SAND2008-0391C

Probing Properties in Single Crystal Nickel Under Sliding Contacts - Tests and Simulations

N. R. Moody

M. J. Cordill, S. V. Prasad

C. Battaile, J. R. Michael, W. W. Gerberich

Sandia National Laboratories, Livermore, CA

Sandia National Laboratories, Albuquerque, NM

University of Minnesota, Minneapolis, MN

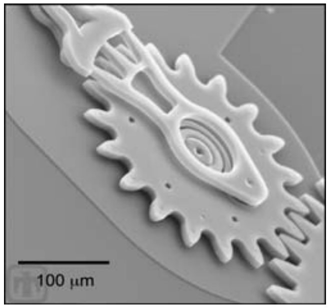
International Symposium on Indentation Behavior of Materials

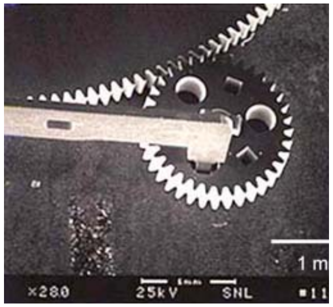
Hyderabad, India

February 4-7, 2008

We have a strong interest in microsystems where performance and reliability must be assured.

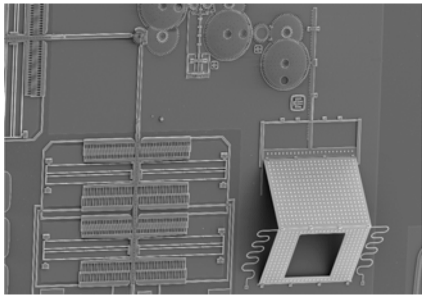
Silicon Surface Micromachine Technology





Nickel Based LIGA Technology

Gears

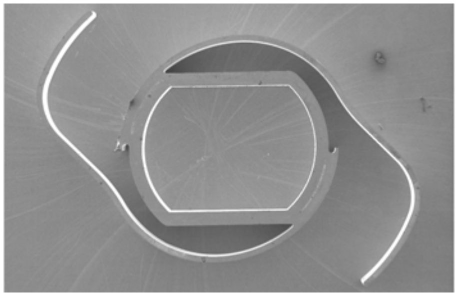


MEMS Mirrors

Common Set of Issues

- high aspect ratios
- friction
- wear
- strength

Issues arise from contact



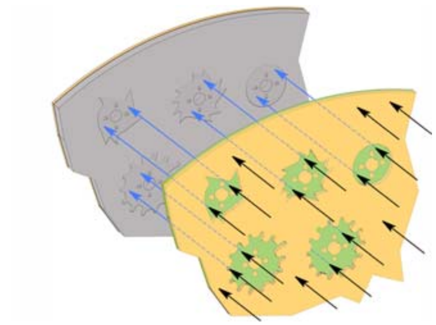
Micro Springs

Material behavior controls performance and reliability.

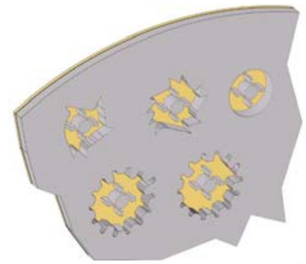
Electrodeposited nickel parts are of special interest.

LIGA: LIthographie, Galvanoformung, Abformung

- X-rays from a synchrotron are incident on a mask patterned with high Z absorbers. X-rays are used to expose a pattern in PMMA, normally supported on a metallized substrate.

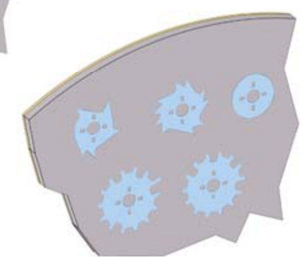


- The PMMA is chemically developed to create a high aspect ratio, parallel wall mold.

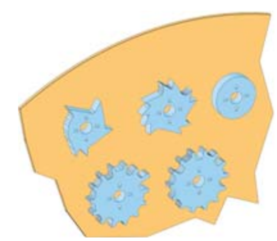


photograph of chrome mask

- A metal or alloy is electroplated in the PMMA mold to create a metal micropart.

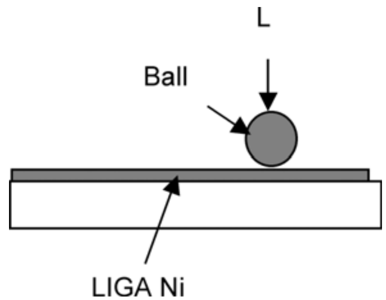


- The PMMA is dissolved leaving a three dimensional metal micropart. This micropart can be separated from the base plate if desired.

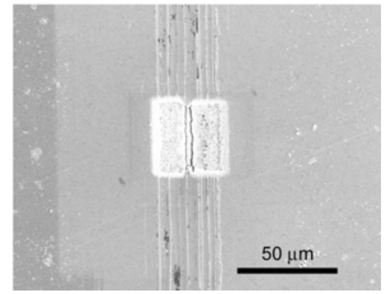


Regions of contact give rise to severe gradients in strain that can change topography and microstructure.

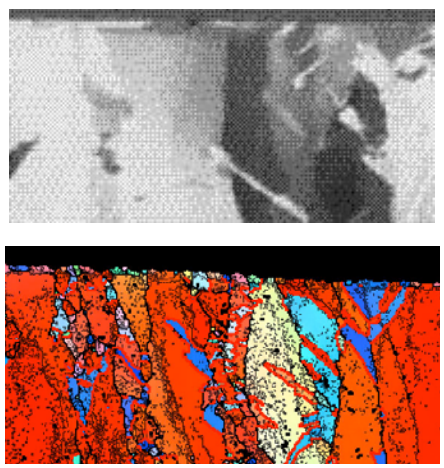
Contact Spring



Wear Scar

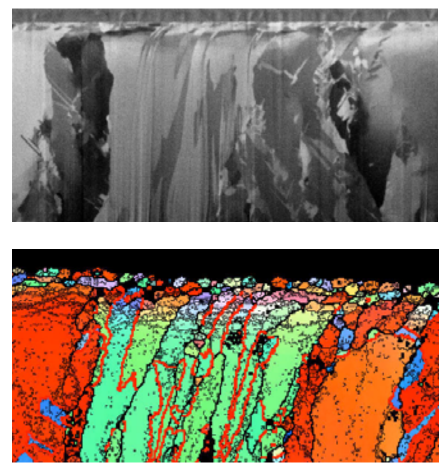


LIGA Ni



Unworn Surface

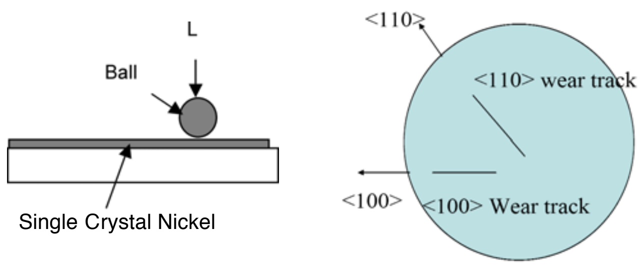
LIGA Ni

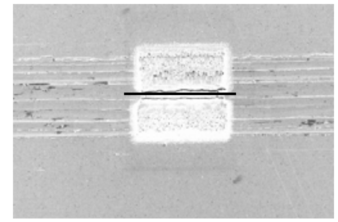


10 gm 1000 cycles

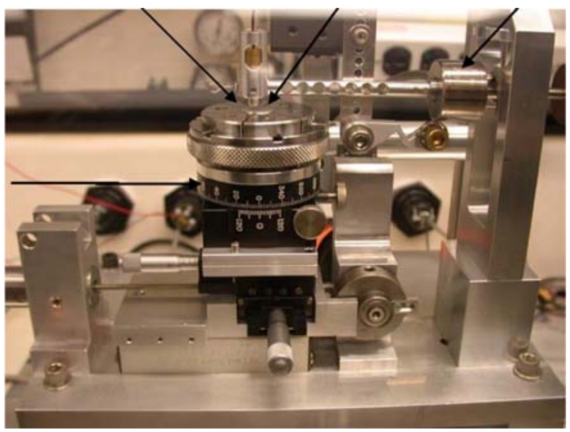
(Prasad, Michael, Christenson, 2003)

A rotary friction test module was used to create [110] and [100] wear tracks on (001) oriented single crystal nickel.

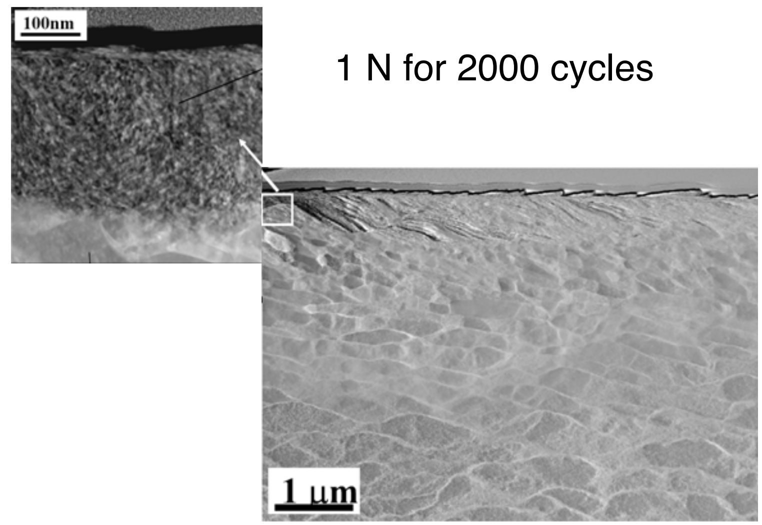




FIB sections taken along the wear tracks



Rotary stage for crystal alignment

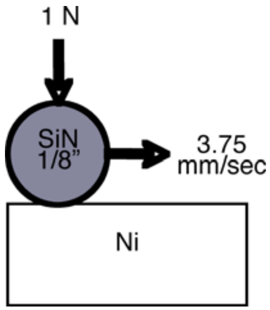


1 N for 2000 cycles

(Prasad, Michael, Battaile, Kotula, 2005)

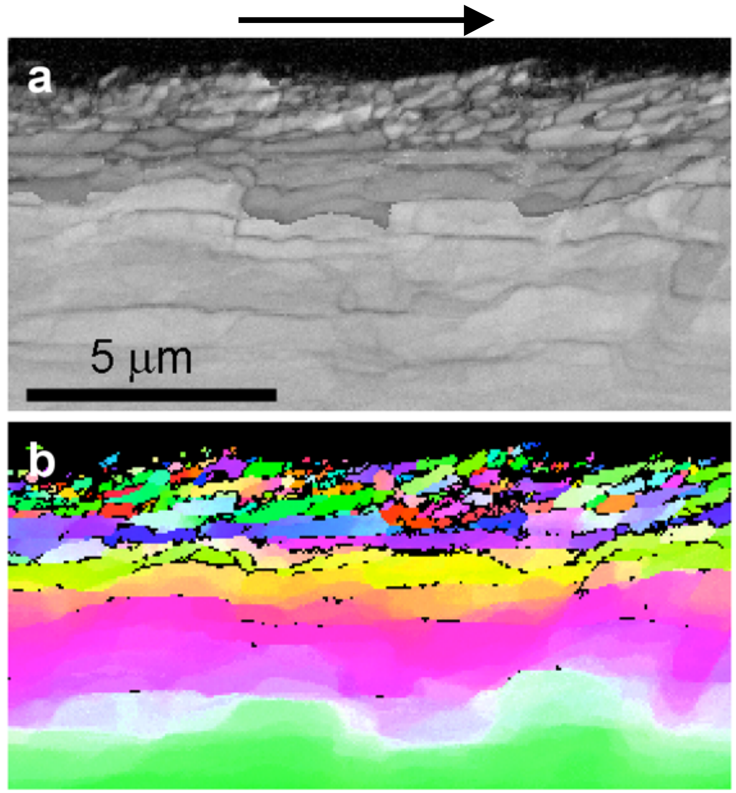
Sliding induced deformation and grain rotation also generated unique nanoscale surface structures in single crystal nickel

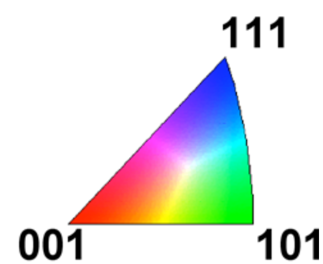
Ball on disk tests show a wear-induced change in structure



(111)[211]

band contrast map





orientation map

(S. Prasad, J. Michael, P. Kotula)

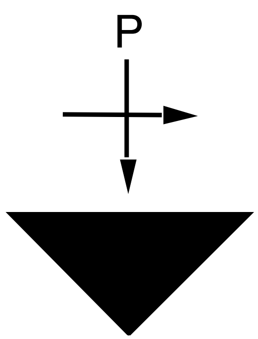
EBSD mapping reveals changes in microstructure and orientation

Purpose

Determine the effects of sliding contacts on properties of single crystal nickel at the nanoscale

Materials

Polished (001), (011), and (111) single crystal nickel samples.

