

Evaluation of Vitreous and Devitrifying Enamels as Hot Forming Lubricants for Aluminum AA5083 Alloy

A.R. Riahi, A.T. Morales, and A.T. Alpas

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The adhesion of aluminum to tool surfaces during the hot forming of sheet aluminum alloys presents challenging tribological problems. Graphite and boron nitride are commonly used as aluminum adhesion mitigating solid lubricants for hot forming processes, but lubricant breakdown in high-stress areas, such as corners and bends, remains an issue compromising the quality of the formed parts as well as the tool life. Low-melting temperature enamels may provide an affordable and easy to apply alternative. In this study, vitreous (amorphous glass) and devitrifying (two phase crystalline glass) layers were deposited on the surface of sheet aluminum samples with a sedimentation technique. Enamel lubrication was effective in preventing aluminum transfer to the steel counterface. Hence, the prospect exists for the use of these enamels as aluminum workpiece lubricants in hot forming operations.

Keywords AA5083 aluminum alloy, adhesion, boron nitride, coefficient of friction, enamels, hot forming lubricants

1. Introduction

The hot forming of aluminum sheets provides flexibility when forming parts with complex shapes, achieving high-production rates at a low cost. However, the adhesion of aluminum to tool surfaces is a drawback of this process. The resulting defects not only cause deterioration of the surface quality of the panels, but also slow down production because the deposits must be removed from the dies. Graphite and boron nitride are among the solid lubricants commonly used to alleviate adhesion problems, but can be difficult to remove and too expensive to use in large quantities (Ref 1) and neither is completely effective in eliminating adhesion of aluminum to the tool. Consequently, new protective lubricants that mitigate aluminum adhesion must be developed. Most of the experiments aimed at studying the aluminum adhesion problem have been conducted at room temperature (Ref 2). In other studies, the high-temperature performances of liquid lubricants have been evaluated (Ref 3-5). Coatings like electroless nickel and tungsten carbide-based cermets have been reported to limit the adhesion of aluminum to die materials at hot forming temperatures (Ref 6), but the

application of these coatings to large die surfaces is both expensive and time consuming.

The use of low-melting-temperature enamels as lubricants in the metal working industry dates back to the 1940s (Ref 7). Various types of glasses have been used as lubricants in the hot extrusion of steel (Ref 7, 8), as well as nonferrous alloys, including nickel-based super-alloys among others (Ti- and Mo-based alloys) (Ref 9-11). The use of glass blankets in the stretch forming of aluminum and magnesium sheets has also been reported (Ref 12, 13). What these reports do not address is whether glass could be used as a lubricant in the hot sheet forming of aluminum alloys. Because enamels have a low shear strength at temperatures above their softening temperature, they could provide a low coefficient of friction (COF) that reduces the adhesion of aluminum to the dies.

The present study explores the potential of vitreous and devitrifying enamels, with and without BN additions, as low-friction adhesion mitigating lubricants to be used between aluminum and steel. Tribological experiments were conducted with the enamel surfaces heated to 470 °C while the sliding steel counterfaces were held at a lower temperature. An AA5083 grade aluminum sheet metal was selected for these experiments since it is used extensively in commercial hot forming processes (Ref 14). Coefficients of friction for the vitreous and devitrified enamels were determined and the mechanisms of material transfer were studied.

2. Materials and Experimental Methods

2.1 Deposition Process of Enamel Layers

Test samples were made of aluminum alloy AA5083 with the following composition (wt.%): 4.4% Mg, 0.79% Mn, 0.17% Fe, <0.2% Si, 0.07% Cr, 0.01% Cu, and the balance aluminum. Two types of enamels were deposited on the aluminum surfaces. These were:

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A.R. Riahi and A.T. Alpas, NSERC/General Motors of Canada Industrial Research Chair, Department of Mechanical, Automotive, and Materials Engineering, University of Windsor, Windsor, ON, Canada N9B 3P4; and A.T. Morales, General Motors Research and Development Center, 30500 Mound RD, Warren, MI 48090-9055, USA. Contact e-mail: ariahi@uwindsor.ca.

- (1) Vitreous enamel (commercial lead borosilicate glass) with a melting point of 1050 °C. The composition range of the enamel powder was (40-50)% PbO, (15-25)% SiO₂, (15-25)% B₂O₃, (2-5)% Al₂O₃, (2-5)% TiO₂, (2-5)% ZrO₂, and (2-5)% SnO₂ (wt.%). This type of enamel is designated as VE in this work.
- (2) Devitrifying enamel which designated as DVE in this work (commercial lead zinc borate glass) has a melting point of 1260 °C. The composition range of the DVE powder was (55-75)% PbO, (10-25)% ZnO, (10-19)% B₂O₃, (2-5)% SiO₂, and (2-5)% CuO (wt.%).

The 25 mm × 25 mm coupons were cut from 1.2 mm thick AA5083 sheets. One surface of each of the samples was ground with 400 grit emery paper and cleaned ultrasonically in acetone. Enamel powders were then applied to the aluminum samples with a sedimentation method where the substrate was placed at the bottom of a cylindrical container filled with ethanol of 50 mm in height. Enamel powders were added to the ethanol at a rate of 0.05 g/cm² of the sample's top surface area. The mixture was stirred and left to settle for 2 h. This process allowed the powder mixture to deposit uniformly on the aluminum surface. The ethanol was extracted and the samples were dried in air. The enamel layers obtained using this process ranged in thickness from 100 to 150 μm. Both VE and DVE layers were prepared in the same way.

