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Effects of Porous Surface Layer on Lubrication Evaluated by Ring Compression Friction Test

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Lubrication is an important parameter in cold working of steels. Metal soap on phosphate coating is the most commonly used lubrication system in industries at present, but the technique is less productive and environmentally hazardous. The authors proposed an alternative lubrication process which makes use of porous layer formed on the surface of the workpiece by oxidization and chemical reduction in the previous paper. The technique, due to the porous layer, enables to hold more liquid lubricant on the surface and consequently to decrease the friction. This research expands the range of application of this technique. The porous layer is applied to three types of steels containing different levels of carbon and its effects are analyzed under three lubrication conditions; unlubricated, machine oil and grease, by ring-compression test. Another important condition, compression speed is investigated by using two types of equipments, a hydraulic press (low speed) or a mechanical press (high speed). This study proves that the porous layer technique is applicable to a range of types of steels reducing the friction coefficient, and at the same time provides some insights to explain the mechanics of the technique. The lubrication effects are explained by the thickness of lubricant held at the interface between the die and the surface of workpiece.

KEY WORDS: porous metal; cold working; lubrication; oil pit; tribology; carbon steel.

1. Introduction

In cold working of steels, phosphate coating is an excellent lubrication technique. However, because of its hazardous wastes and low productivity, alternative lubrication systems are demanded.[1] Various approaches such as use of suitable die materials and special treatments of die surface were proposed. Cold working without lubricant,[2,3] or with limited lubricants[4] were also discussed. As another solution, the authors proposed a new lubrication method using a porous layer formed on the workpiece surface.[5]

The porous layer is created on an iron surface by oxidization and chemical reduction under hydrogen atmosphere as shown in **Fig. 1**.[6] In the chemical reduction process, oxygen in the oxides combines with hydrogen gas producing H_2O while iron oxides on surface transform to pure iron. During the process, oxygen vacancies left aggregate to energy balanced points on the surface forming pores there. The mechanism to form porous layer was discussed in detail by Matthew.[7]

In cold forging, the porous layer is expected to be used as a lubrication technique since more lubricant could be trapped in its pores than flat surface. The effectiveness of this lubrication technique was confirmed with 0.15% carbon steel.[5] Ceron[8] proposed a similar technique where porous Sn layer formed by chemical etching was used with low vis-

cosity lubricant for lubrication, and which showed some successful results. The porous layer used in their study, however, is made of the material different from the matrix, so the technique could cause several problems, such as detachment of the layer and the difference in deformation amount between the layer and the mother material, which our oxidation-reduction technique does not suffer. However the mechanism of the lubrication has not been understood completely yet.

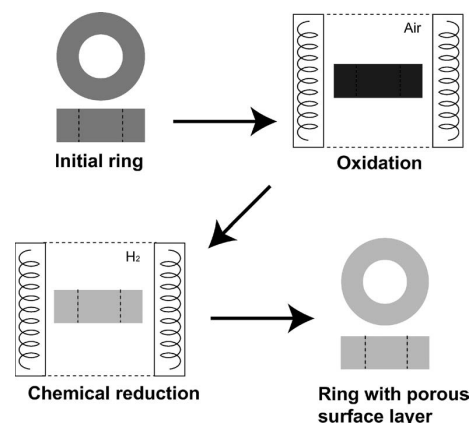In this research, the porous layers are formed on 0.25%,

Fig. 1. Heat treatment to make porous layer on steel surface.

0.45% in addition to conventionally used 0.15% carbon steels by the oxidation-reduction technique. So far, pore formation was reported only on pure iron and 0.15%C steel in ferrous materials, so it is important for industries to confirm that the porous layer creation technique can be applied to other commonly used industrial steels such as 0.25% and 0.45% carbon steels. Furthermore, carbon is the most important element to control the strength of steels but also it is known that it slightly suppresses the oxidation of the steel, and thus, the influence of carbon content on the pore formation is studied. The performance of the porous layer is analyzed by ring-compression test at room temperature under three different lubrication conditions. In the test, the friction coefficient is estimated by the change in inner diameter of ring. As the test is simple and well established, it was used to check the lubrication effects, although surface expansion is not as large as those in most industrial processes. The compression test is also performed on a mechanical press to investigate the dependence on compression speed. The decrease in friction coefficient is correlated by the amount of lubricant on the interface.

