# Gracious Ngaile

Department of Mechanical and Aerospace Engineering, North Carolina State University, Campus Box 7910, Raleigh, NC 27695 e-mail: gngaile@ncsu.edu

# Frank Botz

Diversified Chemical Technologies, 15477 Woodrow Wilson, Detroit, MI 48238

# Performance of Graphite and Boron-Nitride-Silicone Based Lubricants and Associated Lubrication Mechanisms in Warm Forging of Aluminum

Although water/oil-graphite emulsions are widely used in warm forging processes, they carry environmental concerns. In an attempt to replace graphite-based lubricants in warm forging of aluminum alloys, two variants of boron-nitride-silicone lubricants were formulated. The two variants were made by dispersing boron nitride powder in polydimethyl siloxane oil at concentrations of 1% and 8%. The formulated lubricants were initially tested for their thermal degradation characteristics using a thermogravimetric analyzer and compared to the thermal degradation behavior of graphite and silicone oil lubricants. Ring compression tests were then carried out at 260°C and 370°C. Boronnitride-silicone lubricant variants did not show significant difference in performance as die temperature was increased from 260°C to 370°C. This is in contrast to graphite, which performed much better at  $260^{\circ}C$  than at  $370^{\circ}C$ , due to thermal oxidation. On the other hand, silicone oil exhibited the worst performance at  $260^{\circ}C$  and the best performance at 370°C. In both boron nitride lubricant variants, the polydimethyl siloxane facilitated hydrostatic/hydrodynamic lubrication at 260°C, with boron nitride acting as a barrier film that reduced friction. However, the lubrication mechanisms changed at  $370^{\circ}$ C, where the depolymerization of polydimethyl siloxane led to formation of silica due to thermal oxidation. Silica, together with boron nitride, acted as a film barrier with low shear strength. The dual lubrication mechanisms make boron-nitride-silicone lubricants suitable for a wide range of aluminum forging temperatures. [DOI: 10.1115/1.2805432]

Keywords: warm forging, lubricant formulation, graphite lubrication, boron nitride

#### 1 Introduction

The demand for high strength-to-weight ratio has resulted in drastic increase in the use of aluminum alloy forgings in the automotive and aerospace industries. The high strength-to-weight ratio and high corrosive resistance of aluminum alloys make this material ideal for applications where contact with water or humidity cannot be avoided, such as car wheels.

Numerous variables play significant roles in warm forging of aluminum alloys, including (a) temperature control of both the forging stock and the dies, (b) lubrication, (c) forming speeds, and (d) material forgeability. Due to the narrow temperature range in warm forging of aluminum, accurate control of the temperature of the stock and dies is crucial [1]. Furthermore, in order to successfully forge intricate shapes, the aluminum alloy should possess a high degree of forgeability, which has been shown to increase with temperature. For example, the flow stress of aluminum alloy Al 6061 can decrease by 50% as the temperature is increased from  $370^{\circ}$ C to  $480^{\circ}$ C.

To ensure proper flow of material at the tool-workpiece interface, with less propensity for galling and tool wear, good lubrication is essential. The lubricants that are commonly used for warm forging of aluminum are graphite-based derivatives [2–6]. The use of oil-graphite emulsions or water-graphite suspensions, however, is in jeopardy because of environmental concerns and health and safety regulations.

To address the environmental concerns pertaining to the use of graphite, development of high-molecular-weight transparent lubricant or synthetic lubricants both with and without solid lubricants has been carried out [4]. Some of the nongraphite lubricants developed to replace graphite have not gained wide acceptance in aluminum forging because of their high propensity toward galling as compared to graphite [7]. Nishimura et al. [7] investigated the influence of die coating by chemical vapor deposition, and physical vapor deposition where CrN, TiCN, TiAlN, WC, ZrO2, and SiAlON die coatings were used in injection upsetting of aluminum billets against oil-graphite lubricant, water-based boron nitride, and molybdenum disulfide. Molybdenum disulfide resulted in a high friction coefficient for all die coatings. Oil-based graphite and water-based boron nitride exhibited the same performance except for TiCN die coating, which exhibited the lowest friction when water-based boron nitride was used. One of the factors that has hindered the productivity of warm forging of some aluminum alloys is the low forging temperatures, which are usually set to the maximum temperature at which the lubricant can be functional.

