Wear 376-377 (2017) 931–939

Contents lists available at ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

Tribological properties and mechanisms of self-mated ultrafine-grained titanium

J.E. Mogonye, T.W. Scharf*

Department of Materials Science and Engineering and Center for Advanced Research and Technology, University of North Texas, Denton, TX 76203-5017, USA

ARTICLE INFO

Article history:
Received 2 September 2016
Accepted 19 October 2016

Keywords:
Titanium
Severe plastic deformation
Ultrafine grained materials
High frequency reciprocating sliding
Self-mated sliding wear

ABSTRACT

While titanium and its alloys have an excellent balance of properties, they continue to have limited applications owing to their relatively poor wear resistance, in particular for commercially pure titanium. Processing by severe plastic deformation (SPD) has been developed over the last two decades that produces an ultrafine-grained (UFG) microstructure resulting in improved mechanical properties. However, only limited studies are available to date on the wear behavior of SPD-processed UFG metallic materials with many conflicting results in terms of reduced wear rates. Furthermore, the wear behavior and mechanisms of SPD-UFG metallic materials are unknown in self-mated sliding contacts. Here, the friction and wear properties of self-mating UFG titanium in a cross-cylinder high frequency reciprocating contact is reported and compared to coarse grain (CG) titanium. Cross-sectional focused ion beam microscopy, transmission electron microscopy, and Raman spectroscopy studies were performed inside worn surfaces to determine the dry sliding wear-induced structural and chemical evolution responsible for the observed friction coefficients and wear rates. It was determined that the microhardness of UFG titanium increased from 2.6 to 3.4 GPa; however, this increased hardness did not correlate to lowering of friction and wear. Instead, the similar friction coefficients and wear rates of CG titanium and UFG titanium were due to similar wear surface structures and oxide debris composition. Furthermore, wear surface morphologies and cross sections inside the wear tracks revealed that CG titanium had a major shear deformation contribution to wear, but with the wear rates being equivalent, wear is most likely dominated by oxide particle abrasion.

1. Introduction

Severe plastic deformation (SPD) processing, such as equal-channel angular pressing/extrusion (ECAP/ECAE), accumulative roll bonding (ARB), and high pressure torsion (HPT), of metallic materials has received much attention over the last two decades due to the well documented improvement in mechanical properties by strain hardening and grain refinement [1,2]. In particular, the SPD-induced UFG structure (< 1 μm grain size) that results in increased hardness has been a motivating factor to study the wear resistance based on Archard's relationship of increasing wear resistance with hardness. While there have been numerous studies detailing the improvements in mechanical properties with SPD-processed materials, there have been less on the tribological properties, and from these studies there are conflicting results, which a recent review summarized in detail [3]. In the case of commercially pure titanium, six studies [4–9], and subsequent ones since the published review [10–13], report wear resistance either increases, decreases, or remains the same after SPD processing when compared to CG counterparts. While the studies consistently determined that UFG structures lead to increased hardness, the reported wear rates, like the friction coefficient, are a systems property dependent upon a range of factors, both experimental, e.g. counterface material, and wear-induced, e.g. tribofilm formation and debris entrapment. Nevertheless, several factors emerged from the review that appear to influence the wear resistance of SPD-processed materials [3]. In the case of improved wear resistance it was determined that smaller grain size, refined microstructure, higher hardness and strength are contributing factors. In contrast, this improvement in wear resistance can be negated by decreased ductility, low strain hardening capability, high oxidation rate, and strain-induced grain coalescence.

However, none of the tribological investigations on SPD-processed materials to date have studied self-mated SPD-UFG materials in sliding contacts. Therefore, the present study investigates the hardness, friction and wear properties of self-mated SPD-UFG titanium in a cross-cylinder high frequency reciprocating contact,

* Corresponding author.
E-mail address: scharf@unt.edu (T.W. Scharf).

http://dx.doi.org/10.1016/j.wear.2016.10.016
0043-1648/

with comparisons to CG titanium. The high frequency reciprocating tribometer was selected since it can accommodate self-mated SPD processed cross-cylinders, and small multiple stroke lengths can be placed along the cylinder length. In addition, the differences in UFG and CG wear particle size and morphology were studied in these self-mated contacts. Mechanistic studies were also performed with cross-sectional SEM-FIB-TEM and Raman spectroscopy inside worn surfaces and subsurfaces to determine the sliding wear-induced structural and chemical evolution responsible for the friction and wear behavior, and to provide new insights into some of the aforementioned conflicting wear resistance results.