2. Experiment

2.1. Material and Pore Formation

In order to confirm the material properties of the steels used for the experiments, the flow stress curves were obtained by uniaxial compression test of $\phi 10$ mm $\times$ 15 mm cylindrical specimens made of the three types of steels (0.15, 0.25 and 0.45% carbon steels) under good lubrication at room temperature. During the compression test, the load and the height were measured every 1 mm of displacement. After every displacement, the specimen was unloaded, teflon (PTFE) sheet and lanolin were applied to the interface to minimize the friction. Then the specimen was reloaded to next displacement. Two specimens of each steel were tested, and as the both samples showed very close curves the average flow stress curves are presented in **Fig. 2**. The flow stress increases with carbon content, although the work hardening rate does not depend much on carbon content. It is expected that the amount of lubricant on the interface between the workpiece and the die depends on the tool pressure. In other words, a larger amount of lubricant may be
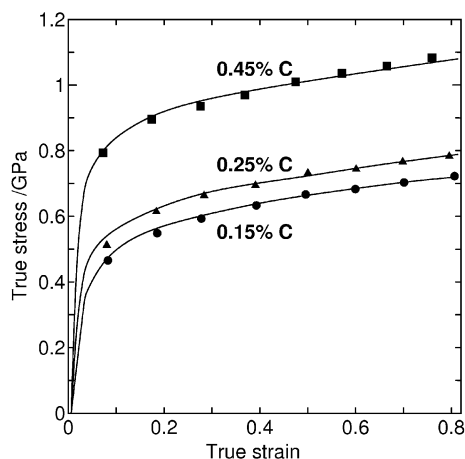
kept on the interface when working with a softer workpiece with lower carbon content. The three steels were compared to reveal the effects of tool pressure on friction.

For ring-compression test, rings with 20 mm outer diameter, 10 mm inner diameter and 5 mm height were machined from the carbon steel bars. The machined rings were polished with emery papers up to #1 500 and degreased. Porous layer was formed on ring surface by the heat treatment as shown in Fig. 1. First, the rings were oxidized for 1.8 ks in air at 873 K in an electric tube furnace. The atmosphere in the furnace was subsequently replaced with hydrogen gas remaining the temperature and under the condition the rings underwent chemical reduction for 600 s. After the heat treatment, the rings were cooled slowly in the furnace to room temperature in a hydrogen atmosphere.

The surfaces of the rings were observed with a scanning electric microscope. X-ray diffraction patterns were measured with Cu-Kα radiation to investigate the components of the surface layer.

2.2. Ring Compression Test

The heat-treated rings were compressed 40% in height under three lubrication conditions; (a) dry (unlubricated), (b) grease (Cosmo Auto grease No. 00, kinematic viscosity of 118 mm^2/s at 313 K) and (c) machine oil (Idemitsu Daphne mechanic oil No. 32, kinematic viscosity of 32 mm^2/s at 313 K). The compression was conducted uniaxially on a hydraulic testing machine with flat dies of tool steel having the average roughness of 0.09 μm at a speed of 1.5 mm/min. After each compression, the dies were polished by #1 500 emery paper and degreased. Compression load was monitored during the deformation. The untreated (nonporous) normal rings were also compressed under the same conditions. The friction coefficient was estimated from the change in the inner diameter of the ring according to the monogram in the reference.[9] The monogram was drawn based on analyses with the energy method. A high-speed compression test was also conducted on a mechanical press with the same dies. The average compression speed was 4 500 mm/min.

3. Results

SEM micrographs of the porous surfaces are shown in **Fig. 3**. Pores with diameters on the order of hundreds of nanometer are formed uniformly on the steel surfaces. The pore diameter increases slightly with carbon content. The thickness of the porous layer t was measured by sectional observation. The thickness is 2.0–2.5 μm as shown on the photographs. The thickness of porous layer decreases slight-

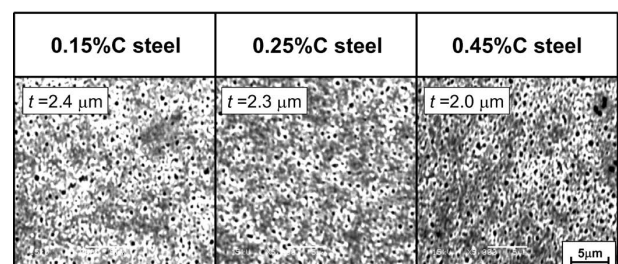**Fig. 2.** Flow-stress of the three steels obtained by repeated compression test.

