	0.63
25	10	0.147	0.30	25	10	0.036	36.65	25	10	0.086	0.22
25	20	0.122	—	25	20	0.035	42.48	25	20	0.075	—
25	30	0.159	0.67	25	30	0.062	21.31	25	30	0.047	4.90
38	10	0.147	0.44	38	10	0.058	1.25	38	10	0.080	0.21
38	20	0.138	0.03	38	20	0.077	0.38	38	20	0.049	4.13
38	30	0.173	0.19	38	30	0.045	1.56	38	30	0.089	0.57
Fn = 3.3 N				Fn = 3.3 N				Fn = 3.3 N			
3.8	10	0.143	0.23	3.8	10	0.148	0.82	3.8	10	0.081	1.48
3.8	20	0.139	4.03	3.8	20	0.12	—	3.8	20	0.088	5.55
3.8	30	0.162	0.31	3.8	30	0.11	0.12	3.8	30	0.087	0.03
25	10	0.152	0.76	25	10	0.056	0.80	25	10	0.055	1.05
25	20	0.156	0.48	25	20	0.044*	1.54*	25	20	0.035	0.52
25	30	0.163	0.41	25	30	0.05	2.00	25	30	0.030	1.55
38	10	0.113	9.02	38	10	0.049	0.14	38	10	0.321	0.03
38	20	0.116	1.36	38	20	0.039	0.27	38	20	0.040	0.34
38	30	0.101	13.26	38	30	0.045	0.19	38	30	0.026	0.69
Fn = 6.3 N				Fn = 6.3 N				Fn = 6.3 N			
3.8	10	0.155	0.42	3.8	10	0.117	0.35	3.8	10	0.086	0.07
3.8	20	0.138	0.06	3.8	20	0.108	—	3.8	20	0.079	0.60
3.8	30	0.165	—	3.8	30	0.103	—	3.8	30	0.078	0.85
25	10	0.137	5.28	25	10	0.036	0.24	25	10	0.059	0.27
25	20	0.122	1.05	25	20	0.061	0.72	25	20	0.029	0.46
25	30	0.121	0.01	25	30	0.059	3.44	25	30	0.035	1.09
38	10	0.100	20.78	38	10	0.047	0.30	38	10	0.230	2.35
38	20	0.106	32.86	38	20	0.039	0.21	38	20	0.023	14.08
38	30	0.090	21.15	38	30	0.046	0.09	38	30	0.073	0.06

*Midpoint condition $\mu_{avg} = 0.044$, standard deviation, $\sigma = 0.009$; $K_{avg} = 1.54$ $\sigma = 1.15$.

