REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
19-05-2010	Final	2/15/08 - 11/30/2009

4. TITLE AND SUBTITLE
FRICTION IN FULL VIEW

5a. CONTRACT NUMBER
FA9550-08-1-0016

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
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5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Northwestern University
Evanston, IL 60201

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
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10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
AFRL-SR-AR-TR-10-0264

12. DISTRIBUTION/AVAILABILITY STATEMENT
Distribution A- Approved for Public Release

13. SUPPLEMENTARY NOTES

14. ABSTRACT

We have used an in situ transmission electron microscopy (TEM) technique to perform tribological investigations on various thin films. Using a Nanofactory HS100 STM-TEM sample holder and Tecnai F20ST TEM (200 kV), we were able to slide sharp probe tips on samples to study the single-asperity behavior. During sliding we were able to simultaneously use the various instrumentation of the TEM, including bright and dark field imaging, electron diffraction, and chemical analysis in the form of EDX and EELS at high resolution.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE		1	Prof. L. D. Marks
					19b. TELEPHONE NUMBER *(Include area code)* 847-491-3996

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We have used an *in situ* transmission electron microscopy (TEM) technique to perform tribological investigations on various thin films. Using a Nanofactory HS100 STM-TEM sample holder and Tecnai F20ST TEM (200 kV), we were able to slide sharp probe tips on samples to study the single-asperity behavior. During sliding we were able to simultaneously use the various instrumentation of the TEM, including bright and dark field imaging, electron diffraction, and chemical analysis in the form of EDX and EELS at high resolution.

The experimental setup [1] allows for three-dimensional movement of an electrochemically etched tungsten tip along the surface of the film, mounted on a standard TEM grid at a 45º angle. After sliding on highly ordered pyrolytic graphite (HOPG) samples prepared by repeated exfoliation, TEM imaging of the probe tip showed adhesion of several graphite layers, directly confirming the hypothesis that graphite-on-graphite sliding is more favorable than tungsten-on-graphite sliding [1]. Electron diffraction also showed that sliding on conformal HOPG layers caused some of those layers to rotate slightly out of alignment. Likely this is because while six-fold symmetry is still preferred, there is a higher energy cost associated with sliding one layer of carbon atoms directly over another.

We performed chemical bonding analysis on the sliding behavior of very low friction "N3FC" and "NFC6" diamond-like carbon films prepared using plasma-enhanced chemical vapor deposition and magnetron sputtering at Argonne National Laboratory. By repeatedly sliding on these films while taking electron energy loss spectra (EELS), we were able to observe and quantify the transformation of sp^3 tetragonal carbon bonds to sp^2 trigonal bonds as a direct consequence of sliding cycles [2]. This information is important in engineering applications where ultra low friction is required, and the out-of-plane bonding character of sp^2 carbon can result in increased adhesion between the sliding surface and counterface.

Experimentally, we presented the direct evidence of tribological recrystallization and grain growth in a polycrystalline gold thin film [3]. Thin films of gold and gold(60%)-palladium(40%) at thicknesses from 3 to 27nm were sputtered at room temperature in a vacuum of 10^{-5} Torr onto lacey carbon films supported on 200 mesh TEM grids (Ted Pella, Inc., USA). The samples were analyzed using a Tecnai F20 G2 under conditions of bright-field (BF) and dark-field (DF). Mechanically induced rapid recrystallization and grain growth at ambient temperature was confirmed under dynamical DF imaging condition. The driving force for mechanically stimulated recrystallization and grain growth originates from the stored energy in the films.

Theoretically, we have formulated an analytical model for friction in terms of dislocation drag forces during metal-on-metal sliding contact. Being purely analytical, the model has predictive power which was found to correspond well with experiment [4]. We also considered the problem of metal-on-metal plowing modeled by dislocation creep. Similarly, the models use contact mechanics and geometry to generate predictions of the friction force without the need for experimentally measured empirical terms. Both of these models correlate well with experimentally-observed trends.

We generalized a model for friction at a sliding interface involving the motion of misfit dislocations to include the effect of thermally activated transitions across barriers in crystalline materials. In this model, we obtained a comparatively simple form with the absolute zero-temperature Peierls barrier replaced by an effective Peierls barrier which varies exponentially with temperature, in agreement with recent experimental observations of thermally activated friction. Going further, we suggest a plausible method for generalizing the frictional drag at a more constitutive level by replacing the Peierls stress in a more general sense where the microstructure (e.g., dislocation density, grain size, asperity shape etc.) is built in. Last, but not least, we point out that when barriers are included the static coefficient of friction becomes larger than the dynamic coefficient of friction, which is an important connection to reality [5].

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