Fig. 3. Photographs of the surfaces after heat treatment.

ly with carbon content possibly because carbon retards oxidation slightly, but it is confirmed that porous layer can be formed on three steels by the heat treatment regardless of the carbon content in steels. The porous layer formed by the oxidisation-reduction technique provides more effective lubrication than normal rough surfaces which are sometimes used for lubrication with lubricants because the porous layer can tightly trap the lubricant in its pores while rough surfaces are being flattened and losing the effects as the forming process progresses.

X-ray diffraction patterns of 0.15% carbon steels (a) before the heat treatment, (b) after the oxidation and (c) after the chemical reduction are shown in **Fig. 4**. Before the heat treatment (a), only peaks of *bcc* α-Fe and those of clay mount are observed. After the oxidation, peaks of hematite Fe_2O_3 and magnetite Fe_3O_4 appear. After the chemical reduction (c), all the peaks of the oxides disappear. This means that the porous layer was formed by deoxidation of the oxide scale and that the porous layer in Fig. 3 is not oxides but α-Fe. This fact is important because α-Fe is ductile metal, so it could follow surface expansion during the compression.

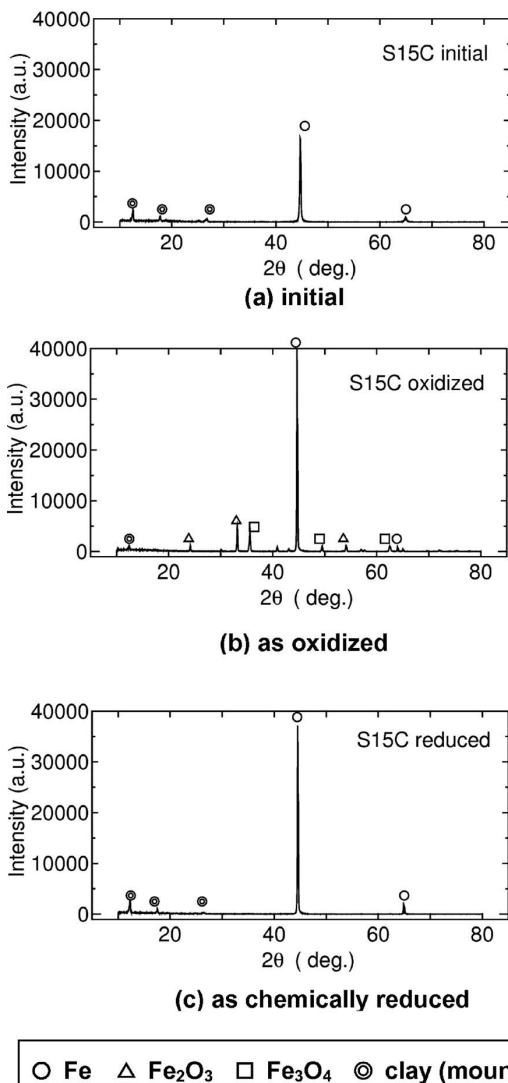The friction coefficients of 0.15% and 0.45% carbon

steels in the low-speed compression test are shown in **Fig. 5**. The coefficients were obtained by averaging three specimens. According to the monogram, the error in reading is less than 0.001 when the friction coefficient is less than 0.2. The deviation among the specimens measured under the same conditions is less than 0.01 when liquid lubricant was used. When the friction coefficient is larger than 0.2, the accuracy of the monogram reading is lower. Thus, the deviation was up to 0.02 under dry conditions.

As a whole, the porous rings show lower friction coefficients than the non porous rings when a lubricant is used whether it is the grease or the oil. This proves that the porous layer can keep larger amount of the lubricant at the interface than non porous surface during the operation and successfully reduce the friction. From the fact that the pores (partly connected to each other) can be observed even after the compression as shown in **Fig. 6**, it can be said that the effects of porous layer lasted until the end of the process.
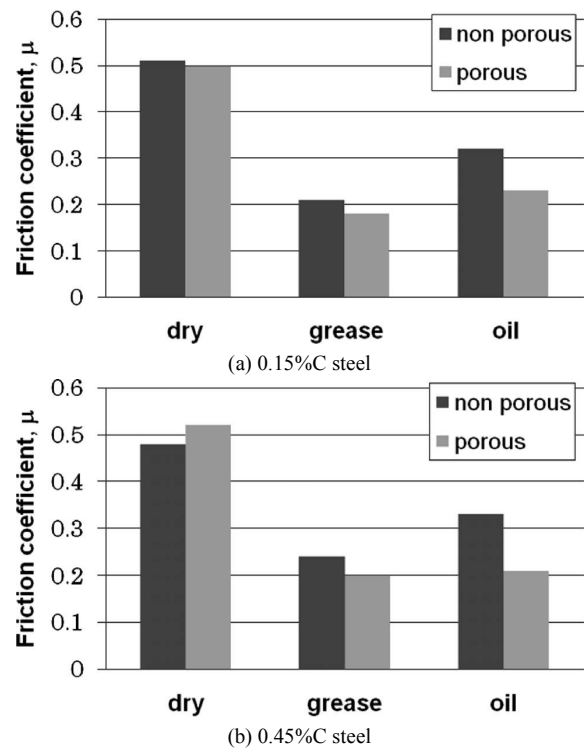

