Experimental Measurements of Surface Residual Stress Caused by Nano-scale Contact of Rough Surfaces

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ABSTRACT

A previous dislocation model analysis predicts that nano-scale contacts of surface steps induce nucleation of dislocations leading to pro-load and anti-load dislocation segregation near the contact surface. Such dislocation segregation generates a sub-layer of tensile residual stress in a much thicker layer of compressive residual stress near the surface. The sub-layer thickness is expected to be about 50 to 100 times the step height. In order to verify the predictions of the model analysis, experiments are carried out on polycrystalline aluminum surface to determine the existence of the tensile sub-layer. The variation of the residual stress along the thickness direction is measured using a newly developed high sensitivity curvature-measurement interferometer. The interferometer measures the curvature change of the back surface of a plate specimen of about 1.9 mm thickness while the contact-loaded front surface is chemically etched. The residual stress distribution measured with sub-nanometer spatial resolution is compared with analytical predictions.

INTRODUCTION

When two rough surfaces are in contact, significant wear occurs due to contact pressing and sliding between the two surfaces. The rate of wear is a function of many parameters including normal force, sliding velocity, temperature, thermal, mechanical and chemical properties of the contact material. And it involves many different mechanisms. Empirical diagrams of wear-mechanisms reveal that at low sliding region, which is the typical working condition for gears, brakes and clutches during the operation of an automobile, plasticity-dominated delamination wear plays the most important role [1]. Thus the understanding of the mechanisms of delamination wear has been highly valued by the wear community.

Among the many causes of delamination wear, surface roughness plays an important role [2-5]. When two rough surfaces are brought together, local yielding and the subsequent localized damage and wear occur at surface asperities far before the system reaches its bulk yielding condition. The relation between surface roughness and wear rate has been studied by many researchers. Most of the previous studies, however, relied on the empirical knowledge constructed from experimental data, with little understanding of the source and mechanisms.

Recently, an effort toward the understanding of the real mechanisms governing plastic deformation of contact surfaces was initiated in the current researchers' group. A dislocation based unit process model consisting of a surface with one step in contact with a flat rigid surface was used to study surface roughness evolution under contact loading [6]. Rice-Thomson's dislocation nucleation criterion [7] was used to model the dislocation nucleation from the surface step. Under contact load, a dislocation nucleates and grows out from the surface step when the nucleation criterion is satisfied. The criterion is based on the driving force on the dislocation which is calculated using conservation integrals. Results of the unit process model show that under a contact load, the surface step induces stress concentration which promotes dislocation nucleation along easy-glide slip planes. Dislocations nucleated on a slip plane inclined away from the step edge are driven to stay only in a thin layer near the surface by the far-field contact loading, while dislocations nucleated along other slip planes easily move away from the surface into the bulk material. The former dislocation is named anti-load dislocation. The segregation of anti-load dislocations near the surface produces net Burgers vector of the dislocations, which generates a sub-layer of tensile residual stress near the contact surface. In contrast, the latter dislocation is called pro-load dislocation which can be easily driven away from the step edge into the bulk by the far-field stress of the contact loading. The anti-load and pro-load dislocations and their segregation are schematically depicted in Figure 1. The segregation of the pro-load dislocations produces a much thicker layer of compressive residual stress away from the contact surface into the bulk. Since cracks form much more easily under a tensile residual stress, this model indicates that the dislocation nucleation around surface steps may be one of the mechanisms causing delamination wear.

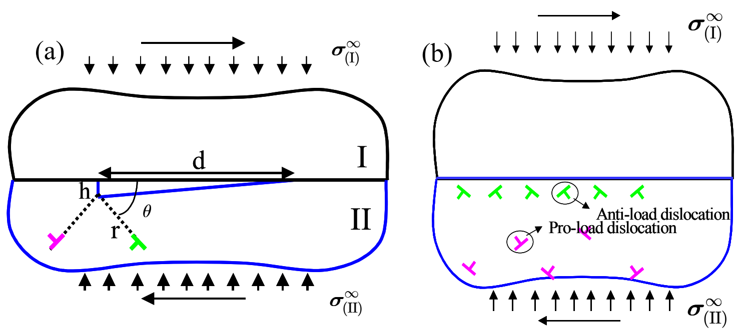


Figure 1 Dislocation nucleation and segregation from surface steps (a) Unit process model [6] (b) Segregation of anti- and pro-load dislocation

In order to verify the predictions of the unit process model and to achieve a good understanding of the mechanisms causing delamination wear, contact compression experiments on rough surfaces were carried out in this paper to study the residual stress development due to nano scale contact. Polycrystalline aluminum surfaces with a nano scale roughness were contact loaded using a smooth stainless steel indenter. The residual stress distribution along the thickness direction was measured using a recently developed curvature interferometer. Chemical etching approach was used to release the residual stress and introduce the time dependent curvature change. Results are used to compare with the predictions from the unit process model.