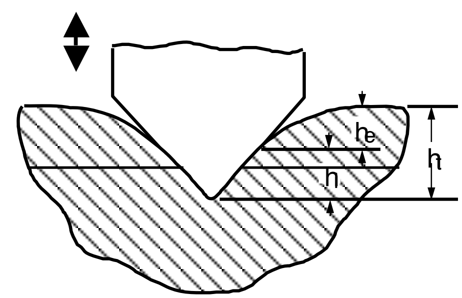


Wear Resistance

Nanoscratch tests using conical diamond tips were used to create wear patterns along selected directions in single crystal nickel samples.

Mechanical Properties

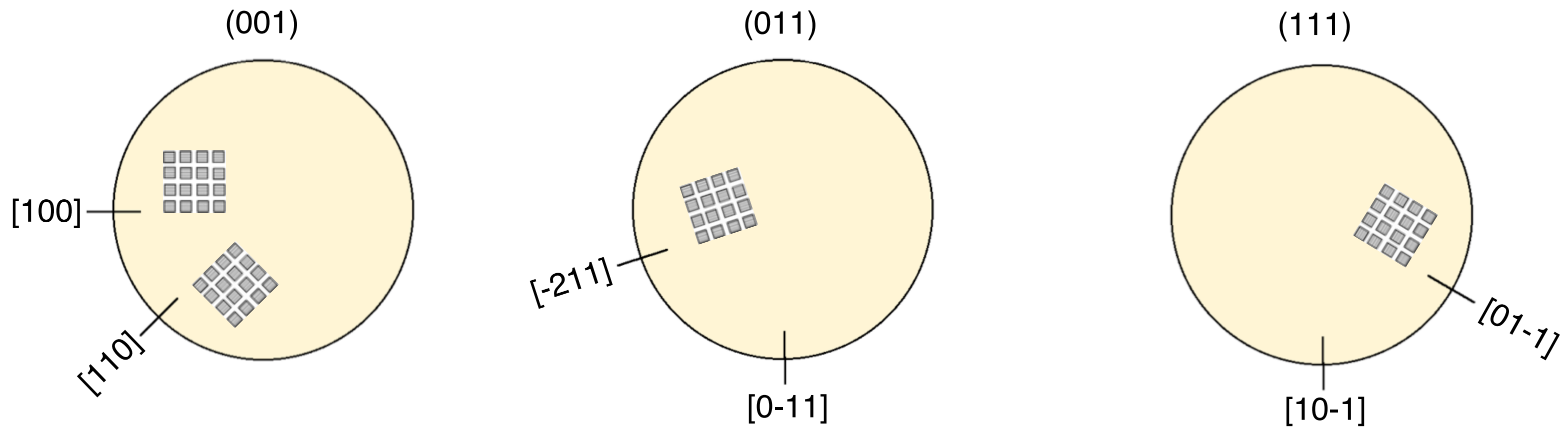
Nanoindentation was used to measure modulus and hardness of the material under the wear patterns.

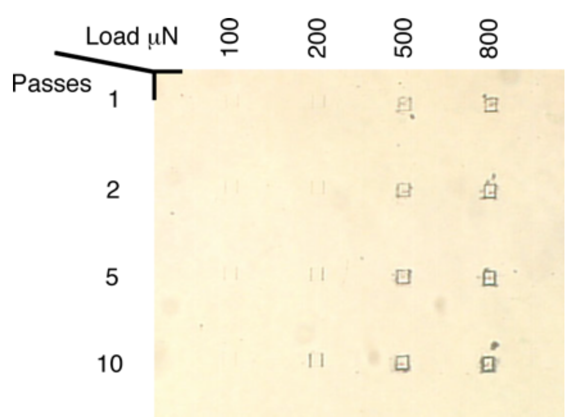


Simulations

Finite element modeling was used to provide insights on deformation processes and changes in structure

Nanoscratch tests were used to create wear patterns in polished nickel single crystals.



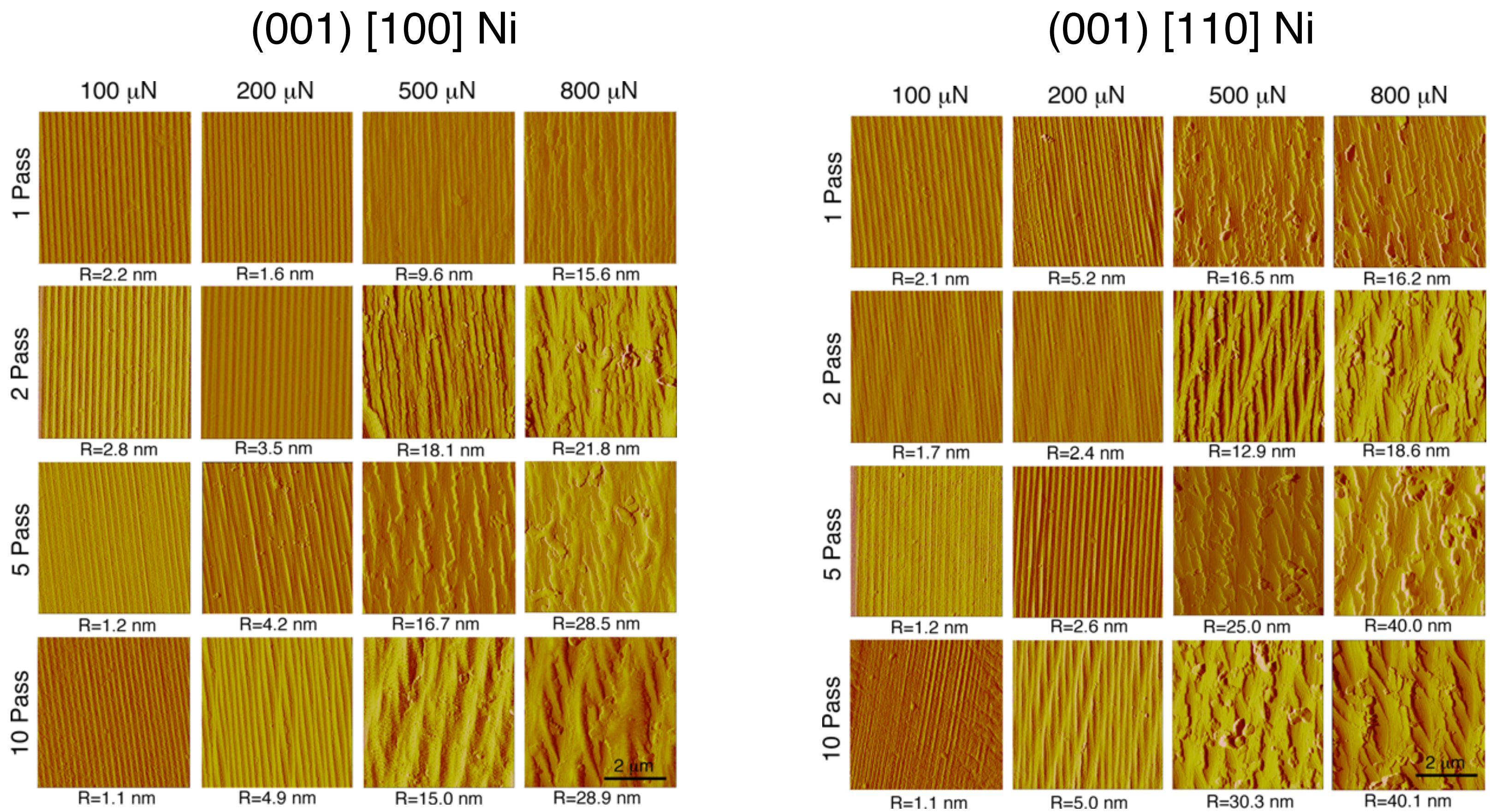


The wear patterns were generated
on a Hysitron Triboindenter
using a 1 μm conical diamond tip
along selected directions

Properties were measured using
a Nano Instruments XP
a DCM head in CSM mode
and a 50 nm radius Berkovich tip

Low load high resolution nanoindentation was used to measure material properties within each wear pattern.

Roughness and deformation increase with applied load and number of scratch passes.

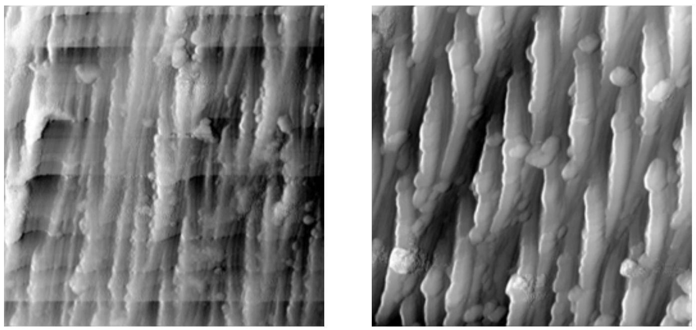


Roughness increases significantly under the highest loads tested.

Modes of deformation vary with the number of scratch passes at high loads.