(a) 0.15%C steel

(b) 0.45%C steel

Fig. 5. Friction coefficients at low-speed compression.

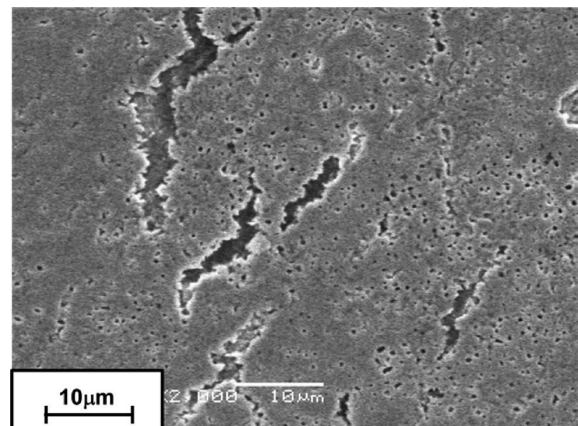**Fig. 4.** Change in X-ray diffraction pattern of 0.15%C steel during heat treatment.

Fig. 6. Photographs of the surface after compression (0.15%C Steel).
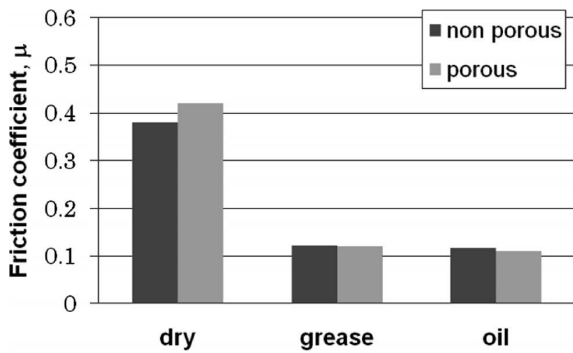
Fig. 7. Friction coefficients at high-speed compression (0.15%C Steel).

Fig. 8. Relationship between friction coefficient and thickness of lubricant on interface.

The oil cases show larger differences between porous and non porous than the grease cases. This means that the porous layer works more effectively when the viscosity of the lubricant is lower. Moreover, the difference is slightly larger for 0.45%C steel than 0.15%C steel. 0.45%C steel causes higher compression stress because of the higher flow stress than 0.15%C steel. This implies the advantageous effects of porous layer become more significant when the operation pressure is higher. These results lead to a conclusion that the porous layer is more effective in the case when lubricant on interface is little, such as when the flow stress of workpiece is higher and/or the lubricant is thinner.

Figure 7 shows the friction coefficients of 0.15% carbon rings in the high-speed compression test. The lubricants are considerably effective to decrease friction. Compared with low speed compression, the friction coefficients at the high-speed compression with lubricants are remarkably lower. However the difference between the porous and the non-porous rings is not as large as that at the low-speed compression. This is supposed to be because larger amount of lubricant are held at the interface even on the non-porous surface due to the high compression speed. This is why the friction coefficients at high-speed compression are considerably lower than those at the low-speed compression.

4. Discussion

Friction between workpiece and tool (die) during compression is still a complicated phenomenon for modelling. The friction is sensitive to processing parameters, *i.e.*, workpiece, tool, lubricant, surface roughness, tool pressure, compression speed, temperature, surface expansion, *etc*. The lubrication behaviour is complicated, but, can be partially explained by the amount of lubricant trapped by hydrodynamic effect.[11] On a porous surface layer, the lubrication may also depend on porosity, pore size, pore morphology, and thickness of the porous layer. As very little experimental data have been published, it is not possible to propose a precise model of lubrication on porous layer at this stage. Here the lubrication effect is explained with the amount of lubricant on the interface.