Additionally, a set of samples with vitreous (VE) and devitrifying (DVE) enamels with BN-rich layers on their top surfaces were prepared. BN (hexagonal) powders were applied to the surface of the enamel samples by pressing BN-lubricated aluminum coupons against the coupon on which enamel layers had been deposited using a pressure of 160 kPa. These samples were then heated in a furnace. The (VE + BN) samples were held at 520 °C for 20 min and the (DVE + BN) samples were treated at 365 °C for 30 min. Figure 1(a) shows the cross-sectional SEM micrograph of a (VE + BN) coating. The coating was intentionally fractured under bending stress to expose the cross-sectional microstructure. The EDS spectrum (Fig. 1b) shows that BN particles were concentrated in the layers adjacent to the top surface of the VE enamel layer (Fig. 1c).

2.2 High-Temperature Tribological Experiments

The friction experiments were conducted using a commercial tribometer (CSM High-temperature pin-on-disc tribometer) with a ball-on-flat configuration. A sliding speed of 2×10^{-3} m/s, and a load of 1.0 N were used. Most tests were carried out for 500 cycles (500 rotations of the lubricated samples in contact with steel balls). Each rotation produced a sliding track of diameter of 10^{-3} m. The tests were conducted against AISI 52100 type steel balls (6 mm diameter) to evaluate the interaction between enamels deposited on AA5083 aluminum samples and steel. During the sheet aluminum hot forming operation, the steel die is usually at a lower temperature. The samples with an enamel layer were heated to 470 °C (a typical hot working temperature for AA5083 aluminum), but the counterface ball was held at 200 °C during the heating stage. The applied load of 1 N generated an average contact pressure of 31 MPa—well above the typical average forming pressure of 2 MPa used in some commercial aluminum hot forming processes (Ref 15). After each experiment, contact surface morphologies were examined using an SEM equipped with EDS.

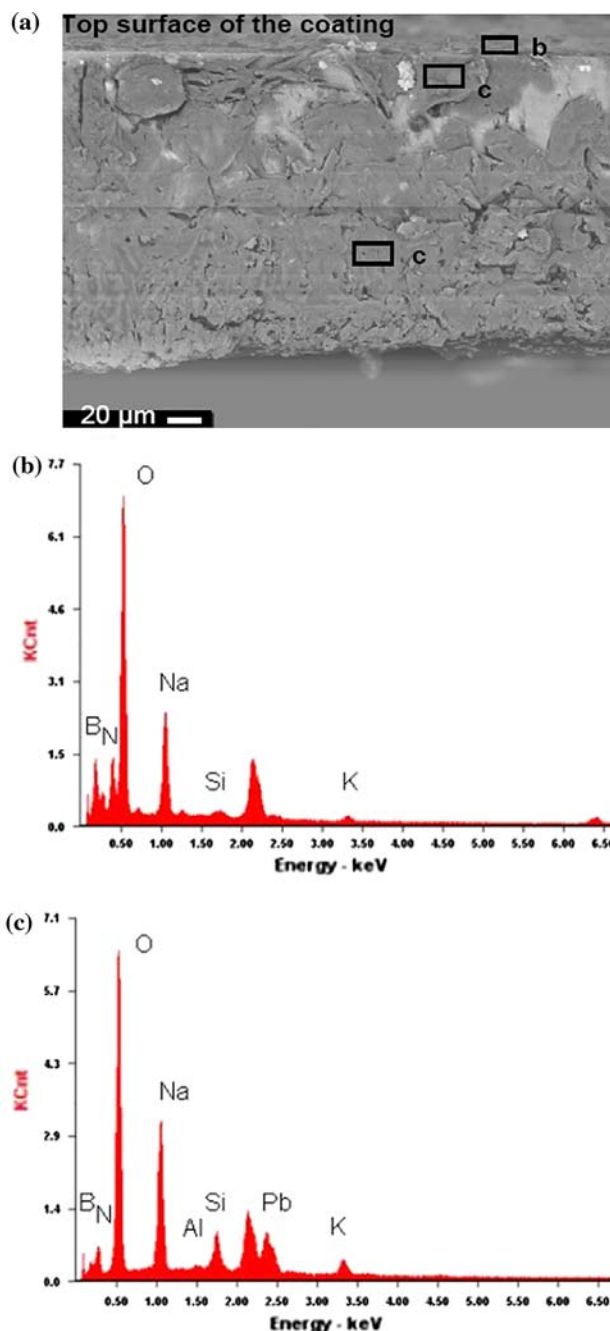


Fig. 1 (a) SEM micrographs of the fractured cross section of the (VE + BN) enamel layer. (b, c) EDS analyses of the areas shown in Plate 1(a) as “b” and “c” (the two areas shown as “c” in Plate 1(a) have the same composition). (The peak at 2.2 keV is for gold used as a conductive coating on SEM specimens)

3. Results

3.1 Measurement of Coefficient of Friction

3.1.1 Vitreous and Devitrifying Enamels. Variations of the COF obtained during experiments conducted at 470 °C using VE type enamels on aluminum sliding against the AISI 5200 steel counterface are shown in Fig. 2. A plot showing the variation of the COF generated between the AA5083 and the AISI 52100 during unlubricated contact at the same

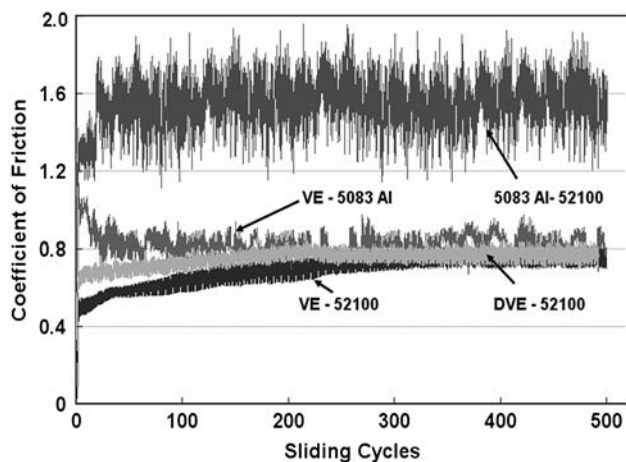


Fig. 2 COF vs. number of cycles for VE and DVE lubricants on AA5083 sliding against AISI 52100 steel and AA5083 aluminum at 470 °C. COF of AA5083 vs. AISI 52100 steel in the unlubricated condition is shown as baseline. DVE lubricated AA5083 vs. 52100 steel is given as reference

