Friction and anti-galling properties of hexagonal boron nitride (h-BN) in aluminium forming

B. Podgornik¹, F. Kafexhiu¹, T. Kosec², J. Jerina³, M. Kalin³

¹Institute of Metals and Technology, Ljubljana, Slovenia

²Slovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia

³Laboratory for Tribology and Interface Nanotechnology, University of Ljubljana, Slovenia

Abstract

In the present work the friction and anti-galling properties of the h-BN in a steel-aluminium contact have been investigated, focusing on the possibility of replacing graphite with the h-BN in the aluminium forming operations. Extruded AA2014 alloy, widely used for the production of complex parts, was used. Investigation included the effect of h-BN powder size and concentration on the level and stability of the coefficient of friction, galling resistance and the quality of the Al surface. Tribological and anti-galling properties at room and elevated temperature of 400°C have been carried out on the load-scanner test rig. Graphite was used as a reference solid lubricant. In the case of the h-BN powder three different particle sizes were used, i.e., 0.5 µm, 5 µm and 30 µm, added to NGL class-2 industrial grease in the concentrations of 5 %, 10 % and 20 %. Results show that white h-BN, as a solid lubricant, is capable of successfully replacing graphite and providing “clean” surface, but only under mild contact conditions. It has similar lamellar structure but inferior load-carrying capacity, with its lubrication and anti-galling properties very much depending on the powder size, concentration and temperature. Contrary to the graphite tribological properties of h-BN deteriorate with

¹Corresponding author; tel.: +386 1 4701930, fax: +386 1 4701939, e-mail: bojan.podgornik@imt.si

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increased concentration but improve with temperature and powder size. Best results are obtained for concentration of 5 % and 30 μm powder size.

Keywords: solid lubricant, boron nitride, Al extrusion, friction, galling.

1. Introduction

In metal forming operations lubricants are essential, not only to reduce friction and effect stress and strain distribution, but also to reduce tool wear, increase the forming limit, improve workpiece surface quality and to ease the release of the finished part [1-3]. Hence, lubrication is critical, especially in hot metal forming. Inadequate lubrication or a breakdown of the lubrication film will cause direct contact between workpiece and the die, followed by transfer and build-up of the softer workpiece material on the tool surface, also known as galling [4]. Galling is a common problem in forming applications that may change the tool geometry, increase the friction force, cause tearing in a severely strained area and lead to workpiece rejection [5]. Several parameters are known to affect galling, with the risk of galling being reduced by decreasing tool surface roughness, minimizing sliding distance, decreasing contact pressure, using low friction coating or by addition of lubricants [6,7].

Automotive industry makes ever increasing demands on reduced weight of car chassis and components thus resulting in extensive use of light-weight materials, including aluminium, magnesium and titanium alloys [8]. Especially aluminium-based alloys present a number of interesting properties, such as low density, corrosion resistance, thermal conductivity, recyclability and good mechanical properties [9]. However, the formability of these metals is lower and much more demanding than that of steel [8,9]. They are also very prone to adhere to the tool's bearing surface [10]. The tribological mechanisms taking place during forming of light

metals are very complex and can involve adhesion, mechanical interaction of surface asperities, ploughing, deformation and fracture of surface oxide layers [10,11]. These mechanisms usually act simultaneously, thus posing considerable challenge and requirements also from the lubrication point of view [3,12]. Therefore, simultaneous development in surface engineering and forming lubricants is crucial [13].

In cold forming lubrication is mainly based on liquid lubricants, which can be water- or oil-based [14]. However, commonly used metal forming lubricants are often flammable, contain active hazardous elements and often require additional cleaning procedures and solvents for removal from the formed surface. Post-cleaning and disposal of these lubricants and solvents can be difficult, costly and environmentally unfriendly [1]. On the other hand, hot metal forming operations make extensive use of solid lubricants which are less demanding from the application point of view. Beneficial action of solid lubricants is mainly related to the lamellar structure and softness of these materials [15]. The most extensively used solid lubricant, found in almost all hot forming applications is graphite, followed by MoS₂, which is due to their low cost, availability and excellent lubrication performance. However, graphite needs moisture for good lubricity, finally resulting in a very dirty and potentially harmful environment [16]. It may also leave dark stains on the formed surface, which can lead to parts rejection. Lubrication properties of graphite have been extensively investigated, explained in detail and well documented [15,17]. However, there are also other compounds with similar structure which were recognized as the potential solid lubricants [18].

