Microstructure and Wear Properties of Friction Induced Nano-crystalline Surface Layers of Carbon Steels

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ABSTRACT

Pin-on-disc sliding friction tests were carried out for 0.45 mass % carbon steels, and TEM microstructure and hardness of the sub-surfaces of the friction specimens were investigated. Particularly effects of friction conditions on the microstructure at the surfaces and wear properties of the friction induced microstructure were studied. It was found that ultra-fine equi-axed grains in the 30 - 50 nm size range were produced in the case of a high friction speed of 5.0 m/s in an air atmosphere. Moreover, nano-crystalline microstructure can be produced in a vacuum atmosphere even if the friction speed was low. The friction induced nano-crystalline surface layers, which exhibited significant high hardness, showed good wear resistance.

INTRODUCTION

Ultra-fine grained (UFG) and nanostructured metallic materials produced by severe plastic deformation (SPD) have attracted growing interest owing to their superior mechanical properties without alloying [1]. SPD processes fall into two major classifications: bulk SPD and surface SPD. Bulk SPD processes, which involve high-pressure torsion (HPT), equal-channel angular pressing (ECAP) and accumulative roll bonding (ARB) [2], are defined as a method of metal forming that is used to impose a very high strain on a bulk solid without introducing any change in the dimensions of the samples [3]. However there is a general tendency for a saturation in grain refinement and recovery (and even recrystallization) by continuing bulk SPD processes [4,5]. On the other hand, surface SPD processes, which include sliding friction [6], shot peening such as surface mechanical attrition treatment (SMAT) [7,8], machining [9], burnishing such as surface mechanical grinding treatment (SMGT) [10] and hammering such as ultrasonic nano-crystalline surface modification (UNSM) [11,12], are usually accompanied by the formation of the nano-crystalline structure at thin surface layers. Principal differences of surface SPD

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methods in comparison to the bulk SPD processes are the localization of plastic deformation near the surfaces and strong gradient of all properties (strain, stress, microstructure, hardness, etc.) [13]. The achievement of the surface nano-crystallization can be attributed to the fact that the near surface straining results in nonhomogeneous plastic deformation with large strain gradient and high density geometrically necessary dislocations [14].

Among these surface SPD methods, sliding friction is one of the most powerful processes for microstructural evolution in the sub-surface, including grain refinement, recrystallization of deformed structure, interactions with environments, transfer and mechanical mixing [15]. Advantages of sliding friction are that friction conditions (speed, load, etc.) can be changed over wide ranges and that friction apparatuses are fairly simple. Moreover, friction induced UFG structure would exhibit potentially high wear resistance resulting from the high hardness at the surface. However the influences of sliding conditions on friction-induced microstructure and wear behavior of friction-induced microstructures have not been clear.

In the present study, pin-on-disc sliding friction tests were carried out for 0.45 mass % carbon steels, and microstructure and Vickers hardness of friction induced structure were investigated. Particularly the effects of friction conditions (friction speed and atmosphere) on the microstructure at the surfaces and wear properties of the friction induced microstructures were studied.

**EXPERIMENTAL**

A common pin-on-disc method, which is schematically illustrated in Fig. 1, was employed in sliding friction tests. A pin, 5 mm in diameter (the contact area diameter was 2 mm) and 20 mm in length, was loaded on a rotating disc, which has a diameter of 60 mm and a thickness of 5 mm. The materials used for the pin and disc specimens were normalized 0.45 mass% carbon steel (JIS S45C). A pin and a disc of the same materials were rubbed each other in an atmosphere of air or a vacuum ($10^{-4}$ Pa). The sliding speed was varied in the range of 0.05 - 5.0 m/s, and the applied load was varied in the range from 10 to 50 N. Both the pin and disc specimens were cleaned ultrasonically in acetone prior to friction testing.

The microstructure of the specimens after rubbing was examined at the longitudinal cross-section of the sub-surface by optical microscopy, TEM (Transmission Electron Microscopy) and STEM (Scanning Transmission Electron Microscopy). The cross-sectional STEM and TEM samples were prepared by FIB (Focused Ion Beam) milling using a standard lift-out technique [16,17]. The Vickers hardness was measured at the cross-section near the surfaces of the specimens with a test load of 0.098 N for 15 s.
The wear properties of the friction-induced microstructure were investigated by using the same pin-on-disc sliding method. In wear tests, the rubbed pin specimens with friction-induced microstructure surface layers were slid against as machined 0.45 mass% carbon steel discs in an atmosphere of air. The sliding speed was 0.1 m/s, and the load was 20 N. The wear amount was evaluated from the pin displacement.

RESULTS AND DISCUSSION

Effects of Friction Speed on Microstructure. Figure 2 shows optical microstructure of the longitudinal cross-section of the sub-surface for a pin rubbed at a load of 20 N in an atmosphere of air. When the sliding speed was low (Fig. 2 (a) and (b)), plastic flow structure in which grains are inclined to the friction direction was formed near the surface. On the other hand, in the case of a high sliding speed of 5.0 m/s (Fig. 2 (c)), a featureless white-etched layer, that is ultra-fine grained (UFG) structure, was observed above the plastic flow structure.