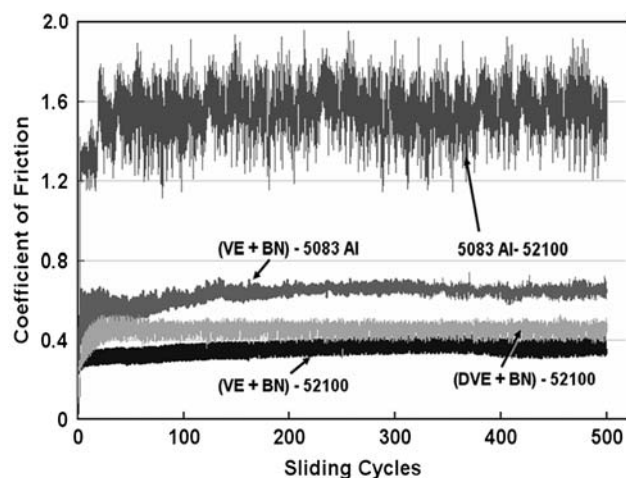


Fig. 3 COF vs. number of cycles for (VE + BN) and (DVE + BN) lubricants on AA5083 sliding against AISI 52100 steel

temperature is given as a reference. The initial COF between the steel and aluminum was 1.30, which later increased to an average of 1.54. The measurements exhibited large fluctuations after the first 50 cycles. The COF curve of the VE sliding against the AISI 52100 was characterized by a gradual increase of the mean COF from 0.50 to 0.72 at 260 cycles, after which the COF remained constant with much smaller fluctuations compared to aluminum-steel contact. VE lubricated AA5083 aluminum coupons were tested against the AA5083 pins (of 6 mm diameter, similar to the steel counterface) to compare the effect of the counterface material. The initial COF value generated against the AA5083 counterface was high (1.2), and then decreased to 0.9, which was still higher than that of the VE lubricant-steel contact. Figure 2 also shows how the COF for the DVE-lubricated aluminum sliding against steel varies with the number of cycles. In this case, the COF increased gradually to 0.77 for the first 200 cycles and maintained this value until the end of the test.

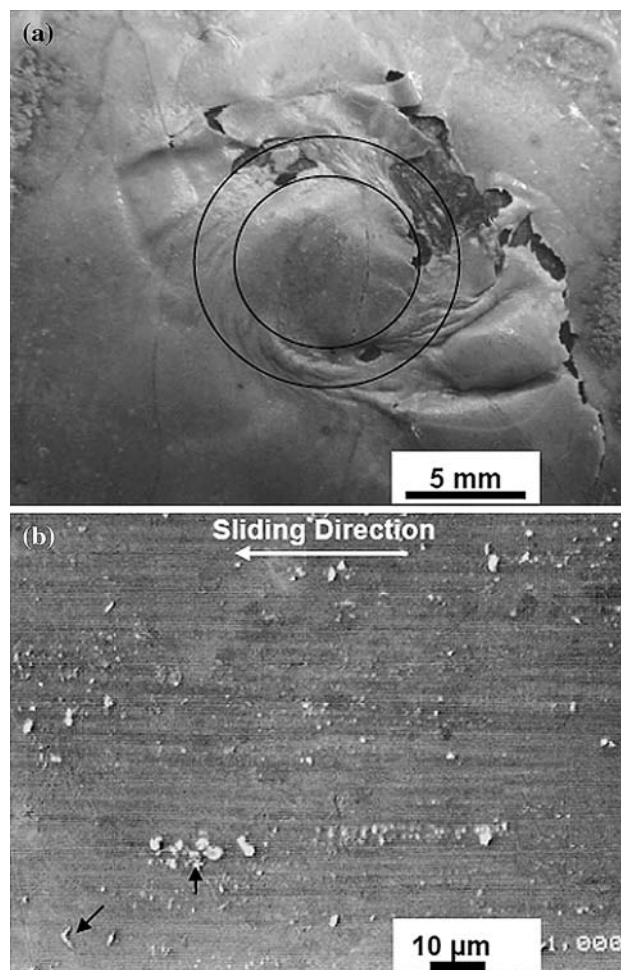


Fig. 4 (a) Macroscopic damage (wrinkling) on the VE lubricant layer in the vicinity of the sliding track after 500 cycles. The sliding track is identified as the encircled area. (b) High-magnification view of the sliding track on the VE lubricant. Cylindrical and spherical debris particles produced from the VE lubricant surface are indicated by arrows

In summary, the use of VE as lubricant reduced the high COF caused by unlubricated aluminum-steel contact by approximately 50%. The COF values of the surfaces lubricated with either enamel, VE or DVE, were not significantly different. The effect of the counterface material was evident from the comparison of VE lubricants tested against steel and aluminum, where the latter produced a higher COF.

3.1.2 Enamels with BN-Rich Layers. The (VE + BN) lubricated aluminum sliding against AISI 52100 steel showed an initial average COF of 0.30 that increased and stabilized at a low constant value of 0.37, as shown in Fig. 3. Therefore, the use of (VE + BN) enamel lubricants with BN-rich layers resulted in a 70% decrease in the unlubricated COF of AA5053-AISI 52100. There was no particular advantage to running the (VE + BN) lubricated surfaces against an aluminum counterface, because this had resulted in a higher COF compared to a steel counterface. The COF of (DVE + BN) lubricated aluminum sliding against AISI 52100 steel also revealed a smooth increase from 0.3 to 0.5 in the first 20 cycles and then stabilized at an average COF of 0.45 through the end of the test.

