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Sliding wear behavior of sub-microcrystalline pure iron produced by high-pressure torsion straining

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ABSTRACT

Sliding wear behavior of sub-microcrystalline pure iron disks produced by high-pressure torsion (HPT) straining was studied using a ball-on-disk configuration, and the effects of the strain in the HPT process and of the duration of wear tests were investigated. It was found that the wear amount decreased with increasing number of turns during HPT, and that the specific wear rate of the initial stage was inversely proportional to the Vickers hardness of the disks. However, for long wear test times, the wear rate became very small for both non-deformed and HPT-processed specimens, because of transition from adhesive shearing wear to sliding process which was caused by (i) grain refinement at the wear-induced layer, (ii) submicron wear particles adhered to the worn surface, and (iii) surface flattening by local asperity deformation. There was no significant difference in microstructure and hardness beneath the worn surface between non-deformed and HPT-processed specimens after the wear tests. Thus HPT improved the wear resistance only at the early stage, and became less effective for long wear tests. This study is the first to show that submicron grains produced by HPT straining were further refined to a size of 100–150 nm by friction shear straining while grain refinement of iron is saturated upon further straining in HPT processing.

1. Introduction

Bulk nano/submicron-structured materials produced by severe plastic deformation (SPD), such as high-pressure torsion (HPT), equal-channel angular pressing (ECAP) and accumulative roll bonding (ARB), have attracted growing interest, owing to their superior mechanical properties [1]. The HPT process, where a thin disk is held between two anvils and subjected to torsional straining under high pressure, is one of the most powerful techniques for preparing ultrafine-grained (UFG) materials [2], and the microstructure and mechanical properties of HPT-processed pure iron and steels have been studied [3–5]. Since wear resistance is a critically important material property for UFG materials intended for structural applications, the wear properties of UFG materials fabricated by SPD processes are of great interest, in view of the potentially high wear resistance resulting from high hardness.

As has been pointed out by Gao et al. [6], however, conflicting results have been reported concerning the effects of SPD processes on wear behavior. Some studies have demonstrated that wear resistance increases as a result of SPD processes [7–13], while others have claimed that it decreases owing to (i) low strain-hardening capability [14–17], (ii) micro-crack formation due to low ductility [14,18,19], (iii) grain recrystallization and growth due to friction heating [20,21], and (iv) intensive adhesion [22]. Another study has reported that SPD processing has no significant effect on the wear behavior [23]. One of the weaknesses of the literature on wear in SPD-processed specimens is that the wear mechanism remains poorly understood because of the limited data obtained by material characterization after the wear tests.

Wang et al. [17] carried out a flat-on-flat reciprocating sliding wear test in ambient air for an HPT-processed Al-1050 alloy. An HPT-processed disk sample with a 10 mm diameter was rubbed against a surface made of 316 stainless steel, giving a contact area of about 78 mm^2. They exhibited that the initial severe wear stage of the HPT-processed Al-1050 alloy was much longer than that of the as-received samples, resulting in a decrease in the wear resistance of the HPT-processed specimens, and claimed that the long severe wear stage was due to the loss of work hardening capacity after HPT straining. However, the subsurface material characteristics after the wear tests were not shown; thus, there was no data on the work hardening behavior in their paper. Zhilyaev and coworkers [19] conducted sliding wear tests for UFG

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copper materials processed by combinations of HPT and ECAP using an oscillating friction tribometer with a ball-on-disk contact configuration. Copper disks with a 10 mm diameter slid against Cr6 steel balls having a diameter of 6 mm at an oscillating stroke of 1 mm. They reported that UFG specimens gave higher wear rates than coarse-grained copper, and concluded that the high wear rates were attributed to brittleness of the UFG copper leading to the development of crack networks. Moreover, they claimed recently that annealing of Cu specimens processed by HPT led to an increase in the grain size and a decrease in the wear rate [24].

On the other hand, Wang's group [25] has also performed scratch tests on HPT-processed pure Ti samples and demonstrated that HPT-processed samples have better wear resistance than coarse-grained Ti. A diamond indenter with a radius of 200 μm was used to perform a reciprocating movement on HPT-processed disk specimens (diameter: 10 mm), and the testing area was located between 2 and 4 mm from the center of the disk. Faghihi et al. [26] performed wear tests using a linear reciprocation ball-on-flat tribometer and showed that the wear resistance increases as a result of HPT. An alumina ball with a 4.75-mm diameter was rubbed against an HPT-processed pure Ti disk with a 12 mm diameter, and the stroke length was 5 mm. Recently, Aal et al. [27] also carried out reciprocated sliding wear tests on an HPT-processed Al–7 mass% Si alloy. A tungsten carbide ball with a diameter of 5 mm was rubbed against an HPT-processed disk with a diameter of 10 mm. The stroke length was 7 mm, and the wear scar position was 2.5 mm from the disk center. Aal et al. reported that the wear mass loss and coefficient of friction were reduced after the HPT process relative to the as-cast samples owing to the increase in hardness through grain refinement. However, the initial microstructure can be modified during friction sliding and thus the grain size and hardness might play a role only in the early stage of wear [19]. Nevertheless, studies on the influence of strain in the HPT process and of the duration of wear tests are limited.