![Figure 2](image.png)

Figure 2. Optical microstructure of a longitudinal cross-section of the sub-surface of the pin rubbed in air at a sliding speed of (a) 0.05 m/s, (b) 0.5 m/s and (c) 5 m/s.

Figure 3 shows STEM microstructure of the longitudinal cross-section of the same pin samples. In the case of a low sliding speed of 0.05 m/s (Fig. 3 (a)), lamella structure was observed. The lamella structure became finer as the worn surface was approached, and the lamella interlayer distance reached approximately 80 nm at the top surface. When the speed was higher of 0.5 m/s (Fig. 3 (b)), ultra-fine lamella structure was formed, and the interlayer distance was approximately 50nm. On the other hand, in the case of 5.0 m/s (Fig. 3 (c)), ultra-fine equi-axed grains in the 30 - 50 nm size range were observed. The nano-crystalline structure could be formed by high strain rate or phase transformation owing to the temperature rising caused by frictional heating [18]. Thus high sliding speeds were favorable to obtain fine microstructure at the frictional surface. BCC structure of α-Fe was identified by the selected area diffraction (SAD) pattern for the each specimen.

The Vickers hardness profiles of the sub-surface of the pins rubbed in air were shown in Fig. 4. It can be seen that the hardness increased as the surface was approached and the sliding speed increased. Obviously the hardness rise is due to the grain refinement caused by frictional severe plastic deformation.
Effects of Atmosphere on Microstructure. Figure 5 is microstructure of the longitudinal cross-section of the sub-surface for a pin rubbed at a low sliding speed of 0.05 m/s at a load of 50 N in an atmosphere of a vacuum. UFG structure at the surface and plastic flow structure underneath the UFG structure were observed. The UFG structure thickness (∼30μm) was thicker than that of the air atmosphere specimen. The UFG (in the 30 - 70 nm size range) were slightly elongated in the friction direction, which implied that the effect of friction straining remained in the structure. The SAD pattern showed nearly-continuous rings, which indicated that the microstructure was composed of BCC polycrystalline with an almost random orientation. The important finding is that nano-crystalline microstructure can be produced even if the friction speed is low in the case of a vacuum atmosphere. This is probably due to the fact that large strain can be introduced at the surface, because the material is easily adhered and difficult to be removed as wear debris from the surface. Since frictional heating is negligible small in the low sliding speed, we confirm that UFG structure is not produced by recrystallization owing to phase transformation caused by frictional heating, but large friction strain and large strain gradient [19].
Figure 5. (a) Optical micrograph of a longitudinal cross-section of the pin rubbed in a vacuum at a sliding speed of 0.05 m/s and Vickers hardness results and (b) TEM dark-field image of the rectangular area marked “A” in (a).

Wear Properties of Friction Induced Microstructure. It is hoped that the friction-induced nano-crystalline structures exhibit high wear resistance owing to the high hardness. The wear properties of the friction-induced microstructures were investigated by using the same pin-on-disc machine, i.e., the pin specimen with the friction-induced microstructure was rubbed against a new S45C disc (as machined). Fig. 6 shows the wear test results of the friction induced microstructure of the pins.

Figure 6. Wear properties of the friction-induced microstructure: (a) wear volume of the pin rubbed in air, (b) wear volume of the pin rubbed in a vacuum.

The pins with the plastic flow microstructures that were produced at relatively-low sliding speeds of 0.05 - 1.0 m/s in an atmosphere of air exhibited high wear in the same way of an as machined pin. On the other hand, the pin with the UFG microstructure that was produced at a high sliding speed of 5.0 m/s in an atmosphere in air exhibited low wear. Moreover, the pin with the UFG microstructure that was produced at low speeds of 0.05 – 0.5 m/s in a vacuum atmosphere exhibited low wear. As shown above, ultra-fine grains in the 30 - 50 nm size range had been produced in these specimens. Therefore we conclude that the friction-induced nano-crystalline structure shows high wear resistance. However the plastic flow microstructure did not exhibit good wear resistance. This might be due to the severe wear test conditions in the present study.

SUMMARY

Pin-on-disc sliding friction tests were carried out for 0.45 mass % carbon steels, and microstructure and Vickers hardness of friction induced structure near the surface were investigated. The following conclusions were obtained:
When friction tests were conducted at low sliding speeds (0.05 and 0.5 m/s) in an atmosphere of air, plastic flow structure in which grains are inclined to the friction direction was formed near the surface. On the other hand, in the case of a high sliding speed of 5.0 m/s, ultra-fine equi-axed BCC structure grains in the 30 - 50 nm size range were observed.

Nano-crystalline microstructure can be produced in a vacuum atmosphere even if the friction speed was low. It was confirmed that UFG structure is not produced by recrystallization owing to phase transformation caused by frictional heating, but large friction strain and large strain gradient.

The friction induced nano-crystalline structure showed good wear resistance due to the significant high hardness.

REFERENCES