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Effects of grain refinement and strength on friction and damage evolution under repeated sliding contact in nanostructured metals

T. Hanlon[a,b], A.H. Chokshi[a,1], M. Manoharan[b], S. Suresh[a,*]

[a]Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA
[b]GE Global Research Center, Niskayuna, NY 12309, USA

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Abstract

The early stage sliding contact fatigue behavior of nanocrystalline materials, with average and total range of grain sizes well below 100 nm, was studied. The evolution of friction and damage during repeated sliding contact in the nanocrystalline metals and alloys was systematically compared and contrasted with that in ultrafine-crystalline and microcrystalline materials so as to develop a broad perspective on the effects of grain size on sliding contact fatigue. Some critical experiments were performed to separate the effects of material strength and grain size on friction and damage evolution. Over the range of materials examined, strength rather than grain size appeared to dominate the steady-state friction coefficient and damage accumulation, each diminishing with substantial increases in material strength.

Keywords: Nanocrystalline; Ultra-fine-crystalline; Equal channel angular pressed

1. Introduction

The potential use of nanocrystalline (nc) materials in load-bearing structural applications inevitably will depend on their resistance to damage and failure under repeated loading conditions. Currently, however, there is a relative paucity of experimental data pertaining to these properties within the nc grain size regime. In a companion paper [1], we examine the mechanical fatigue characteristics of nc metals and alloys in terms of grain size, load ratio as well as relative propensity for crack initiation and crack propagation. This study was initiated to probe the sliding contact fatigue behavior of several nc (average grain size typically smaller than 100 nm) and ultra-fine-crystalline (ufc) materials (average grain size between 100 and 1000 nm), with direct comparisons made to their microcrystalline (mc) counterparts (average grain size above 1000 nm), wherever appropriate.

Several previous studies have been initiated to characterize abrasion and wear properties of mc materials [2–6]. However, the complexity of the mechanical damage evolution process has, in general, led only to empirical results without associated fundamental trends. While there are no simple relationships between properties such as wear performance and hardness (for example), it has been proposed that scratch testing provides a more accurate means of characterizing a single abrasive event [7–10]. Repeated scratch testing, where a single region of the surface is exposed to multiple contact passes, is extremely useful in terms of monitoring damage evolution [11,12], and has previously been employed to study the wear behavior of plasma sprayed coatings and hard metals [13–15].

The objective of this study was to determine the effects of grain size on friction evolution and damage accumulation under contact fatigue conditions. Because sliding contact entails significant compression and shear at asperity contacts, large plastic strains are expected, even at low applied loads. Plastic deformation, and the associated energy dissipation, can therefore contribute significantly to the level of friction during sliding. As a result, critical experiments designed to isolate the effects of grain size reduction from the associated increase in strength were performed. The present study seeks to deal with a number of

* Corresponding author. Tel.: +1 617 253 3320; fax: +1 617 253 0868.
E-mail address: ssuresh@mit.edu (S. Suresh).
[1] Permanent address: Department of Metallurgy, Indian Institute of Science, Bangalore, India.

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novel issues. First, recent progress in the study of mechanical deformation of nanocrystalline metals and alloys has provided a fundamental knowledge base with which the repeated sliding contact response, which is presently unknown, can be explored. Second, with the recent advances in quantitative probes for imposing and monitoring frictional sliding events at the nano-scale, it is now feasible to develop a more fundamental and quantitative understanding of the resistance of nanocrystalline materials to damage induced by repeated sliding contact.