Different studies indicate [1,19-21] that the boron based compounds such as the hydrogen borate, borate esters, boric acid and alkali-borates are among the best lubrication additives when it comes to forming aluminium alloys. They exhibit very low friction as a consequence of low friction layers generated by tribochemical reaction and show strong interatomic bonding and

rigidity of layers thus preventing scuffing and galling. Boric acid for example was found to form chemically bonded films on the oxidized surface of aluminium and its alloys [1]. Another low friction boron compound is a hexagonal boron nitride (h-BN). The h-BN also has a lamellar crystalline structure which is similar to those of graphite and MoS₂, but is white in color and superior in terms of thermal stability [22]. In the h-BN lamellar structure the bonding among molecules within each layer is covalent, whereas the bonding between adjacent layers are weak Van der Waals forces [23]. This kind of structural properties of the h-BN provides easy shear along the basal plane of the h-BN. It was also found that even single-layer h-BN can show low friction [24]. These properties make the h-BN a good solid lubricant candidate. Although h-BN micro- and nano-particles are well-known solid lubricant additives providing low friction and slow wear rate [15,19,25], not many reports exist on the performance of the h-BN in a high temperature forming applications.

The aim of the present work was to determine friction and anti-galling properties of the h-BN in steel-aluminium contact at room and elevated temperature and to investigate the effect of h-BN powder size and concentration. Special focus was on the possibility of replacing graphite with h-BN in aluminium forming application's.

2. Experimental

2.1 Materials and lubricants

Al-alloy used in this investigation to evaluate lubrication and anti-galling properties of h-BN was extruded AA2014 alloy with a hardness of 135 HB (Wolpert Dia testor 3b). It is a high strength aluminium alloy used for complex machine parts. The chemical composition of the alloy was the following (mass content in %): 93.02 % Al, 0.07 % Cr, 4.30 % Cu, 0.18 % Fe, 0.74 % Mg, 0.88

% Mn, 0.72 % Si and 0.09 % Zn. Specimens in the shape of discs ($\phi 42 \times 8$ mm) were cut from the extruded bar and the surface was polished to an average surface roughness (R_a) of ~ 0.05 μm . As a counter material a hardened DIN 100Cr6 bearing steel balls ($\phi 10$ mm) and vacuum heat treated Dievar hot work tool steel (Böhler Uddeholm) cylinders ($\phi 10 \times 100$ mm) were used. 100Cr6 steel balls were commercial bearing balls with a hardness of 60 HRC and roughness $R_a = 0.02$ μm . Hot work tool steel cylinders (0.35 % C, 0.2 % Si, 0.5 % Mn, 5.0 % Cr, 2.3 % Mo, 0.6 % V) were first vacuum heat treated, using austenitizing temperature of 1000°C and double 2h tempering at 610°C , followed by fine surface grinding and polishing. Vacuum heat treatment and polishing resulted in cylinders hardness of 46 HRC (Rockwell 4JR, Instron B2000) and average roughness (R_a) of 0.10 μm , properties typical for Al-forming dies.

As the base lubricant, a commercially available lithium-based NLGI class 2 grease was used, made from the lithium 12-hydroxy-stearate thickener, high-quality mineral oil, corrosion inhibitors and anti-oxidants. Base grease had a dropping point of 190°C and density of 940 kg/m^3 . Natural graphite powder with 96 % purity and an average particle size of ~ 5 μm was used as a reference solid lubricant, which was mixed with the base grease in 5 %, 10 % and 20 % concentrations, respectively. In the case of the h-BN solid lubricants, commercial powders with 98.5 % purity (Lowerfriction Lubricants, Canada) were employed, using three different particle sizes; 0.5 μm , 5 μm and 30 μm . All h-BN powders were found to be highly crystalline with all peaks of hexagonal boron nitride and complete three-dimensional ordering, as shown by the XRD analysis [21]. The h-BN powders were then mixed with the base NLGI-2 grease in concentrations of 5 %, 10 % and 20 %, respectively. Mixing of the solid lubricant powder into the base lithium grease was done manually, preheating grease to 50°C and stirring for about 10 minutes. For all mixtures uniform distribution without solid particles agglomeration was obtained. The lubricants' designations and description are given in Table 1.

