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Effect of grain size on the micro-tribological behavior of pure titanium processed by high-pressure torsion

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ABSTRACT

The micro-wear behavior of commercial pure Ti was investigated before and after processing by high-pressure torsion (HPT) to provide comparisons over a range of grain sizes. The HPT-processed Ti had an average grain size of ~130 nm while the as-received and HPT plus annealed samples had grain sizes of ~8.6 μm and ~607 nm, respectively. The results show all Ti samples have a similar dynamic coefficient of friction but different wear mechanisms. Wear of the coarse grained (CG) Ti showed extensive plastic deformation and wedge formation which produced large wear debris whereas wear of the ultra-fine grained (UFG) Ti was dominated by abrasive wear mechanisms and produced small wear debris. In addition, the UFG Ti showed a more homogenous wear grooving and a lower wear rate than CG Ti which suggests that UFG Ti is more suitable for wear applications.

1. Introduction

Processing by severe plastic deformation (SPD) is defined formally as any method of metal forming, conducted under a hydrostatic pressure, that may be used to impose a very high strain on a bulk solid without introducing any significant change in the overall dimensions of the sample and with the capability of producing exceptional grain refinement [1]. It is now widely established that SPD processing produces ultra-fine grained (UFG) materials possessing unique mechanical and physical properties [2]. Potential industrial applications of SPD-processed materials include precision instruments, biomedical implants and micro-electro-mechanical systems (MEMS) devices [3–5]. However, during the service life of moving contacts, damage is often caused by wear particles that are generated from the interface, especially in the field of electronic engineering and precision instruments.

Considering the inevitability of wear to components moving against each other in close contact, there has been considerable interest in testing the wear behavior of SPD-processed materials [6–13]. Nevertheless, there is conflicting evidence on the effect of SPD processing on the wear resistance of materials, where this is due in part to the different materials studied but primarily it is due to the inherent complexity of wear when two materials contact over a large area.

Some earlier work focused on the nature of the microscopic wear mechanisms and the generation of wear particles in micro- or nano-contacts [14]. An in situ scanning electron microscopy (SEM) wear study via micro- or nano-tribology test systems was one of the most effective methods and several different types of test systems have been developed. An in situ SEM video tape recording (SEM-VTR) tribosystem was developed and used to study the micro wear mechanisms of steel, ceramics and coatings. Several different wear modes, and the transitions between them, were plotted on wear maps [14–16]. Atomic force microscopy (AFM) was also employed to scratch samples in a much lower load range. Recently, a cost effective tribometer was developed which was used to study the micro-scratch behavior of WC/Co hard metals and coatings [17–19]. This test system was subsequently further developed so that it can perform wear tests with a load range between 5 mN and 250 mN, thereby filling the gap between AFM and the traditional wear testers.

Abbreviations: AFM, atomic force microscopy; CG, coarse grained; COF, coefficient of friction; CP, commercial purity; HPT, high-pressure torsion; IFM, infinite focused optical microscopy; MEMS, micro-electro-mechanical systems; OM, optical microscopy; PBS, phosphate buffer solution; SEM, scanning electron microscopy; SPD, severe plastic deformation; TEM, transmission electron microscopy; UFG, ultra-fine grained.

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Titanium and its alloys are attractive candidate materials for applications from aerospace to sports and biomedical due to their high strength to weight ratio, excellent corrosion resistance and good biocompatibility [20]. Alloys such as Ti–6Al–4V are most often used for biomedical applications, but nevertheless the alloy elements released from Ti alloys may cause long-term health problems [21–23]. Therefore, attempts have been made to produce UFG pure Ti with a comparable high strength to Ti–6Al–4V and a much improved biocompatibility [24–28]. Recent studies also reported the superior osteoblast cell compatibility of UFG pure Ti substrates produced via SPD processing over coarse grained (CG) pure Ti and Ti–6Al–4V substrates, thereby making the UFG pure Ti even more attractive for biomedical applications [27,29].

Recently, attention has been directed to studies of the tribological behavior of Ti processed via SPD [6,25,30,31]. Initially, it was reported that UFG Ti had a lower coefficient of friction than CG Ti under various conditions [25], but this result was not consistent with a later study which revealed a similar coefficient of friction around 0.6 for both CG Ti and UFG Ti [6]. This conflict may be due to the different experimental setups used, as the indenter of the friction tester in one study rotated near the centre of the Ti sample [25] whereas in the other study the indenter reciprocated on the surfaces of the Ti samples [6]. Nevertheless, all studies showed the same trend for the wear mechanism, with the SPD processing inhibiting adhesion and leading to wear by micro-abrasion. Two recent studies reported a similar or slightly worse wear resistance for UFG Ti compared to CG Ti under very low contact pressures, and this was attributed to the formation of an oxide layer which masks the effect of the microstructural change [30,31]. Tribocorrosion tests in a phosphate buffer solution (PBS) demonstrated a better performance for UFG Ti processed by high-pressure torsion (HPT) compared to CG Ti under the tribocorrosion conditions [28]. Furthermore, wear tests performed in a liquid nitrogen environment showed that Ti with the smallest grain size exhibits the best wear resistance due to a higher strength and lower ductility at this very low temperature [32].

