

M.C. OH*, H. SEOK*, H.-J. KIM**, B. AHN*[#]**DE-LUBRICATION BEHAVIOR OF NOVEL EBS BASED ADMIXED LUBRICANT IN ALUMINUM P/M ALLOY****ZACHOWANIE NOWEGO LUBRYKANTU NA BAZIE EBS W TRAKCIE SPIEKANIA PROSZKU STOPU ALUMINIUM**

The objective of the present research is to develop a novel lubricant for Al-Cu-Mg P/M alloy and to address the effects of the lubricant and compaction pressure on sintered properties. A lubricant mixture consisting of Ethylene Bis Stearamide, Zn-Stearate, and fatty acid was newly developed in this study, and the de-lubrication behavior was compared with that of other commercial lubricants, such as Ethylene Bis Stearamide, Zn-Stearate, and Al-Stearate. Density and transverse rupture strength of sintered materials with each lubricant were examined, respectively. The microstructural analysis was conducted using optical microscope.

Keywords: admixed lubricant, compaction pressure, powder metallurgy, sintering, Al-Cu-Mg alloy

1. Introduction

Recently, the application of lightweight alloys has been greatly stimulated as the demand for fuel efficiency significantly increases in automotive and transportation industries. Al and Al alloys are most popular and widely used in lightweight engineering structures and components. Especially, Al alloys produced by powder metallurgy (P/M) processes are successfully replacing heavy Fe-based P/M alloys for manufacturing automotive parts [1, 2].

However, in Al P/M processes, there are two factors interfering with proper sintering process. First, air-atomized Al powders are often oxidized, resulting in the formation of Al₂O₃ layer on the surface which is hardly removed at the P/M sintering temperature. Another issue in the Al P/M process is residual lubricants between powder boundaries. Solid lubrication is generally employed to reduce internal friction between powder particles, to allow easier ejection of the compact from the die, and to reduce die wear improving life of P/M die. In general, the admixed lubrication is more productive and economical process than die-wall lubrication in powder metallurgy (P/M) process. However, in admixed lubrication, residual lubricants between powder particles interfere with inter-particle diffusion during sintering process, resulting in deterioration of mechanical properties of sintered Al P/M alloys [2-9]. In the present study, a newly developed lubricant mixture is proposed to improve sintered properties of Al-Cu-Mg P/M alloy, and its de-lubrication behavior is investigated.

2. Experimental

Commercial grade air-atomized Al, Cu and Mg powders were mixed to form the composition of Al-6Cu-0.5Mg (wt.%), and blended using 3D tubular mixer for 30 min. Al-Cu alloys are generally strengthened by θ (Al₂Cu) precipitates [10, 11], and the addition of Mg enhances diffusibility and improves sintered properties of Al P/M alloys by destroying Al₂O₃ layers on Al powder surface forming MgAl₂O₄ spinel structure [12].

A new solid lubricant mixture was prepared in the composition of 20 wt.% Zn-stearate, 15 wt.% diamide and balanced Ethylene Bis Stearamide (EBS). About 1 wt.% of the lubricant was introduced into the Al-6Cu-0.5Mg alloy powder in ad-mixed condition. The ejection properties and de-lubrication properties of the new lubricant were compared with those of conventional lubricants, such as, EBS, Al-stearate, and Zn-stearate. For the comparison of ejection properties, the sliding pressure between powders and die-wall and the stripping pressure between lower punch and die-wall were directly measured from compaction press. To determine, the de-lubrication behavior, thermal gravimetric analysis (TGA) and derivative thermal gravimetric analysis (DTG) were performed under a pure N₂ gas with a constant flow rate of 100 ml/min and heating rate of 10°C/min.

