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ALUMINUM FORMING***

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ABSTRACT

Preliminary research in our laboratory has demonstrated that boric acid is an effective lubricant with an unusual capacity to reduce sliding friction (providing friction coefficients as low as 0.02) and wear of metallic and ceramic materials. More recent studies have revealed that water or methanol solutions of boric acid can be used to prepare strongly bonded layers of boric acid on aluminum surfaces. It appears that boric acid molecules have a strong tendency to bond chemically to the naturally oxidized surfaces of aluminum and its alloys and to make these surfaces very slippery. Recent metal formability tests indicated that the boric acid films formed on aluminum surfaces by spraying or dipping worked quite well; improving draw scale performance by 58 to 75%. These findings have increased the prospect that boric acid can be formulated and optimized as an effective boundary lubricant and used to solve the friction, galling, and severe wear problems currently encountered in cold-forming of aluminum products. Accordingly, the major goal of this paper is to demonstrate the usefulness and lubrication capacity of thin boric acid films formed on aluminum surfaces by simple dipping or spraying processes and to describe the lubrication mechanisms under typical metalforming conditions. We will also examine the nature of chemical bonding between boric acid and aluminum surfaces and develop new ways to optimize its

performance as an effective boundary lubricant.

INTRODUCTION

One effective way to increase fuel efficiency in modern automobiles is to further reduce their overall weights. Weight reduction can be achieved by using lightweight aluminum alloys, especially in automotive body parts and structures. However, forming these alloys into automotive body parts may be difficult, mainly because of a combination of the high galling tendency of aluminum alloys, the extreme pressure requirements of metal-working operations, and the inability of conventional lubricants to prevent galling under such increasingly stringent conditions.

At Argonne National Laboratory (ANL), we discovered that boric acid is one of the slipperiest solids known (providing friction coefficients as low as 0.02) [1]. Benchtop friction and wear studies have demonstrated that boric-acid based compounds exhibit extremely low friction coefficients

when applied to the surface of metals and ceramics. Figure 1 illustrates how dramatically the friction coefficient decreases when boric-acid powders are introduced at the interface between an M50 steel pin sliding against an M50 steel disc under dry sliding conditions at room temperature [2]. Initially, the friction coefficient is approximately 0.8, typical of steel against steel, however when the boric-

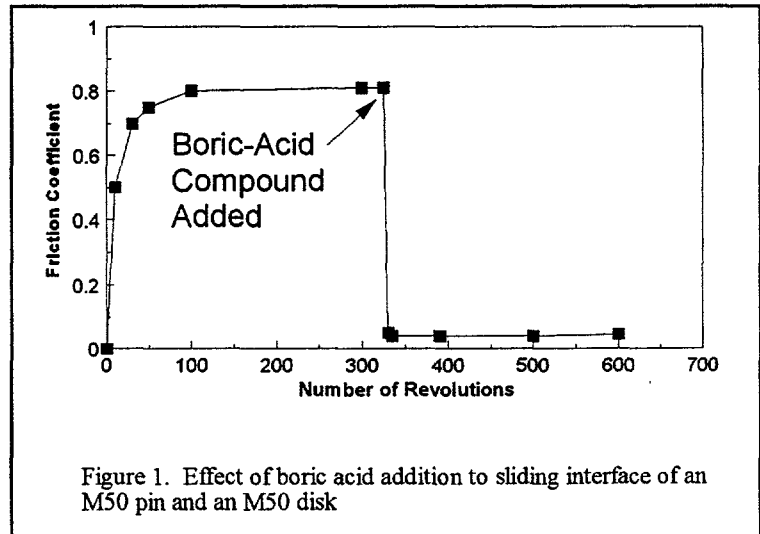


Figure 1. Effect of boric acid addition to sliding interface of an M50 pin and an M50 disk

acid powder is added to the interface, the friction coefficient drops to 0.05 or less. Detailed investigations indicated that friction coefficient decreases further with the increasing contact stresses, suggesting that boric acid could be a potential extreme pressure lubricant for cold-

The results of crystal chemical studies have revealed that the lubrication mechanism of boric acid is mainly associated to its layered crystalline structure [4]. The atomic layers of boric acid align themselves parallel to the direction of sliding motion and slide easily over each other to provide low friction. Strong interatomic bonding and rigidity of layers prevent direct metal-to-metal contact, thus inhibiting scuffing and/or galling.