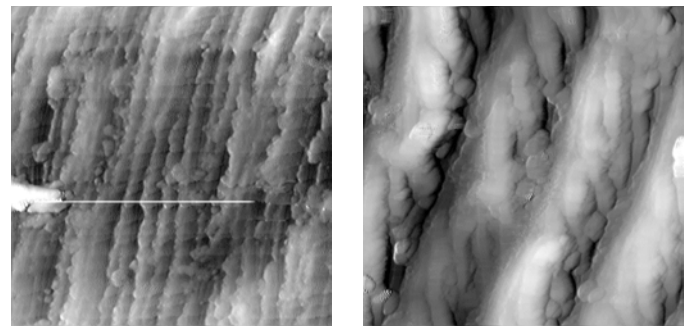
500 μN

1 Pass 5 Pass

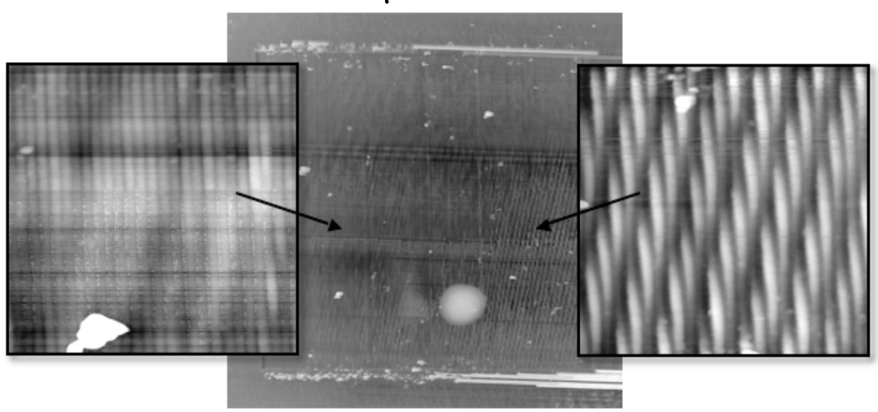


800 μN

1 Pass 10 Pass



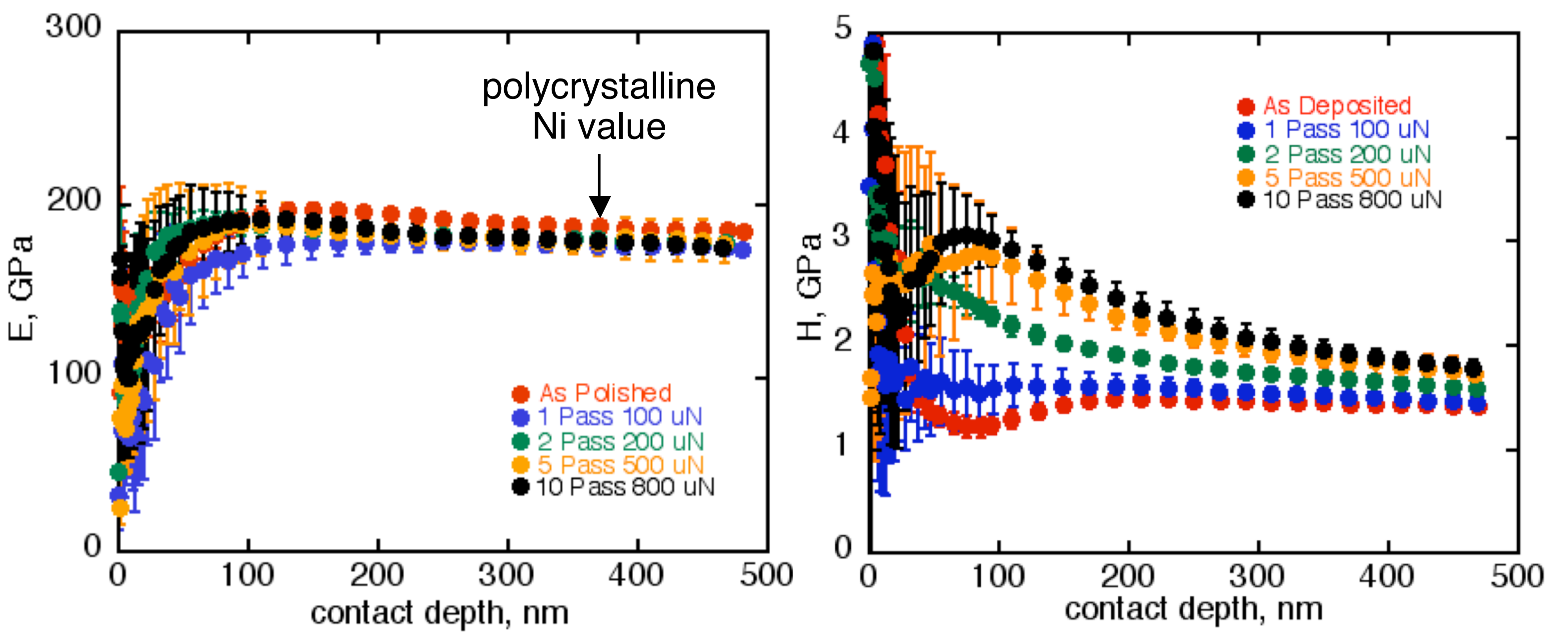
200 μN 1 Pass



Deformation modes vary within wear patterns at low applied loads.

Modulus values are similar to polycrystalline nickel. Hardness increases with load and number of passes.

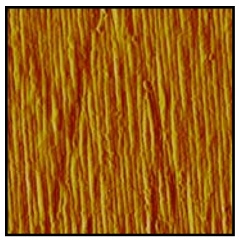
(001)[110] Ni



Roughness had a strong effect on near surface elastic modulus and hardness values.

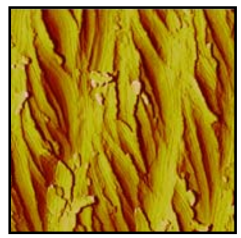
Hardness variations are a product of asperity geometry and property gradients.

R=2.8 nm

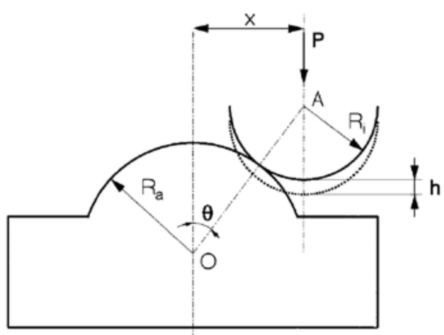


100μN/1 Pass

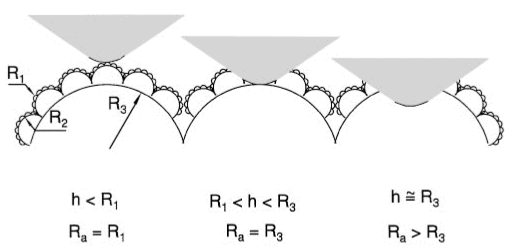
R=26.4 nm



800μN/10 Pass



Single Asperity



Multiple Asperities

Near surface variations in hardness can be attributed to:
- surface chemical effects
- indentation size effects
- property variations with depth
- surface roughness

Surface roughness results from large strains and local strain rate responses during wear pattern generation

Asperities created during wear are high stress and strain energy structures

Probing properties introduces additional factors.

(Bobji and Biswas, 1999)

Experimentally we can separate geometric and material through contact area to a first order correction.

Experimentally these terms are separated using a P/S^2 approach assuming a constant elastic modulus

$$S = \frac{2}{\sqrt{\pi}} E^* \sqrt{A} \;:\; H = \frac{P}{A}$$

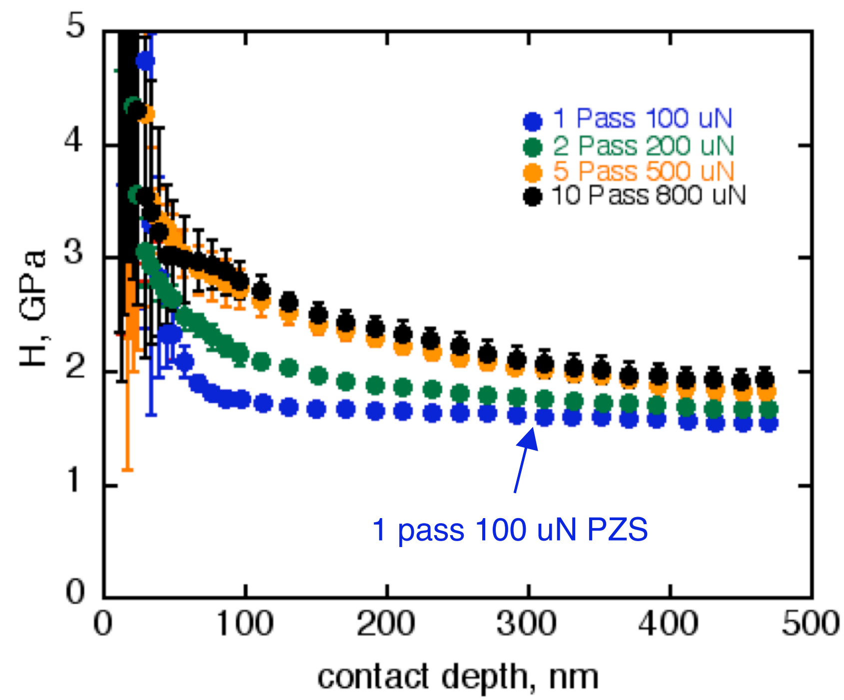
with

$$\frac{1}{E^*} = \left[\frac{\left(1 - \nu_i^2\right)}{E_i} + \frac{\left(1 - \nu_s^2\right)}{E_s} \right]$$

$$\frac{P}{S^2} = \frac{\pi P}{4AE^*} = \frac{\pi H}{4E^*}$$

(Page, Pharr, Hay, Oliver, Lucas, Herbert, Riester, 1998)

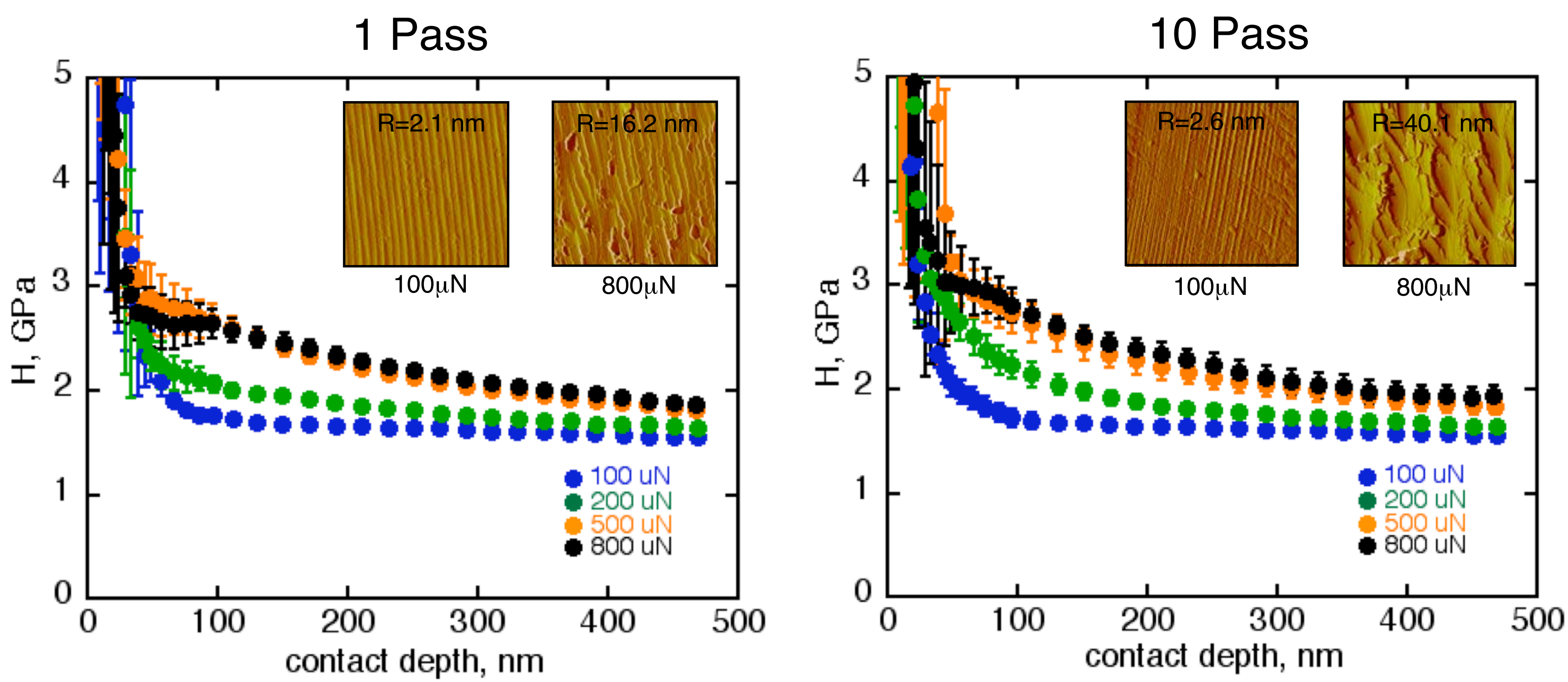
(001)[110] Ni



Corrected hardness values exhibit a well-defined increase with applied load and number of lateral scratch passes.

