

Evaluation of new cold forging lubricants without zinc phosphate precoat

Mark Gariety^a, Gracious Ngaile^b, Taylan Altan^{a,*}

^aEngineering Research Center for Net Shape Manufacturing, The Ohio State University, 339 Baker Systems, 1971 Neil Ave, Columbus, OH 43210, USA

^bDepartment of Mechanical and Aerospace Engineering, North Carolina State University, Raleigh, NC 27695, USA

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Abstract

Zinc phosphate coatings plus metal soap lubrication system is required in nearly all steel cold forging operations. However, the chemical byproducts of this lubricant system are difficult to dispose of and have a negative environmental impact. In order to replace zinc phosphate based lubricants partially or completely, candidate lubricants were sought from lubricant manufacturers worldwide. The performance evaluation of these lubricants was conducted using the double cup backward extrusion test developed at the Engineering Research Center for Net Shape Manufacturing (ERC/NSM). With the use of the commercial FEM code DEFORM, friction factor calibration curves, i.e. cup height ratio vs. punch stroke, were established for different friction factor values. By matching the cup height ratio and the punch stroke from experiment to that obtained from FE simulations, the friction factor of the lubricants was determined. Three lubricants; namely, MEC Homat, Daido AquaLub, and MCI Z-Coat, were found to perform comparable to or better than zinc phosphate.

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1. Introduction

In cold forging operations, pressures as high as 2500 MPa are developed at the tool–workpiece interface. In addition, the interface temperature may reach several hundred degrees centigrade and the surface expansion may reach 3000% [1]. Thus, the lubricants used in cold forging may be subjected to very severe conditions. Failure to withstand the above-mentioned conditions implies failure to satisfactorily form the desired part and may lead to galling and/or die failure. A good lubrication system is essential for cold forging processes and it is a determining factor for making the process competitive with other manufacturing processes.

Since the mid 1930s, nearly all steel cold forging processes have used a zinc phosphate coating plus metal soap based lubrication system in order to withstand the severe conditions described above. The use of zinc

phosphate coating, however, has a negative environmental impact [2–4]. Thus, environmentally friendly lubricants capable of replacing zinc phosphate coating based lubrication systems are needed.

1.1. The zinc phosphate coating and soaping process for steel

When the term zinc phosphate coating is used, it refers to a two-part system consisting of a pre-coat and a lubricant. The function of the pre-coat is to prepare the surface of the billet for subsequent coating with the lubricant. This pre-coat, zinc phosphate, is commonly referred to as the conversion coating. In the case of cold forging, the lubricant may either be an extrusion oil or a soap. Because of a physical or chemical reaction that occurs between the lubricant and the pre-coat, the lubricant adhesion to the billet is improved. This is what allows the lubricants to withstand the high-pressure sliding conditions and large surface expansions that are common in cold forging operations. Fig. 1 shows the general structure of such a lubrication system. The treatment sequence to obtain this lubrication

*Corresponding author. Fax: +1 614 292 7219.

E-mail address: altan.1@osu.edu (T. Altan).

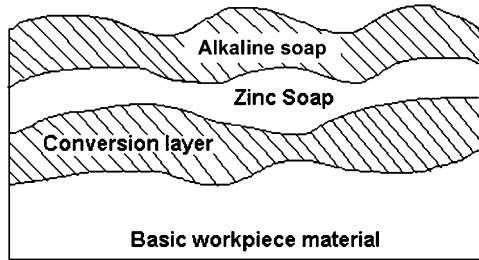


Fig. 1. Structure of zinc phosphate lubrication system [1].

Table 1
Zinc phosphate treatment sequence of steel billets

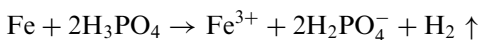
Type of operation	Operation
Cleaning	Mechanical cleaning Degreasing Rinsing with cold water Pickling Rinsing with cold water Rinsing with warm water added activators
Phoscoating	Phoscoating Rinsing with cold water Neutralizing
Lubrication	Lubricating with soap, MoS ₂ , etc. Drying

system on carbon steel billets for cold forging operations is shown in Table 1 [1,5].

The cleaning operations are designed to remove heavy scale and grease from the billet surface. The mechanical cleaning methods include shot blasting and peeling. Shot blasting provides a better surface for adhesion of the zinc phosphate coating and the lubricant. Chemical cleaning, or degreasing, operations commonly utilize alkalines (water soluble salts) like caustic soda, soda, silicates, phosphates, and borates. The baths for these solutions must be heated above 50 °C [1].

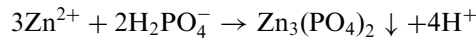
The pickling operation prepares the surface of the billet for the zinc phosphate coating process and is usually completed with sulfuric acid, but hydrochloric acid is also used. Sulfuric acid is easier to handle, but the bath must be heated to 40–60 °C, whereas the hydrochloric bath may be kept between room temperature and 40 °C [1].