More recent studies in our laboratory have revealed that boric acid has a strong tendency to form chemically bonded films on the oxidized surfaces of aluminum and titanium alloys. This observation raises the prospect of developing an effective boundary lubricant for use in cold forming of aluminum and its alloys. In this paper, we provide experimental data that demonstrate the lubricity of boric acid films formed on the surfaces of 6061 and 6111 aluminum alloys under sliding contacts and instrumented metalforming conditions. Specifically, the main objective of this study is to demonstrate the usefulness and lubrication capacity of thin boric acid films for aluminum forming. In our experimental work, we explored the lubrication capacity of boric acid powders, bonded thin boric acid layers, and thin films formed by dipping or spraying processes. We will also describe the lubrication mechanisms under typical metalforming conditions.

EXPERIMENTAL PROCEDURES:

Test Materials: For standard pin-on-disk experiments involving powder and bonded-film lubrication, we used M50 bearing balls (9.55 mm in diameter) and 6061 and 6111 aluminum disks. The bearing balls were highly polished and had a surface roughness value that was better than 0.01 μm center-line-average (CLA). The nominal hardness of ball material was about 61 in the Rockwell C scale. The aluminum disks were hand-ground on a grinding wheel down to 0.2 μm CLA roughness level. Both the balls and the disks were ultrasonically cleaned in methanol and acetone bath for 5 min each and blow dried before storing in a desiccator until the actual tests or surface treatment.

Friction and Wear Tests: Sliding friction and wear tests were performed under contact loads of 5, 10, and 20 N (creating initial peak contact stresses of 25, 32, and 40 ksi, respectively) at a sliding velocity of 21 mm/s for a distance of 125 m. The relative humidity of the laboratory room varied between 30 and 70% and the test temperature was 23°C. The principle of the pin-on-disk test machine used for friction and wear evaluation is shown in Figure 2.

Principle of pin-on-disk wear test

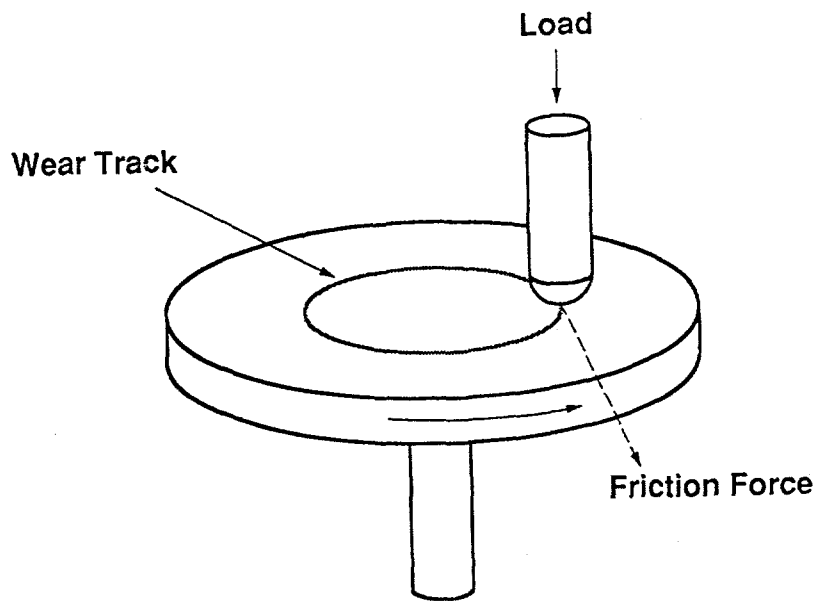


Figure 2. Schematic illustration of contact geometry of pin-on-disk test apparatus.