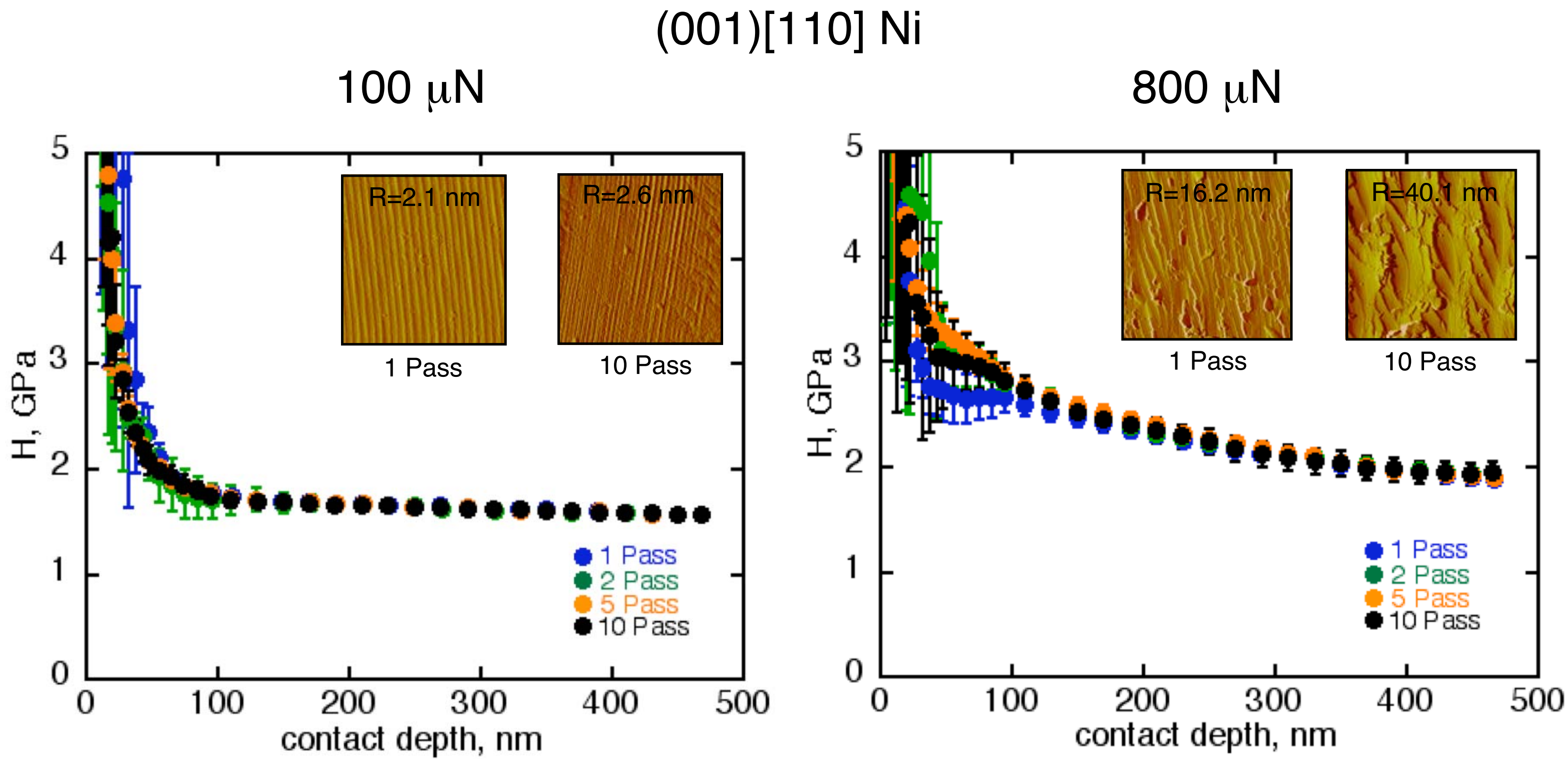
Increasing applied load leads to a significant increase in hardness and the extent of near surface deformation.

(001)[110] Ni



The effect was similar after one pass and after 10 passes.

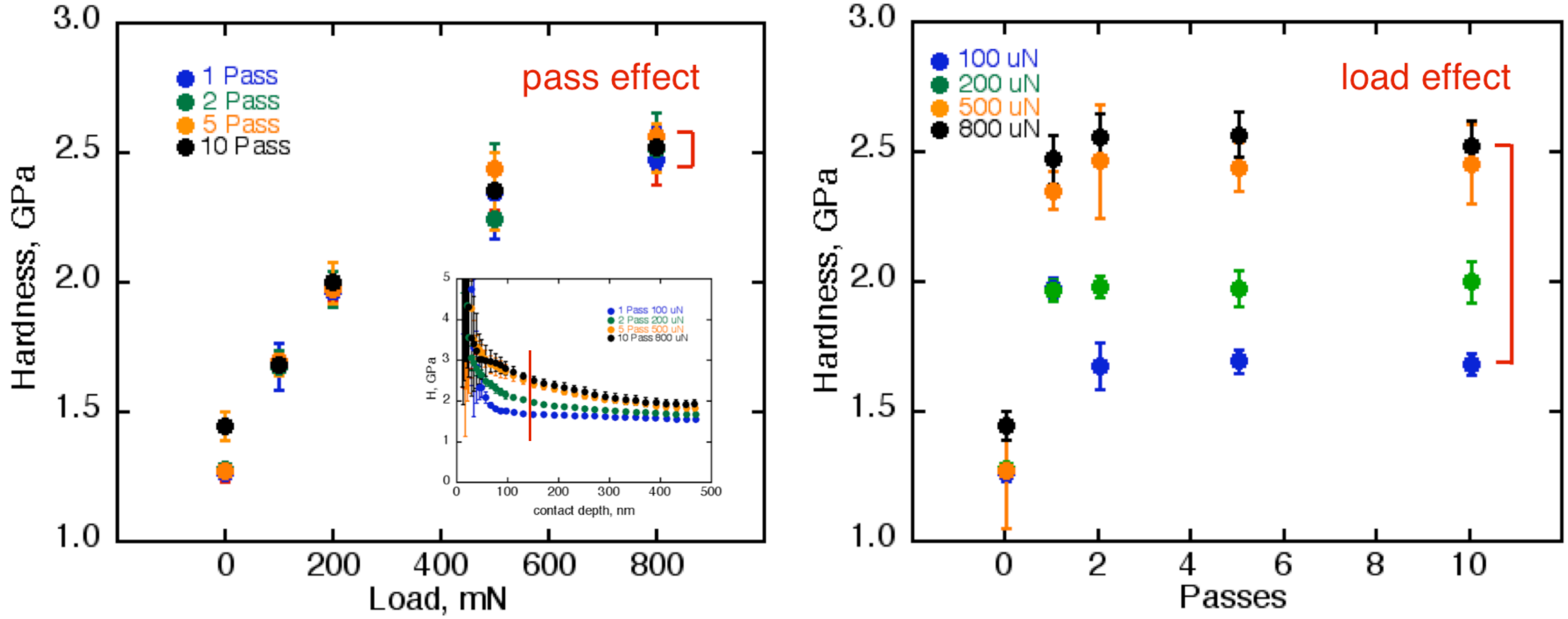
An increase in the number of lateral scratch passes has no apparent effect on hardness.



The effect of wear pattern generation on material hardness appears to be primarily through applied load.

Cross plots show that applied load leads to a significant increase in hardness within the wear patterns

(001)[110] Ni

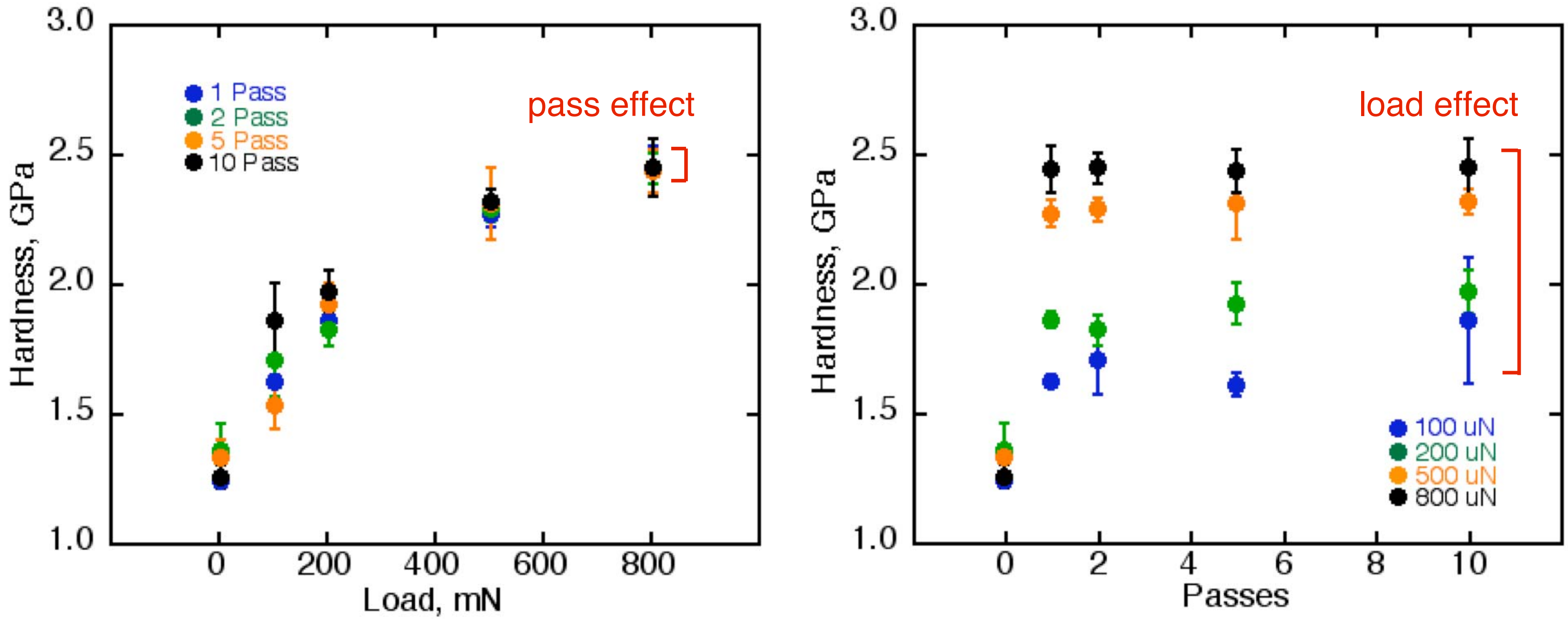


Data taken at a depth of 150 nm.

The lack of a cyclic scratch pass effect contrasts with the change in wear surface appearance.

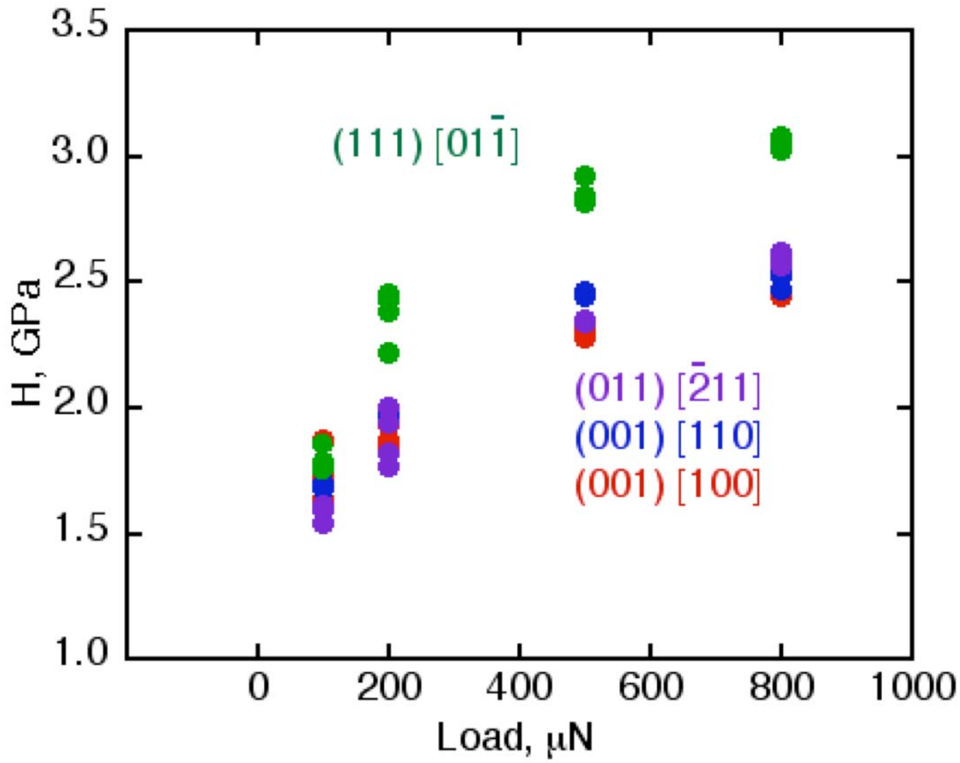
Cross plots for the [100] direction show the same effect of load and cyclic passes as the [011] direction.

(001)[100] Ni



The number of passes had no measurable effect on hardness.

Cross plots for (001)[100] and (011)[-211] show the same effect of load and cyclic passes as (001)[110] direction.