The present investigation was initiated to study the microtribological behavior of commercial purity (CP) titanium after processing by HPT with and without annealing. The purpose of the study was to understand and compare the scratch behavior of samples having different grain sizes. The coefficient of friction (COF) of SPD-processed material was compared with the as-received material. A worn surface examination by SEM and infinite focus optical microscopy (IFM) was used to identify the nature of the wear mechanisms and also to evaluate the volume loss.

2. Experimental material and procedures

A CP titanium alloy (Grade 2) was used in this investigation with the following impurities in wt.%: 0.015%H, 0.1%C, 0.25%O, 0.03%N and 0.3%Fe.

Prior to micro-scratch testing, some of the materials were subjected to HPT processing using the procedure described in detail elsewhere [33]. For HPT, specimens having diameters of 10 mm were sliced from the as-received billets and ground with abrasive paper to thicknesses of ~0.8 mm. The HPT processing was conducted at room temperature on a facility having two anvils with circular flat-bottom depressions at the centres. The depression depth was 0.25 mm and the diameter was 10 mm. The processing was performed under a pressure of 3.0 GPa for 10 revolutions. After HPT processing, some of the HPT-deformed samples were annealed at a temperature of 500 °C for 10 min.

Following HPT, all samples including as-received samples were prepared with 4000# SiC abrasive papers followed by polishing with 1 μm diamond paste which gave each sample an average roughness R_a of 15 ± 5 nm. After cleaning with acetone, the samples were subjected to micro-scratch testing using a new micro-scratch tester developed at the National Physical Laboratory in the UK. As shown in Fig. 1, the key feature of this micro-scratch system is the flexure design which was based on a three dimensional cage with an eight-bar kinematic chain. When the probe is brought into contact with the sample surface, a compression force is generated by deflection of these eight bars. At the same time, the normal force capacitance plate measures the deflection of the bars and thereby indicates the extent of bending. The position of the probe is adjusted by applying a feedback signal to a piezoelectric actuator mounted under the flexure to keep the compression force constant. Similarly, the friction force generated during the wear test is measured using the vertical capacitance plate. The micro-scratch system can carry out wear tests with load ranges of 5–250 mN, a stroke length of up to 8 mm and a speed range of 15–220 μm s^{-1} [34].

These scratch tests were performed at room temperature (18 ± 2 °C) under a relative humidity of 50 ± 5%, under loads of 100 or 200 mN for 1, 2, 5, 10, 50, 100 or 200 turns. These two loads were designed to lie within the capacity of the micro-scratch system and to bring sufficient plastic deformation to the samples so that the wear behavior of the different samples would be readily recorded. A diamond indenter with radius of 200 μm was used to create a micro-scratch of 2 mm length on the disk surface. The testing area was located between 2 and 4 mm away from the centres of the disks and the indenter carried out a reciprocating movement on the sample surfaces with a speed of 0.1 mm/s.

The Vickers microhardness, Hv, was measured using a Matsuzawa Seiki MHT-1 microhardness tester with a test weight of 1000 g and a dwell time of 15 s. The microstructures of the samples were investigated by optical microscopy (OM) and transmission electron microscopy (TEM). Specimens for OM were fine ground by 4000# SiC abrasive paper and then etched with a solution of $HF:HNO_3:H_2O = 2:3:10$ in volume percentage. The foils for TEM were produced by twin-jet chemical polishing at a temperature of −30 °C in a solution of 5% perchloric, 35% butanol and 60% methanol.

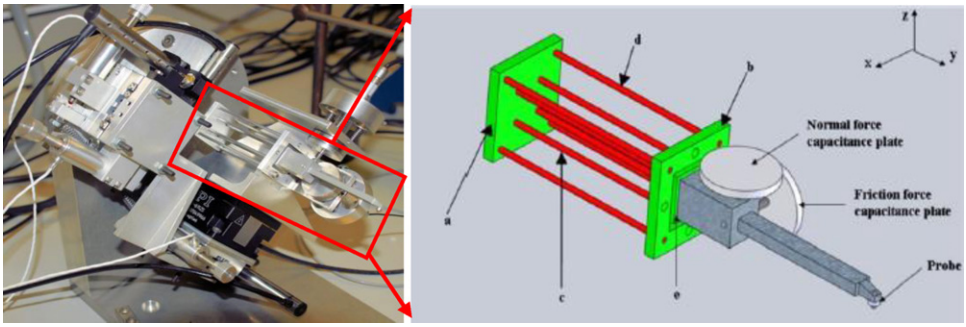

Fig. 1. The overall schematic of micro-scratch test system: (a) the free plate in the flexure element; (b) the mounting plate supported on the piezoelectric actuator; (c) is inner flexure bars; (d) outer flexure bars; (e) the probe lever support [34].