Mater. Res. Soc. Symp. Proc. Vol. 1021 © 2007 Materials Research Society 1021-HH05-09

Nanoripples Formation in Single Crystals

Megan E. Pendergast, Ramakrishna Gunda, and Alex A. Volinsky
Mechanical Engineering, University of South Florida, 4202 E Fowler Ave ENB118, Tampa, FL, 33620

ABSTRACT

Normally a wear test results in a trench formed of the removed material. A phenomenon of nanoripples formation has been observed in KBr and InSb single crystals undergoing wear experiments in an ultra-high vacuum atomic force microscope. While similar experiments using AFM cantilevers in ambient environment did not exhibit ripples, scanning KBr single crystal in air with a diamond tip in the Hysitron Triboindenter formed surface ripples 100 nm high, 1 micron apart. It has been observed that the initial nanoripples height and period increases with the number of repeated scans. For a leveled KBr sample, nanoripples formed uniformly throughout the scan width, although they originated at the bottom of the sloped sample and progressed up the slope. A tip velocity threshold of about 10 microns/second was determined under which no rippling was observed.

Several mechanisms may be responsible for the ripples formation: including dislocation dynamics, chatter, piezo hysteresis, and others. Most likely there is a combination of effects, with a clear differentiation between nanoripples origination and propagation.

INTRODUCTION

The field of nano-mechanics has seen a drastic increase in interest in nanoscale patterns formation over the past few decades. With implementation of tools like the Ultra High Vacuum Atomic Force Microscope (UHV-AFM), and Depth Sensing Nano-indentation, mechanical and tribological properties can be investigated at the micro and nano scales. This has led to research of nanoscale patterns. The Hysitron nanoindentor can perform nanoindentation, but has an added surface scanning feature. This surface scanning allows manipulation of a material topographical characteristic.

In a typical wear experiment a hard material is scanned over the tested material surface, resulting in a wear rate measurement in terms of the removed material depth as a function of normal applied load and number of wear cycles. When applying 2-10 μN forces while continuously scanning over the same area, we observed periodic ridged pattern, oriented perpendicular to the tip motion direction. Example of this pattern can be seen on the macro scale in nature in instances like wind blown sand dunes in deserts, on the ocean floor, and even on the Mars surface.

The nanoscale rippling is not restricted to just wear-induced patterns; recent literature shows the same formations in processes such as ion sputtering [1]. Ion milling produces a strikingly similar ripple pattern during Ga ion beam erosion of the silicon surface. The ions were bombarded at normal angles to the silicon and a combination of Focused Ion Beam and Scanning Electron Microscopy was used to measure the erosion of silicon and thus ripples propagation [2].

The appearance of these nano-ripples during mechanical wear tests performed on single crystals is described in this paper. These ripples formed at the bottom of a wear trench after repeated scanning. Original experiments performed in the UHV AFM of 10^{-10} Torr succeeded at producing ripples on InSb (100) single crystal with repeated scanning of the surface in AFM using a silicon nitride tip. This scanning caused surface reconstruction of wavy patterns forming perpendicular to the tip movement, coinciding with the $\langle 1\bar{1}0 \rangle$ InSb crystallographic direction [3]. Scanning for 100 passes produced ripples 30 nm high and 130 nm wide.

Analogous UHV-AFM experiments performed on KBr and Al single crystals by another group were instrumental in the inspiration for ambient environment experiments [5]. Those UHV-AFM experiments had the cantilever tip of the AFM move repeatedly over a single scan line. This resulted in periodic pile-up structures forming around that scan line. Additionally, nanoripples were formed in square areas perpendicular to the scanning direction. In both cases normal load was in the range of 10-30 nN. The distance between ripple peaks was found to correlate with the tip radius (larger tip, larger spacing). In the presence of topographic features such as cleavage steps, the early ripples were distorted by these features but as scanning continued, aligned themselves perpendicularly as the cleavage steps were worn down. Attempting to perform the same experiments using regular AFM outside the vacuum did not exhibit any ripple formation. This may be associated with the fact that UHV-prepared surfaces have higher surface energies than surfaces with adsorbed species in ambient environment.

We made an attempt to produce the wear ripples in single crystals in the ambient conditions using the Hysitron nanoindenter with a Berkovich diamond tip. However, the mechanism by which these patterns form and how the ripple pattern formation is influenced by the experimental conditions is poorly understood. Several mechanisms are identified as possible causes of the ripple formation and need to be investigated.