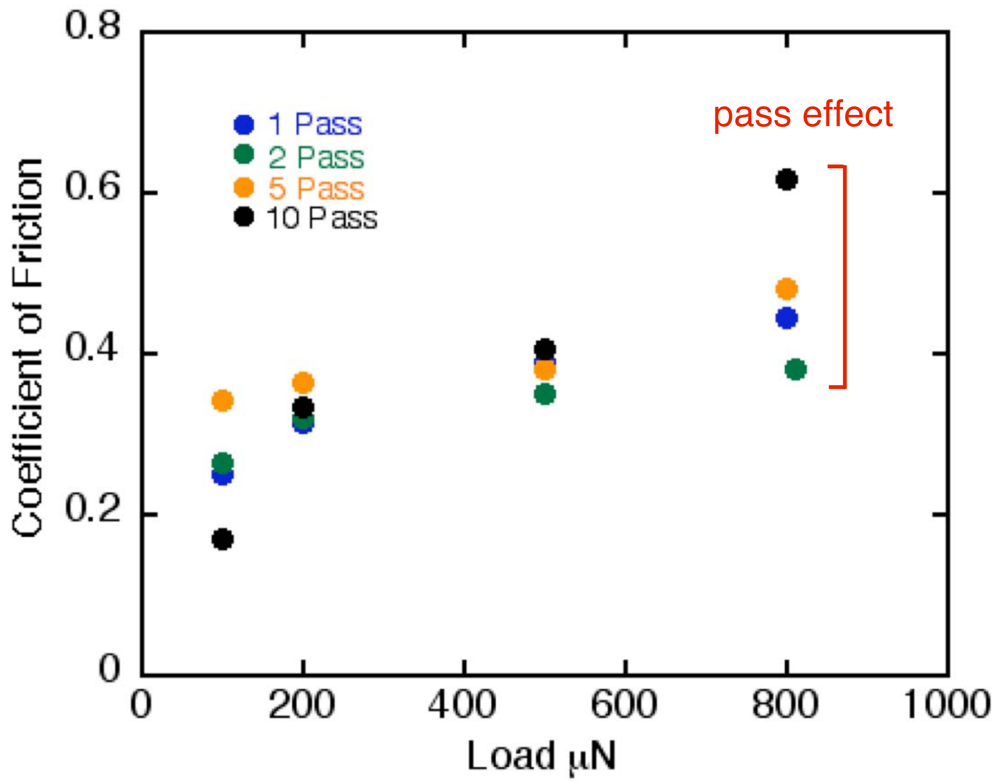


Data taken at a depth of 150 nm.

Measured hardness values were higher in (111)[01-1] patterns.

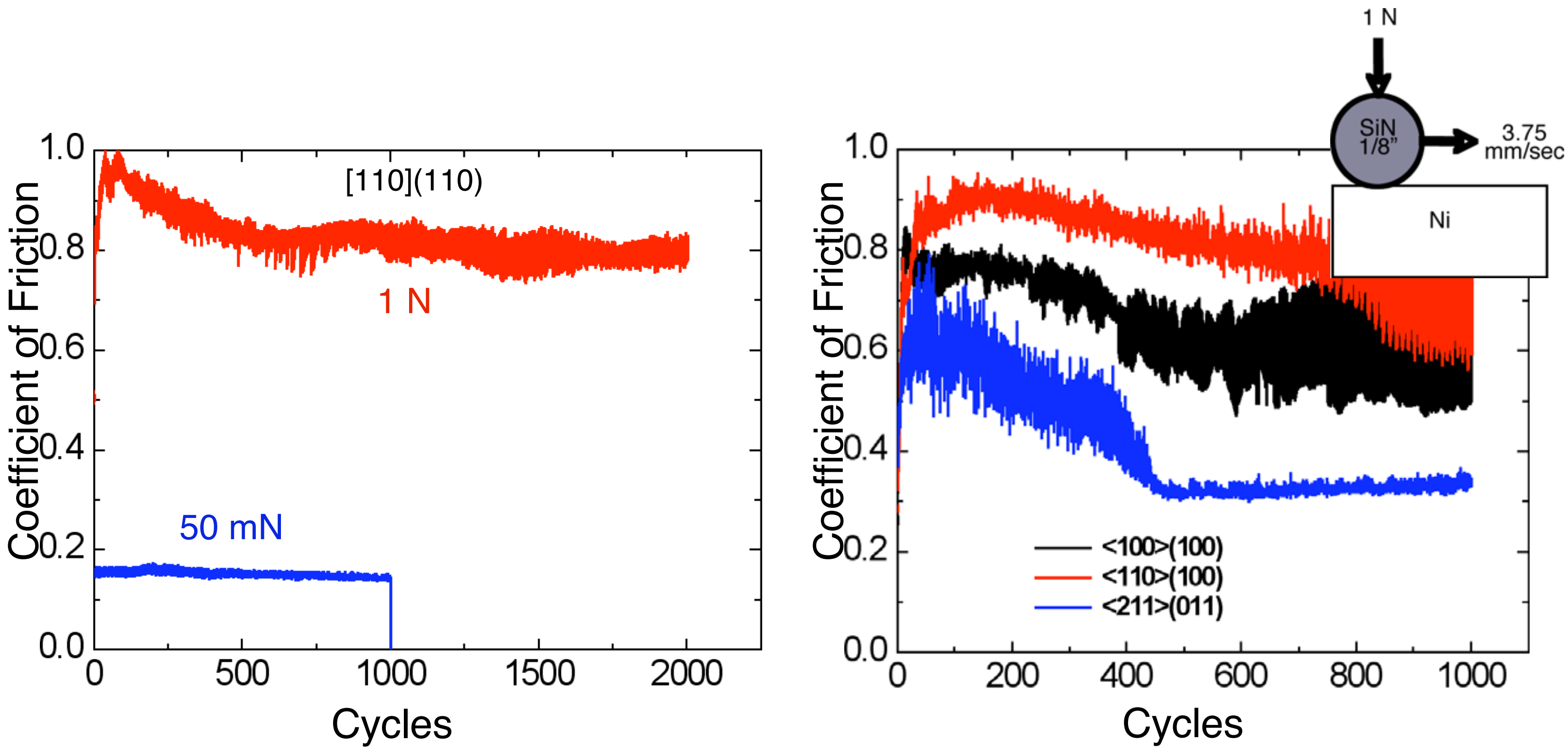
The coefficients of friction for single crystal nickel also exhibit a general increase with hardness.

(001)[100] Ni



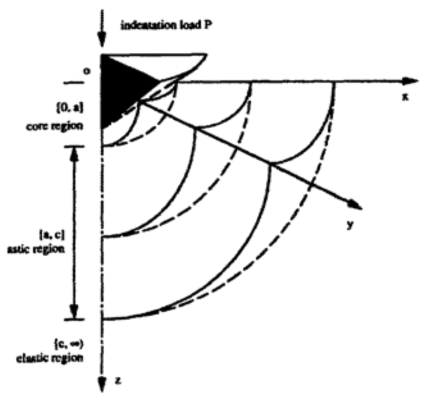
There is a stronger correlation with surface roughness than hardness.

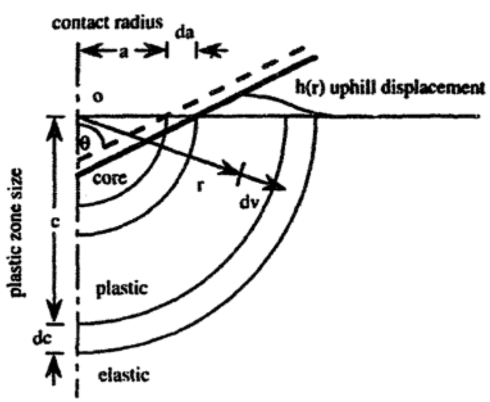
Coefficients of friction increase with increasing applied load and number of cycles during the first 50 cycles.



A strong effect of orientation develops at high applied loads

A simple algorithm describes the evolution of deformation under rigid indenter contact.





Assuming stress and strain states maintain radial symmetry

$$\frac{da}{dc} = \frac{\sigma_o}{E}\left[3(1-\nu)\frac{c^2}{a^2} - 2(1-2\nu)\frac{a}{c}\right]$$

where

$$c = \sqrt{\frac{3P}{2\pi\sigma_o}} \qquad \sigma_o = H/3$$

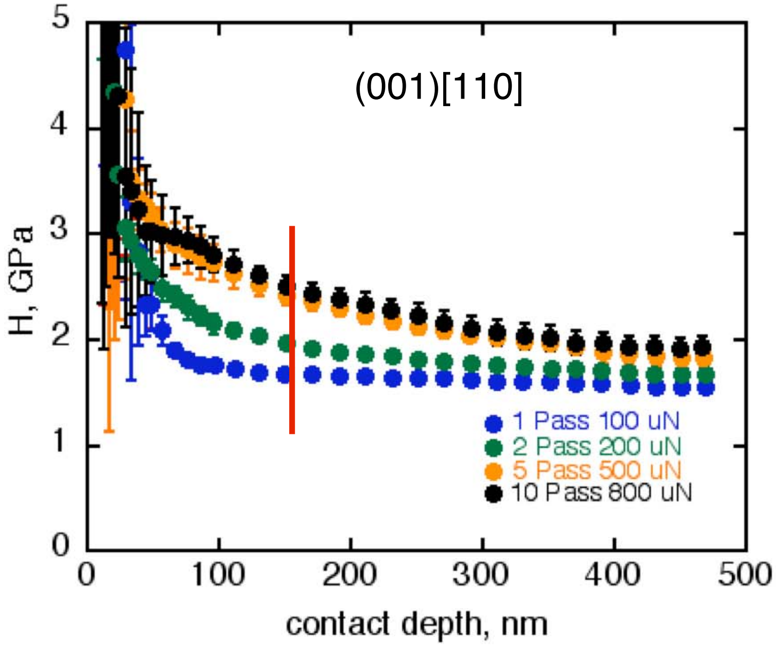
giving plastic radial strains

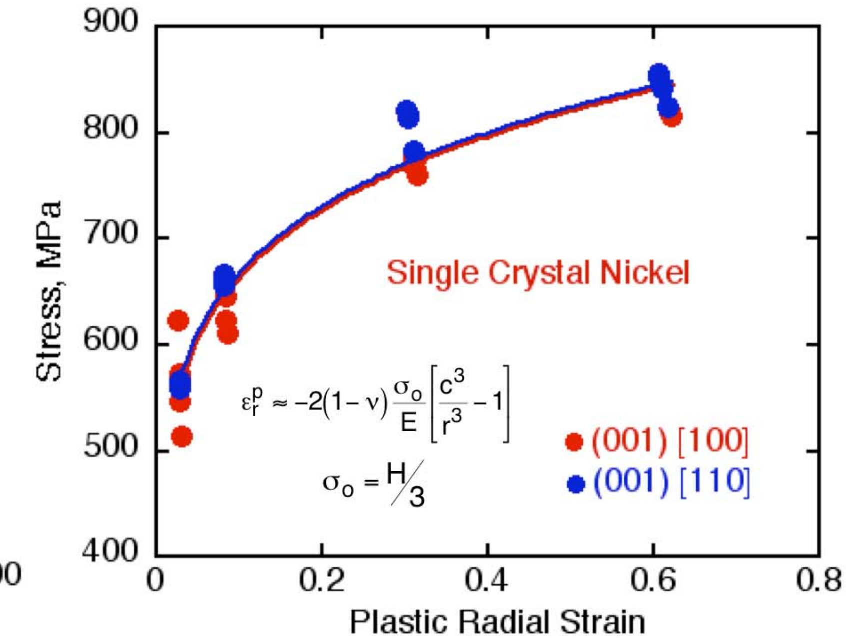
$$\varepsilon_r^p \approx -2(1-\nu)\frac{\sigma_o}{E}\left[\frac{c^3}{r^3} - 1\right]$$

(Harvey, Huang, Venkataram, Gerberich, 1993)

It also applies to deformation under cyclic contacts and from single and multiple cycles of lateral scratches.

Stress and strain were determined at 150 nm within the plastic zone assuming the ISE was constant for all tests.