In the zinc phosphate coating operation, a zinc phosphate coating is formed on the steel surface. First the steel billet is brought into contact with a zinc phosphate solution that has a pH of approximately 2.0 and is heated to 40–90 °C depending on the chemicals added to the solution. Upon contact, a pickling reaction occurs as follows [1,5]:



As iron is dissolved from the surface, the zinc phosphate coating process, bonding of zinc phosphate to the billet

surface, begins. This reaction occurs as follows [1,5]:

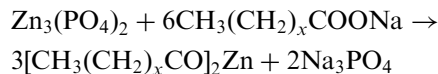


Also, the zinc phosphate solution contains oxidants that act as accelerators. These oxidants help convert the Fe²⁺ ions, formed previously, into Fe³⁺ ions. The Fe³⁺ ions then combine with phosphate and precipitate as sludge. The oxidation of Fe²⁺ to Fe³⁺ can be accelerated by the addition of sodium nitrite or sodium chlorate to the zinc phosphate solution. The billet is often rinsed with cold water following the zinc phosphate coating process in order to neutralize the remaining phosphoric acid [1,5].

The properties of the zinc phosphate coating are determined by [1,5,6]:

- Zinc phosphate solution concentration and aging.
- Type of accelerators used.
- Process temperature.
- Process time.

In conventional cold forging operations, when a zinc phosphate coated billet is emerged in a soap-based lubricant bath (70–80 °C), a chemical reaction occurs. Most soap-based lubricants contain sodium stearate. The reaction between the zinc phosphate and the sodium stearate creates a zinc soap that is bonded to the zinc phosphate coating (Fig. 1). This reaction is described as follows [1,5]:



After removing the billet from the bath, the soap must be allowed to dry. As Fig. 1 shows, not all of the sodium soap is converted to zinc soap. By controlling the concentration of the soap bath, the thickness of the soap layer can be controlled. However, care should be taken because if the soap layer becomes too thick, it could build up on the tools and cause dimensional accuracy problems [1].

1.2. Problems associated with the zinc phosphate coating lubrication system

The zinc phosphate coating lubrication systems used in cold forging present several problems [7].

- It is costly to apply and remove the zinc phosphate layer. Several baths at temperatures between 40 and 90 °C containing different solutions are necessary.
- After the zinc phosphate coating process, the baths become polluted with heavy metals like lead and cadmium. The wastewater treatment and the baths result in solids, which contain heavy metals, oils, and other pollutants. Most of this waste cannot be reused and thus becomes hazardous waste.
- The mechanical properties of the base material that the zinc phosphate coating is applied to are affected. Zinc

phosphate can increase corrosion and diffuse into the workpiece material during heat treatment. This is a common cause for surface embrittlement.

2. Survey of candidate lubricants for replacement of zinc phosphate based coating lubrication systems

A survey of cold forging lubricant manufacturers revealed three candidate lubricants for the replacement of zinc phosphate coating lubrication systems. These included: MEC Homat, Daido AquaLub, and MCI Z-Coat. Due to confidentiality, only non-proprietary information is given below regarding the preparation and formulations of these lubricants.

2.1. MEC homat lubricant

MEC Homat lubricant is a water-based lubricant whose main components are metal compounds and organic sulfur compounds [8]. The billet surface treatment is achieved in three stages; shot blasting, lubricant application, and drying.

The mechanism of solid lubricant film formation is via thermo-chemical reaction as depicted in Fig. 2. In this process the sulfur radicals caused by tribo-reaction with fresh surface of iron forms a film of iron sulfide with good lubrication effect. The application of this lubricant requires that the billet be shot blasted, following by lubricant application, and finally drying.

2.2. Daido aqualub lubricant

The AquaLub lubricant is based on phosphoric compound which has adsorption ability onto metallic surface and solid lubricants. Fig. 3 shows the holding mechanism. The generic chemical compositions are: (a) calcium compound 5–10%, (b) water soluble inorganic salt 1–5%, (c) phosphorous organic compound 0.5–1%, (d) lubricant surfactants 5–10%, (e) synthetic alcohol 5–10%, (f) water insoluble inorganic salts 5–10%, and water 60–80% [9]. The application of this lubricant requires shot peening to prepare the billet surface before lubricant treatment.

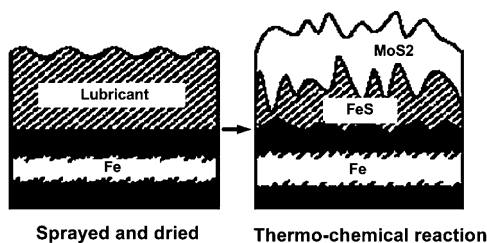


Fig. 2. Mechanism of lubricant layer formation.

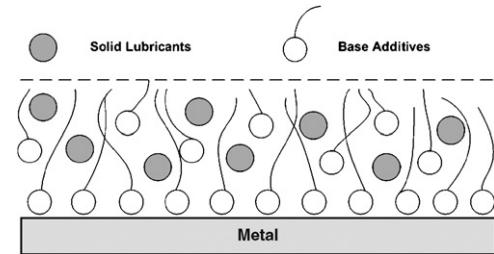


Fig. 3. Structure of Aqua lubrication system.