APPLICATION OF CONTACT LOADING

The circular polycrystalline aluminum samples used in this experiment have a 30 mm diameter and 1.9 mm thickness. The samples were cut from an original 8” computer hard disk of the same thickness with mirror like finish on both sides. AFM scan reveals that the roughness of the sample surface is in the range of several nano meters. The stainless steel indenter has a diameter of 50 mm, big enough to cover the whole sample surface, and was mechanically polished with Al_2O_3 slurry down to 0.3 μm. Before starting the contact experiments, simple tension experiments were performed to measure the mechanical properties of the material. The tensile strength and Young’s modulus were found to be 120 MPa and 70 GPa, respectively. In order to study the effect of loading levels on the residual stress development, different loading levels including 0, 20, 40, 50, 60 and 80 MPa were chosen to be applied on different samples. An Instron 4505 Tensile/Compressive testing machine is used to do the initial tensile testing and to apply the contact loading through the indenter onto the top surface of the sample, while the bottom surface of the sample was supported by a PMMA block. Therefore, hard contact only occurs at the top surface where localized plastic deformation was introduced under the surface asperities, while the bottom surface remained elastic by transferring the load into the compliant PMMA block.

RESIDUAL STRESS MEASUREMENT USING A CURVATURE INTERFEROMETER

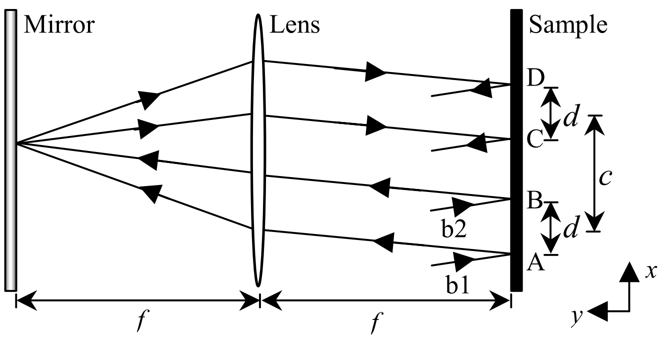


Figure 2. Schematic of the curvature interferometry

In order to measure the residual stress caused by the contact loading, a new curvature interferometer was developed by utilizing the interference between two laser beams repeatedly reflected from the sample surface. The setup is schematically shown in Figure 2, which consists of a convex lens, a reflecting mirror, and a reflective sample surface arranged such that the sample surface and the mirror are located on the two focal planes of the lens.

With this setup, the sample surface becomes the image plane of itself for the lens, i.e., points D and C are images of points A and B. A light beam b1 incident at point A always reaches point D, while another light beam b2 incident at point B always arrives at point C regardless of their incident angles. After repeated reflections from the sample surface, the two beams b1 and b2 accumulate a path length difference, Δ, proportional to the curvature, κ, of the sample, such that $\kappa = 1/R = \Delta/(2cd)$. The optical path length difference, Δ, corresponds to a phase difference of $2\pi\Delta/\lambda$ between the two beams, where λ is the wavelength of the laser. The phase difference can be obtained by measuring the interference between the two beams reflected from positions C and D. As a result, the curvature of the sample surface can be determined from the phase difference. When sample curvature changes with time, the real time curvature variation, $\kappa(t)$, can be monitored by measuring the variation of the path length difference, $\Delta(t)$. With a slight modification, this setup is also capable of measuring the absolute curvature.