For the powder lubricated cases, we initiated a test on our pin-on-disk machine using a pair of a 6061 alloy disk and a M50 steel pin and let the test proceed under a load of 10 N until reaching a steady state friction value. The M50 pin had a hemispherical cap of 127 mm radius in order to establish a point contact at the start of sliding tests. The surface roughness of the disk was approximately $0.2 \mu\text{m}$ CLA, whereas the pin had a surface roughness of about $0.005 \mu\text{m}$ CLA. Initially, we allowed the pin to slide against the disk without any lubrication until a steady state regime is established. Subsequently, we fed manually fine boric acid powders (particle size; 20-50

μm) into the sliding interface of the test pair.

Metal Formability Tests: Instrumented metalformability tests were performed by a commercial Interlaken test machine whose main features and test procedures are described in details in Refs. 5 and 6. It provides a means for measuring relative friction as well as ranking sheet metal lubricants. Briefly, this tester uses a a short stroke clamping actuator and two dimensional tooling that allows the die to be opened and the specimens removed without a large clamping stroke. A schematic illustration of this tester is shown in Figure 3. Rectangular strips of aluminum (9.5"X0.5"X0.03") are used as the test pieces which are firmly clamped around the edges of the stretching punch and the saps is developed entirely at the expense of specimen thickness. Prior to testing two parallel lines of 0.5" are scribed in the center region of the strip. Specimen surface is cleaned and boric acid lubricant is applied on the clean and dry surfaces by two simple processes: (1) dipping in a water solution of boric acid and (2) spraying concentrated boric acid solution directly onto the test pieces. The samples were then dried in open air. The boric acid coated specimens were locked in the die to ensure a pure stretching operation. The punch in metalformability machine stretches the specimen until it fails. By measuring the scribe before and after the experiment, the percentage increase in length of the specimen is determined by using the formula: Relative change of length = $[(L_1 - L_0)/L_0] \times 100$; where L_1 and L_0 are the final and initial scribe lengths. Two to three tests were run under each condition to check the reproducibility of the friction and wear data.

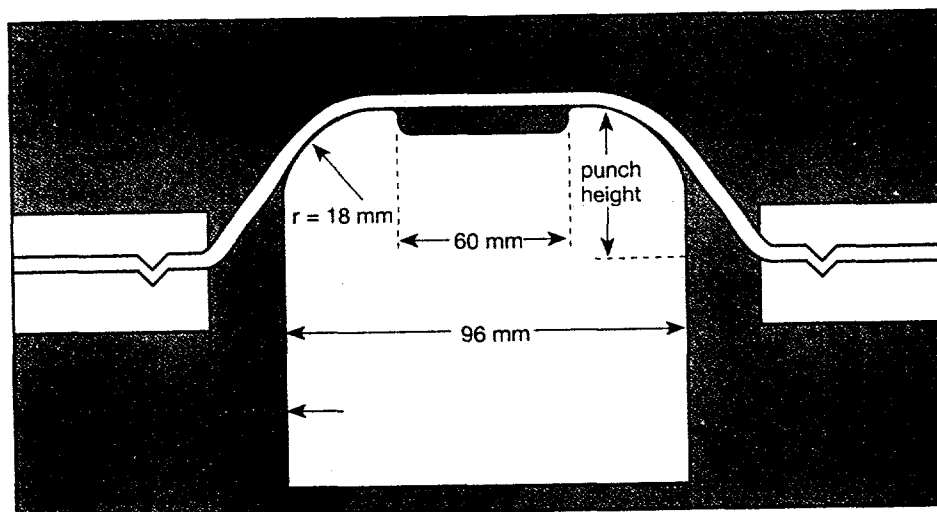


Figure 3. Schematic illustration of metal formability machine.