2. Experimental methods

2.1. Materials

As received cylindrical specimens of commercially pure titanium, α titanium hexagonal close-packed structure, were of varying lengths with initial diameters of either 12.0 mm or 5.4 mm for CG titanium and UFG titanium, respectively. The UFG titanium was processed by shear extrusion, similar to equal channel angular extrusion, a severe plastic deformation process that results in microstructural grain refinement [14,15]. In these two structures, the measured titanium grain size varied from 8 to 20 μm for CG titanium, determined by electron backscatter diffraction, and 50 nm to 2 μm for UFG titanium, determined by precession electron diffraction [14,15]. To accommodate sample sizes in the reciprocating tribometer fixtures, which has up to 6.35 mm diameter acceptance, cylinders were cut to length using a wire electrical discharge machine (EDM) in a deionized water dielectric bath. Subsequently the CG specimen diameter was reduced to near 6.35 mm using the wire EDM and then polished down to a diameter of $\leq$6.35 mm on a lathe using silicon carbide paper to remove the EDM recast surface layer that could otherwise bias the friction and wear results. Final polishing of the CG samples was completed with 600 grit silicon carbide paper until the surface finish was visually similar to the UFG samples, which did not undergo any diametrical dimension changes for testing. Prior to characterization, all sample surfaces were cleaned by ultrasonication in acetone for 10 min followed by 10 min in propanol then blown dry under nitrogen gas flow.

2.2. Hardness and wear testing

Microhardness measurements were conducted on a HMV Shimadzu micro hardness tester. Samples approximately 5 mm thick were cut from the cylinders prepared for friction and wear testing using a diamond saw. The samples were then mounted in a thermoset plastic and the surfaces polished to a final finish using 1200 grit silicon carbide paper. A Vickers geometry diamond was used under a normal load of 1 kgf (9.807 N) with a 10 sec hold at peak load. A total of 9 individual indents were made on each specimen with a minimum spacing of 300 μm between indents. Indentation size diagonals were measured using a digitally calibrated 40X objective.

Self-mated titanium friction and wear tests were performed on a Compass Instruments, Inc. high frequency reciprocating rig (HFRR) using custom-built cylindrical fixtures. These fixtures, shown in Fig. 1, allow for reciprocated sliding motion in a crossed-cylinder configuration with one cylinder stationary and the other reciprocating. This contact geometry results in a circular point contact for the stationary cylinder that allows for initial contact stress evaluation by Hertz's equations for maximum contact stress and shear stress. Although the CG and UFG samples were of different radii, similar wear contact conditions were achieved for both by using equivalent initial Hertz stress conditions by varying the applied dead weight/normal force, F_N. Three separate initial maximum Hertzian stresses of nominally 250, 500, and 1000 MPa were used for each material pair during HFFR testing at a frequency of 20 Hz, shown in Table 1. The 500 MPa maximum contact stress condition was also conducted at a reciprocating frequency of 50 Hz to assess friction dependence on sliding velocity. The total sliding distance for each HFRR test was 216 m and each test condition was repeated at least three times.

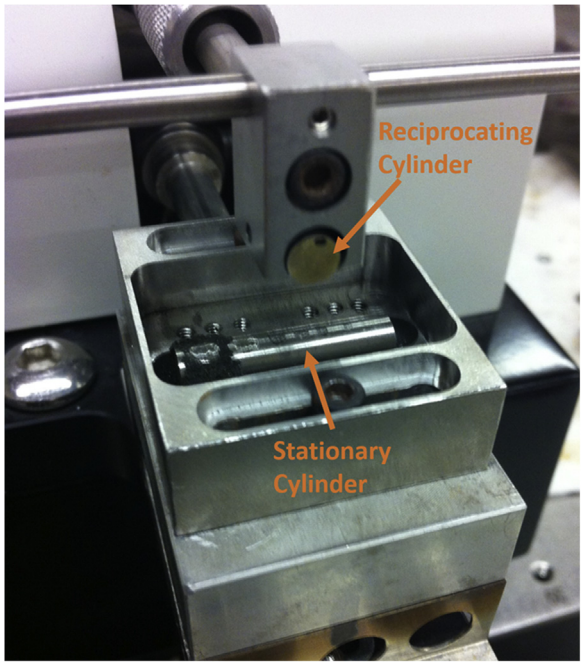

Fig. 1. Photograph of high frequency reciprocating sliding tribometer with UFG titanium cylinders (5.4 mm diameter) mounted in custom-built crossed-cylinder fixtures.

Specific wear rates were quantified for each HFFR test condition as the material volume removed normalized by the product of the applied normal load and total sliding distance. Wear volumes post HFFR testing were calculated from micro stylus profilometry data collected using a Veeco Dektak 150. Profilometry scans were performed across the midpoint of the wear scar and perpendicular to the direction of sliding for stationary cylinders and parallel to the direction of sliding for reciprocating cylinders. These wear scar cross sectional profilometry scans were extrapolated to wear volume according to the approximate functional solution to the volume integral of intersecting crossed cylinders provided by Warburton and Bradford [16]. A MATLAB user input script was used to calculate the circular fit to the worn area values from the measured micro stylus profilometry data.

2.3. Wear debris particle characterization

Loose wear particle debris from both CG and UFG titanium samples were collected from the bottom stationary cylinders after 20 Hz reciprocating frequency testing for the three loading conditions. The loose particles were then introduced to a solution of 20% ethanol and 80% deionized water at a nominal density of 1 mg/mL by measuring wear debris mass by microbalance. The wear particle debris solutions were then ultrasonicated for two hours to assist break up of conglomerations of particles. Further sonication and shaking of vials was utilized until there was no observable particle sediment in the vials. Immediately following the final ultrasonication step 2 mL of the suspension was