The Al-6Cu-0.5Mg powders with various lubricants (new EBS-based lubricant, EBS, Al-stearate, and Zn-stearate) were compacted under pressures of 150, 300, and 600 MPa and then sintered at 610°C for 60 min. in a pure N₂ gas, respectively. For the comparison of sintered properties with respect to various lubricants, standard transverse rupture strength (TRS)

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was measured using a universal testing machine (Instron Series 3360). Microstructural characterization was performed on the sintered materials using an optical microscope (Olympus BX41M).

3. Results and discussion

In general P/M processes, after the compaction pressure is released, the green compact must be pushed out of the die. In the initial stage of ejection, there is a peak pressure required to initiate ejection of the green compact which is termed as a stripping pressure generated by friction between lower punch and die wall due to infiltrated powders between them. After the peak pressure, there is a sliding pressure to sustain the ejection motion generated by friction between green compact and die wall [13, 14].

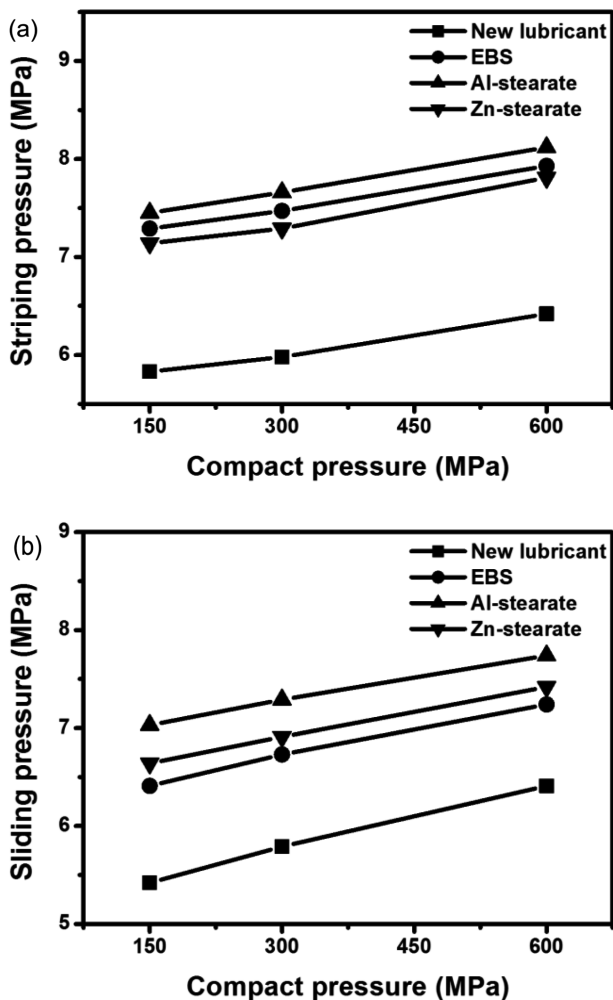


Fig. 1. Ejection characteristics of Al-6Cu-0.5Mg P/M alloy depending on the lubricants and compaction pressures: (a) stripping and (b) sliding pressures during ejection

Fig. 1 shows the relationship between applied compaction pressure and both stripping and sliding pressures during ejection with various lubricants. Both pressures increase as compaction pressure increases, meaning that higher ejection forces are required with higher compaction pressures, regardless of types of lubricant used. In each compaction pressure, all commercial lubricants give rise to similar ejection pressures shown

both in Fig. 1(a) and 1(b). On the other hand, the new lubricant mixture developed in this study significantly reduced both the stripping and sliding pressures more than 15% in all compaction pressures. In the new lubricant, appropriate combination of EBS and Zn-stearate provide a balanced reduction of both stripping and sliding pressures. Also, the addition of diamide in the new lubricant appears to diminish surface frictions, resulting in the enhanced ejection properties. This result indicates that the new lubricant in the present study has great potential as a novel lubricant in commercial manufacturing of P/M components.