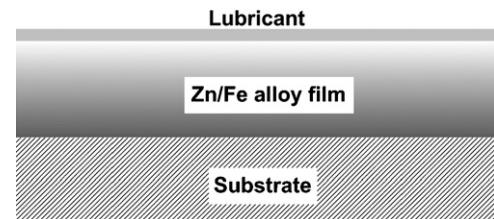


Fig. 4. Structure of Z-Coat lubrication system.

Table 2
Z-Coat treatment sequence of billets

Type of operation	Operation
Cleaning	Degreasing and descaling
Z-Coat	Mechanical coating of “Z-Iron” via blasting machine
Lubrication	With soap, oil, or MoS ₂ ,

2.3. MCI Z-Coat-based lubricant

MCI Z-Coat lubricant forms a zinc/iron film on the surface of the billet. This film is porous and can be combined with forging oils, metal soaps, or molybdenum disulfide [10]. The structure of Z-Coat lubrication system and billet treatment sequence are given in Fig. 4 and Table 2, respectively.

Common to all the three candidate lubricants discussed above is that the lubricant film is firmly bonded to the surface by mechanical means. The absence of major chemical reactions in the billet treatment process has reduced the amount of hazardous waste that could be generated as compared to zinc phosphate coating based lubrication systems. Furthermore, the absence of chemical baths have simplified and reduced the time for billet treatment.

It should, however, be noted that further development efforts for these new lubrication systems are still needed to ensure that the lubricity levels are competitive to zinc phosphate coating based lubricant system, particularly, for severe deformation processes.

3. Double cup backward extrusion tribo-test

There are various tribo-tests developed to date used for evaluation of cold forging lubricants. These include; ring

compression test, spike test, ball penetration tests, etc. In this study, however, the double cup backward extrusion test was used. As compared to other commonly used tests such the ring compression test, the double cup backward extrusion test can mimic severe deformation with very high surface enlargement occurring in actual severe cold forming operations.

3.1. Test set-up

The double cup backward extrusion test is a combination of single cup forward and single cup backward extrusion processes. Fig. 5 shows the experimental setup, while Fig. 6 shows a cross-sectional view of the tooling. The container and the lower punch are fixed on the bed of the press and the upper punch is fixed on the ram of the press. In this test, the upper punch moves downwards while the bottom punch and the container are kept stationary. The diameters of both punches are the same. The upper cup is formed by a backward extrusion process and the lower cup is formed by a forward extrusion process. The simultaneous action of the two punches inside the cylindrical container generates the two cups. The maximum surface expansion generated in this test may reach as high as 500%.

The purpose of the double cup backward extrusion test is to establish a correlation between the ratio of the extruded cup heights and the friction conditions present at the

billet–punch and billet–container interfaces. With such a correlation, the existing friction conditions can be quantified. The friction conditions at these interfaces are expressed as a number known as the friction factor, m , which varies between 0 and 1 with $m = 0$ representing a frictionless interface and $m = 1$ representing sticking friction. The friction factor, m , is defined as follows: [11].

$$\tau = mk = m \frac{\bar{\sigma}}{\sqrt{3}},$$

where τ is shear stress, k is the shear strength, and $\bar{\sigma}$ is the flow stress of the workpiece material.

The friction conditions at the billet–container and billet–punch interfaces control the ratio between the backward and forward extrusion (upper and lower cup heights). Studies on friction conditions in cold forging with this test method have shown the cup height ratio to be extremely sensitive to the friction factor [12,13]. Thus, by comparing the cup height ratio and punch stroke to the friction factor calibration curves obtained through FEM, the friction factor of various lubricants can be quantified and compared.

3.2. Determination of friction calibration curves by FEA of the double cup backward extrusion test

Finite element (FE) simulations of the double cup backward extrusion tests were completed in order to understand the parameters affecting metal flow in the test and to obtain friction factor calibration curves. The friction factor calibration curves will be used later to identify the friction factor of the tested lubricants.

3.2.1. Conditions for the FE simulations

The commercial finite element code DEFORM-2D was used for the simulations. The general conditions for the model are shown in Table 3. Because the double cup backward extrusion test involves bulk material deformation, the constant shear friction model ($\tau = mk$) was used in the simulations. The material properties were determined using the uniform compression test and expressed in the form of a power law, $\bar{\sigma} = K\bar{\epsilon}^n$, where K is the strength coefficient and n is the strain-hardening exponent [14]. Fig. 7 shows the finite element model.

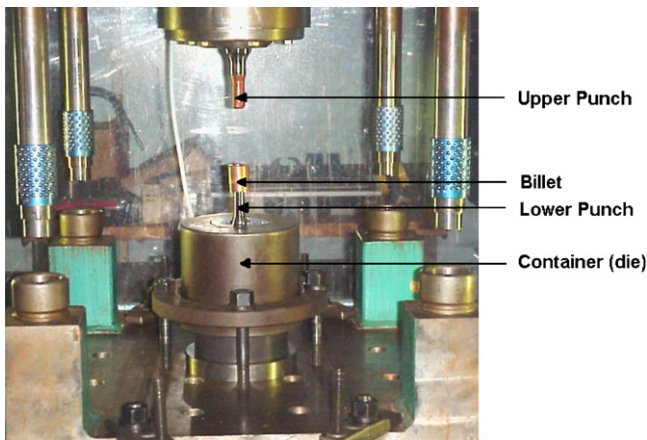


Fig. 5. ERC/NSM double cup backward extrusion test tooling.