The aim of this study is to formulate a lubricant based on dispersion of boron nitride powder on silicone oil that can be used for warm forging of aluminum. The formulated lubricant should withstand temperatures above the failure level of water-/oil-based graphite, which is widely used in industry.

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Fig. 1 Structure of hexagonal boron nitride structure

#### **2** Lubricant Formulation

Boron-nitride-silicone based lubricant combines the desirable characteristics of boron nitride and silicone oil, in anticipation that the formulated lubricant will be superior to graphite lubricants used in warm forging of aluminum alloys such as Al 6061.

Boron nitride is an advanced ceramic material with valuable chemical and thermal properties. The boron nitride considered in this study is the hexagonal layered lattice, as shown in Fig. 1. Similar to graphite, boron nitride has lattice layers that may be shifted easily. In contrast to graphite, boron nitride has high thermal stability and oxidation resistance. Whereas graphite oxidizes at  $370^{\circ}$ C, boron nitride oxidizes at  $1000^{\circ}$ C [8]. Thus, the temperature level at which boron nitride can still be effective is far greater than graphite. On the other hand, silicone is a polymeric compound based on a chain structure of alternating silicon and oxygen atoms with organic side chains (Fig. 2). The side chain is a methyl group in the dimethyl silicones, but other silicones may instead have longer alkyl chains. Silicones are thermally stable up to  $540^{\circ}$ C [9].

**2.1 Mechanism of Lubrication.** The boron nitride in the boron-nitride-silicone lubricant provides a physical barrier between tool and deforming material as the workpiece surface expands. Silicone oil provides good lubrication in the hydrostatic/hydrodynamic friction regime. Because of higher-temperature stability, silicone oil will also perform better under more severe conditions. However, at warm forging temperatures, the viscosity of silicone are expected, silicone by itself may provide insufficient separation and protection from wear between the tool and workpiece surfaces. Thus, the silicone oil is intended to reduce the internal friction in the barrier film created by the deposition of boron nitride on a surface. This barrier film will exhibit internal friction between the platelets as they slide over one another under pressure. The silicone oil will provide internal lubrication for the



Fig. 2 General structure of silicone

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Table 1 Lubricants analyzed

Lub. No.	Lubricant	Lubricant composition
1	Water graphite	Graphite (20%)
2	Silicone oil	Polydimethyl siloxane, viscosity: 350 cS at 25°C
3	Boron-nitride- silicone-1	Boron nitride 1%, polydimethyl siloxane, viscosity: 50 cS at 25°C
4	Boron-nitride- silicone-2	Boron nitride 8%, polydimethyl siloxane, viscosity: 350 cS at 25°C

barrier film and will reduce the friction between the boron nitride platelets and between the boron nitride and metal surfaces.

**2.2** Composition of Boron-Nitride-Silicone Variants. Two variants of boron-nitride-silicone were formulated. In variant one, polydimethyl siloxane and hexagonal boron nitride powder were mixed. The viscosity of the siloxane was 50 cS at  $25^{\circ}$ C, and the level of boron nitride in the mixture was kept at 1% by weight. In variant two, polydimethyl siloxane and boron nitride powder were mixed such that the viscosity of the silicone oil was 350 cS at  $25^{\circ}$ C, and the boron nitride level was 8%. This was intended to be a higher-viscosity formulation with better high-temperature performance.

# **3** Testing Methodology

**3.1 Determination of Thermodegradation of Lubricants.** The formulated lubricants boron-nitride-silicone-1 and boronnitride-silicone-2, together with graphite and silicone oil lubricants (Table 1), were first analyzed for thermal degradation by measuring the weight loss of the sample as a function of temperature under a nitrogen atmosphere. A TA Instrument TGA-2950 thermogravimetric analyzer was used under an increase of  $10^{\circ}$ C/min from room temperature to 600°C. The samples were purged with nitrogen gas at a flow rate of 85 ml/min. The sample weights for water graphite, boron-nitride-silicone-1, boronnitride-silicone-2, and silicone oil were 8.1920 mg, 9.2340 mg, 8.6460 mg, and 9.7220 mg, respectively.