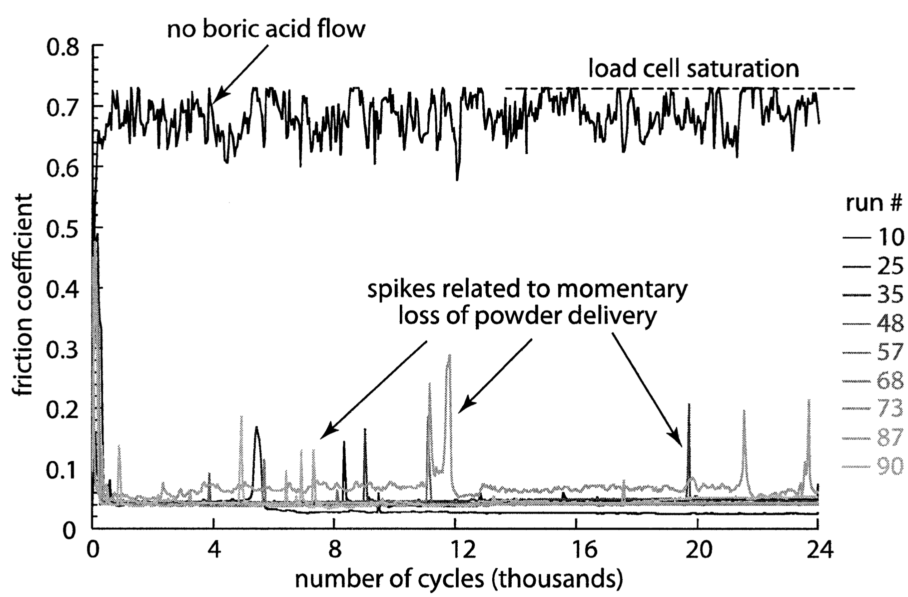

Fig. 4—Plots of the friction coefficient versus cycle number for the 9 repeat tests and the control, which had no boric acid in the gas flow. The experimental conditions were Fn = 3.3 N; Ω = 2,000 RPM; D = 25.4 mm; and f = 20 g/min.

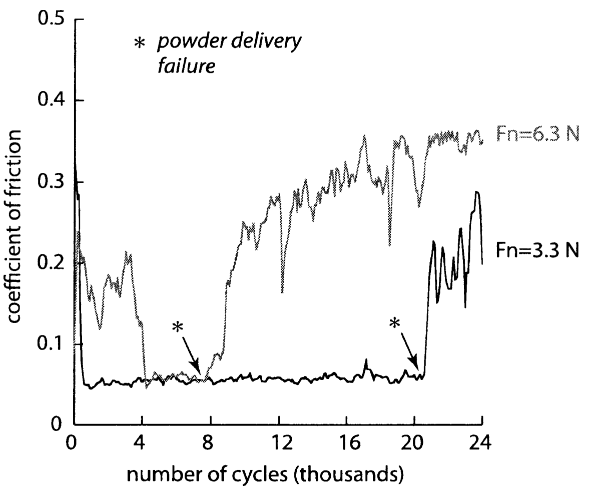

Fig. 5—Plots of the friction coefficient versus cycle number for the two experiments that had terminal blockages in the boric acid delivery during the experiment. The wear track diameter was 25 mm, the surface speeds were 5.0 m/s, and the normal loads are identified in the plot.

occurring in only a few tens of revolutions. This suggests that the rate of deposition of boric acid is precariously close to the removal rate of the solid lubricant surface films, which is consistent with the experimental observations of friction spikes as shown in Fig. 4.

Figure 6 contains three plots of the friction coefficient for various normal loads, sliding speeds, and powder flow rate while holding all other variables constant on each plot. It is apparent that normal load and flow rate do not have any significant effect on the coefficient of friction. Further, the lower sliding speeds had higher friction coefficients than the higher sliding speeds tests under the same conditions. Competitive rate models for the deposition and removal of the boric acid film were investigated in an attempt to explain these phenomena. However, these models all suggest that the removal rate should increase as sliding speeds increase, leading to an increase in friction; while the experimental data show the opposite trend. This implies that either the removal rate of the boric acid films is suppressed with increasing sliding speed or the film formation rate is enhanced as the speed increases.

It is hypothesized that the nitrogen and boric acid stream was not the primary mechanism of boric acid transport to the pin contact; rather, the fluid flow generated across the rotation of the disc sample entrained the boric acid and delivered it to the pin contact. This classic problem was solved by von Karman *(13)*, *(14)*, who proposed a similarity solution for the air flow in the region above a spinning disk and showed that the air is pulled down at the center of the disk and expelled radially at its periphery. The flow is assumed to be laminar if Re $< 300{,}000$, where the Reynolds number is given by the product of the peripheral speed, V, and radius, R, divided by the kinematic viscosity, ν, i.e., $\mathrm{Re} = (VR)/\nu$. For these experiments, the highest Reynolds number at the contact was 4,000. Rogers and Lance *(15)*, *(16)* provided a numerical solution to this