Table 1: Description of lubricants used in the investigation

Solid Lubricant	Particle size	Concentration	Designation
Base NLGI-2 grease			L
Graphite	5 μm	5 %	G-5
		10 %	G-10
		20 %	G-20
h-BN	0.5 μm	5 %	B05-5
		10 %	B05-10
		20 %	B05-20
	5 μm	5 %	B5-5
		10 %	B5-10
		20 %	B5-20
	30 μm	5 %	B30-5
		10 %	B30-10
		20 %	B30-20

2.2 Tribological testing

Lubrication properties and effect of the h-BN powder size and concentration on the coefficient of friction and wear rate at room temperature were investigated using reciprocating sliding ball-on-flat contact configuration (Fig. 1a). In reciprocating sliding tests the 100Cr6 steel ball was loaded against the stationary Al-alloy disc, with the lubrication grease containing solid lubricant particles applied on the disc surface prior the test. Tests were performed for 10 min at a frequency of 2.5 Hz and amplitude of 10 mm, which resulted in an average sliding speed of 0.05 m/s and total sliding distance of 30 m. Testing load was 4.5 N, corresponding to a maximum Hertzian contact pressure of 500 MPa. The test results and lubrication performance of the h-BN at room conditions were evaluated in terms of the average coefficient of friction and the Al-alloy disc wear volume and wear rate, measured by 3D white-light optical interferometry (Alicona, InfiniteFocus G4).

A high temperature performance and the ability of the h-BN solid lubricant to prevent galling and Al-alloy transfer to the steel cylinder were evaluated under a single-pass progressively loading sliding contact using Load-scanning test rig (Fig. 1b). Tests were performed at a constant sliding speed of 0.01 m/s, and at room and elevated temperature of 400°C. These conditions were aimed at simulating the Al extrusion process. Although the load-scanning test doesn't directly correspond to the extrusion its cross-cylinder configuration allows a gradual increase in load during a single stroke, with each point along the contact path of both specimens corresponding to a specific load and displaying a unique contact history [26]. In the current case cross-cylinder configuration consisted of AA2014 cylinder, which was forced to slide against a stationary hardened tool steel cylinder within a load range of 10 – 2,800 N. In the case of high temperature tests Al-alloy cylinder was heated by means of two temperature-controlled heating elements installed in the specimen holder (Fig. 1b). Anti-galling properties of the h-BN were determined by monitoring the coefficient of friction as a function of load, and by examining the contact surfaces after sliding and defining critical loads for galling initiation (L_{C1} ; Fig. 2a) and Al-transfer

layer build up (L_{C2} ; Fig. 2b). Furthermore, volume of the transferred Al-alloy on the tool steel cylinder surface and surface roughness of the Al-alloy cylinder sliding track were also analyzed using 3D white-light optical interferometry.

Fig.1: Tribological test configurations; (a) reciprocating sliding test and (b) load-scanning galling test

Fig. 2: Critical load definition for (a) galling initiation (L_{C1}) and (b) Al-alloy transfer layer build-up (L_{C2})

Before each test both contact specimens were ultrasonically cleaned in ethanol and dried in air, and about 50 mg of grease applied on the contact surface of the Al-alloy specimen before it was brought into the contact. All the tests were performed in a normal laboratory atmosphere, with each test repeated at least three times to ensure relevant results.

3. Results and discussion

3.1 Reciprocating sliding contact

An average coefficient of friction and Al-alloy disc wear rate for room temperature reciprocating sliding tests are shown in Fig. 3. In the case of the base lithium grease (L) average coefficient of friction was 0.17, with the steady state conditions reached after about 60 seconds or 2-3 m of sliding. However, throughout the test local lubrication film breakthroughs occurred as indicated by the friction spikes (Fig. 4). In terms of wear, the base lithium grease itself provided complete protection of the hardened bearing steel ball against wear when in contact with the AA2014 alloy disc. On the other hand, for base grease Al-alloy disc wear rate after 30 m of sliding was $4.9 \cdot 10^{-9} \text{ mm}^3/\text{Nm}$. As shown in Fig. 5 abrasive wear combined with plastic deformation was the prevailing wear mechanism.

In a highly loaded contacts and under starved lubrication which is typical for forming applications solid lubricants and solid lubricant additives are often required to prevent metal-to-metal contact and to protect contact surfaces from galling and wear [27]. However, under low loads and mild contact conditions experienced in the current reciprocating sliding tests the solid lubricants were found to have only limited and rather negative effect, as shown in Fig. 3. In the case of the graphite containing grease (G) similar friction level, wear mechanism and wear rates were observed as for the pure grease. Although graphite provided smoother sliding without

lubrication film breakdowns (Fig. 4) and about 5 % lower Al-disc wear rate, positive effect was limited to a lower graphite concentrations (5 % and 10 %). High graphite concentration of 20 % resulted in intensified lubrication film breakdowns, increased coefficient of friction (0.18), longer running-in and higher wear rate ($5.2 \cdot 10^{-9} \text{ mm}^3/\text{Nm}$).