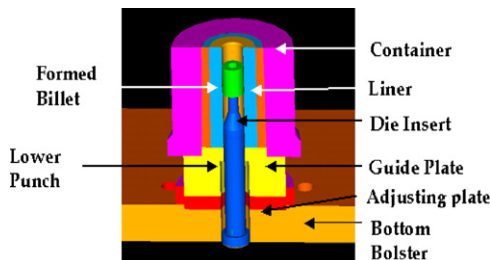


Fig. 6. Cross-sectional view of double cup backward extrusion tooling.

Table 3
FE simulation parameters

Condition	Description
Material	AISI 8610
Matl properties	$\bar{\sigma} = K\bar{\epsilon}^n$; $K = 690$ MPa, $n = 0.14$
Billet height	31.75 mm (1.25 in.)
Billet diameter	31.75 mm (1.25 in.)
Friction model	Shear friction
Friction factors	$m = 0.00, m = 0.02, m = 0.03, m = 0.04, m = 0.05, m = 0.06, m = 0.065, m = 0.07, m = 0.08, m = 0.09, m = 0.10, m = 0.15$
No. of elements	1500

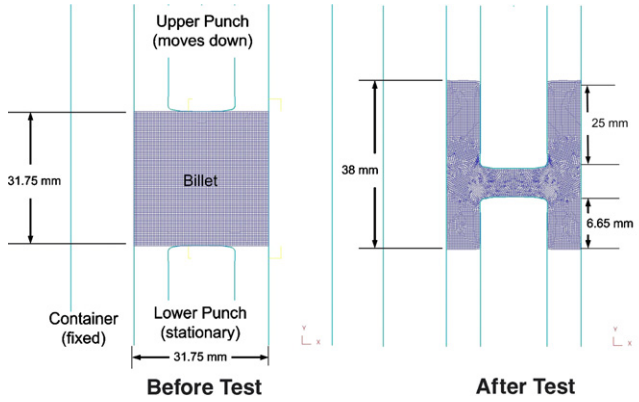


Fig. 7. FE model ($m = 0.1$).

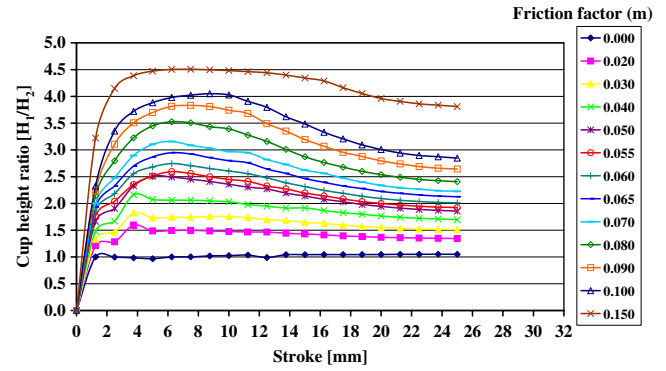


Fig. 9. Friction factor calibration curves.

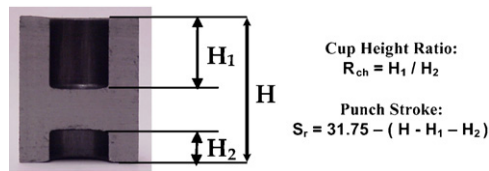


Fig. 8. Cup height ratio and punch stroke definition.

3.2.2. FE simulation results and discussion

The cup height ratio, R_{ch} , and the punch stroke, S_r , are defined in Fig. 8. The cup height ratio is an indication of lubricity. As the friction factor increases, so does the cup height ratio. Thus, if there was no friction, the cup heights would be the same and the cup height ratio would be equal to one.

Using the nodal coordinates from the FE simulation, the cup height ratio was determined at various stroke lengths. Fig. 9 shows the friction factor calibration curves where the cup height ratio was plotted versus the punch stroke. By matching the cup height ratio and punch stroke from an actual double cup backward extrusion test to the calibration curves, the friction factor for a given lubricant can be determined.

Fig. 9 shows that the cup height ratio increases with increasing friction factor. It also shows that the cup height ratio increases with increasing stroke up to a maximum value, and then gradually starts to decrease until a stroke of 20 mm is reached. At this point, the cup height ratio becomes constant with increasing stroke for all friction factor values. Thus, a punch stroke greater than 20 mm should be used in the actual experiments for the tool and billet dimensions of the ERC/NSM test setup.