The thickness of lubricant held on the interface h^* between non-porous workpiece and tool can be calculated by fluid dynamics.[10] Oyane and Osakada derived the film thickness h^* in compression of a plate as,[12]
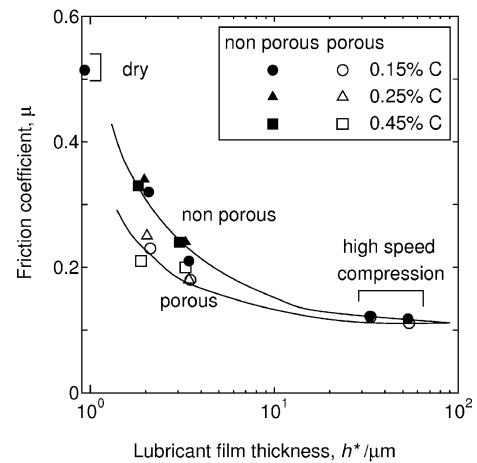
$$h^* = \sqrt[3]{\frac{6\mu U L^2}{p}}$$

where, p: tool pressure, η: lubricant viscosity, U: compression speed, L: distance from the edge.

In this study, the density of the machine oil used was 0.8647 g/cm³. The density of the grease was assumed to be unity.

The lubricant thickness h^* varies along the radial direction. In order to compare the amount of the lubricant, the thickness at the middle point between the inner and outer edges was calculated as the representative value. In other words, L was estimated by {(outer diameter)–(inner diameter)}/4. This is taken because the slab (elementary) analysis predicts that the neutral line locates at the middle of the inner radius and the outer radius. p was substituted with the mean pressure calculated from the experimental compression load. Although the film thickness obtained may not be very accurate due to these rough assumptions, it is assumed to be sufficient for qualitative discussion. The thickness of lubricant increases with increasing viscosity of lubricant and with decreasing tool pressure. The relationship between the friction coefficient obtained by ring compression and the lubricant-film thickness calculated is shown in **Fig. 8**.

In both cases of porous and non-porous rings, the friction coefficient decreases with increase in thickness of lubricant. Under the same thickness of lubricant, the friction coefficient is lower on porous rings than that on nonporous ones. In addition, compared at the same level of the friction coefficient, the thickness of lubricant is about 1 μm thinner on the porous specimens than on non-porous ones. This means that the porous ring requires 1 μm thinner lubricant film to provide the same lubrication effects as the non porous ring. The 1 μm difference in film thickness roughly corresponds to the excess amount of lubricant held in the pores calculated as,

The porosity 40% × The average thickness of the porous layer 2.3 μm =0.92 μm ~ 1 μm.

This implies that the amount of lubricant held in the porous layer is effective as an extra lubricant layer to reduce the friction on the interface. Therefore effects of the porous surface layer are significant in the cases of thin lubricant, high

pressure and low speed.

From this study, it is proved that the porous layer creation method is usable to a range of steels with different levels of carbon content. This fact is important to industry because carbon is the most important element for steels. Not all steels has been checked, and oxidation of stainless steels or silicon steels may be difficult, but at least this lubrication technique is applicable to most structural steels. The porous surface layer provides less friction than a normal plain surface when liquid lubricant is supplied. This is also a significant discovery, even though further studies are required for industrial applications. The tool pressure in ring compression test is neither very high and the surface expansion of specimens is nor very large. The effects of porous layer need to be investigated by more severe tests, such as spike test and double-cup extrusion. Galling and wear should be also assessed, since the decrease in friction by the porous layer with liquid lubricant may extend tool life. These examinations will be taken in next stage.

5.　Conclusions

0.25%, 0.45% as well as 0.15% carbon steels have been heat-treated to form porous layer on the surface. The friction coefficient of the porous layer with lubricant has been examined in a ring compression test at room temperature.

(1)　The porous layer is successfully formed by oxidization and chemical reduction at 873 K on the surfaces of all three carbon steels.

(2)　In low-speed compression, lubricant on porous steels reduces the friction coefficient more than on normal nonporous surface. The effect of the porous layer with lubricant is more obvious when the viscosity of the lubricant is lower and/or when the flow stress of the workpiece is higher.

(3)　In high-speed compression, the porous surface layer has negligible effect because there is surplus lubricant on the interface.

(4)　The above-mentioned lubrication behavior can be explained consistently with the thickness of lubricants held on the interface.

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