2. Materials and experimental details

In order to develop broad trends pertaining to the sliding contact fatigue response of metals and alloys as a function of grain size and composition, a variety of metallic materials were chosen for investigation. The materials investigated in this study include electrodeposited, fully dense nc and ufc pure Ni (with comparisons made to a fully dense pure mc Ni), equal channel angular pressed (ECAP) pure Cu, ECAP pure Ti, and an electrodeposited nc Ni–W alloy. Full details of the structure and properties of the pure Ni [16,17], ECAP Cu [18], and Ni–W alloy [19] examined below are presented elsewhere. The average grain sizes of the nc, ufc, and mc pure Ni were 30 nm, 300 nm, and 10 μm, respectively. In addition, two Cu samples of 99.999% purity were subjected to 5 ECAP cycles, with one subsequently annealed at 473 K for 1 h. Measurements via dark-field TEM revealed average grain sizes of 340 ± 20 and 400 ± 20 nm for the as-received and annealed ECAP Cu, respectively [18]. The 0.2% offset yield strength values were found to be 340 and 110 MPa in the as-received and annealed conditions, respectively. Commercially pure Grade 2 Ti billets subjected to zero (mc Ti) or eight (ufc Ti) ECAP cycles had average grain sizes of 23 μm and 300 nm, respectively, while that of the electrodeposited nc Ni–W alloy examined was ∼10 nm [19]. In-plane specimen dimensions for all contact fatigue experiments in pure Ni, Ni–W, and Ti were 15 mm × 15 mm, while ECAP Cu specimens measured ∼4 mm in diameter. The thickness of each pure Ni sample measured between 100 and 150 μm, Ni–W specimens were ∼50 μm in thickness, and all Ti and Cu specimens were of considerably greater thickness (1 μm and ∼3 mm, respectively).

An instrumented microindenter was used to conduct all sliding contact fatigue experiments. Force transducers mounted on either side of the indenter tip monitored tangential loads generated during scratch experiments, and a spherical sapphire tip (1/16-inch diameter) was used in each test. Tips were replaced after every experiment to avoid material transfer between samples.

Initial contact was made between polished specimens (thoroughly cleaned in an ultrasonic ethanol bath) and the indenter tip, after which a prescribed normal load was applied as the sample stage was displaced at a constant rate of 25 μm/s. The normal load was ramped to its maximum value over the first 50 μm of stage motion, and remained constant thereafter. A 500 μm scratch at the maximum normal load was introduced, followed by a 15 μm tip retraction from the surface. The sample stage was subsequently replaced to its initial position, and the scratch was repeated in this unidirectional fashion, up to 98 times. Normal and tangential loads were acquired throughout the entire length of the scratch. Focused ion beam (FIB) microscopy was performed following scratch tests in the nc, ufc, and mc Ni to characterize changes in the microstructure.

3. Results

Fig. 1a illustrates the early stage evolution of friction at a normal indentation load of 1.25 N, as a function of the number of unidirectional scratch passes in the pure and alloyed electrodeposited Ni. A 'steady-state' friction coefficient was reached after approximately 40–60 passes in all four materials. Fig. 1 clearly illustrates a reduction of steady-state friction with grain refinement. Comparison in nc Ni and solid-solution strengthened Ni–W indicates

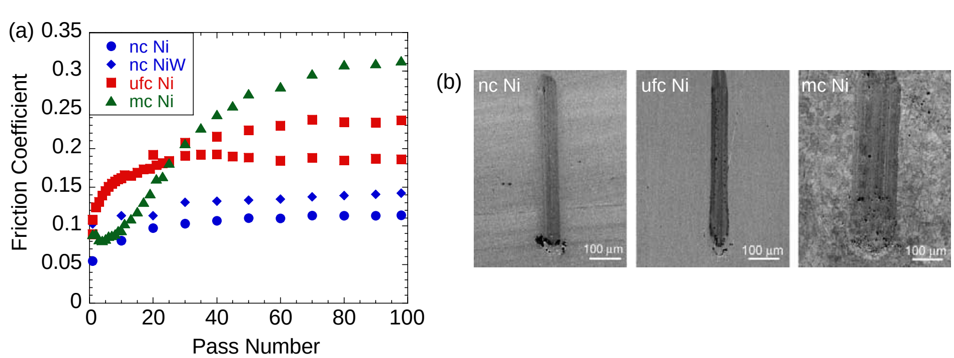

Fig. 1. (a) Early stage friction evolution as a result of repeated unidirectional scratching of nc, ufc, and mc pure Ni, as well as solid solution strengthened nc Ni–W, at a normal load of 1.25 N. Only minor variations in the friction coefficient were observed between pure and tungsten alloyed nc Ni. (b) SEM images of the scratch paths produced in nc, ufc, and mc pure Ni at a normal load of 1.25 N after 98 passes. Debris accumulation within the boundaries of the wear region exhibits a positive correlation to grain size.