Fig. 3: Reciprocating sliding test results for base NLGI-2 grease (L), Graphite (G) and h-BN lubricants at different concentrations and powder size (B)

Fig. 4: Coefficient of friction for room temperature sliding tests

Fig. 5: Typical SEM micrographs of AA2014 disc wear track tested at room temperature reciprocating sliding contact; (a) pure grease, (b) graphite G-5 and (c) h-BN B30-5

When using the h-BN solid lubricant powder three different powder sizes were used 0.5, 5 and 30 μm , respectively. In general, the use of the h-BN resulted in higher friction (0.17 – 0.18) and wear rate, especially when using high concentration of 20 % ($k = 7.0 - 7.5 \cdot 10^{-9} \text{ mm}^3/\text{Nm}$). However, the h-BN powder size also has a considerable influence. The highest and the most unstable friction was observed for the smallest h-BN powder size (0.5 μm), which also resulted in the highest wear rates. On the other hand, as the h-BN powder size increases from 0.5 to 30

μm wear rate for low concentrations steadily reduced from $5.3 \cdot 10^{-9} \text{ mm}^3/\text{Nm}$ to $4.0 \cdot 10^{-9} \text{ mm}^3/\text{Nm}$, thus providing the lowest wear rate and best surface protection, as shown in Figs. 3 and 5.

These results indicate that under room temperature and mild contact conditions the h-BN can provide comparable lubricating properties to the graphite, which may be contributed to similar lamellar structure [21,22]. However, sufficiently large particles of h-BN need to be used (above 5 μm) in order to facilitate easy sliding of solid lubricant's layers and surface protection. This would become increasingly important at large stress and strain rates experienced in the metal forming processes. On the other hand, contrary to severe contact conditions found in metal forming, solid lubricant particles may even impair tribological properties when used in low load and low shear rate situations. As shown by the current results, presence of solid lubricant particles in the grease leads to a higher friction, which is due to the higher solid lubricant shear strength compared to grease and base oil [18]. Higher the h-BN concentration, more pronounced is the friction increase for the steel/Al contact. Furthermore, for small solid lubricant particles and/or high concentrations (above 10 %) particles may start to agglomerate and deteriorate lubricating performance of base lubricant thus leading to increased wear [3].

3.2 Galling resistance

Results of the anti-galling properties evaluated in terms of critical load for galling initiation (L_{C1}) and Al-alloy transfer layer formation and build-up (L_{C2}) are shown in Fig. 6; for room temperature in Fig. 6a and for elevated temperature of 400°C in Fig. 6b. In the case of room temperature tests and pure lithium grease (L) first signs of Al-alloy adhesion on the tool steel cylinder surface were observed at a critical load of $\sim 700 \text{ N}$ and Al-alloy transfer layer build-up at 1400 N. As indicated by the coefficient of friction (Fig. 7a) which gradually increases from 0.12

to 0.25 as the load exceeds 2500 N, there was a gradual transfer and accumulation of Al-alloy on the surface of tool steel cylinder. By adding a graphite powder into the lithium grease similar critical loads but higher and less stable coefficient of friction were obtained when using graphite concentration of 5 %. However, as the graphite concentration increased to 10 % and 20 %, coefficient of friction was reduced to below 0.2 and critical loads L_{C1} and L_{C2} increased for ~40 % and even above 70 %, respectively. These results confirm positive action of a graphite solid lubricant when used under severe metal forming conditions and high loads. However, in this case sufficient concentration needs to be used to provide effective contact separation and galling protection. At low concentrations (≤ 5 %) action of graphite is restricted to some individual contact spots, manifested in unstable friction.

Although under mild reciprocating sliding h-BN shows comparable or even better tribological properties than graphite, this was not the case for room temperature contacts characterized by the severe plastic deformation. As shown in Figs. 6a and 7a, the use of the h-BN solid lubricant as an additive results in higher friction (0.2 – 0.4) and almost instantaneous galling initiation at a critical load even below 500 N and Al-alloy transfer layer build-up below 900 N, way inferior to the graphite or even pure lithium grease. The only exception was 5 μm size h-BN powder used in 5 % concentration. It gave similar friction and about 20 % improvement in galling resistance compared to the pure grease.