The physical arrangement of the curvature interferometer for residual stress measurement is shown in Figure 3. A cube beam splitter, BS1, was used to produce two parallel beams from a single incident He-Ne ($\lambda = 628\, nm$) laser beam. The two parallel beams were then directed to the back surface of the sample (unloaded side) by the second beam splitter, BS2. After reflecting back from the sample surface, the two beams were focused onto a reflective mirror, M2 by the focal lens. The reflected beams from the mirror were

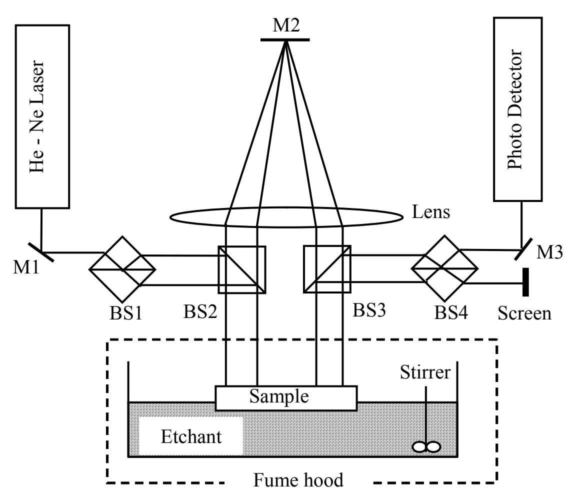


Figure 3. Experimental setup.

then sent back through the lens to the sample surface. After the second reflection from the sample surface, the two beams were steered by beam splitter, BS3, to a mixing beam splitter, BS4, to generate two sets of interference signals (180° out-of-phase). One set of the interferometric signal was detected by a photodetector and digitized using an Agilient Multi channel Data Logger 34970A connected with computer. The other set of signal was directed towards either a screen or a second photodetector as a reference.

In order to measure the residual stress at different distances from the contact surface, the loaded side of the sample was chemically etched using an aluminum etchant consisting of 16 parts phosphoric acid, 2 parts deionized water, 1 part acetic acid and 1 part nitric acid. The etching rate of this etchant was calibrated to be 110 nm/minute. The gradual removal of the material released the residual stress and caused the curvature change of the sample. To guarantee uniform removal of the materials and avoid localized reactions, a magnetic stirrer was used to stir the etchant during the measurement. To avoid exposure to the toxic acids and contamination of the lab environment, a compact bench top fume hood was used to contain the chemical etching setup and remove the toxic vapors and gases. The clear nature of the hood material introduced no interruptions into the optical measurements and allowed real time observation of the etching process.

Once the interferometric signal was obtained, simple plate bending theory was used to relate the time dependent curvature variations to the thickness dependent residual stress distribution in the sample. The Stoney [8] equation took a very simple form for this experiment

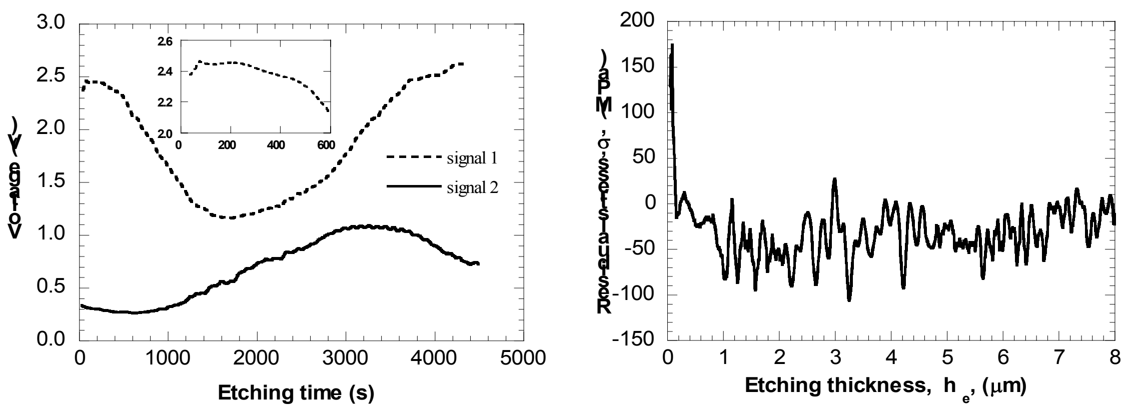
$$\sigma(h_e) = \frac{Eh^2}{6(1-\nu)}\frac{d\kappa}{dh_e} = \frac{Eh^2}{12cd(1-\nu)}\frac{d\Delta}{dh_e},$$

where σ is the thickness-dependent in-plane residual stress, h_e is the etching thickness which can be obtained from the etching rate and etching time, E and ν are the Young's modulus and Poisson's ratio, respectively, h is the sample thickness, c, d, κ, and Δ are the same as defined earlier. For all the measurements in this paper, $h = 1.9$ mm, $c = 10.5$ mm, $d = 5.5$ mm, $E = 70$ GPa, and $\nu = 0.3$.