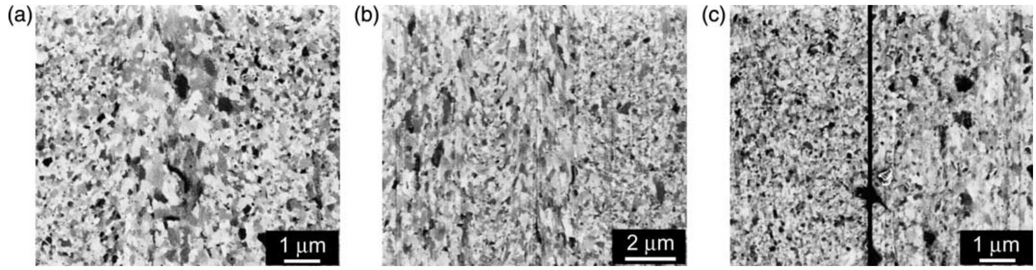

Fig. 2. FIB images of scratches produced in pure Ni at a normal load of 1.25 N. (a) nc Ni, 10 passes, (b) nc Ni, 98 passes, (c) ufc Ni, 50 passes. The interface between damaged (right) and undamaged (left) material is shown in (c). Images (a) and (b) are fully within the confines of the scratch. Local increases in grain size were observed with an increasing number of sliding contact fatigue cycles.

a relative insensitivity of the friction coefficient to the level of alloying.

Fig. 1b displays the microscopic damage evolution following 98 repeated scratches in nc, ufc, and mc Ni, at a normal load of 1.25 N. In each case, the accumulated debris within the nc Ni wear path was considerably less than that in the ufc or mc Ni, after an equivalent number of passes. The width of the scratch path diminishes with decreasing grain size, as a result of increasing material hardness.

Fig. 2 clearly illustrates regions of coarsened grains within the wear paths of nc and ufc Ni, after a limited number of contact fatigue cycles. A similar change in grain size was difficult to detect in mc Ni, as the entire width of the scratch path spanned only a few grains.

A separate set of experiments, designed to isolate the effects of material strength from those due solely to grain refinement, was performed on the ECAP Cu system described above. Samples of as-received and annealed ECAP Cu were subjected to a similar scratch testing protocol to that carried out in the electrodeposited Ni. A normal load of 1.0 N was imposed through a 1/16-inch diameter spherical sapphire tip, with varying numbers of unidirectional passes (500 μm in length). Fig. 3 illustrates the evolution of friction as a function of the number of contact fatigue cycles.

In an attempt to gauge the level of hardness (and to a first approximation, the local strength) inside and outside the damaged regions, both Cu samples were subsequently subjected to instrumented nanoindentation with a normal load of 7×10^{-3} N. Fig. 4 illustrates the variation in hardness between the as-received and annealed samples both far from and within the confines of the scratch paths. While the hardness of the annealed ECAP Cu is noticeably below that of the as-received material outside of the damaged region, there appears to be no significant variation between the two materials within the scratch.

Additional contact fatigue experiments were conducted on the mc and ufc ECAP pure Ti system described above. Scratches 500 μm in length were introduced in both specimens with a normal load of 1.25 N. Fig. 5a illustrates the early stage friction evolution as a function of pass number for the ufc and mc pure Ti. The mc material exhibits slightly higher in friction coefficient, with respect to the ufc Ti. The difference between the two is minimal, however, relative to that observed between mc and nc electrodeposited Ni. A significantly higher amount of wear debris was found within and around the scratches introduced in the mc and ufc Ti, relative to all other materials examined (Fig. 5b and c).

4. Discussion

For tribological applications, a higher scratch resistance and reduced friction coefficient are typically desired [20]. It is apparent from the results obtained in pure Ni that a reduction in grain size can result in a diminishing coefficient of friction. It was necessary, however, to determine whether this effect was grain size or strength dominated, as strength typically increases with grain refinement.