Fig. 10 shows FEA-generated plots of the forging load ratio (extrusion load divided by load at zero friction). The extrusion loads for friction factor $m = 0.05, 0.1$ and 0.15 were divided by the extrusion load for non-friction conditions, i.e., $m = 0.0$. For the friction range of $m = 0.05–0.15$ the extrusion load ratio varies from 1.0 to 1.1. This shows that at lower friction range the extrusion load is

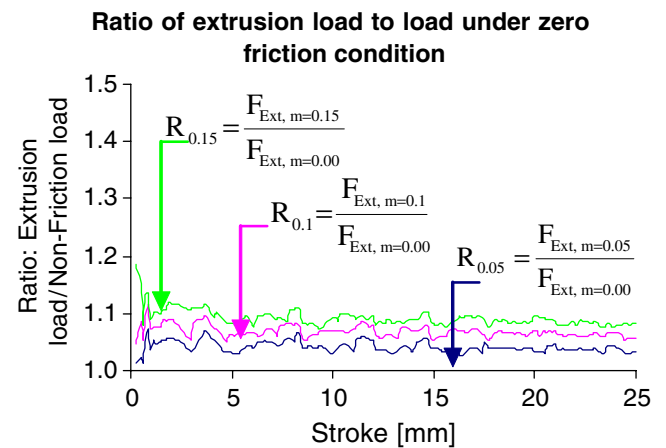


Fig. 10. Forming load sensitivity to friction in double cup extrusion test—FEA.

insensitive to friction. It should be noted that at the same friction range the ratio of cup height varies from 1.0 to 4.5. Thus, for accurate evaluation of lubricants the cup height ratio should be used.

4. Evaluation of candidate lubricants by the double cup backward extrusion test

4.1. Experimental procedures

The experiments were completed with billets made from AISI 8610 grade alloy steel supplied by Piper Impact. The specimens were obtained from the same rod, 31.75 mm in diameter. The specimens were cut to a length of 31.75 mm each. The chemical composition of the billet material is shown in Table 4. Table 5 shows the experimental matrix, where 20 samples were tested for each lubricant.

4.1.1. Lubricant application and surface characterization

The billet treatment for Zinc Phosphate coating + soaping was done by Piper Impact Co. using the standard procedures as discussed in Section 1.1. MCI Z-coat was also applied by the lubricant manufacturer, Metal Coating International (Table 2).

Table 4
Chemical composition of AISI 8610

C	Ni	Cr	Mo	Mn	Si
0.1%	0.55%	0.5%	0.25%	0.7–0.9%	0.15–0.3%

Table 5
Experimental matrix

Lub. no.	Lubricant	No. of samples
1	Zinc phosphate coating + soap	20
2	MEC Homat	20
3	Daido AquaLub	20
4	MCI Z-Coat	20

The MEC Homat and Daido lubricants were applied by staff at the ERC/NSM. The following procedures were followed for the application of the MEC Homat lubricant.

- Agitate the lubricant until it was homogeneous and without trapped air bubbles.
- Heat the lubricant to 40 °C.
- Clean the billets with a standard degreasing agent.
- Apply the lubricant to the billet with a brush.
- Allow the lubricant to dry on the billet.

The same procedures were followed for the application of the Daido AquaLub lubricant except that it was applied at room temperature instead of 40 °C.

Optical micrographs of the billet surfaces coated with the four lubricants were taken before the experiments (Fig. 11). The figure shows that the lubricants coated the billet surfaces evenly.

4.1.2. Tests

The experiments were conducted using a 160 ton CNC hydraulic press with a ram speed of 10 mm/s and a 21 mm punch stroke.

Following each test, the container and the punches were cleaned and checked for galling/scratching in order to insure the same conditions existed for every test. When the cup temperature returned to room temperature, the cup height ratio and the punch stroke were calculated by measuring the upper and lower cup heights as well as the total extruded part height with a caliper (Fig. 8).

4.2. Results and discussion

By plotting the average cup height ratio (obtained from 20 specimens) and the average punch stroke (obtained from 20 specimens) on the friction factor calibration curves, the average friction factor for each lubricant was estimated as shown in Fig. 13. Fig. 12 shows extruded cup sample for each lubricant. All samples were axis-symmetrically extruded such that the cup height variations within a single cup was insignificant. The cup height ratios, however,

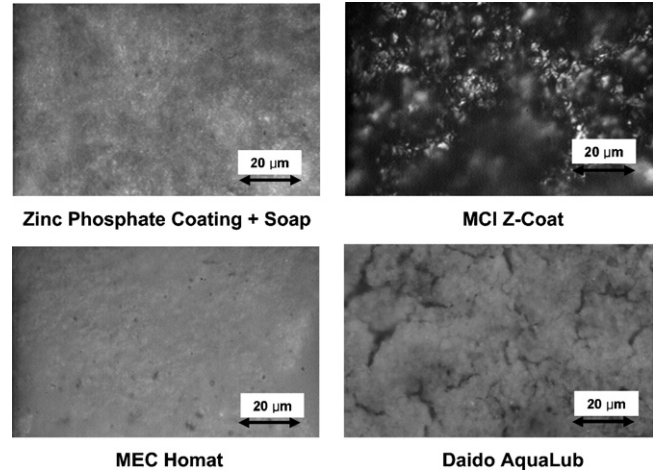


Fig. 11. Optical micrographs of lubricated billet surfaces before experiments.