Scanning electron microscopy (SEM) was used to assess the uniformity and surface morphology of the boric acid films. A micro-laser Raman spectroscope was also used for chemical characterization of sliding surfaces. The Raman instrument was operated with a HeNe laser having a wavelength of 632.8 nm with an output power of 25 mW and spot size of 2 μm .

RESULTS

Figure 4 shows the friction coefficient of the M50/6061 test pair before and after boric acid is added to sliding interface. As is evident, the steady-state friction coefficient of the pair is about 0.6 before boric acid addition, but with the introduction of boric acid powders to the sliding interface, the friction coefficient drops sharply to a value of 0.05. This simple experiment demonstrates the

impressive lubrication capacity of bulk boric acid.

Figure 5 shows the sliding friction performance of bonded boric acid films on 6061 aluminum alloy under 5, 10, and 20 N loads. In these tests, M50 balls were used as the counterface material. It is clear that boric acid films on the surface provide very low friction coefficients to the sliding

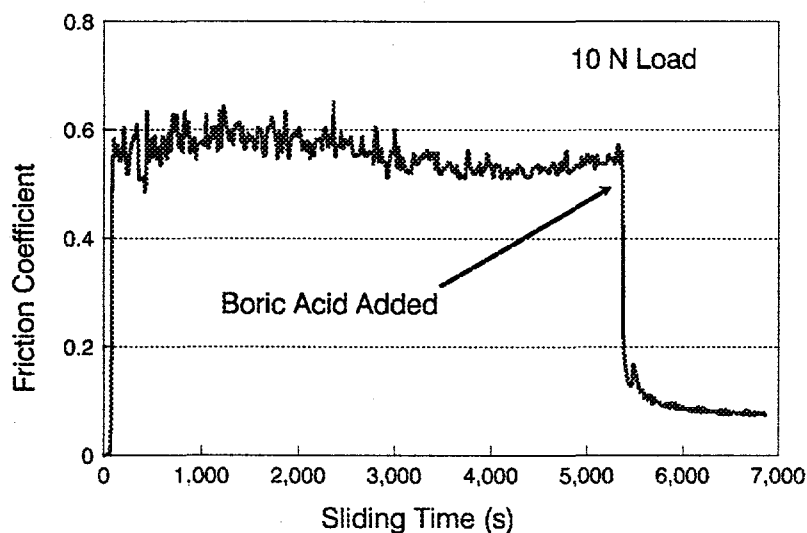


Figure 4. Sliding friction performance of M50 pin against 6061 Al alloy before and after lubrication by boric acid powders.

interface. Depending on the load, the friction coefficient varies between 0.04 and 0.06. In general, the higher the contact load the lower the friction coefficient. This experiment further verifies that boric acid indeed a good solid lubricant for sliding contact applications.