The results obtained in the ECAP Cu system, where material strength was varied at a relatively constant grain size, indicate that strength reduction has a deleterious effect on both the scratch resistance and friction coefficient. The steady-state friction coefficients of the annealed and as-received material are comparable, apparently due to the equivalence in hardness (and to a first approximation local

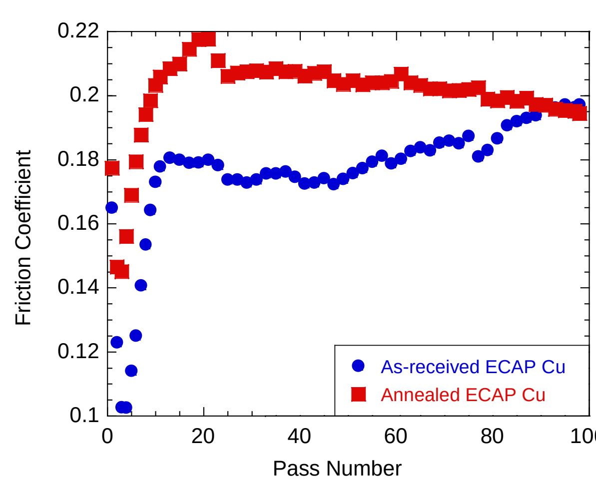

Fig. 3. Friction evolution during unidirectional sliding contact fatigue of ECAP pure Cu in its as-received and annealed (473 K for 1 h) conditions. Normal load=1 N.

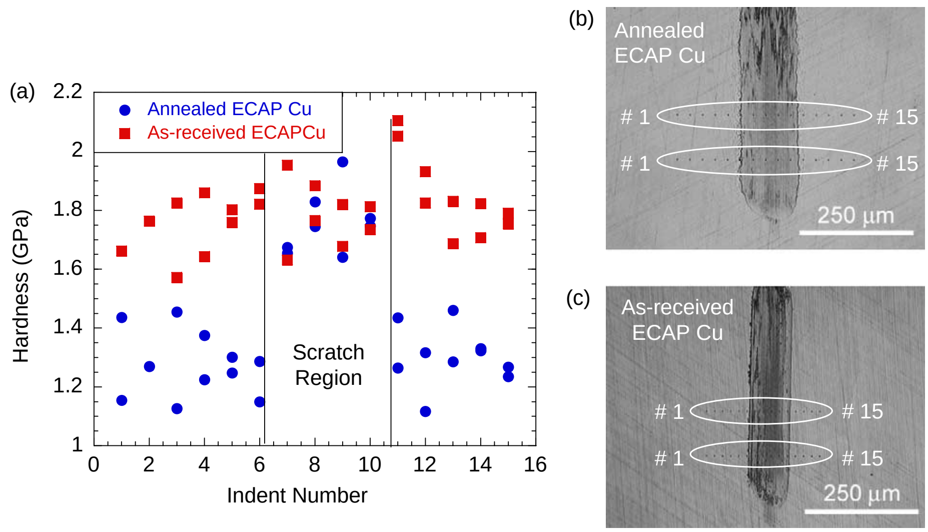

Fig. 4. (a) Hardness variation as a function of position across the damaged and undamaged regions of (b) annealed and (c) as-received ECAP Cu. Each row of indents contains 15 measurements, labeled '1'–'15' for reference in (a).

strength) within the confines of the damaged regions. The large plastic strains associated with sliding contact are known to produce a positive hardness gradient in Cu, where a higher local hardness exists at the surface, within the damaged region [21]. This observation was confirmed in the current experiments, and its effect on friction evolution indicates that strength, rather than grain size, is the determining factor with regard to the friction response.

In addition, the yield strength variation between pure mc and ufc ECAP Ti was substantially smaller (by a factor of 3.7) than that between the pure mc and nc Ni investigated. The steady-state friction coefficients observed in the former (Fig. 5) were only moderately different, even with a grain size variation of nearly two orders of magnitude. This relative lack of disparity supports the above contention that strength, and not grain size per se, dominates the friction response. The level of wear debris produced during abrasion events is also assumed to be strength dominated, considering its apparent insensitivity to the large variation in grain size between mc and ufc Ti. The amount of debris observed in the nc Ni, however, was substantially less than that in the ufc or mc Ni, under equivalent loading conditions. Again, the strength variation between mc and nc Ni is much larger than that between mc and ufc Ti. It is therefore reasonable to assume that damage accumulation, in terms of material removal and wear debris, is also dominated by material strength, rather than grain size.