Another shortcoming of the literature has to do with the wear test method. It is known that the shear strain in HPT increases linearly with the distance from the center of the disk sample [28,29] and that the hardness also depends on the distance from the center [3–5]. In the flat-on-flat sliding wear test, the effect of the disk position on hardness is not taken into account. Although the hardness distribution seems nearly homogeneous across the disk sample area owing to the high number of revolutions in the HPT process, a problem still remains in that the strain varies with distance from the disk center.

In the present work, the dry sliding wear properties of HPT-processed pure iron were investigated using a ball-on-disk friction method. Sliding wear is most frequently encountered in engineering components in comparison with other situations such as rolling, fretting or erosive wear. A ball was loaded on a rotating HPT-processed disk so that the rubbing could be performed at a constant distance from the center of the disk. The advantage of this method is that the effects of strain can be detected directly, unlike in reciprocating sliding wear tests [17,19,24–27]. The aims of this work were to investigate the effects of strain (i.e., the number of turns in the HPT process) and of the wear test time on the wear behavior, and to assess the wear resistance of HPT-processed sub-microcrystalline pure iron. Furthermore, the worn surface topography and the subsurface metallurgical changes taking place during wear tests were studied to clarify the wear behavior.

2. Experimental

2.1. Materials

The starting material was pure iron (11C, <30Si, <30Mn, <20P, <3S, <2B, 8N, 14O, 300Al, <20Ti, <30Cr, <30Cu, mass ppm), which was annealed at 1273 K for 1 h in a pure Ar atmosphere. Optical microscopy revealed that the average grain size in this material was $\sim$500 μm. The material was cut and polished into disks 10 mm in diameter and 0.85 mm in thickness in preparation for HPT straining.

The HPT process was carried out using anvils with a depression 0.25 mm in depth and 10 mm in diameter. Details of the HPT process have been described elsewhere [30]. Since the flat bottom of the depression was roughened to increase the frictional force between the disk and anvil, no slip occurred during HPT straining. A disk specimen was placed between the stationary upper anvil and rotational lower anvil and torsion-strained at a rotation speed of 0.2 rpm under a pressure of 5 GPa at room temperature. The number of turns (N) was varied between five conditions: $N=1/4$, 1/2, 1, 5 and 10. For comparison, $N=0$ specimens, which were only compressed at 5 GPa but not torsion-strained, were prepared as well. The average grain size of the $N=0$ specimen was about 300 μm. For wear tests, burr, which was formed around the periphery of the HPT-processed disk owing to material flow in the radial direction, was removed by grinding, and then the flat surface of the disk was mechanically polished with #400 emery paper to achieve an average surface roughness (Ra) of less than 0.4 μm, as measured by a Accretech Surfcom 130A stylus surface profilometer.

The microstructure of HPT-processed disks was characterized using a Jeol JSM7001F scanning electron microscope (SEM) equipped with a field emission gun operating in the backscatter electron (BSE) mode. To prepare SEM samples, a disk was cut in half, forming a centered vertical section along the torsional axis, which was polished with emery papers and a diamond-lapping film on the order of 1 μm, followed by chemical–mechanical polishing with 0.04 μm colloidal silica for 30 min. SEM observation was performed at an accelerating voltage of 15 kV and a working distance of 4 mm. Vickers hardness measurements were carried out on the centered vertical section of the HPT-processed disks by using a Mitutoyo HM-102 tester with an applied load of 0.98 N for 15 s.

2.2. Wear test method

Wear properties of the HPT-processed pure iron were investigated using a ball-on-disk friction method, illustrated in Fig. 1. A ball, 6 mm in diameter, was loaded on a rotating HPT-processed disk in a chamber. The rubbing track diameter was 5 mm. The ball material used was cemented carbide (94 mass% WC, 6 mass% Co) with a hardness of Hv 1745, which is one of the most widely used materials in tribo-systems because of the high wear resistance. The ball surface was polished to a mirror-like finish. The disk and ball specimens were cleaned ultrasonically in acetone prior to wear testing. Wear induced oxide surface layers often mask the effect of strengthening by UFG structure [23]. Thus the air in the chamber was evacuated to a vacuum on the order of 10^{-4} Pa, and then replaced by pure Ar (99.999%) gas at atmospheric pressure in order to minimize tribo-oxidation. All wear tests were carried out at a temperature of 15–25 °C in a pure Ar atmosphere. The sliding speed was 1.57 mm/s (the rotation speed of the disk: 6 rpm), and the applied load was 40 N. The reason for choice of this low sliding speed was to minimize the effects of frictional heat. The rather high load, which corresponds to an initial mean Hertian contact pressure of 1.5 GPa, was chosen so that the native oxide layers of the specimens would be removed by wear immediately. Three