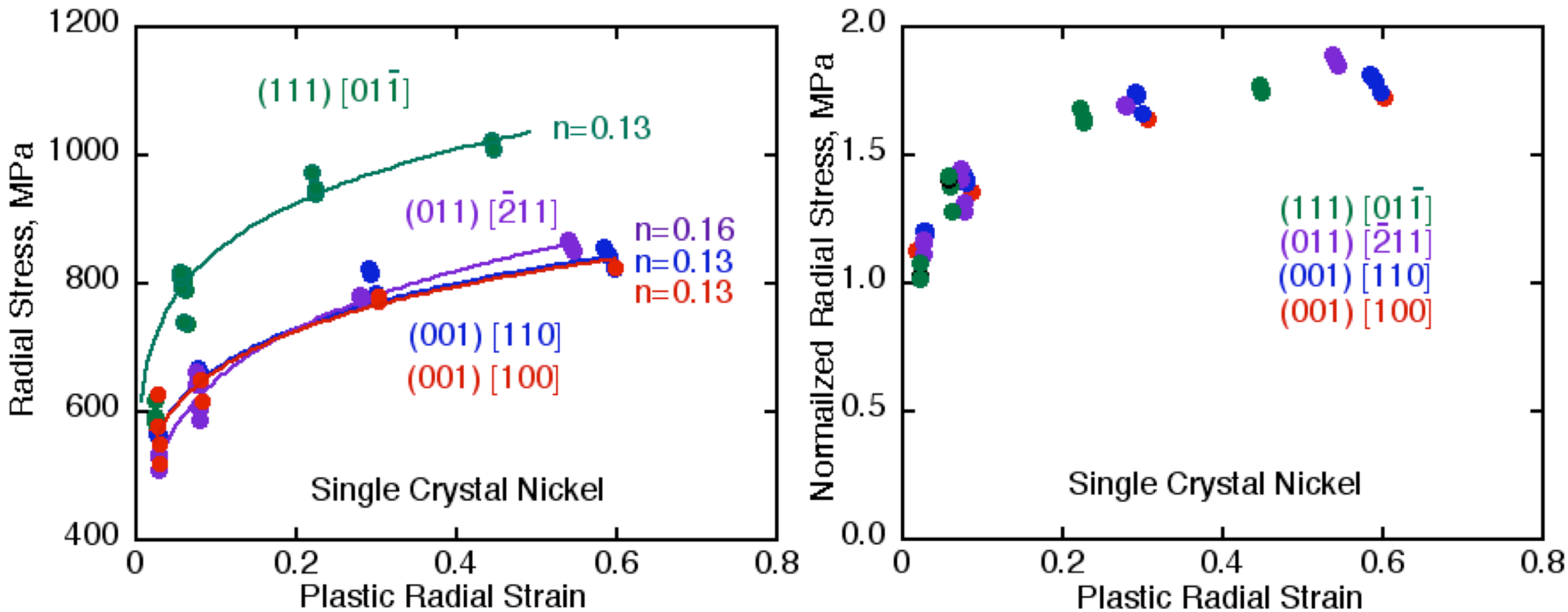




Data taken at a depth of 150 nm.

There is a strong hardening effect with essentially the same behavior along the [100] and [110] directions.

Stress-strain curves vary with crystal orientation.

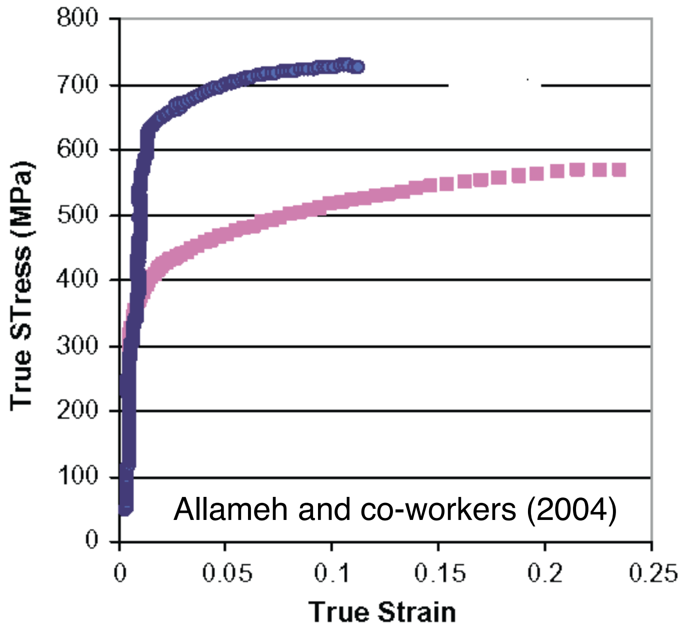


Data taken at a depth of 150 nm.

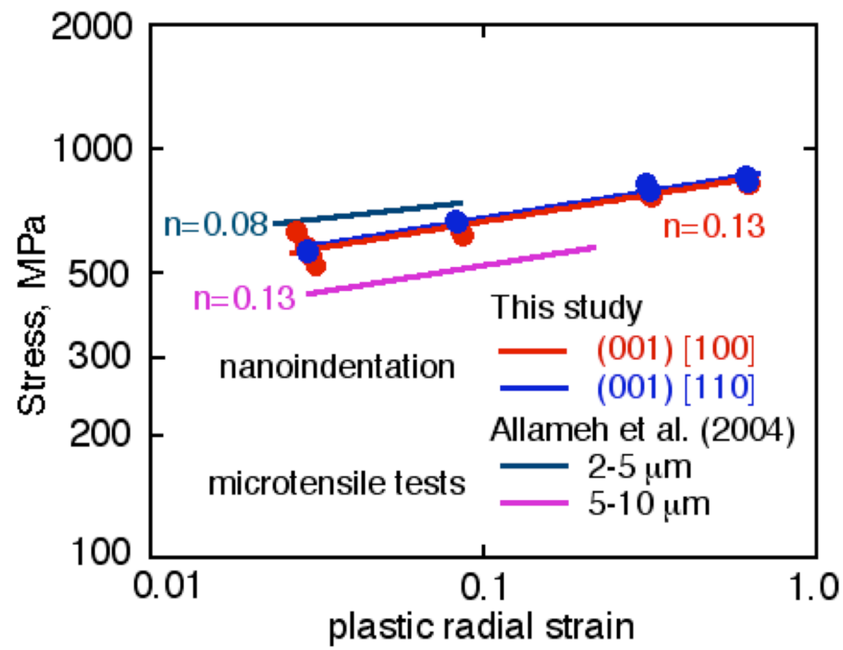
Hardness accounts for most observed orientation effects.

Stress and strain data and work hardening response from nanoindentation tests complement microtensile results.

electrodeposited Ni

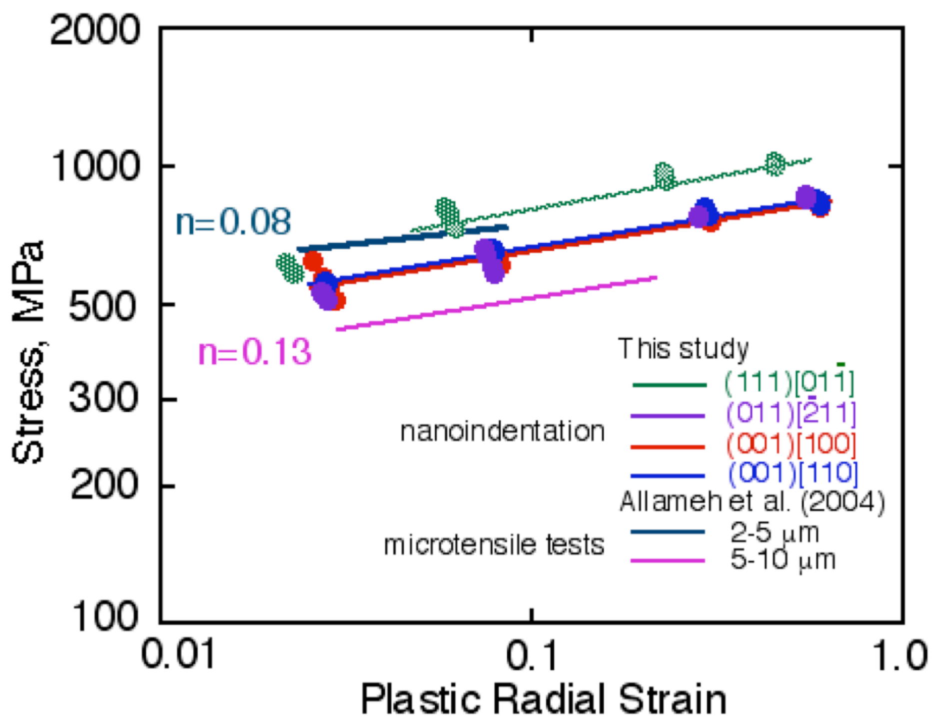


Strong (001) texture



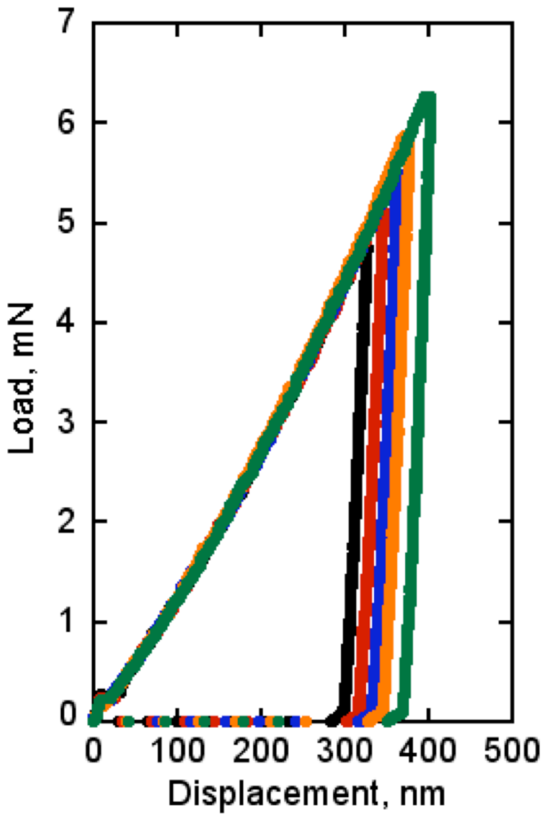
The single crystal work hardening response under nanoindentation follows tensile test results.

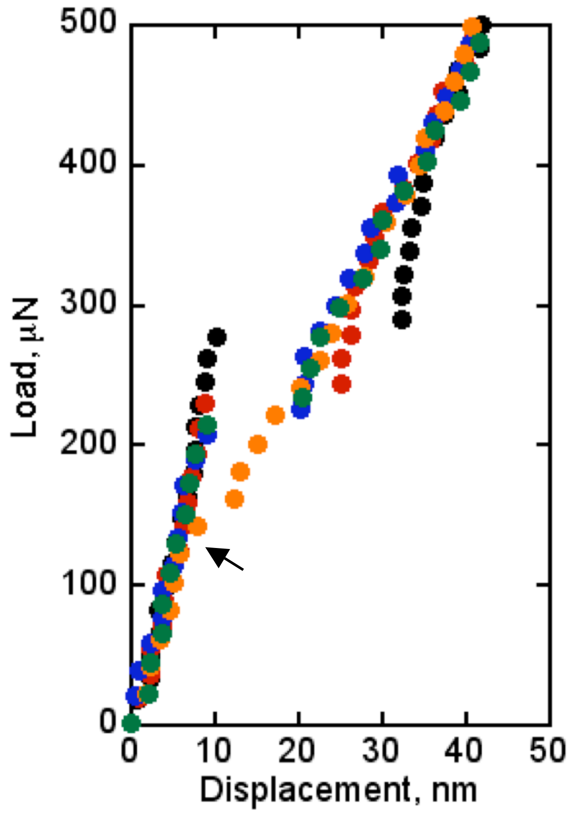
Stress and strain data from the (001) and (011) crystals superimpose.



The (111) stresses are higher than for other orientations but work hardening follows other orientations and electrodeposited Ni.

Conical tip indentation reveals inflections at low loads and excursions at higher loads.