Figure 3 shows the thermogravimetric analyzer curves. The weight of water-based graphite dropped sharply to 25 wt % at 100°C, but did not significantly drop further as the temperature was raised to 600°C. The thermogravimetric analyzer curve for silicone oil shows that the oil was stable at up to  $250^{\circ}$ C, gradually decreased in wt % between  $250^{\circ}$ C and  $400^{\circ}$ C, and had its wt % drop sharply to zero between  $400^{\circ}$ C and  $570^{\circ}$ C. This is due to decrease in viscosity as a function of temperature, which led to volatilization of low-molecular-weight silicone. Boron-nitride-silicone-1 lubricant exhibited a different thermal degradation pattern from silicone oil. A rapid drop in wt % of boron-nitride-silicone-1 is observed between  $250^{\circ}$ C and  $450^{\circ}$ C. From



Fig. 3 Thermogravimetric analysis curves of water graphite, silicon oil, boron-nitride-silicone-1, and boron-nitride-silicone-2

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Fig. 4 (a) Experimental setup and (b) scheme of top and bottom dies

 $450\,^{\circ}$ C to  $600\,^{\circ}$ C, the weight of boron-nitride-silicone-1 gradually decreased to 1%. The residue of 1% is the boron nitride. The volatilization of boron-nitride-silicone-1 started at a lower temperature than for silicone oil because of low viscosity of boron-nitride-silicone-1 (Table 1). The boron-nitride-silicone-2 exhibited a similar thermal degradation pattern to that of silicone oil. This is because boron-nitride-silicone-2 contained silicone oil with a viscosity of 350 cS. Boron-nitride-silicone-2, however, shows a slightly higher thermal degradation temperature than for silicone oil because it contained 8 wt % of boron nitride. At 600 °C, a residue of 10 wt % was observed. Most of the residue is believed to be boron nitride.

# 3.2 Performance Evaluation of Lubricant Using Ring Compression Test

3.2.1 Experimental Setup and Test Procedures. The lubricant variants were tested using the ring compression test, which is capable of varying die temperature up to 450°C and a maximum billet temperature of 1000°C. A schematic of the experimental setup is shown in Figs. 4(a) and 4(b). The die temperature and billet temperature can be controlled to  $\pm 3$  deg. In the ring compression test, a flat ring-shaped specimen is compressed to a predetermined height reduction. The internal and external diameters of the compressed ring are very sensitive to the friction conditions at the tool-ring interface. If friction is high, the internal diameter is reduced as deformation increases, but if friction is low, the internal diameter is increased as deformation increases. To determine shear friction factor for a lubricant, the measured reduction in the hole is superimposed on friction calibration curves (see Fig. 7). These calibration curves are determined using finite element analysis.

A 150 ton hydraulic press was used to run the tests. The dies for the ring compression test were all made from AISI A2 steel and were hardened to 60RC. In addition to the shear friction factors obtained from the ring test surface, chemical analysis of the ring samples was conducted using scanning electron microscopy,



Fig. 5 Specimens before experiment

which was coupled with energy dispersive X-ray spectroscopy to determine the degradation of lubricant as a function of temperature and deformation.

The experimental procedures were as follows: The height, inner diameter and outer diameter, of the ring sample were recorded. The specimens were heated on an electric furnace to  $430^{\circ}$ C. Immediately after spraying lubricant on the heated dies (bottom and top dies), the specimen was placed on the bottom dies and compressed to different reduction levels at a ram speed of 5 mm/s. For each lubricant test condition, three samples were used. The tests used die temperatures of  $260^{\circ}$ C and  $370^{\circ}$ C.

3.2.2 Specimen Preparation and Application of Lubricants. The ring specimens were machined from a 38 mm diameter rod of aluminum 6061. The specimens were cut to a ring height of 12.5 mm and drilled and reamed to a final inner diameter of 19.20 mm. Figure 5 shows the ring samples before the experiment. The average surface roughness  $R_a$  for the specimen was about 4  $\mu$ m. The lubricants that were tested in this study are given in Table 1. All the lubricants were applied to the bottom and top dies using a pressure spray gun that was set at 0.83 N/mm<sup>2</sup>.