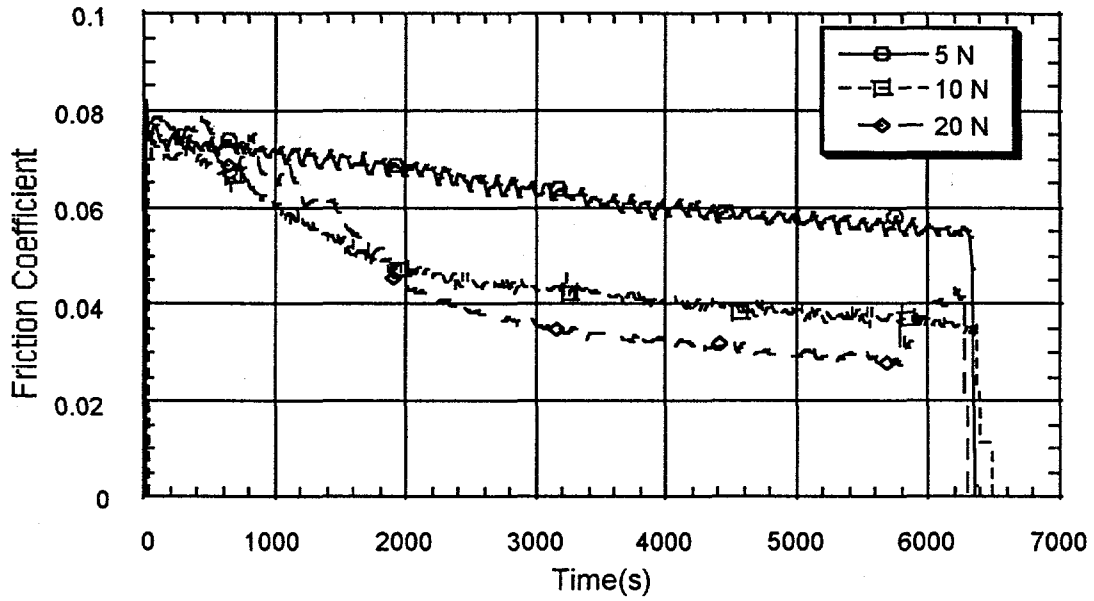


Figure 5. Sliding friction performance of M50 pins against boric acid coated 6061 Al alloy under 5, 10, and 20 N loads.