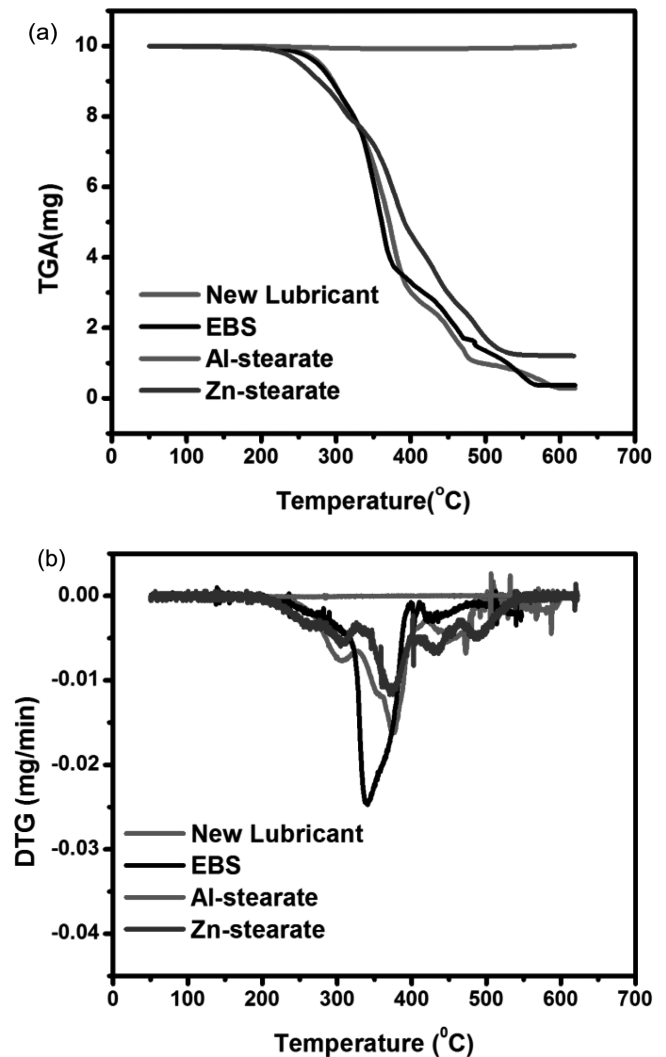


Fig. 2. Decomposition behavior of the lubricants examined by (a) TGA and (b) DTG thermal analyses

Fig. 2 shows the results of TGA/DTG analysis for various lubricants only. In Fig. 2a, TGA result shows most of lubricants exhibited rapid weight loss above $\sim 200^{\circ}\text{C}$ except the Al-stearate. This implies that three lubricants, new lubricant, EBS, and Zn-stearate, start decomposition and evaporate at temperatures of 200°C and above. The weight loss behavior of these three lubricants was similar with respect to temperatures. However, in case of Al-stearate, it is apparent that there is no perceptible weight loss as temperature increases. Al-stearate is decomposed into Al and stearic acid, and the stearic acid evaporates at elevated temperatures. However, remaining Al is combined with oxygen originated from the oxide layers of

alloy powders or from the atmosphere, and forms Al_2O_3 during sintering process [15, 16]. Also, weights of both the new lubricant and EBS were obviously reduced to about zero when temperature reached 610°C , meaning that those two lubricants were totally decomposed and evaporated at that temperature. In case of Zn-stearate, however, 10% of the original weight still exists at 610°C , because metallic Zn remains while other volatile organic phases evaporate after decomposition of Zn-stearate. In Fig. 2b, DTG result shows the optimum range of decomposition temperature. Most of lubricants exhibited a significant rate of weight change between 300 and 400°C , while the weight of Al stearate remained constant in all temperature range. The instant rate of the new lubricant was lower than that of EBS, but higher than those of all other lubricants.