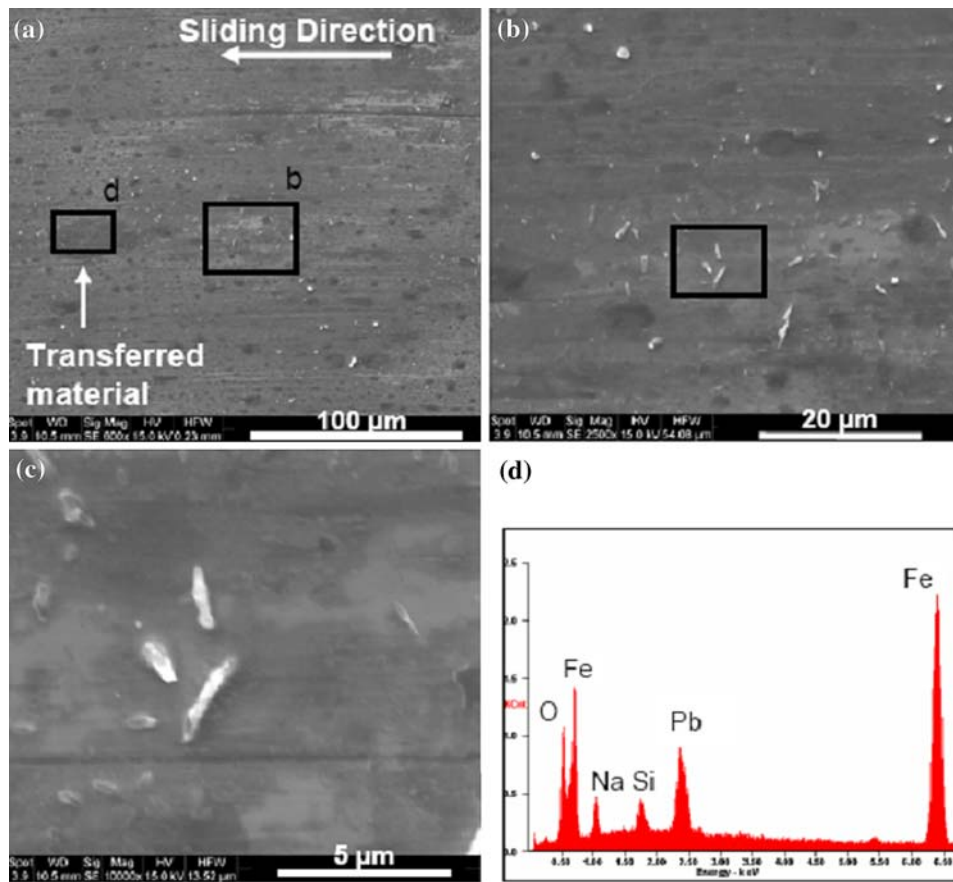


Fig. 5 (a) Material transfer on the AISI 52100 steel counterface from the VE-lubricated aluminum. (b) Magnified view of area shown in Plate 5(a) as “b”. (c) Magnified view of area inside the frame in Plate 5(b) showing debris particles. (d) EDS analysis of the material transferred to the counterface from the area indicated as “d” in Plate 5(a)

3.2 SEM Observations of Lubricant Surfaces and Counterfaces

At the end of the sliding test, it was observed that the VE film on the AA5083 aluminum was partially removed during sliding. However, no drastic increase in COF curve (Fig. 2) that would indicate metal-to-metal contact was observed. The detached parts of the film at and around the wear track are shown in Fig. 4(a), which also reveals that the damage was the result of the deformation and macroscale ‘wrinkling’ of the lubricant layer at 470 °C. Figure 4(b) shows a high-magnification SEM secondary electron image of a typical area within the sliding track on the VE-lubricated film. The surface was fairly smooth and featureless, with the exception of longitudinal deformation lines extending parallel to the sliding direction, and small (1-3 μm) spherical debris particles generated during wear. The corresponding surface morphology of the AISI 52100 steel ball counterface is shown in Fig. 5(a). This micrograph, together with the high-magnification micrographs in Fig. 5(b) and (c), show that material transfer to the counterface surface occurred with fine debris particles producing a cylindrical morphology (with lengths 5-10 μm). The material that transferred to the ball surface was identified by EDS as containing Pb, Si, Na, and O, demonstrating that the transfer layer consisted of the same components as the VE (Fig. 5d). The cylindrical debris particles shown in Fig. 5(b) and (c) were formed as a result

of the rolling action of the fragments that were detached from the transferred VE layers.

SEM and EDS investigations were carried out on BN-rich enamel lubricated surfaces against steel (Fig. 6) and aluminum counterfaces (Fig. 7) to study the effect of the BN addition. Figure 6(a) shows the sliding track produced on the sample with VE lubricant and a BN-rich top layer (VE + BN) sliding against the AISI 52100 steel. The image shows a fine debris on the sliding surface, but no sign of wrinkles like those seen in the VE lubricant film without BN. The debris on the sliding track is a mixture of VE and BN particles generated during sample preparation. The material, transferred to the counterface (Fig. 6b) had the same composition as the (VE + BN) lubricant layer. The transferred material accumulated both in the sliding direction and sideways, i.e., normal to the sliding direction by following the slope of the curvature of the contact area at the tip of the pin indicating that the transferred material had low-shear strength. A dark and compact deposit consisting of BN powder accumulated at the back (with respect to the sliding direction of the enamel-lubricated aluminum sample) of this interlaced transfer layer.