EXPERIMENTAL RESULTS

The main objective of the current experiment is to verify the existence of the sub-surface tensile layer as predicted by previous dislocation model [6]. If such a tensile layer indeed exists, one expects to see a reversal in the interferometric signal and hence the measured curvature value. In order to unambiguously identify such reversals, a quarter-wave plate was inserted into one of the two interfering laser beams before they reach the mixing beam splitter, and two linear polarizers aligned with 90° difference in their orientations were placed in front of the photodetectors. Such arrangement introduces a 90° phase shift between the two signals. The signals were acquired by two identical photodetectors. The etching results of a sample loaded to 60 MPa for an etching time of 70 minutes are shown in Figure 4.

As expected, the two signals show an exact 90° phase shift indicated by the fact that when signal 2 reached its peak value around 2000 sec, signal 2 was in its fastest changing region. Comparing the magnitudes of the two signals at critical phase angles, such as 0, $\pi/4$, $\pi/2$ and π, indicates that the first peak of signal 1 corresponds to a reversal of phase-difference variation. Since the most reliable part of a sinusoidal curve is the fast changing part instead of the plateau part. The sensitive parts of the two signals were selectively combined to give the final residual stress vs. etching thickness result as shown in Figure 4b. For an etching period of 70 minutes, a total thickness of 8 μm was removed from the loaded side of the sample. The residual stress showed a tensile value of 175 MPa near the contact surface, while a much lower compressive stress around 50 MPa was observed within a much thicker layer up to 6 μm. The tensile sub-layer extended to a thickness around 146 nm.



(a) (b)

Figure 4. Results of a sample loaded to 60 MPa: (a) interferometric signal, the inset shows the expanded view of the initial part of signal 1; (b) surface residual stress vs. etching thickness.

For the total 10 samples tested at 6 loading levels, the results are summarized in Table 1. The results of 0 load level shows that compressive residual stress exists even in unloaded samples which may be developed during the manufacturing process. Although the magnitudes of the residual stress and the thickness of the tensile layer have a large fluctuation and error bar (typically 10-15%) due to experimental and numerical noise (numerical differentiation), the existence of the reversals in the interferometric signals for samples loaded above 40 MPa is definite. The tensile layer thickness for the current samples spreads in a range of 83-222 nm, which is close to the values predicted by the analysis of the unit process dislocation model [6].

Table 1. Observations of reversals in the etching data and the associated residual stress and thickness.

Loading level	Whether or not exists reversal	Thickness at reversal (nm)	stress level (MPa)
0	No.		-60
0	No		-93
20	No		-100
40	No.		-163
40	Yes	128	132
50	Yes	169	55
60	Yes	146	175
60	Yes	83	154
80	Yes	222	254
80	Yes	117	60

DISCUSSION AND CONCLUSIONS

Motivated by the predictions of a recent dislocation model analysis, contact loading experiments were performed to determine the existence of a sub-surface tensile layer caused by rough surface contact. A new curvature interferometer was developed to measure both time-dependent and absolute curvature of the samples. Using simple plate theory, the residual stress in the sample was calculated from the measured curvature change generated by relaxing the near-surface stresses with chemical etching. In the previous work, a unit process model was used to study the dislocation activity around surface steps under a contact compression loading. The model predicts the existence of a sub-surface tensile layer whose thickness is in the range of 50-100 times the step height. The current experiment used samples with random roughness in the range of a few nano meters (root mean square roughness) which is in the same range as used in the previous analysis. A sub-surface tensile layer was observed for loadings above 40 MPa. The measured tensile layer thickness in the range of 83-222 nm confirmed the previous dislocation model predictions. The current experiment suggests that even if the nominal loading stress is below the bulk yielding stress, localized yielding can still occur under the surface asperities, which results in the formation of the sub-surface tensile layer. The results obtained from different loading levels suggest that there exists a critical load only above which a sub-surface tensile layer can be formed. This critical load can be a function of surface roughness, background residual stress and material properties such as yield strength. The existence of such sub-surface tensile layer is expected to play an important role in the wear mechanisms associated with gears, engines and clutches, as these parts of the automobile usually undergo a normal contact loading during the operation of the system. Residual tensile stress in the layer introduces cracks which then contribute to the removal of materials.

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