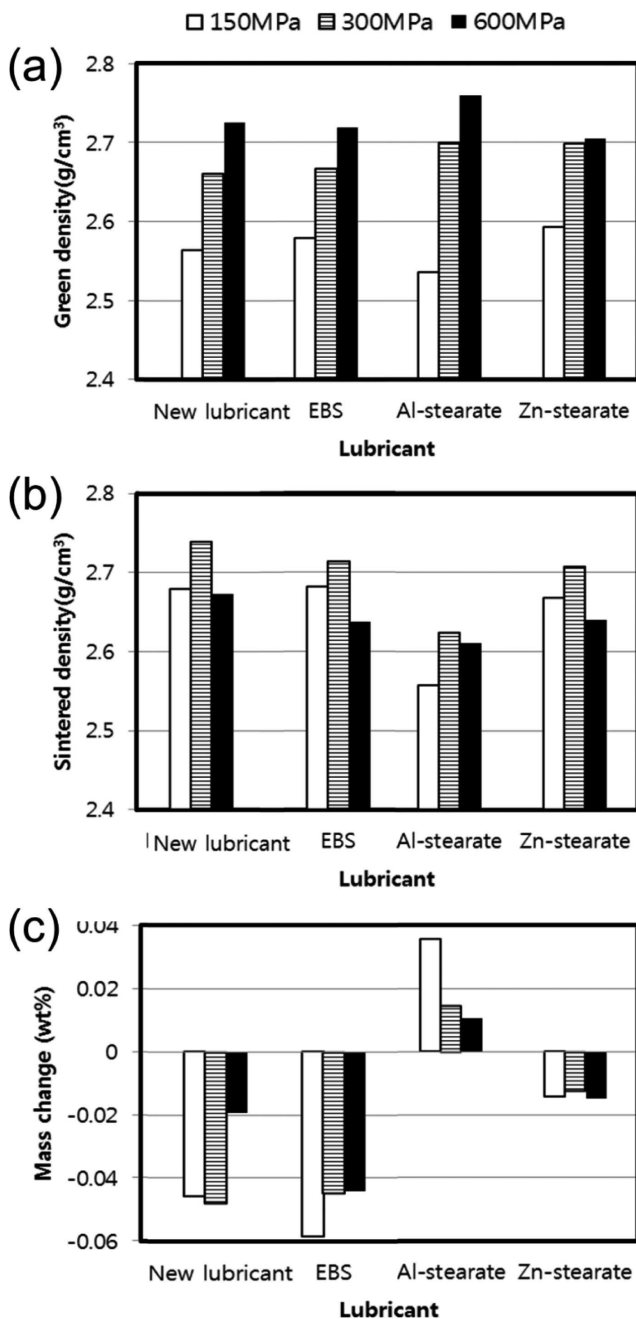


Fig. 3. Density variation depending on the lubricants and compaction pressures: (a) green density, (b) sintered density, and (c) weight differences between green compacts and sintered materials

Fig. 3 shows density variations depending on the lubricant types and the compaction pressures. As shown in Fig. 3a, green density with all four lubricants significantly increased from 150 to 300 MPa compaction pressures, while the increment was relatively small from 300 to 600 MPa. The maximum green density was achieved when Al-stearate lubricant was used and compacted at 600 MPa. After sintering, density variations exhibited different behavior than green density, as shown in Fig. 3b. Density generally increases when green compacts are sintered, however for the materials compacted at 600 MPa in this study, the density decreased significantly after sintering. For the Al-6Cu-0.5Mg alloy system, the compaction pressure of 600 MPa is in the pressure range where the powders are plastically deformed exhibiting relative green density above 97% of theoretical density [17, 18]. In this pressure range, internal pores after compaction are primarily closed ones, impeding the de-lubrication during sintering process. Therefore, the residual trapped lubricant which has much lower density than metallic alloy powders significantly reduces the total density. In case of compactions pressures of 150 and 300 MPa, the pressures are in the ranges where the powders undergo elastic deformation and local plastic deformation, respectively. Therefore, those materials after sintering become more densified because of sufficient de-lubrication through open and partially open pores, respectively.