EXPERIMENTAL DETAILS

The rippling effect observed in the UHV AFM was replicated in the ambient environment using the Hysitron Triboindenter. The tool is capable of repeated surface scanning producing AFM-like images. It can scan up to 80x80 μm, making 256 passes of an area with a rigidly supported diamond tip. Initial experiments on a freshly cleaved KBr sample using a Berkovich diamond tip and a normal load of 2 mN produced ripples in the ambient environment after 20 scans of the 10x10 μm area. The ripples formed perpendicular to the tip motion direction, as observed in the UHV-AFM ripples as well. An additional similarity to the UHV-AFM ripples on KBr is the wearing down of cleavage steps as the experiment progresses. Distinct cleavage steps were significantly worn down in both experiments. The mechanical properties of KBr were measured and are presented in Figure 1. Partial unloading technique was compared with quasistatic indentation used to measure KBr reduced modulus and hardness. A movie constructed of single image scans of KBr single crystal rippling due to scanning at 1 Hz can be seen online [6]. The scan size is 4 μm.

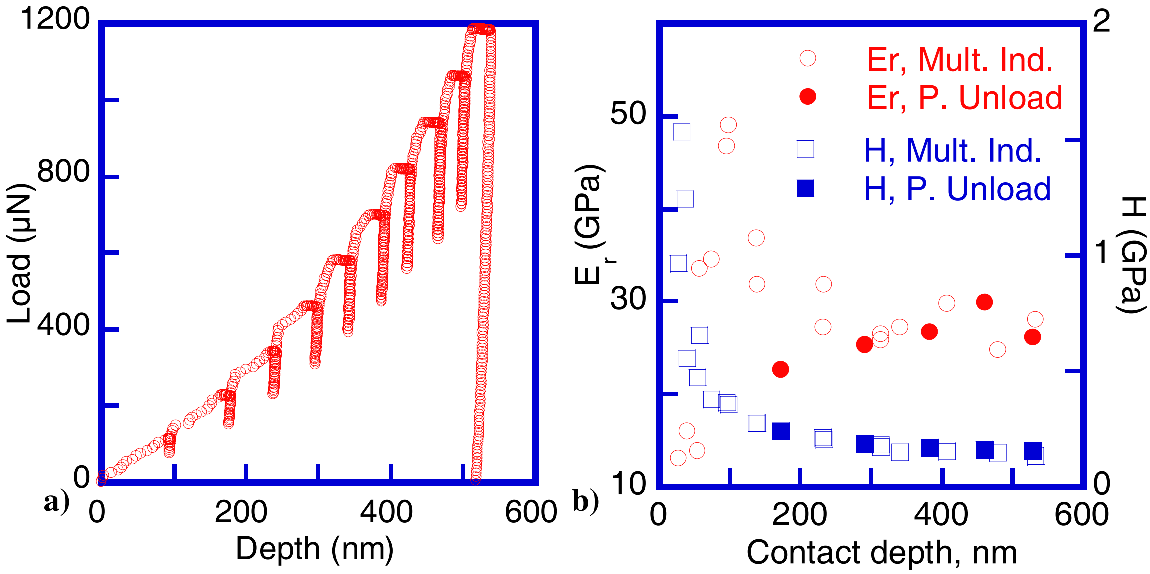

Figure 1. a) Partial unload data for KBr; b) KBr reduced modulus and hardness.

Additional wear experiments preformed on other materials using Hysitron nanoindentor also produced ripples. Single crystal aluminum, seen in Figure 2 produced rippling, forming perpendicular to the tip direction as well. Figure 2 is a 30 μm image with the 10 μm square test area shown before scanning in Figure 2a, and after scanning for 1000 cycles in Figure 2b. The periodicity of the ripples increases with the number of scans.

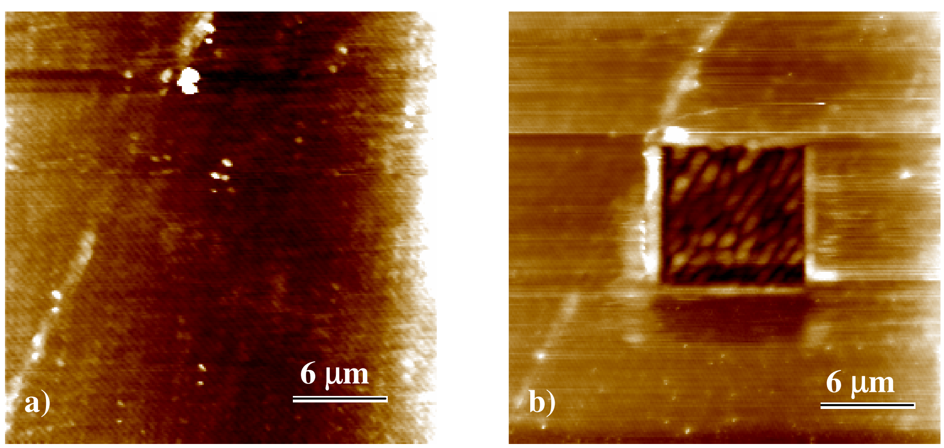

Figure 2. a) Topography of Al single crystal before the wear test (20 nm Z scale) and b) nanoripples formed after 1000 scans (45 nm Z scale).