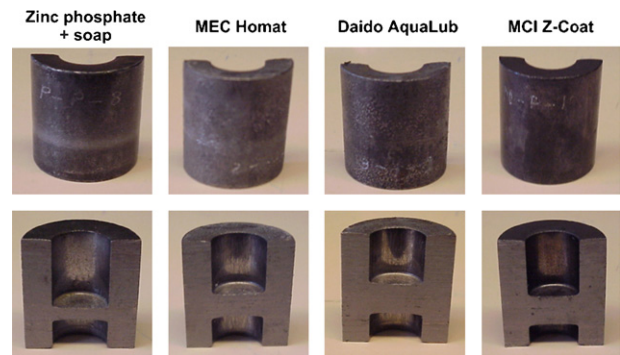


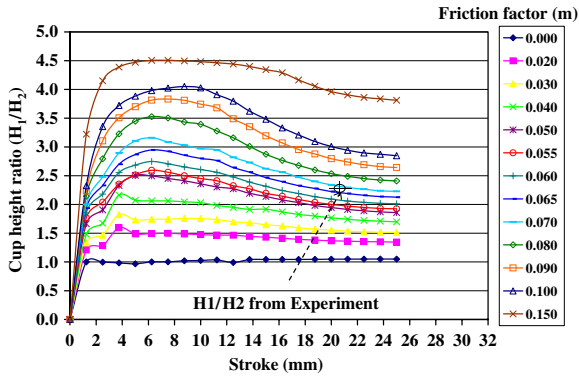
Fig. 12. Extruded cup samples.

varied from one sample to the other and the degree of variation was a function of the lubricant used.

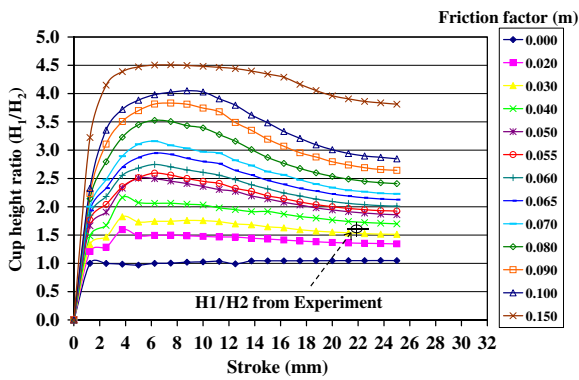
Performance of zinc phosphate coating+soap: The average cup height ratio, H_1/H_2 , for the Piper Impact Zinc phosphate coated billets tests was measured to be 2.247. In addition, the average real stroke was 21.52 mm. By plotting these values on the friction factor calibration curve, the average friction factor for this lubricant was estimated to be $m = 0.065$ (Fig. 13a). The friction factors varied from $m = 0.060$ to 0.070 for the 20 samples tested.

Performance of MEC Homat: The average cup height ratio, H_1/H_2 , for the MEC Homat tests was measured to be 1.600. In addition, the average real stroke was 21.87 mm. By plotting these values on the friction factor calibration curve, the average friction factor for this lubricant was estimated to be $m = 0.035$. (Fig. 13b). The friction factors varied from $m = 0.030$ to 0.040 for the 20 samples tested.

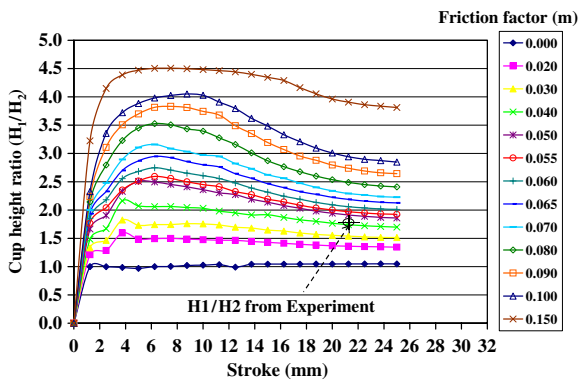
Performance of AquaLub: The average cup height ratio, H_1/H_2 , for the Daido AquaLub tests was measured to be 1.730. In addition, the average real stroke was 21.46 mm. By plotting these values on the friction factor calibration curve, the average friction factor for this lubricant was estimated to be $m = 0.040$ (Fig. 13c). For the 20 samples tested the friction factors varied from $m = 0.025$ to 0.055.



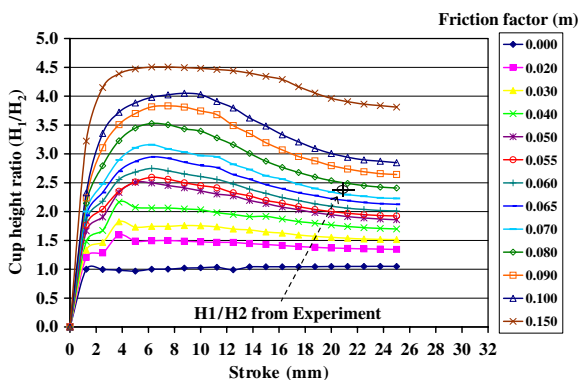
(a) Zinc Phosphate Coating + Soap



(b) MEC Homat



(c) Daido AquaLub



(d) MCI Z-Coat

Fig. 13. Determination of friction factors for the four lubricants tested.