#### 4 Test Results and Discussion

4.1 Performance Evaluation Based on Shear Friction Factor. The friction calibration curves discussed above show friction factor *m*, which quantifies lubricant effectiveness. The friction shear law was used because it is more relevant for bulk metal forming than Coulomb's law. The friction shear law can be expressed as  $\tau = m\overline{\sigma}/\sqrt{3}$ ,  $(0 \le m \le 1)$ , where  $\tau$  is the shear stress, *m* the friction shear factor, and  $\overline{\sigma}$  the flow stress of deforming material. Figure 6 shows photographs of selected ring samples for the four lubricants tested. Visual observation of graphite-lubricated ring samples shows that varying the die temperature from 260°C to 370°C resulted to a drastic change in the hole size. At 370°C, the hole of the ring sample decreased from 19 mm to about 0.2 mm, implying severe friction with increase in temperature. The reverse is observed with silicone oil.

At  $260^{\circ}$ C, the lubricant performed poorly, but at  $370^{\circ}$ C, a drastic improvement is observed, implying that lubricity increases with temperature. Boron-nitride-silicone-1 and boron-nitride-silicone-2 showed slight decrease in friction factor as temperature increased from  $260^{\circ}$ C to  $370^{\circ}$ C, with boron-nitride-silicone-2 outperforming boron-nitride-silicone-1.

To obtain actual shear friction factors, the experimental data were superimposed on friction calibration curves. Figure 7 shows the experimental data for ring height reduction (Re) varying from 40% to 65% when the tests were conducted at die temperature of 260°C. The test data for die temperature of 370°C are shown in Fig. 8. The friction calibration curves were obtained by carrying out nonisothermal finite element simulation using 2D-DEFORM software package. All the experimental conditions were incorporated into the simulations. The simulation model setup included die temperature levels of 260°C and 370°C, ring sample temperature of 430°C, and ram speed of 5 mm/s.

The average shear friction factor values are plotted in Fig. 9 for

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Fig. 6 Deformed samples with graphite, silicone oil, boronnitride-silicone-1, and boron-nitride-silicone-2 (Re=60%)

both die temperatures. Water-based graphite exhibited an average friction factor of m=0.36 at a die temperature of  $260^{\circ}$ C. However, at a die temperature of  $370^{\circ}$ C, the friction factor went as high as m=0.92. At a die temperature of  $260^{\circ}$ C, silicone oil lubricant exhibited an average shear friction factor of m=0.48, the highest of any lubricant at that temperature. Interestingly, at  $370^{\circ}$ C, silicone oil exhibited a friction factor of m=0.23, the lowest of any tested lubricant at that temperature. The average friction factor for boron-nitride-silicone-1 at  $260^{\circ}$ C was m=0.45. A slight improvement was observed when the die temperature was increased to  $370^{\circ}$ C, where boron-nitride-silicone-1 exhibited a friction factor of m=0.43. Boron-nitride-silicone-2, which performed better than boron-nitride-silicone-1 at all temperatures, exhibited a friction factor of m=0.3 at  $260^{\circ}$ C and m=0.28 at  $370^{\circ}$ C.



Fig. 7 Friction factors for four lubricants tested at a die temperature of 260  $^\circ\text{C}$ 

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Fig. 8 Friction factors for four lubricants tested at a die temperature of 370 °C

#### 4.2 Performance Evaluation Based on Surface Quality

4.2.1 Surface Chemical Analysis and Surface Morphology. Surface analysis of the tested ring samples was carried out in order to study the morphology of the remaining lubricant film on the ring surface and also to observe the lubricant chemical elements on the ring surface. A scanning electron microscope coupled with energy dispersive spectroscopy was used. Figure 10 shows examples of spectral analysis of water graphite and boronnitride-silicone-2 and locations where X-ray and micrographs were taken.

The influence of die temperature on the lubricant film morphology was examined via scanning electron microscope, as shown in Fig. 11. The micrographs for graphite-lubricated specimens show some difference in surface morphology between die temperatures of  $260^{\circ}$ C and  $370^{\circ}$ C. With boron-nitride-silicone-2 and silicone oil, there is also an apparent difference in the morphology of the remaining lubricant film on the ring samples. Both boron-nitridesilicone-2 and silicone oil show abundant tiny white flakes on the surface at  $370^{\circ}$ C. These tiny flakes are hardly visible at  $260^{\circ}$ C. Although boron-nitride-silicone-2 contained silicone oil and boron nitride from the micrographs, it is difficult to determine whether any flakes originated from boron nitride. It is clear, however, that the formation of these tiny flakes significantly enhanced lubrication performance for boron-nitride-silicone-1, boron-nitridesilicone-2, and silicone oil.