Fig. 6: Effect of solid lubricant type, size and concentration on critical loads for Al-alloy galling initiation (L_{C1}) and transfer layer build up (L_{C2}); (a) room temperature and (b) 400°C

Fig. 7: Coefficient of friction during load-scanning test against AA2014 Al-alloy; (a) room temperature and (b) 400°C

Al-alloy and above all lower friction. However, as shown in Figs. 6b and 7b considerable improvement is only obtained when using high graphite concentrations. By using 20 % graphite concentration critical load for galling initiation and Al-alloy transfer layer build-up increased for 2-4 times, from ~18 to 35 N and from 25 to 100 N, respectively. Furthermore, at elevated temperature of 400°C graphite in 20 % concentration provided very low and stable friction (~0.05) maintained in a broad load range (Fig. 7b). Contrary to room temperature, the h-BN tests show some beneficial properties when used at elevated temperature, although still not approaching graphite properties. As shown in Fig. 7b, the h-BN results in about 2 times lower friction and ~40 % better galling resistance as compared to pure lithium grease (Fig. 6b). Similar to reciprocating sliding test results effect of h-BN increases with the h-BN powder size and diminishes with increased concentration. The best results against AA2014 alloy at elevated temperature were obtained for large h-BN powder size (30 µm) and concentrations of 5-10 %.

Results of the adhered Al-alloy volume analysis (Fig. 8) performed on the tool steel cylinder wear track and the surface roughness of the mating AA2014 alloy cylinder (Fig. 9) confirm galling results. In the case of room temperature testing and pure lithium grease (Figs. 8a and 10a) $\sim 6 \cdot 10^{-4} \text{ mm}^3$ of Al-alloy has adhered to the tool steel surface at a load of 1000 N and $\sim 12 \cdot 10^{-4} \text{ mm}^3$ at 1400 N. Average surface roughness of the generated surface on the Al-alloy cylinder was $\sim 0.25 \text{ µm}$ (Fig. 9). Graphite gave steady improvement in galling resistance on one hand, reducing volume of adhered Al-alloy for ~20 % and 50 % as the graphite concentration increased to 5 % and 10 %, respectively, but resulted in increased Al-cylinder surface roughness. However, at 20 % practically no adhered material could be observed (Fig. 10b) and surface roughness of Al-alloy cylinder wear track dropped to $\sim 0.15 \text{ µm}$. The h-BN on the other hand resulted in ~60 % more Al-alloy being adhered on the tool steel surface (Fig. 10c), with the

volume increasing as the concentration of the h-BN powder is raised from 5 to 20 %. The exception is 5 μm size h-BN powder in 5 % concentration, giving results comparable to 10 % graphite concentration (Fig. 10d). Otherwise, volume of adhered Al-alloy increases with increased h-BN powder size. Correspondingly also surface roughness of the mating Al-alloy cylinder wear track deteriorates, increasing S_a from 0.25 μm to $\sim 0.60 \mu\text{m}$, which aggravates with h-BN powder size (Fig. 9).

At elevated temperature the graphite as a solid lubricant additive had similar beneficial effect, reducing the volume of adhered Al-alloy (Fig. 8b) and improving Al-alloy cylinder surface roughness (Fig. 9) as the concentration increases. Although h-BN could not match properties of graphite it still resulted in 15-30 % lower volume of Al-alloy adhered during high temperature sliding and similar Al-cylinder wear track surface roughness (0.25 – 0.30 μm) when used in low concentration of 5 %. However, if h-BN particles are too small ($< 5 \mu\text{m}$) they might lead to impairment of lubrication conditions, thus resulting in more intense Al-alloy transfer (Fig. 8b) and consequently increasing mating Al-alloy surface roughness (Fig. 9).

Fig. 8: Volume of adhered Al-alloy during (a) room and (b) elevated (400°C) load-scanning test

Fig. 9: Surface roughness of mating Al-alloy cylinder wear track

Fig. 10: 3D surface topography analysis of adhered Al-alloy; (a) pure grease, (b) graphite G-20, (c) h-BN B05-20 and (d) h-BN B5-5

A h-BN might have lamellar structure similar to graphite which provides easy sliding and low friction. However, it shows much lower load carrying capacity and poor surface protection against adhesion of Al-alloy at room temperature and high loads. On the other hand, its tribological properties and galling protection will improve when used at elevated temperatures.

4. Conclusions

The h-BN solid lubricant has lamellar structure similar to the graphite, but it shows different tribological behaviour and inferior load-carrying capacity. Using the h-BN as a solid lubricant additive in mild low load contacts results in the higher friction compared with the use of graphite but has comparable wear rates, which become superior when employing large h-BN powder size and low concentrations. However, in severe contact conditions with large shear rates typical for aluminium forming operations h-BN can't match graphite. It results in higher friction and inferior galling resistance. At room temperature the only exception improving lubricating and anti-galling properties of base grease against Al-alloy is 5 μm size h-BN powder at 5 % concentration. On the other hand, properties of h-BN improve at elevated temperatures,

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especially when using large powder size, but contrary to graphite it needs to be kept at low concentrations.

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