The weight variations during sintering depending on the lubricant types and the compaction pressure are shown in Fig. 3c, representing weight changes between green compacts and sintered materials. The weight of material with Al-stearate lubricant was increased after sintering, while that of the materials with other lubricants decreased. Again, as mentioned above, Al phase decomposed from Al-stearate was immediately combined with adjacent oxygen and formed Al_2O_3 . This reaction results not only in the increase of weight, but also in expansion of the volume. Therefore, the overall density decreased after sintering when Al-stearate was used, as shown in Fig. 3b, although the weight increases after sintering. The results of sintered density and weight change account for excellent properties of the new lubricant.

To evaluate the effect of various lubricants on mechanical behavior of sintered materials, the results of TRS tests are shown in Fig. 4. For each case of lubricants, general trend in TRS values depending on the compaction pressures is uniform: highest TRS at 300 MPa, medium at 150 MPa, and lowest at 600 MPa. For the materials compacted at 600 MPa, as discussed above, closed pores containing residual lubricants deteriorated the strength. Also, when temperature arises during sintering of Al alloys, solid phase sintering is started at temperature between 300 and 400°C , while lubricant is not completely removed yet. As a result, remaining open pores in materials compacted at 600 MPa are even closed, trapping the lubricant. The materials with the new lubricant exhibited the highest sintered strength in all ranges of compaction pressures, as shown in Fig. 4. The enhanced strength from the new lubricant is attributed to better de-lubrication behavior during sintering due to the addition of new binder which is decomposed at relatively lower temperatures.

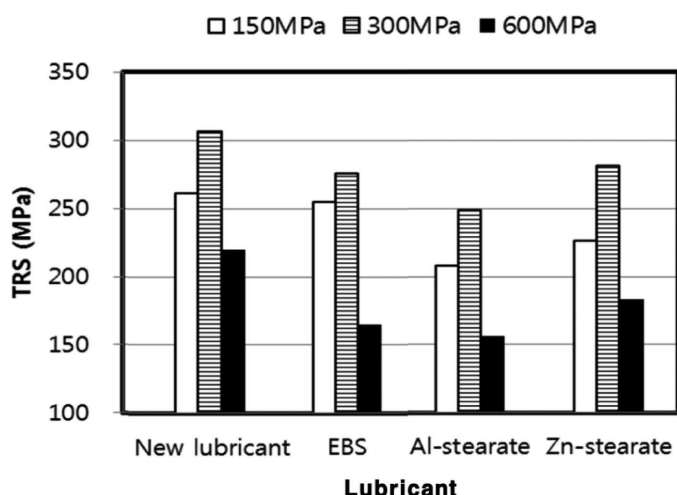


Fig. 4. TRS variation of sintered materials depending on the lubricants and compaction pressures

Fig. 5 exhibits optical micrographs of sintered materials compacted at 600 MPa and lubricated with EBS, Al-stearate, and Zn-stearate. For the material lubricated with EBS in Fig. 5a, small amounts of residual lubricants found in inter-particle areas, while the materials with Al-stearate in Fig. 5b and Zn-stearate lubricants in Fig. 5c appear to contain relatively large amounts and sizes of residual lubricants found in pores. In general, at higher compaction pressure, contact areas between powders are increased so that the inter-particle necking areas are readily developed into closed pores, resulting in poor de-lubrication properties [19]. Decomposition temperature of EBS is approximately 50°C lower than that of other lubricants so that disintegration of lubricant during sintering occurs ear-

lier when EBS lubricant is used. Therefore, the amount of residual lubricants is significantly less than other lubricants.

Fig. 6 shows microstructural variations of the material with newly developed lubricant in this study with respect to the compaction pressures. When compacted at 150 and 300 MPa, very small amount of lubricant remained after sintering, as shown in Fig. 6a and 6b. As discussed above, at the compactions pressures of 150 and 300 MPa, the powders undergo elastic deformation and local plastic deformation, respectively. Therefore, the lubricant are sufficiently decomposed and discharged through open or partially open pores so that the powder compacts can be more densified. However, when compacted at 600 MPa where the powders undergo plastic deformation, more lubricants were found at particle boundaries than at 150 and 300 MPa, as shown in Fig. 6c.