In order to study the lubricant film composition at different temperatures and reduction, spectral analysis was carried out. Four sampling locations were used for each condition. The at. % from four locations were averaged to fairly represent the lubricant film composition. Figures 12(*a*) and 12(*b*) show the lubricant chemical elements observed on the ring surface lubricated with



Fig. 9 Influence of temperature on water graphite, boronnitride-silicone-1, boron-nitride-silicone-2, and silicone oil

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Fig. 12 Lubricant chemical elements for graphite (a) at  $260^{\circ}$ C and (b) at  $370^{\circ}$ C

Fig. 10 Spectral analysis and location where micrographs were taken

graphite at  $260^{\circ}$ C and  $370^{\circ}$ C, respectively. The aluminum chemical element represents the exposed aluminum sample within the lubricant film depth. At  $260^{\circ}$ C, the ring surface had 80% atomic graphite (carbon). Only about 20% atomic aluminum can be observed from the ring sample material. This shows that at this tem-

Lubricant	260°C	370°C
Graphite	100 шт 27-400-об	100 µm
Boron-Nitride- Silicone-1		μ <u>100 μm</u>
Boron-Nitride- Silicone-2	100 µm kotsas so del isto 100 µm	
Silicone oil	15-100-um 40/5million 1000 1000 1000 1000	

Fig. 11 Influence of temperature on lubricant film morphology for water graphite boron-nitride-silicone-1, boron-nitride-silicone-2, and silicone oil (Re=60\%)

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Fig. 13 Lubricant chemical element for boron-nitridesilicone-2 (a) at 260 °C and (b) at 370 °C

perature, graphite lubricant film was still intact, confirming the low friction factor obtained in the ring compression (see Fig. 9). The surface chemistry at 370°C shows a drastic change. Only small percentages of carbon—20 at. % (Re=40%) and 50 at. % (Re=60%)—are observed. Figure 12 also shows a significant increase in percentage of atomic aluminum. This indicates that at 370°C, most of the graphite was depleted, leading to the very high friction factor of m=0.92.

The surface chemistry of boron-nitride-silicone-2 is given in Fig. 13. The lubricant elements detected were carbon, oxygen, and silicon. At 260°C, carbon was about 45 at. %, oxygen 14 at. %, and silicon 10 at. %. Aluminum from the ring sample was 30 at. %. When the die temperature was increased to 370°C, the at. % of lubricant elements decreased, while the at. % of aluminum increased from 30% to 50%. Despite the decrease in at. % of the lubricant chemical elements as die temperature increased from 260°C to 370°C, the shear friction factor dropped from m=0.30 to m=0.28. This suggests that different lubrication mechanisms may have occurred at the different temperatures, as shall be discussed in the next section.

A similar scenario displayed by boron-nitride-silicone-2 is observed with silicone oil (Fig. 14). At 260°C, the lubricant chemical elements carbon, oxygen, and silicon exhibited average at. %



Fig. 14 Lubricant chemical element for silicone oil (a) at  $260^{\circ}$ C and (b) at  $370^{\circ}$ C

of 50, 18, and 17, respectively. Aluminum from the ring sample shows 7 at. % at Re=40% and 22 at. % at Re=60%. As with boron-nitride-silicone-2, when the die temperature increased to 370°C, the atomic percentage of lubricant chemical elements dropped, while the percentage of elemental aluminum exposed on the surface from the ring sample material increased significantly. In contrast to boron-nitride-silicone-2, the shear friction factor improved considerably with increase in temperature, as seen in Fig. 9. Similar to boron-nitride-silicone based lubricants, different lubrication mechanisms/conditions were triggered by the temperature increase.

4.2.2 Influence of Temperature on Lubricant Chemical Reaction and on Lubrication Mechanisms. The thermogravimetric analysis of the four lubricants under nitrogen atmosphere indicated that graphite does not degrade as temperature increases. As can be observed in Fig. 3, the weight of water-based graphite remained constant at 25 wt % in the temperature range from  $100^{\circ}$ C to  $600^{\circ}$ C. The thermogravimetric analysis also suggested that silicone oil will exhibit uniform lubrication characteristics up to a temperature of  $400^{\circ}$ C, beyond which the lubricity will drop sharply until 570°C, when there will be no lubricant at the toolworkpiece interface. The ring test results, however, exhibited opposite trends on the performance of these lubricants.