From Hertz,

$$1/E^{*2} = 9P^2 / 16R\delta^3$$

$$1/E^* = (1-\nu_i^2)/E_i + (1-\nu_s^2)/Es$$

$$\tau_{max} = 0.47P/\pi a^2$$

For excursions,

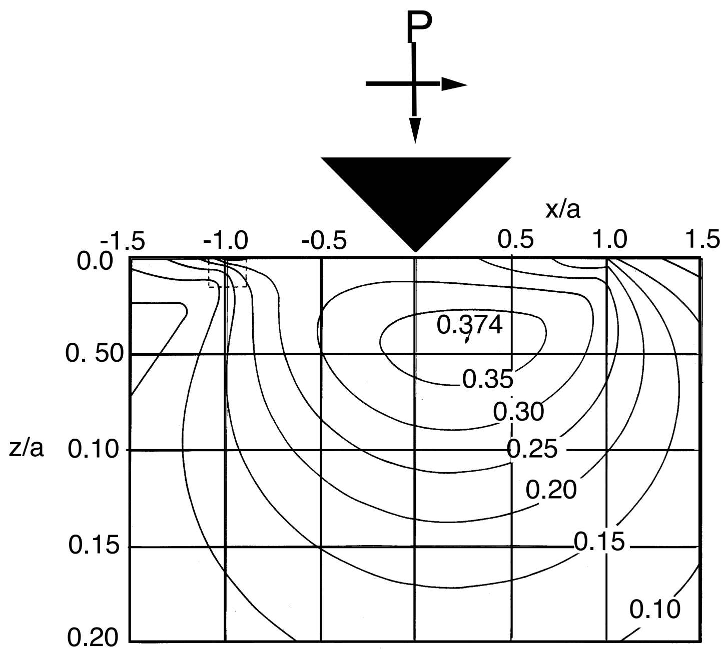
P=140 μN
δ=7.8 nm
R=1 μm
a=125 nm

E=175 GPa
τ_{max}=1.3 GPa

Inflections correspond to dislocation nucleation.
Excursions correspond to multiple dislocation emissions.

Inflections occurred at loads down to 80 μN

Superposition of normal and lateral forces during the scratch tests increases the maximum shear stress and dislocation nucleation.



Curves of $\sqrt{J_2}/p_o$ for μ=0.25

G. M. Hamilton (1983)

Hamilton obtained explicit solutions for stresses beneath a sliding normal loaded Hertzian contact.

For μ=0.25 and ν=0.3

$$p_o = 3P/2\pi a^2$$

$$\tau_{max}(\text{sliding}) = 0.374 p_o$$

Compared to

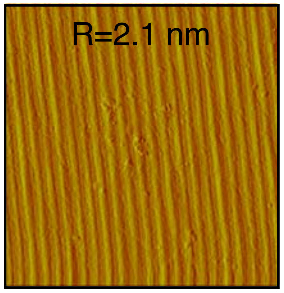
$$\tau_{max}(\text{normal}) = 0.31 p_o$$

Giving a ratio of

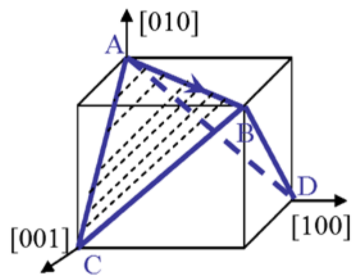
$$\tau_{max}(\text{sliding})/\tau_{max}(\text{normal}) = 1.2$$

Stresses under sliding contacts at relatively low loads are sufficient to trigger extensive dislocation nucleation in the single crystals.

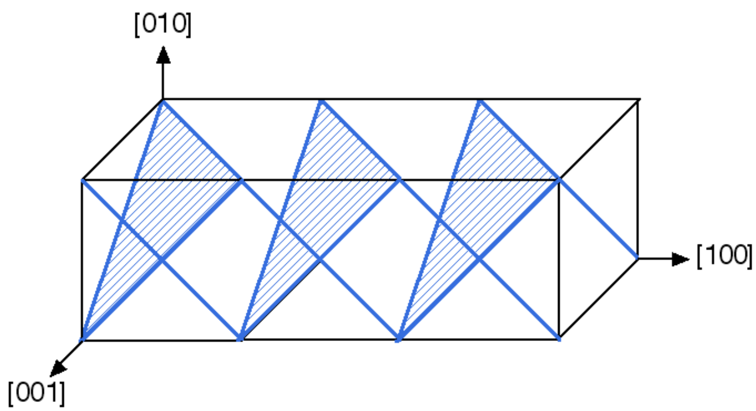
Dislocation generation leads to extensive slip band formation.



100 μN 1 Pass



Single pass sliding contact creates a nanocrystalline pattern of slip bands along the scratch track



Slip on multiple systems during creation of a wear pattern creates a nanocrystalline-like structure

(Prasad, Michael, Battaile, Kotula, 2005)

Multiple passes at high loads lead to a randomly oriented nanocystalline structure

Modeling the effects of wear provides insights on deformation processes and changes in structure

- **Experimental Observations**
  - Change in crystallographic orientation and texture
  - Change in microstructure during single crystal Ni wear

(S. Prasad, J. Michael, P. Kotula)

- **Finite Element Simulations**
  - Elastoplastic Constitutive Model for FCC Metals

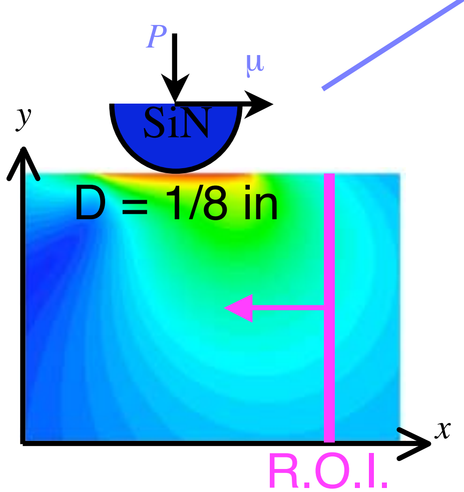
- **Simplified Crystal Plasticity for Cyclic Wear**
  - Reduction of the Elastoplastic Constitutive Model

- **Extended Model for Asperity Contact**
  - Extension of the Simplified Model to Include Asperities

(C. Battaile)

Elastoplastic Model for FCC Metals

Frictional
Elastic
Contact
Stresses



Constitutive Model

$$\tau_{rss} = \frac{1}{2}\sigma : (\mathbf{d} \otimes \mathbf{n} + \mathbf{n} \otimes \mathbf{d})$$

$$\dot{\gamma} = \dot{\gamma}_o \left(\frac{\tau_{rss}}{\tau_{crss}} \right)^m$$

$$\dot{\mathbf{D}}_p = \frac{\dot{\gamma}}{2}(\mathbf{d} \otimes \mathbf{n} + \mathbf{n} \otimes \mathbf{d})$$

$$\Delta\varepsilon_p = \Delta t \sqrt{\frac{2}{3}(\dot{\mathbf{D}}_p : \dot{\mathbf{D}}_p)}$$

$$\tau_{crss} = \tau_o + A\left[1 - \exp\left(-\frac{n}{A}\varepsilon_p\right)\right]$$

$$\dot{\mathbf{D}}_e = \dot{\mathbf{D}} - \dot{\mathbf{D}}_p$$

$$\sigma = \mathbf{C} : (\dot{\mathbf{D}}_e \Delta t)$$

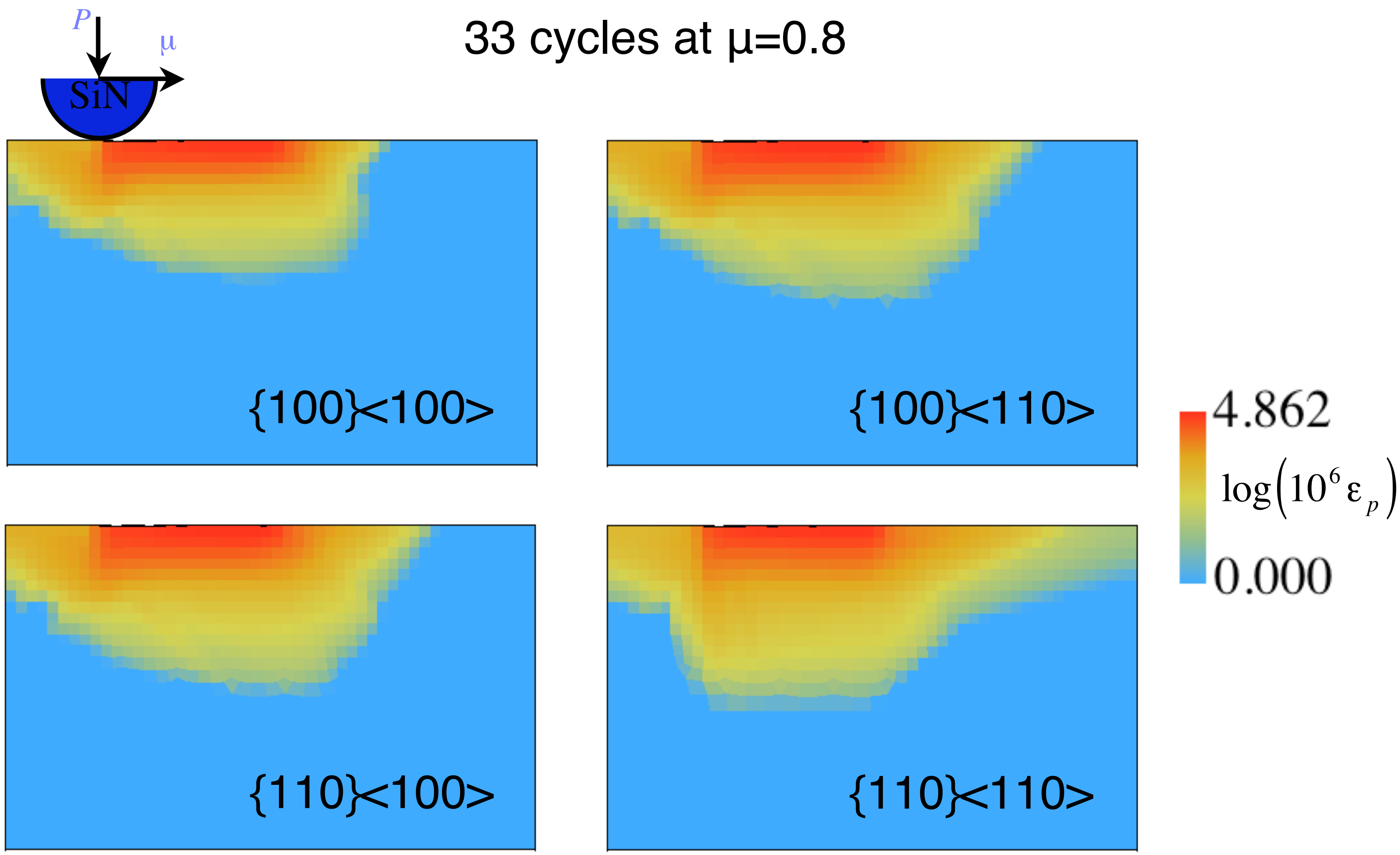
Mechanical
State

Material
Properties

Updated
Stress

Employed a simplified plasticity model for high cycle wear

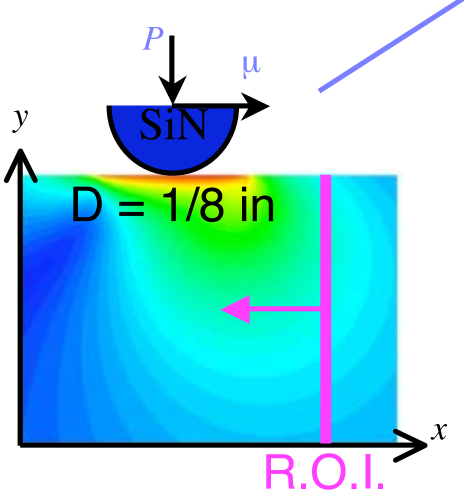
Plastic zone size varies with crystallographic orientation



Simulations show a strong plastic strain gradient forms under sliding contacts that follows experimental observations

Employed a simplified plasticity model for high cycle wear

Frictional Elastic Contact Stresses[3]