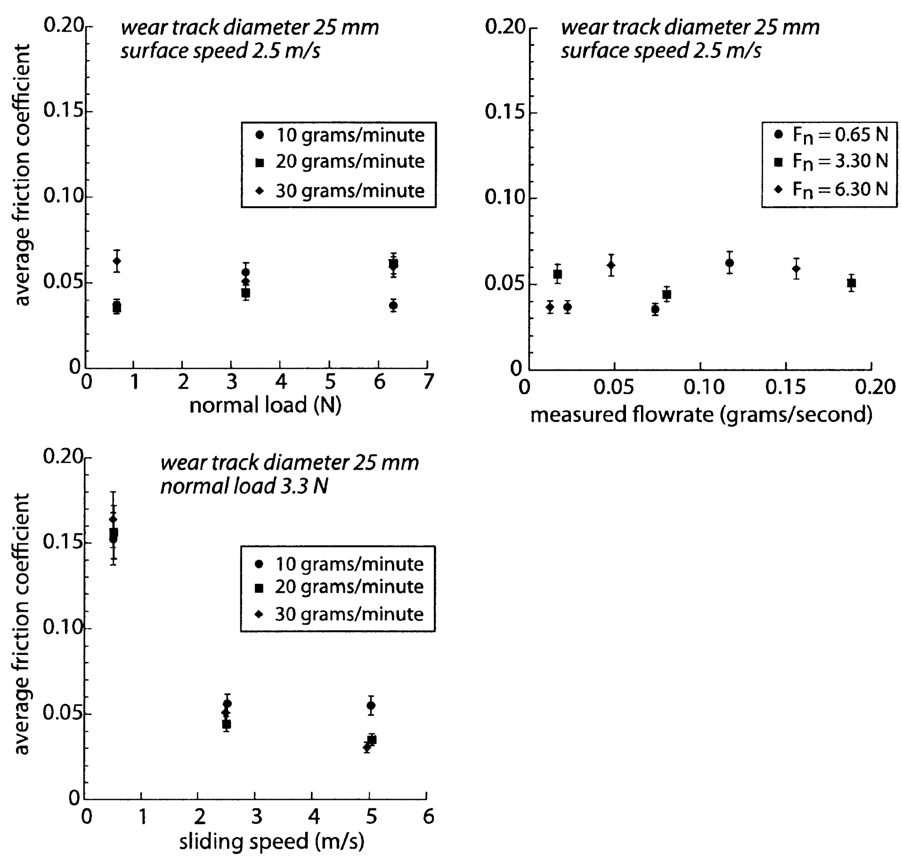

Fig. 6—Plots of the average friction coefficient versus normal load, flow rate, and sliding speed (the experimental conditions are noted in each plot).

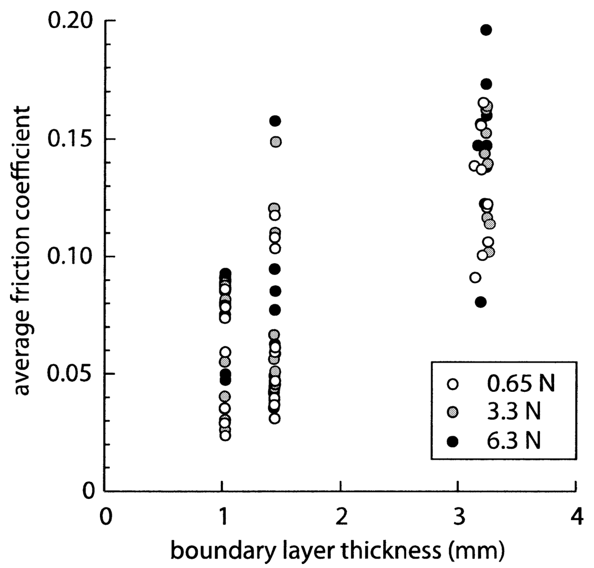

Fig. 7—The average friction coefficients for all experiments plotted versus the boundary layer thickness. The data are separated for the various normal loads as indicated.