The failure of graphite lubricant is attributed to thermal oxidation. The dies on which the lubricants were sprayed were preheated to  $260^{\circ}$ C and  $370^{\circ}$ C, and immediately after spraying, a ring sample preheated to  $430^{\circ}$ C was placed on the dies just before the ring compression test. The whole process (from spraying to ring compression) took about 5 s.

Within this time span, the following were possible reactions involving oxygen, carbon dioxide, or water.

$$\frac{1}{2}O_2 + C \Rightarrow CO \tag{1}$$

$$O_2 + C \Longrightarrow CO_2 \tag{2}$$

$$C + CO_2 \Rightarrow 2CO \text{ (multiplying reaction)}$$
 (3)

$$C + H_2O \Rightarrow CO + H_2$$
 (water gas reaction) (4)

These reactions are exothermic and favored thermodynamically. As discussed in Sec. 2, graphite is susceptible to oxidation at about  $370^{\circ}$ C. The products from the above reactions may have been free to escape within the 5 s, considerably reducing the amount of effective graphite on the die surface as seen in the chemical analysis. Because pressurized air was used to spray the lubricant, the atmosphere within the vicinity of the test was rich in oxygen, hence catalyzing the reaction.

In aluminum warm forging applications, the reactions could be suppressed to make graphite more effective at higher temperature, as by minimizing the process time or spraying graphite using other means that will not enrich oxygen at the forming zone.

The thermogravimetric analysis for silicone oil showed that above 400°C, the lubricant will deteriorate significantly. The chemical analysis and micrographs showed that at the die temperature of 370°C, tiny white flakes are formed on the ring surface, suggesting that thermodynamically induced chemical reactions take place. The tiny white flakes seem to have enhanced the lubricant performance. Thermo-oxidative degradation of silicone in the presence of air gives results much different from the degradation in nitrogen. Grassie and Macfarlane [10] and Camino et al. [11] showed that in polydimethylsiloxane reactions, oxygen can catalyze the depolymerization reaction of silicone to volatile cyclic oligomers, which leads to lower temperatures that trigger weight loss. Camino et al. [11] found that weight loss commences at a temperature of 290°C, as opposed to the 400°C observed in the thermogravimetric analysis experiment under nitrogen (Fig. 3). Camino et al. [11] also found that oxidation takes place on

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oligomers in gas and observed very fine powder of silica. Thus, the white tiny flakes observed in the ring compression tests, particularly at  $370^{\circ}$ C, are silica particles. The thermal reaction that took place in the presence of oxygen can be represented as follows [11]:



The interaction of oxygen during the degrading condensation phase depends on how oxygen diffusion and solubility compete against degradation and product evaporation. Since boron-nitridesilicone-2 had similar silicone oil as a base fluid, it is inferred that formation of silica took place in the same manner. However, boron-nitride-silicone-2 contained 8% boron nitride and this lubricant performed better at all temperature levels. The boron nitride composition may have been responsible for better lubricant performance at 260 °C, because silicone oil alone performed worse at this temperature.

The following are possible lubrication mechanisms for the formulated boron-nitride-silicone-1 and boron-nitride-silicone-2. At 260°C, silicone oil was in a fluid state, facilitating hydrostatic/ hydrodynamic lubrication, and acted as internal lubricant for borone nitride, which inhibited metal-to-metal contact. At 370°C, the silicone oil was no longer in a fluid form but had depolymerized to form silica particles. Thus, the lubrication mechanism was entirely dependent on gliding of silica and boron nitride particles at the tool-workpiece interface. The formation of silica particles further enhanced the performance of boron-nitride-silicone based lubricants.

In warm forging of aluminum applications using the formulated boron-nitride-silicone lubricants, enrichment of oxygen at the tool-workpiece interface is desirable because it catalyzes the formation of silica particles. Chemical reaction and formation of silica are a function of time and temperature. In this study, however, optimal reaction time was not investigated.

#### **5** Conclusions

The performance of water-based graphite, silicone oil, boronnitride-silicone-1, and boron-nitride-silicone-2 lubricants was studied. These lubricants were tested at different temperatures relevant for warm forging of aluminum. The ring compression test, thermogravimetric analyzer, and scanning electron microscope, coupled with energy dispersive spectroscopy, were used for testing and performance analysis.