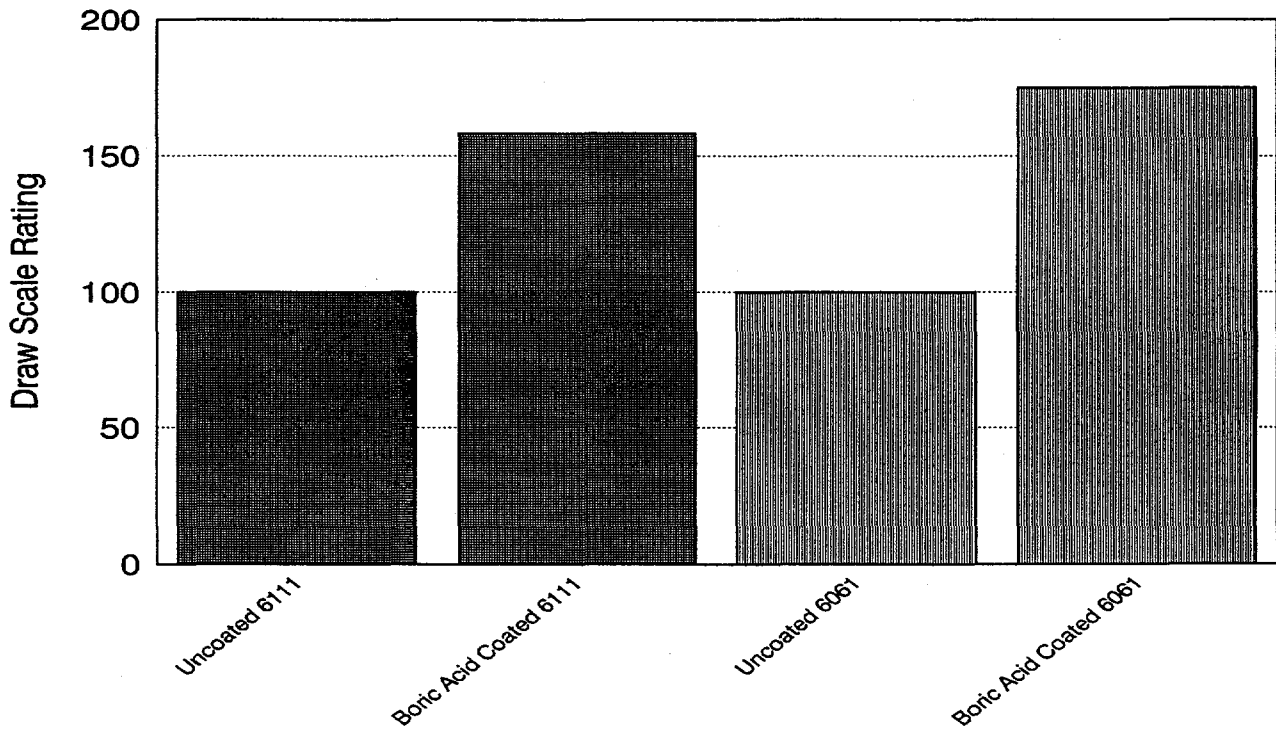


Figure 6. Draw scale ratings of uncoated and boric acid coated aluminum alloy work pieces.

Figure 6 shows the results of metal formability tests. In this chart, draw scale ratings of two surfaces lubricated by simple dipping and spraying are compared with the results of unlubricated metal strips. Again, boric acid lubricated surfaces attain superior performance as reflected by higher drawscale ratings (assessed by $[(L_1-L_0)/L_0] \times 100$).

DISCUSSION

Fig. 4 demonstrates that the friction coefficient of an M50 pin against bare 6061 aluminum alloy is initially rather high. During this initial (unlubricated) stage, the levels of vibration and noise were also high. Visually, we noticed severe wear damage on disk surface. However, as soon as we introduced some boric acid powders to the sliding interface, the friction coefficient dropped precipitously and the level of noise and vibration became undetectable. Fig. 5 further demonstrates that as long as we have a bonded boric acid film on the surface, the friction coefficient becomes very low. Also, Fig.5 reveals that the friction coefficient decreases with increasing contact pressure, thus implying that the lubricating capacity of boric acid increases under heavier loads. In short, these simple experiments demonstrate that boric acid is an excellent solid lubricant. Once applied to a surface, it can lower friction.

Visual inspection of the sliding M50 steel balls/pins and 6061 aluminum alloy surfaces revealed that the wear scars and tracks were covered by a thin layer of boric acid powder. Some particles had accumulated around the circular wear scars or along the edges of the wear tracks. Chemical analyses by micro-laser-Raman spectroscopy revealed that these thin layers were made of boric

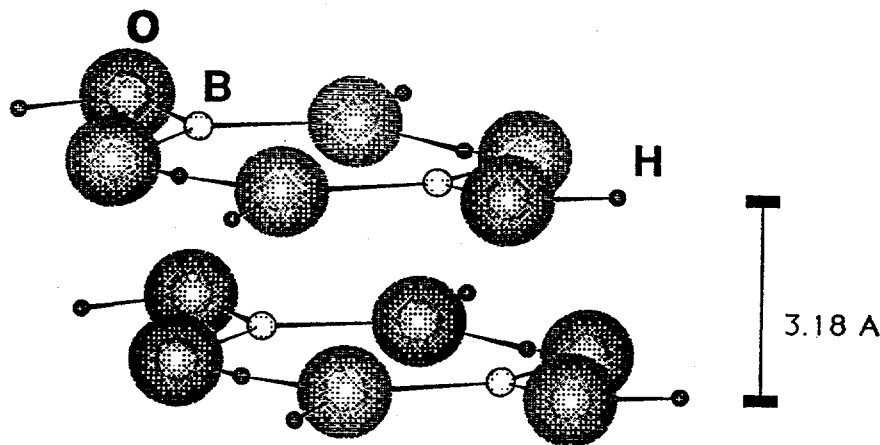
acid. Specifically, the spectra showed very strong Raman lines at approximately 497 and 879 cm^{-1} which are the characteristic Raman lines for boric acid [7].

The results of the metal formability tests further demonstrated the excellent lubricating capacity of boric acid by revealing high draw scale ratings for test pieces coated with boric acid. Specifically, as shown in Fig. 6, the surfaces coated with a thin layer of boric acid performed quite well. Draw scale ratings for the baseline (uncoated) 6111 and 6061 test pieces were 100, but the ratings of boric acid coated 6111 (by dipping) and 6061 (by spraying) were 158 and 175, respectively. This means 58 to 75% improvements in draw scale rating for boric acid lubricated surfaces.