Performance of MCI Z-Coat: The average cup height ratio, H_1/H_2 , for the MCI Z-Coat tests was measured to be 2.368. In addition, the average real stroke was 21.56 mm. By plotting these values on the friction factor calibration curve, the average friction factor for this lubricant was estimated to be $m = 0.075$ (Fig. 13d). For the 20 samples tested the friction factors varied from $m = 0.65$ to 0.080.

Performance ranking of the lubricants is shown in Fig. 14. In this figure the cup height ratios, R_{ch} , are plotted on the ordinate and the friction factor, m , is plotted on the abscissa. It should be noted that these values are an average of 20 tested specimens. The aim of the successful double cup backward extrusion operation is to reach processes with a minimum cup height ratio and a minimum friction factor. Therefore, in this figure, the lubricant with the best performance is located closest to the origin. To further clarify the results, Fig. 15 presents the friction factors, for the four lubricants tested, in bar graph format.

It is evident from error bar shown in Fig. 15 that AquaLub had the highest variations in the cup height

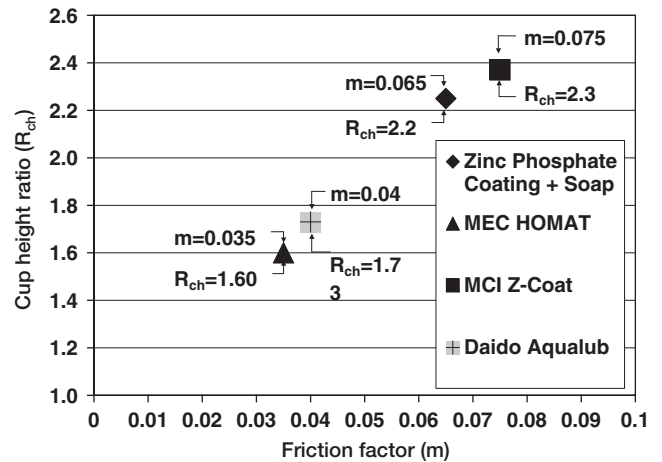


Fig. 14. Lubricant performance diagram.

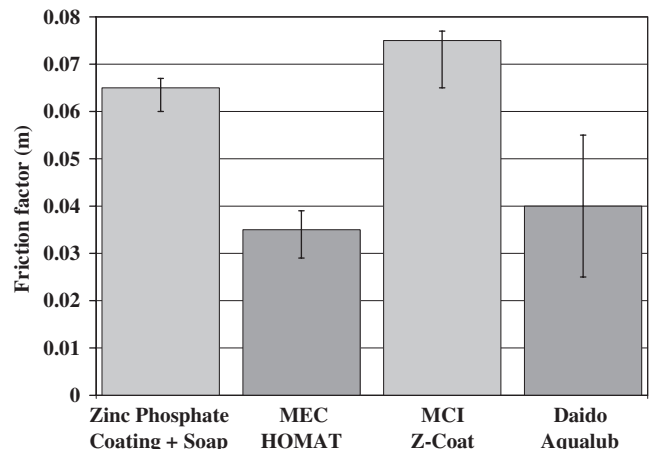


Fig. 15. Friction factors of the four lubricants tested.

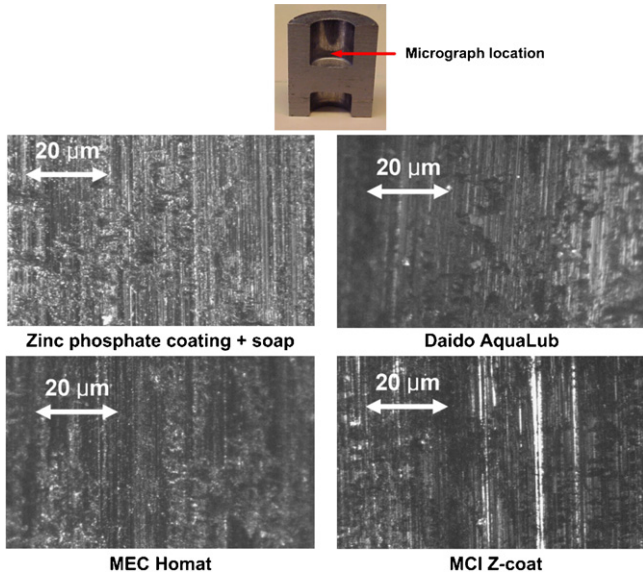


Fig. 16. Optical micrographs of the upper cup after tests.

ratio/friction factors among the four lubricants tested. One of the attributing factors is the fact that shot peening was not carried out to prepare billet surface before lubricant application. This may have caused uneven adherence of the lubricant to the billet. Also, as compared to MEC Homat, no preheating of the lubricant was done for AquaLub. Preheating MEC homat lubricant at 40 °C was carried out to ensure that the lubricant is thoroughly mixed. Furthermore, the initial lubricant temperature helped to accelerate the drying process resulting to more evenly distributed lubricant film on the billet surface.