The sliding track generated on the (VE + BN) lubricated material as it rubbed against the AA5083 counterface is shown in Fig. 7(a), for the purpose of comparing the effect of the damage produced by an aluminum counterface with the steel counterface. As Fig. 3 revealed, the (VE + BN) lubricant run

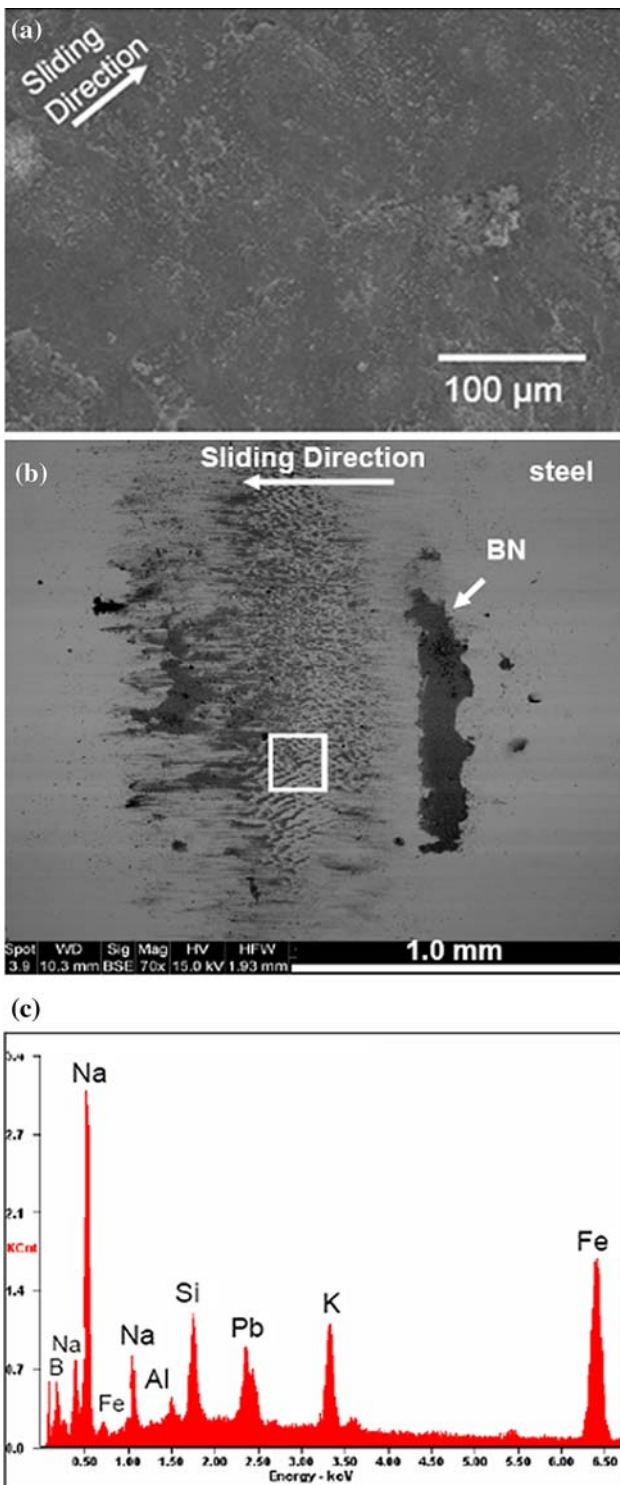


Fig. 6 (a) Sliding track of the (VE + BN) lubricated aluminum sliding against AISI 52100 steel. (b) The material transferred to the AISI 52100 steel counterface. The arrow shows sliding direction of the enamel-lubricated surface of aluminum sample. (c) EDS analysis of the material transferred to the counterface from the area inside the frame in Plate 6(b)

against aluminum resulted in a higher COF when compared to the steel counterface. Consistent with the high COF, the contact surface of the (VE + BN) running against aluminum exhibited signs of more severe damage as indicated by the cracks that