The highly slippery nature of boric acid can be explained as follows. As elucidated in a series of previous research articles [2-4] boric acid crystallizes in a layered triclinic crystal structure (see Fig. 7). The atoms on each layer are closely packed and strongly bonded to each other. The atomic layers themselves are 0.318 nm apart from each other and held together by weak van der Waals forces [4]. In a sense, the layered-crystal structure of boric acid is similar to those of MoS_2 or graphite. Accordingly, the very lubricious nature of boric acid is governed by its layered crystal structure. Under shear stresses of sliding contact, the crystalline layers align themselves parallel to the direction of relative motion; once so aligned, they can slide over one another with relative ease and thus provide the low friction coefficients shown in Figs. 4 and 5.

The nature of bonding between boric acid and naturally oxidized surfaces of aluminum alloys remains a mystery, but based on some crystal chemical knowledge, we speculate that the strong bonds are most probably due to the presence of some ionic bond character within the crystalline

structures of both the aluminum oxide and boric acid materials. Detailed fundamental studies are currently underway and will elucidate the nature of bonding between aluminum surfaces and boric acid lubricant.



INTERLAYER BONDING : van der Waals

Figure 7. Layered crystal structure of boric acid.

SUMMARY

In metalforming practices, a lubricant is expected to reduce friction and wear, prevent material transfer or pick-up, and improve surface quality. Furthermore, new lubricants should be non-flammable, environmentally safe, easy to apply and remove. Boric acid appears to meet these requirements. It can be applied to a surface by simple dipping or spraying process and it can be washed away or rinsed clean on a water jet after a metalworking operation. Application of boric

acid on a sliding or deforming surface can result in low friction, hence improved lubrication. Low friction is a direct consequence of boric acid, mainly because of its layered-triclinic-crystal structure. Under shear stresses, the crystalline layers can align themselves parallel to the sliding direction and, once so aligned, can slide one over another to provide low friction.

REFERENCES

1. A. Erdemir, R. A. Erck, and J. Robles, "The Relation of Hertzian Contact Pressure to Friction Behavior of Self-Lubricating Boric Acid Films," *Surface Coatings and Technology*, Volume 49 (1991) page 435.
2. A. Erdemir, G. R. Fenske, R. A. Erck, F. A. Nichols and D. E. Busch, "Tribological Properties of Boric Acid and Boric-Acid-Forming Surfaces. Part II. Mechanisms of Formation and Self-Lubrication of Boric Acid Films on Boron- and Boric Oxide-Containing Surfaces," *Lubrication Engineering*, Volume 47 (1991) page 179.
3. A. Erdemir, G. R. Fenske, and R. A. Erck, "A Study of the Formation and Self-lubricating Mechanisms of Boric Acid Films on Boric Oxide Coatings," *Surface and Coatings Technology*, Volume 43/44 (1990) pp. 588-596.
4. A. Erdemir, "Tribological Properties of Boric Acid and Boric-Acid-Forming Surfaces. Part I:

Crystal Chemistry and Mechanism of Self-lubrication of Boric Acid," *Lubrication Engineering*,
Volume 47 (1991), page 168.

5. D. Houcque, A. E. Dampts, New Standard Draw Scale simplifies Lubricant Ranking,
Metforming Magazine, October 1996.

6. R. M. Harycki, K. E. Gasper, R. J. Smola, F. I. Saunders, J. M. Garrett, R. H. Wagoner, "A new
Method for Ranking Sheet Metal Lubricants," *Metforming Magazine*, November 1994, pp. 39-47.

7. R. Janda and G. Heller, "IR- und Ramanspektren Isotop Markierter Tetra- und Pentaborate,"
Spectrochim. Acta, 36A, 997-1001 (1981).