Figs. 14 and 15 clearly show that the MEC Homat lubricant and the Daido AquaLub lubricant obtained lower friction factors than the zinc phosphate coating+soap under the given conditions (i.e. interface pressure, sliding velocity, surface enlargement, etc). In particular, MEC Homat exhibited lower friction factor by 45% in comparison to the zinc phosphate coating+soap and the Daido AquaLub exhibited lower friction factor by 40% in comparison to the zinc phosphate coating+soap.

To study the performance of the lubricants in preventing galling optical micrographs were taken at the upper cup where maximum surface enlargement occurs (Fig. 16). The figure shows that with the exception of MCI Z-Coat, the surface morphologies are very similar. The specimens coated with MCI Z-Coat show slightly more scratches in the upper cup than do the other three lubricants. Also both the punches and the container (die) did not show any sign of galling for all the specimens tested.

5. Conclusions

Presently, most cold forging processes require a zinc phosphate coating based lubricant; however, there are a

number of problems with this lubrication system. These problems include: hazardous waste disposal, high equipment and energy costs associated with billet treatment, and human health risks. Three potential lubrication systems that do not contain zinc phosphate coating were evaluated. The following conclusions were drawn from the results of this study:

- MEC Homat and Daido AquaLub performed better than the zinc phosphate coating+soap while MCI Z-Coat performed similar to the zinc phosphate coating+soap under the tested conditions (i.e., interface pressure, sliding velocity, surface expansion, etc).
- There were no signs of galling on the punches or containers after any of the tests.
- The absence of major chemical reactions in billet treatment for MEC homat, AquaLub and MCI Z-Coat has reduced the amount of hazardous waste that could be generated as compared to zinc phosphate coating systems.
- MEC Homat, Daido AquaLub, and MCI Z-Coat show promise as replacements for zinc phosphate coating lubrication systems.

It should be noted, however, that further testing and development efforts for these new lubrication systems are still needed. Additional tribo-tests at elevated temperatures levels (100–250 °C) and different ram speeds are important to ensure that the lubricity levels of these new lubricants are competitive to zinc phosphate coating based lubricant systems, particularly, for more severe deformations beyond the severity emulated by the double cup backward extrusion test used in this study.

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References

- [1] N. Bay, The state of the art in cold forging lubrication, *Journal of Materials Processing Technology* 46 (1994) 19–40.
- [2] M. Takeuchi, F. Ikesue, N. Kashimura, Development of environmentally friendly lubricant with high performance and simple treatment for cold forging, *Advanced Technology of Plasticity 1* (1999) 383–390.
- [3] H. Saiki, G. Ngaile, Recent work on tribology in forging, *Proceedings of International Symposium on Advanced Forming and Die Manufacturing Technology*, Pusan, Korea, September 1999, pp. 37–48.
- [4] L. Dubar, J.P. Bricout, C. Wierre, P. Meignan, New surface processes for cold forging of steels, *Surface and Coatings Technology* 102 (1998) 159–167.

- [5] J. Donofrio, Zinc phosphating, *Metal Finishing* 98 (2000) 57–73.
- [6] L. Lazzarotto, C. Marechal, L. Dubar, A. Dubois, J. Oudin, The effects of processing bath parameters on the quality and performance of zinc phosphate stearate coatings, *Surface and Coatings Technology* 122 (1999) 94–100.
- [7] D. Schmoeckel, M. Rupp, More environment-friendly cold massive forming—production of steel without zinc phosphate layer, Symposium “Latest Developments in Massive Forming,” in Fellbach near Stuttgart, 1997, pp. 183–200 (in German).
- [8] “Water Lubricants for Cold Forging: MEC Homat,” *Technical Bulletin of MEC International*, 2000.
- [9] Replaceable Lubricants to Phosphate/Soap Treatment, *Technical Bulletin of Daido Chemical Industry Co.*
- [10] Zinc–Iron Alloy Film Formed by NCZ’s Unique Blasting System—Z-Coat, *Technical Bulletin of Metal Coatings International*, 1995.
- [11] J. Schey, *Tribology in Metalforming*, American Society for Metals, Metals Park, OH, 1983.
- [12] A. Buschhausen, J.Y. Lee, K. Weinmann, T. Altan, Evaluation of lubrication and friction in cold forging using a double cup backward extrusion process, *Journal of Materials Processing Technology* 33 (1992) 95–108.
- [13] A. Forcellese, F. Gabrielli, A. Barcellona, F. Micari, Evaluation of friction in cold metal forming, *Journal of Materials Processing Technology* 45 (1994) 619–624.
- [14] R. Dixit, G. Ngaile, T. Altan, Measurement of flow stress for cold forging, Report no. PF/ERC/NSM-01-R-05, 2000.