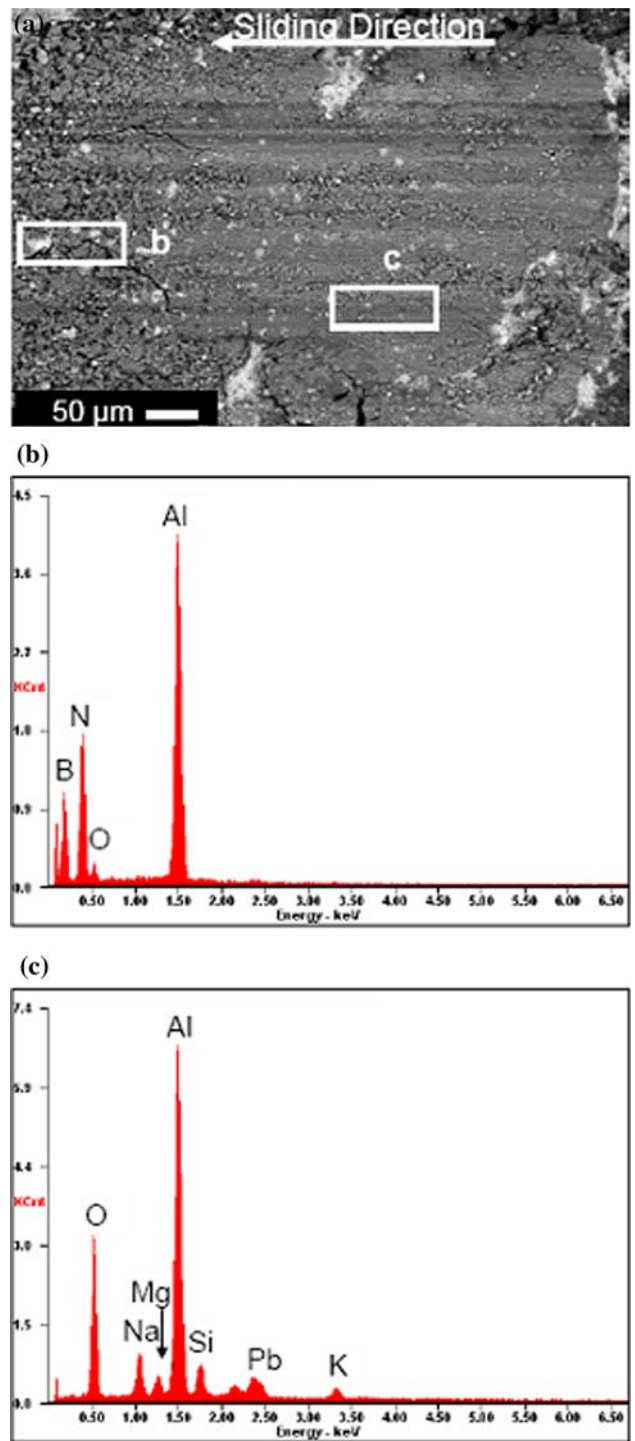


Fig. 7 (a) Sliding track on the (VE + BN) lubricated aluminum sliding against AA5083 aluminum. (b) EDS analysis of BN on the enamel surface in the area indicated as “b” in plate 7(a). (c) EDS analysis of the aluminum transferred from the counterface to the enamel surface in the area indicated as “c” in Plate 7(a)

were formed normally along the sliding direction marked in Fig. 7(a). In addition, material transfer from the aluminum counterface occurred both near these cracks (Fig. 7b), and in other locations of the contact surface (Fig. 7c).

It is also instructive to examine the surface morphologies of the DVE-lubricated aluminum coupons: An SEM micrograph

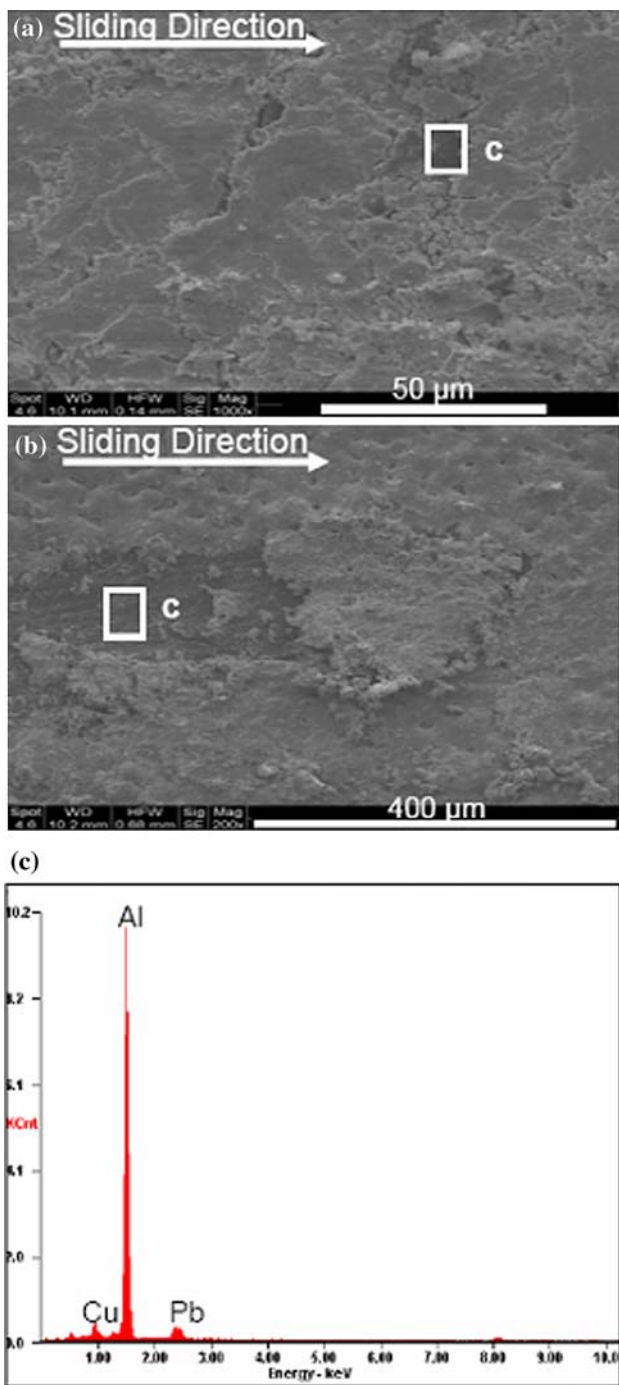


Fig. 8 (a) SEM micrograph from the sliding track on the DVE-lubricated aluminum sliding against AISI 52100 steel. (b) In some areas, the lubricant layer became detached from the aluminum substrate surfaces. (c) EDS analysis of the exposed substrate in the areas indicated as “c” in Plates 8(a) and (b)

from the sliding track of the DVE-lubricated samples tested against AISI 52100 steel is shown in Fig. 8(a), where the DVE lubricant layer shows fragmentation and spalling of small pieces that could be attributed to the low ductility of the DVE lubricant layer at 470 °C. The detached enamel layers left behind patches of exposed aluminum (Fig. 8b and c). Like all other enamels, DVE lubricant transfer to the steel counterface was observed, as shown in Fig. 9(a–d). Observations made on

the surface of the steel counterface in contact with the DVE lubricant indicated that the lubricant was transferred to the counterface in the form of thin layers that accumulated on top of each other. These layers can be seen in the area where they start to spall off from the surface of the counterface (Fig. 9b).