DISCUSSION

Dislocations in the crystal structure of the material are a possible cause of the ripple initiation. Low energy is required to induce a dislocation in single crystal (~10 eV per Burgers vector) [7]. Additionally, because the tip is sliding across the surface, there is a shear force component as well. The addition of this force could be enough to induce dislocations. Load-displacement discontinuities at 5 nm of depth with a 20 μN normal load were observed in indentation curves. With the addition of a shear component, 2 μN normal load could be enough to induce dislocations.

Nanoscale chatter is also being investigated in that the tool tip could be inducing a forced or self-excited vibration expressed in the ripples formation. All experiments were performed at low gain settings. Additionally, in macroscale processes such as milling and turning where chatter is often seen, the tool tip is not removed from the sample, creating a regeneration of waviness similar to the results seen in the Hysitron [8].

Another interesting aspect of the initial ripples formation is sample tilt. If there is a topographical slope to the surface area, the lowest point in that area is where the ripples will initially propagate from, as can be seen in Figure 3. When the tip encounters a sloped surface, the piezo can not react instantly to the topographical change and is subject to acceleration down the slope, and then a quick deceleration when encountering the edge of the specified image and is forced to travel back up the slope. This response time in the piezo could cause a "digging in" of the tool tip on the sample when it is quickly decelerated at the bottom of the slope.

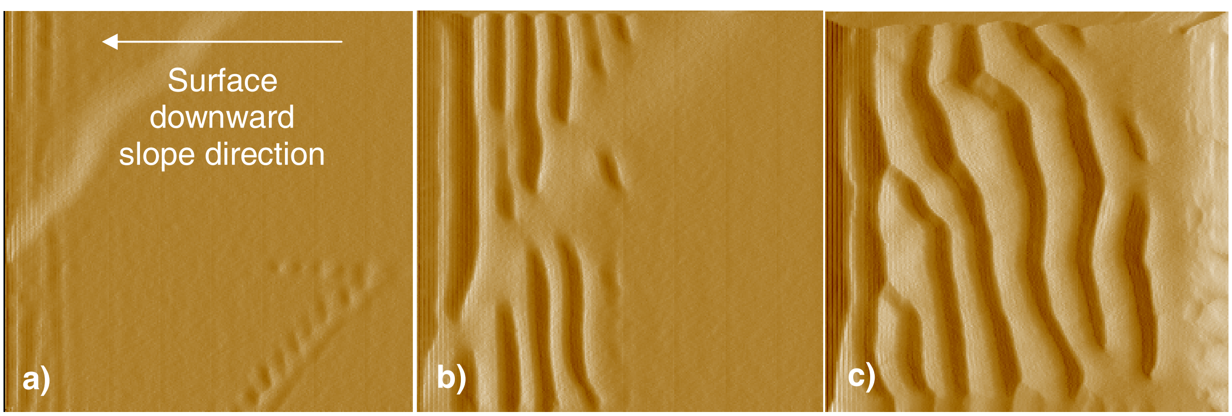

Figure 3. The effect of sample tilt on the formation of ripples. Initiation of nanowear ripples at the sample tilt a) after 5 scans, b) after 100 scans, and c) after 250 scans using Hysitron Triboindenter.

To further study the piezo effects in the Hysitron nanoindentor, scan lines from the tip tracking the sample surface were considered. This data can be seen in Figure 4. The first image (Figure 4 a) is taken at a scan rate of 0.1 Hz, and the bottom image (Figure 4 b) is taken at 3 Hz. There is a drastic difference in the amount of horizontal shift with regards to the scan rate. When the speed of the tool tip increases, the response time of the piezo can not accommodate the surface feature change and the corresponding overshoot is seen in Figure 4b. The feedback is kept at open loop during all experiments, also those in UHV AFM. The piezo is made of Barium Titanate oxide and it is "easier" for the piezo to extend than contract, which is a mechanical property of this material. This effect is displayed below in terms of the large shift when the tip is traveling up the slope. This tendency of the tool tip to overshoot and undershoot the actual surface topography is most likely responsible for the ripple propagation. Not only does is continue to "dig deeper" into the troughs and slopes, but this motion also serves to increase the periodicity of the ripples, an effect observed on all samples that produced ripples. Figure 5 shows the amount of horizontal shift taken at different points along the sample (high points, low points, relatively un-sloped points) and the dependence of the shift on the scan rate.