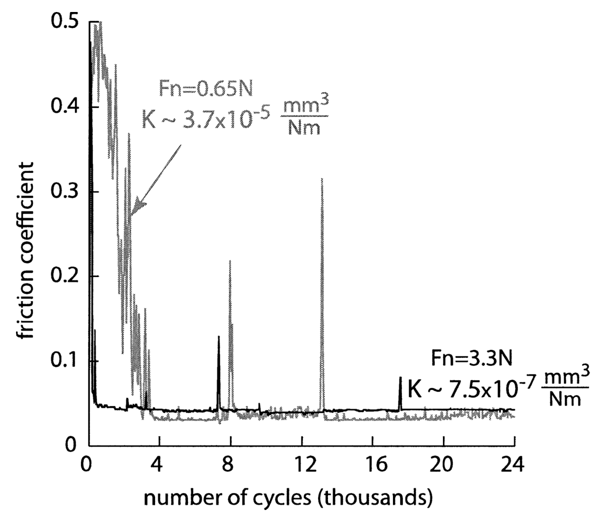

Fig. 8—Plots of the friction coefficient versus cycle number for the two experiments that had the shortest and longest transients to low friction coefficient. The wear track diameter was 25 mm, the surface speeds were 2.5 m/s, and the normal loads are identified in the figure.

problem and demonstrated that the boundary layer thickness, δ, is relatively constant across the disk and varies inversely with the square root of the angular speed, ω (in rad/s), i.e., $\delta = 5.4\sqrt{\nu/\omega}$, where $\nu = 1.5 \times 10^{-5}$ m^2/s is used for the kinematic viscosity of air. The entire data set is plotted versus the calculated boundary layer thickness in Fig. 7. There is a definite trend in the data suggesting that the thinner and higher speed flows are more efficient at delivering boric acid to the contact.

The wear rate of the pin sample for the unlubricated stainless steel contact was measured to be $K = 5 \times 10^{-4}$ mm^3/(Nm). As shown in Table 2, all of the experiments showed greatly reduced pin wear rates. There were a number of experiments where no damage could be detected on the pin surface; such tests are indicated by dashes in Table 2. The lowest measured wear-rate was $K = 7.5 \times 10^{-7}$ mm^3/(Nm) for the 25 mm diameter, 3.3 N load, 2.5 m/s sliding speed, and 0.078 g/s boric acid flow rate conditions. This is a greater than a 500 times improvement in wear resistance over the unlubricated condition.

Substantial, although unsuccessful, effort was made to correlate wear-rate with friction coefficient and other experimental variables. The only qualitative explanation that is offered for the variations in wear rate is that the majority of the material removal occurred during the startup transients. These tests were initiated on nascent surfaces and the boric acid delivery was expected to generate and replenish a surface film in situ. The startup transient varied widely from test to test although all tests eventually reached low friction coefficient. Figure 8 shows the friction coefficient traces for the tests with the shortest and longest times to low friction coefficient. These experiments were both at a radial position of 2.5 mm and a sliding speed of 2.5 m/s, although they were at two different normal loads. The experiment with the shortest transient had a wear rate of 7.5×10^{-7} mm^3/(Nm) and the test with the longest transient had a wear rate of 3.7×10^{-5} mm^3/(Nm). The ratio of the volumes lost between the shortest and longest transient was 0.1 (i.e., the test with a shorter transient lost 10% of the material of the test with the longest transient), while the ratio of the frictional energy dissipated during the transient region is about 1/3.

CONCLUSIONS

Boric acid has been identified as a potential solid lubricant due to its lamellar molecular structure. Further, it is abundant and environmentally benign, with no known health risks to humans. These experiments clearly indicate that powder delivery of boric acid is a viable technique for providing in situ lubrication for concentrated metal contacts. This technique can reliably produce friction coefficients less than $\mu = 0.1$ for a self-mated 302 stainless steel contact and can reduce wear rates by 100 times or more. The method of powder delivery can be improved and future directions for this technology should focus on delivering powder to the contacts with minimal lubricant waste.

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