4. Discussion

The experiments were exploratory in nature and conducted with the purpose of providing insight into the role of material transfer from the contact surfaces of aluminum-lubricated by enamels. Comparison of the COF data obtained by testing various enamels produced as a part of this work against both steel and aluminum counterfaces at 470 °C provided an effective way to rank their performances. According to Fig. 2, the use of VE lubricants on AA5083 aluminum samples reduces the COF by 50% in comparison to the COF of the unlubricated aluminum samples sliding against the same AISI 52100 steel counterface. The main premise of enamel lubrication at 470 °C is that vitreous enamels have low-shear strength at this temperature. They also demonstrate low-adhesion tendencies when in contact with the steel surface, as can be observed in Fig. 5(a), where only a few isolated spots on the counterface exhibited evidence of transferred lubricant films. The almost featureless and uniform deformation pattern observed on the contact surface of the VE lubricant layer (Fig. 4b) is evidence of its capacity to act as an effective lubricant by carrying the applied load and distributing it homogeneously on its surface. Eventually, however, the VE lubricant layer displayed buckling type failure revealing the wrinkles seen in Fig. 4(a). Even so, after 500 sliding cycles the COF (Fig. 2) did not increase in a way to indicate metal-to-metal contact.

The lubrication of the aluminum surface with DVEs was also effective in reducing the COF (Fig. 2), albeit the DVE lubrication produced a slightly higher COF (0.77). It was somewhat surprising to observe that compared to VE, sliding damage on DVEs contact surface was more severe (Fig. 8a, b) and there was more enamel transfer to the counterface. In fact, Fig. 9(a) shows how the tip of the counterface surface was almost entirely covered by a transferred DVE film. The layered structure of the transferred film revealed that in the area shown in Fig. 9(b), material accumulation on the counterface occurred in the form of transfer of much thinner enamel sheets being transferred one after the other from the enamel surface. In this respect, the transfer mechanisms of both VE and DVE display similarities. The cylindrical debris of the transferred VE on steel suggests that thin sheets of enamel layers had been deposited and then rolled to assume this particular morphology. The salient point of metallographic observations is that both VE and DVE are helpful in preventing aluminum-steel contact and reducing the COF. Metal-to-metal contact was prevented during the friction experiments by using either of the lubricant types, although the stress applied was an order of magnitude higher than the average compressive stress applied during hot forming. Thus, prospects for the use of enamels as lubricants in hot forming operations exist. Attention should be paid to the fact that the friction appears to be controlled by enamel transfer to the steel, and enamel adhesion to the steel prevents a further reduction in COF. A smaller amount of enamel transfer to the steel translates into smaller COF, in favor of VE lubricants.

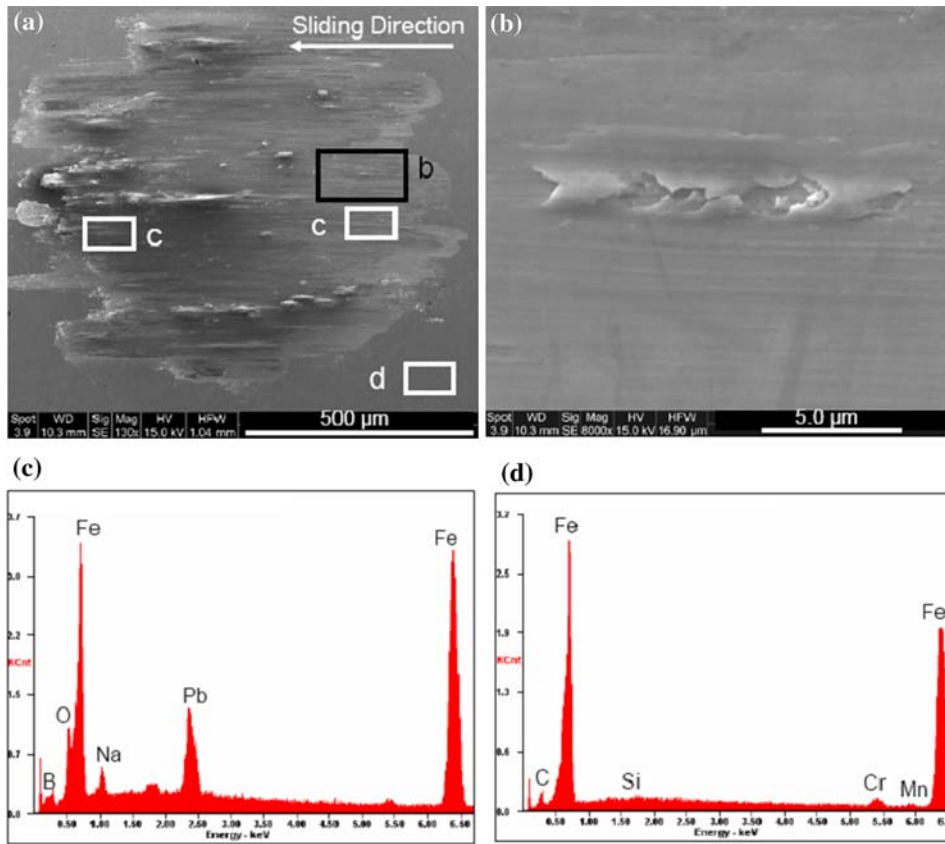


Fig. 9 (a) Material transfer from DVE to the AISI 52100 steel counterface. (b) Magnified view of the area indicated as “b” in Plate 9(a) shows the layered nature of the transferred material in the blistered area. (c) and (d) EDS analysis of the transferred material and steel counterface in the areas “c” and “d” in Plate 9(a)