4. Conclusions

An EBS-based new lubricant mixture for Al-6Cu-0.5Mg P/M alloy was successfully prepared and compared with other commercial lubricants. The new lubricant mixture significantly reduced both stripping and sliding pressures during ejection of the powder compacts, attributed to the presence of diamide in the new lubricant diminishing surface frictions. According to thermal analyses, most lubricants completely evaporated around 600°C, whereas no weight loss was observed for the Al-stearate lubricant because the Al decomposed from Al-stearate simultaneously reacted with oxygen forming Al₂O₃. De-lubrication was promoted through open pores when the compaction pressure was relatively low (150 and 300 MPa), however when compacted at higher pressure

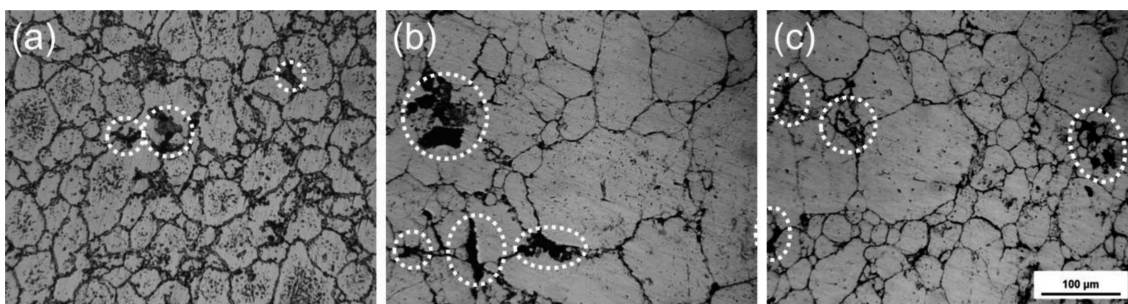


Fig. 5. Optical micrographs of sintered materials lubricated with (a) EBS, (b) Al-stearate, and (c) Zn-stearate lubricants, and compacted at 600 MPa. The yellow dotted circles indicate residual lubricants after sintering process

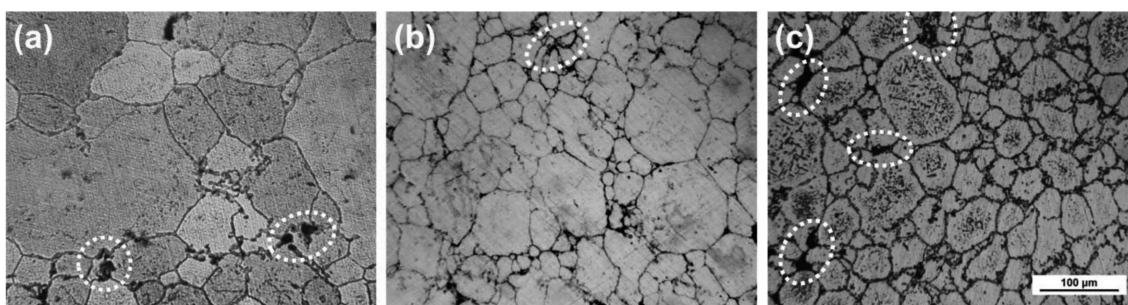


Fig. 6. Optical micrographs of sintered materials with the new lubricant and compacted in the pressures of (a) 150, (b) 300, and (c) 600 MPa. The yellow dotted circles indicate residual lubricants after sintering process

(600 MPa), the lubricants were trapped within closed pores, resulting in deterioration of flexural strength. The greatest sintered density and strength were achieved when the powder was compacted at 300 MPa with the new lubricant mixture proposed in the present study, possessing great potential as a reasonable replacement of conventional lubricants.

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