A separate study has shown that tungsten alloying additions can significantly improve the hardness and scratch resistance of nc Ni [19]. In the present study, an

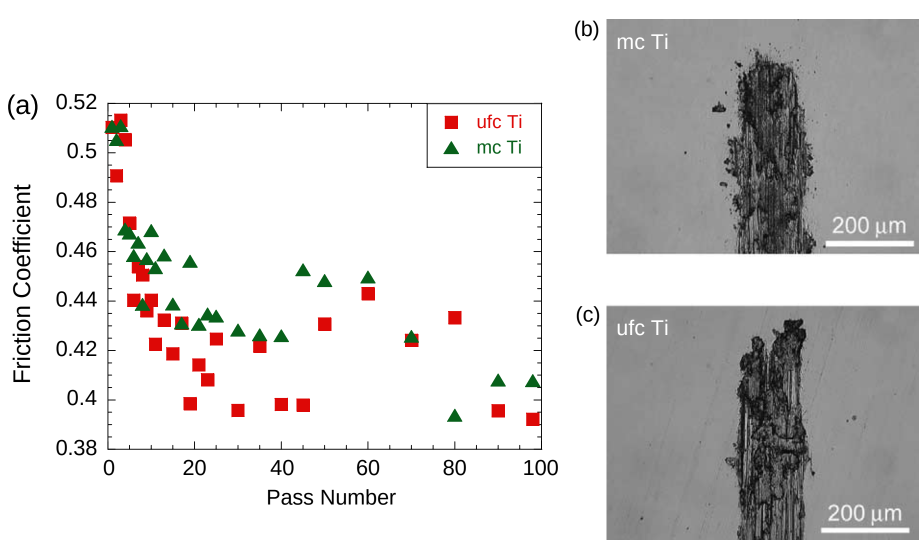

Fig. 5. (a) Early stage friction evolution as a result of repeated unidirectional scratching of ufc ECAP and mc pure Ti with a 1.59 mm (1/16-inch) diameter sapphire spherical indenter at a normal load of 1.25 N. Corresponding damage accumulation after 98 passes is shown for (b) mc pure Ti and (c) ufc ECAP pure Ti.

electrodeposited nc Ni–W alloy (13 at.% W) displayed a 14% increase in hardness, relative to the strongest pure nc Ni. Scratch tests performed at a normal load of 1.25 N on the same nc Ni–W alloy indicate a relative insensitivity of the friction coefficient to alloying content (Fig. 1). The nc Ni–W and pure nc Ni display a similar friction response over the full range of imposed contact fatigue cycles. The potential therefore exists to utilize alloying as an additional (or alternate) means of improving the contact damage resistance, without adversely affecting the friction response. Further studies are necessary to confirm the generality of such trends.

Finally, it is important to recognize that the grain size within the scratch region of the nc and ufc Ni is unstable, as evidenced by the images in Fig. 2. These results are in agreement with similar findings in the same material subjected to cyclic indentation, where grain growth was observed within and around the indented region [21]. This observed instability is of considerable practical interest for tribological applications. An unstable grain structure may result in variable surface properties, as the deformed material progresses from the nc to the ufc or even mc regime. Considerable attention must therefore be devoted to stabilizing the grain size under contact loading conditions.

5. Conclusions

The early stage sliding contact fatigue response of several nc, ufc, and mc material systems has been investigated. The effects of grain size, material strength, and alloying additions on friction and damage accumulation were characterized. Based on the above set of critical experiments, it was concluded that friction and damage evolution were dominated by material strength rather than grain size per se, over the range of materials investigated. In addition, alloying effects appeared minimal in terms of their influence on the friction coefficient for the nc Ni–W alloy studied, presumably because the hardening was insubstantial. Given that both friction and damage evolution were dominated by material strength, it appears that traditional strengthening mechanisms, rather than grain refinement in the nc regime, could provide a more viable (economical) means of improving tribological resistance.

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