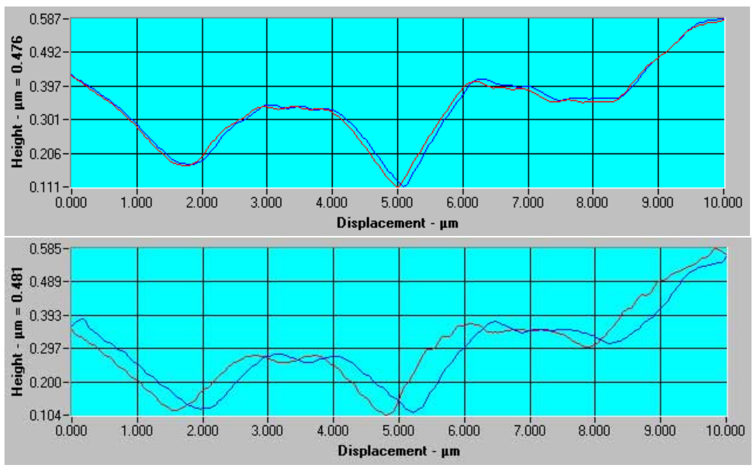

Figure 4: Scan lines taken during wear experiments on KBr showing hysterisis effect at a scan rate of a) 0.1 Hz and b) 3Hz.

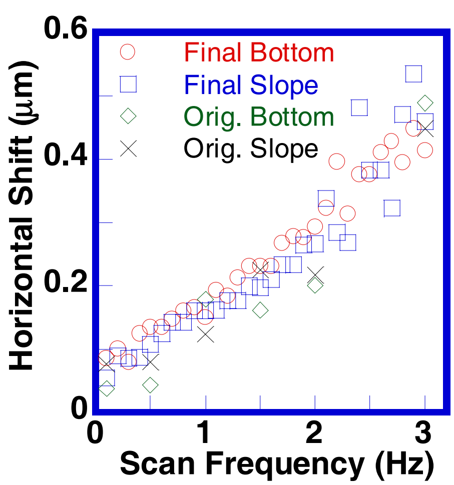

Figure 5: Horizontal shift in different surface features as a function of the scan rate before and after multiple wear cycles.

This tip motion and its contribution to the ripples formation is very applicable to larger scale ripple patterns, such as sand dunes and ocean floor ripples. In sand dunes the wind gains kinetic energy flowing down a trough and picks up sand from the bottom of the trough. Once the wind continues up out of the trough it loses some of the kinetic energy and is forced to release some of the sand, making the ripple pattern increase in periodicity as seen at the nanoscale in our experiments [9]. The tip is just a different medium, as water is in ocean floor ripples, and wind in the sand dunes.

CONCLUSIONS

The nanoripples in single crystalline materials that were previously limited to form in UHV AFM were successfully replicated in the ambient environment using the Hysitron Triboindenter. The exact cause of this ripple formation and propagation is under investigation. With additional testing on single crystal and polycrystalline materials, the aim of discovering the ripple formation and its subsequent dependence on scanning parameters is to be expounded. It is abundantly clear that there is a difference in ripple pattern initiation and its further propagation as seen in the influence of scanning on the ripple periodicity. Future work includes detailed experiments and modelling of the nanowear ripple patterns formation.

ACKNOWLEGDEMENTS

This work is supported by the National Science Foundation (CMMI-0631526), the division of Design, Manufacture and Industrial Innovation, and the Marie Curie Host Fellowship for the Transfer of Knowledge under the project "Nano-Engineering for Expertise and Development – NEED" within the 6th EU Framework Program. The authors would also like to thank Prof. Marek Szymonsli's group for collaboration in this project, and Oden Warren and Lance Kuhn from Hysitron Inc. for valuable discussions.

REFERENCES

1. P. F. A. Alkemade, Phys. Rev. Lett. (2006) 96, 107602
2. S. Habenicht and K. P. Lieb, Phys. Rev. Lett. B (2002) 65, 115327
3. M. Szymonski, F. Krok, B. Such, P. Piatkowski, J.J. Kolodziej, "Ripple Creation on InSb (001) Surface Ions vs. AFM Tip", 3rd EFS Nanotribology Conference, Lisbon, Portigal, 18-22 September, 2004
4. M. Pendergast, A.A. Volinsky, submitted to Appl. Phys. A 2007
5. A. Socoliuc, E. Gnecco, R. Bennewitz, and E. Meyer, Phys. Rev. B (2003) 68, 115416
6. World Wide Web: http://www.eng.usf.edu/~volinsky/KBr.gif
7. N.I. Tymiak, D.E. Kramer, D.F. Bahr, T.J. Wyrobek, and W.W. Gerberich, Acta Mater., Vol. 49, pp. 1021-1034, 2001
8. P. Zelinski, "Chatter Control for the Rest of Us", Modern Machine Shop Magazine, October 2005
9. U. Valbusa, C. Boragno, and F.B. de Mongeot, J. Phys.: Condens. Matter 14, (2002) 8153