The following conclusions are drawn from this study.

 Boron-nitride-silicone-2 exhibited the best performance among all lubricants tested at the die temperature of 260°C, with an average shear friction factor of m=0.3. This was followed by graphite and boron-nitride-silicone-1. Silicone oil had the worst performance at 260 °C, exhibiting a shear friction factor of m=0.48.

- At 370°C, silicone oil exhibited the best performance with a shear friction factor of *m*=0.23, implying that the performance of silicone oil improves significantly with increase in the die temperature. Depolymerization of silicone oil at higher temperature is enhanced by thermo-oxidation, leading to formation of tiny white flakes of silica. The formation of silica has shown to significantly enhance lubrication.
- The performance of graphite lubricant dropped significantly as the die temperature was increased. The shear friction factor increased from m=0.36 at 260°C to m=0.92 at 370°C.
- The formulated boron-nitride-silicone-1 and boron-nitride-silicone-2 lubricants did not vary significantly in performance as die temperature was increased from 260°C to 370°C. Both boron-nitride-silicone-1 and boron-nitride-silicone-2 contained polydimethyl siloxane and boron nitride, though at different percentages. At 260°C, the polydimethyl siloxane facilitated hydrostatic/hydrodynamic lubrication, with boron nitride acting as a barrier film, thus leading to low friction. At higher temperature (370°C), the lubrication mechanisms changed as polydimethyl siloxane depolymerized, leading to formation of silica due to thermal oxidation. Silica together with boron nitride acted as a film barrier with low shear strength. The dual lubrication mechanisms make boron-nitride-silicone based lubricants suitable for a wide range of aluminum forging temperatures.
- The thermogravimetric analysis under nitrogen atmosphere revealed that both graphite and silicone oil can exhibit different lubrication characteristics if conducted in air, due to thermo-oxidation and other chemical reactions.

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#### References

- Kuhlman, G. W., 2005, Bulk Forming ASM Handbook, ASM International, Materials Park, OH, Vol. 14A, pp. 299–316.
- [2] Nishimura, T., Sato, T., and Tada, Y., 1995, "Evaluation of Frictional Conditions for Various Tools Materials and Lubricants Using the Injection-Upsetting Method," J. Mater. Process. Technol., 53, pp. 726–735.
- [3] Siegert, K., Kammerer, M., Keppler-Ott, T. H., and Ringhand, D., 1997, "Recent Development on High Precision Forging of Aluminum and Steel," J. Mater. Process. Technol., 71, pp. 91–99.
- [4] Saiki, H., 1997, "Tribology in Warm and Hot Forging," *JSTP International Seminar on Precision Forging*, Osaka, Japan, Mar. 31–Apr. 1.
  [5] Forcellese, A., and Gabrielli, F., 2000, "Warm Forging of Aluminum Alloys: A
- [5] Forcellese, A., and Gabrielli, F., 2000, "Warm Forging of Aluminum Alloys: A New Approach for Time Compression of the Forging Sequence," Int. J. Mach. Tools Manuf., 40, pp. 1285–1297.
  [6] Sheljaskow, S., 2001, "Tool Lubricating Systems in Warm Forging," J. Mater.
- [6] Sheljaskow, S., 2001, "Tool Lubricating Systems in Warm Forging," J. Mater. Process. Technol., 113, pp. 16–21.
- [7] Nishimura, T., Sato, T., and Tada, Y., 1996, "Evaluation of Anti-Galling Characteristics by Observation of Adhesion Morphologies Using Injection Upsetting," J. Mater. Process. Technol., 62, pp. 235–241.
- [8] Rudolph, S., 2001, "Boron Nitride Release Coatings," Seventh Australian Asian Pacific Conference, The Minerals, Metals, and Materials Society, Tasmania, Australia, Sept. 23–26.
- [9] Lansdown, A. R., 1994, *High Temperature Lubrication*, Mechanical Engineering Publication Limited, London, p. 60.
- [10] Grassie, N., and Macfarlane, G., 1978, "The Thermal Degradation of Polydimethylsiloxanes," Eur. Polym. J., 14, pp. 875–884.
- [11] Camino, G., Lomakin, S. M., and Lazzari, M., 2000, "Polydimethylsiloxane Thermal Degradation, Part I, Kinetic Aspects," Polymer, 42, pp. 2395–2402.

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