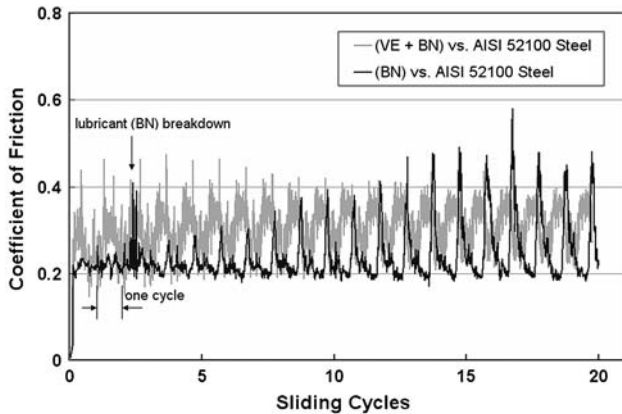


Fig. 10 Initial COFs of BN and (VE + BN) lubricated AA5083 samples sliding against AISI 52100 during the first 20 cycles

In practice, the deposition of enamels on the steel-forming die could provide an alternative strategy. A bench test simulation of this situation was carried out using AA5083 aluminum pins sliding against the VE enamel-lubricated surfaces, but the results did not support of this practice. Not only was the COF of the VE lubricant running against aluminum higher than that of the steel counterface (Fig. 2), but also an associated problem of aluminum deposition to the contact surface of the lubricant arose (Fig. 7). Most of the

damage occurred during the first five cycles (Fig. 2), during which time a high COF in excess of 1.0 was recorded.

Incorporating BN powders with the enamel powders and hot pressing them to produce enamel films with BN-rich top layers resulted in formation of lubricants with COF values that were almost half of those without the BN. As shown in Fig. 3, the COF of the (DVE + BN) lubricant was 0.45 and the (VE + BN) lubricant has a COF of 0.37. Thus, the (VE + BN) type enamel-based lubricant had a COF comparable to that of the commercial hexagonal BN (0.3-0.4) (Ref 6). In practice, BN powders can be sprayed on the enamel layers before the hot forming process of aluminum sheet. Therefore, (VE + BN) type enamels offer a convenient way of lubricating aluminum surfaces during hot forming. No aluminum transfer to the surface of the (VE + BN) lubricant was observed (Fig. 6a). Meanwhile, among all the enamels tested, the smallest amount of lubricant transfer to the steel surface (Fig. 6a) occurred in the case of the (VE + BN) lubricant, consistent with its low and stable COF during the long sliding cycle (500 cycles) tests.

Considering the tribological conditions during an actual hot forming operation, short cycle friction experiments simulate the contact between the forming die and workpiece on which a lubricant layer is applied. As a result, the low COF and antisticking properties of the lubricants applied to the aluminum work-piece prove important during the first few sliding cycles. According to the results presented above, enamel-based lubricants work better when used on the surface of an AA5083 aluminum sheet run against the tool steel.

In an attempt to compare the frictional performance of the (VE + BN) lubricated AA5083 sample surfaces with those lubricated with BN (hexagonal) that has not been mixed with enamel, short cycle pin-on-disc tests were conducted against AISI 52100. These tests that were conducted at 470 °C produced the COF data shown in Fig. 10 for the 20 sliding cycles after the initial contact. Accordingly, conventional BN-lubricated surfaces suffer 'lubricant breakdown,' which is displayed in the form of 'friction spike' after only a couple of sliding cycles (Fig. 10). The COF displays a reduction after this, but eventually exhibits a constantly increasing trend in which oscillating friction spikes dominate the sliding behavior after 13-14 cycles, a sign of the occurrence of metal-to-metal contact. On the other hand, the COF of the (VE + BN) lubricated samples sliding against AISI 52100 shows high-friction peaks of the order of 0.4, but this is likely a surface roughness effect due to the high-initial surface roughness of enamels ($R_a = 15 \mu\text{m}$). As the test proceeds however, the peak COF does not increase, indicating that the lubricant remains intact and provides steady friction behavior. This observation suggests that the application of the (VE + BN) lubricants to the aluminum surface may be more advantageous when compared to conventional BN lubrication by spraying, because the BN layers on the enamel surfaces provide protection against metal contact and maintain a stable COF during the initial contact cycles.

5. Summary and Conclusions

A simple sedimentation technique was used to deposit vitreous (VE) and devitrifying (DVE) enamel layers on the surface of AA5083 aluminum alloy samples. In addition, BN-rich layers were generated at the top surfaces of both VE and DVE enamels by mixing enamel powders with BN powders then by hot pressing the mixtures. Elevated temperature pin-on-disc tests were performed to evaluate the suitability of enamels as low friction and aluminum adhesion mitigating lubricants for hot forming. The following conclusions were obtained:

- (1) The COFs of VE and DVE layers on an AA5083 aluminum alloy measured against AISI 52100 steel at 470 °C were 0.72 and 0.77, respectively, with both VE and DVE proving effective in reducing the high apparent COF (1.54) of unlubricated aluminum-steel contact at this temperature.
- (2) No aluminum transfer occurred from the VE and DVE lubricated AA5083 surfaces to the AISI 52100 steel proving that metal-to-metal contact was prevented during the friction experiments by using either of the lubricant types, even though the applied stress during the tests was an order of magnitude higher than the average compressive stress applied during hot forming.
- (3) Enamel transfer to the counterface may be responsible for controlling the COF. The transfer occurred by

adhesion and accumulation of enamel sheets that either became rolled in the shape of cylindrical debris or spalled off layer-by-layer. A small amount of enamel transfer correlated with a low-COF value.

- (4) Aluminum surfaces lubricated by a mixture of (VE + BN) run against AISI 52100 steel showed a further 40% reduction in COF (0.37) compared to VE-lubricated surfaces without BN. (DVE + BN) lubrication also provided a relatively low COF (0.45).
- (5) Compared to conventional BN lubrication, (VE + BN) type lubricants did not exhibit 'lubricant breakdown' but provided stable COF values during short cycle sliding tests leading to the prospect that their use in hot forming operations may prove beneficial.

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