Mechanical State

Material Properties

Simplified Model[1,2]

$$\tau_{rss} = \frac{1}{2}\sigma : (\mathbf{d}\otimes\mathbf{n} + \mathbf{n}\otimes\mathbf{d})$$

$$\dot{\gamma} = \dot{\gamma}_o \left(\frac{\tau_{rss}}{\tau_{crss}}\right)^m$$

$$\dot{\mathbf{D}}_p = \frac{\dot{\gamma}}{2}(\mathbf{d}\otimes\mathbf{n} + \mathbf{n}\otimes\mathbf{d})$$

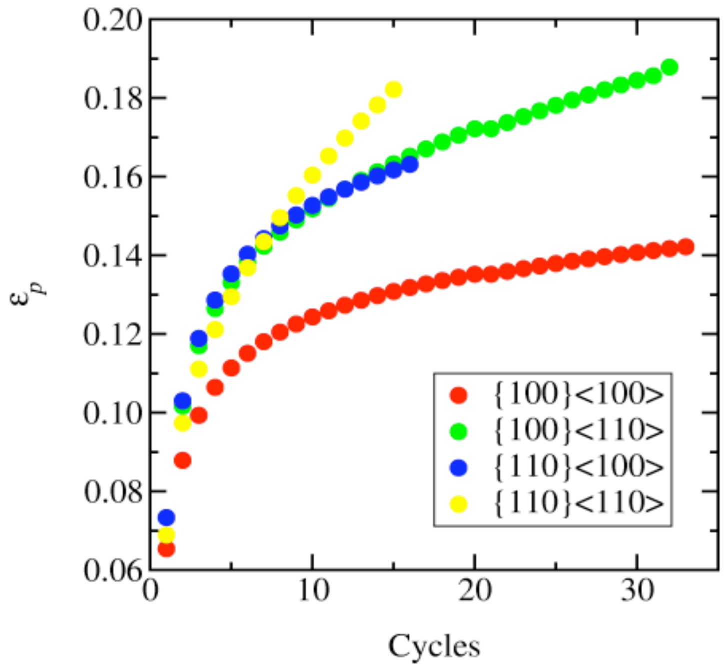
$$\Delta\varepsilon_p = \Delta t\sqrt{\frac{2}{3}(\dot{\mathbf{D}}_p : \dot{\mathbf{D}}_p)}$$

$$\tau_{crss} = \tau_o + A\left[1 - \exp\left(-\frac{n}{A}\varepsilon_p\right)\right]$$

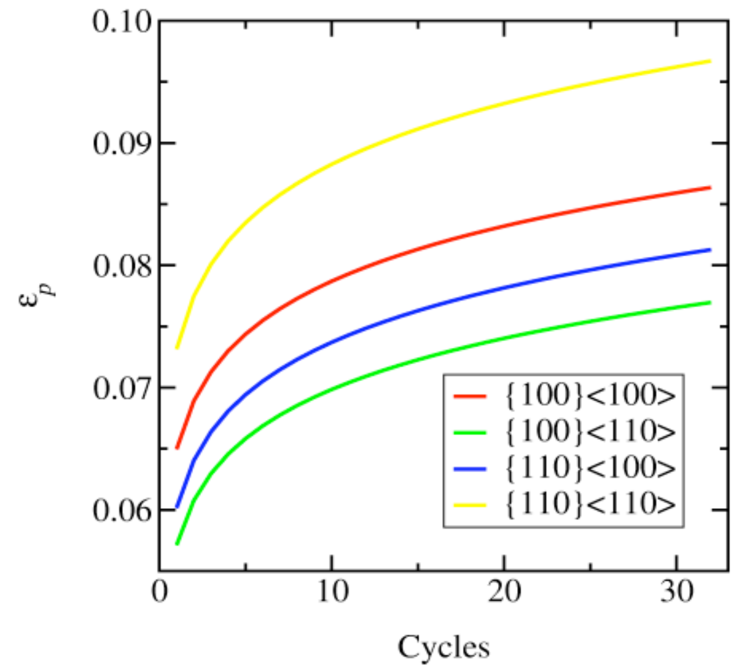
1. R.J. Asaro and J.R. Rice, "Strain Localization in Ductile Single Crystals," *J. Mech. Phys. Solids* **25** (1977) 309-38.
2. E. Voce, "The Relationship Between Stress and Strain for Homogeneous Deformation," *J. Inst. Metals* **74** (1948) 537-62.
3. D.A. Hills, D. Nowell, and A. Sackfield, *Mechanics of Elastic Contacts*, Oxford 1993.

Simplified plasticity model predicts a weaker dependence on orientation than Finite Element Analysis

Finite Element Analysis



Simplified Constitutive Model



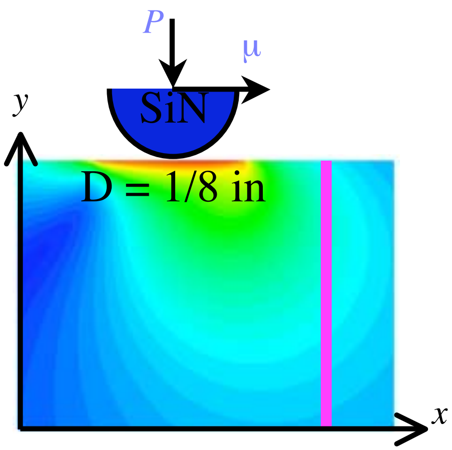
Depth of 350 nm

Ball on disk tests and nanowear tests:
Show evidence of higher strains

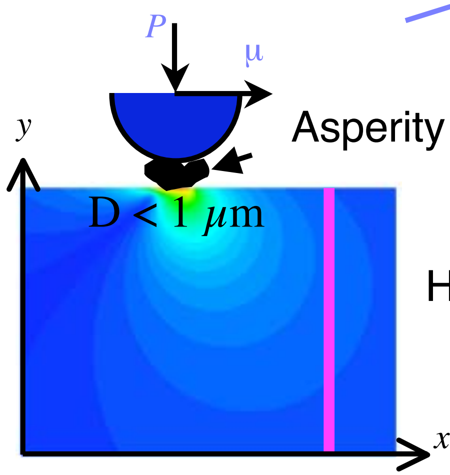
The predicted strains do not correlate with changes in microstructure.

Added asperity contact to the simplified constitutive model to more accurately describe sliding contact

Frictional Elastic Contact Stresses[3]

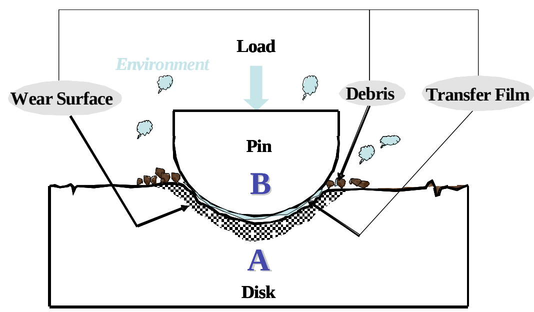


- or -



Asperity / Debris

Higher Stress, Localized



Mechanical State

Material Properties

Simplified Model[1,2]

$$\tau_{rss} = \frac{1}{2}\boldsymbol{\sigma} : (\mathbf{d} \otimes \mathbf{n} + \mathbf{n} \otimes \mathbf{d})$$

$$\dot{\gamma} = \dot{\gamma}_o \left(\frac{\tau_{rss}}{\tau_{crss}} \right)^m$$

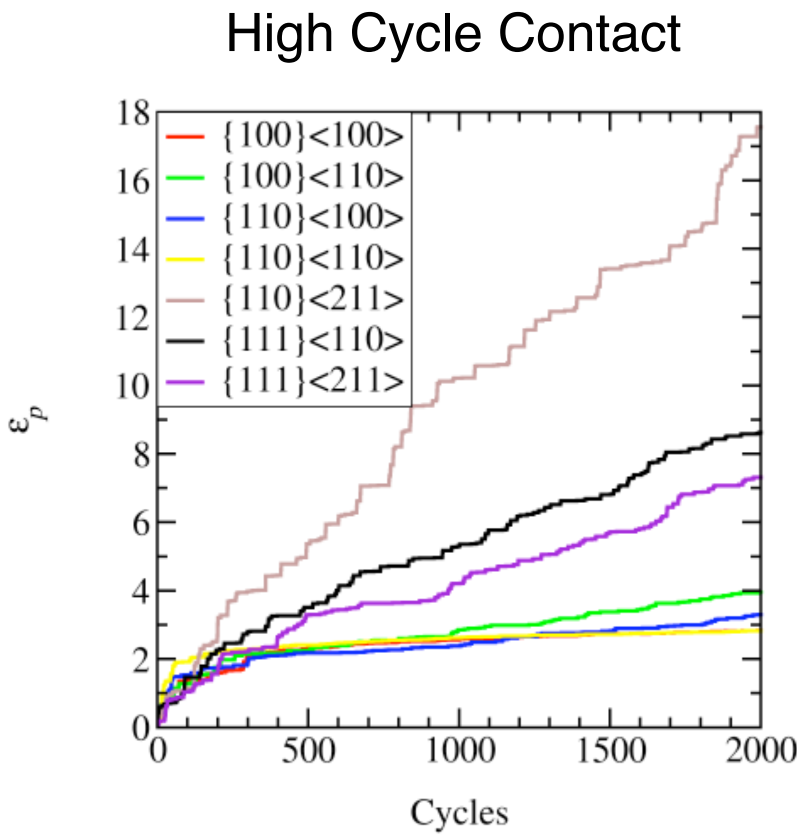
$$\dot{\mathbf{D}}_p = \frac{\dot{\gamma}}{2}(\mathbf{d} \otimes \mathbf{n} + \mathbf{n} \otimes \mathbf{d})$$

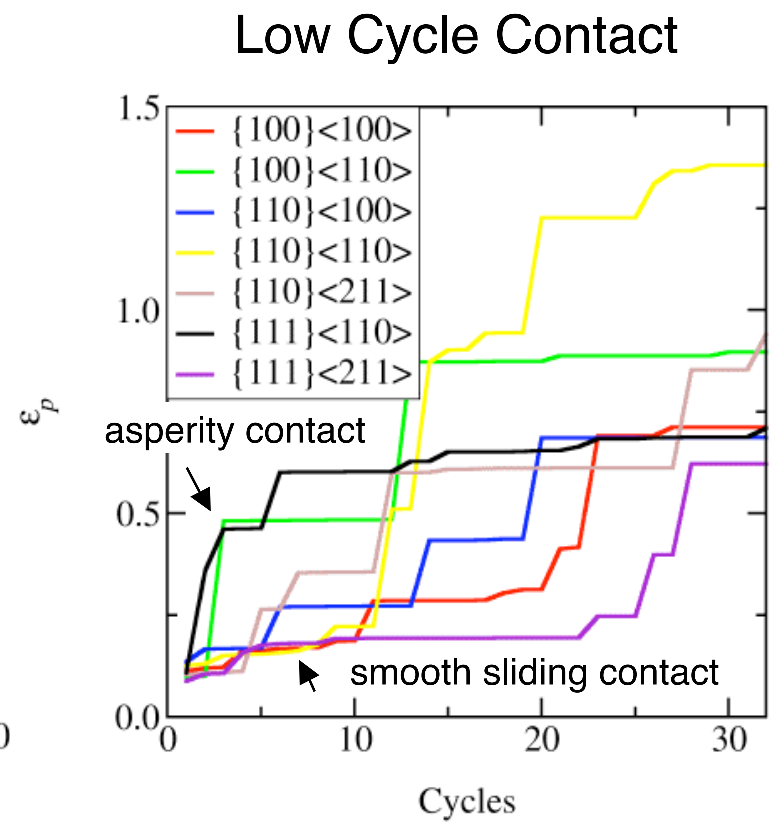
$$\Delta\varepsilon_p = \Delta t \sqrt{\frac{2}{3}(\dot{\mathbf{D}}_p : \dot{\mathbf{D}}_p)}$$

$$\tau_{crss} = \tau_o + A\left[1 - \exp\left(-\frac{n}{A}\varepsilon_p\right)\right]$$

1. R.J. Asaro and J.R. Rice, "Strain Localization in Ductile Single Crystals," *J. Mech. Phys. Solids* **25** (1977) 309-38.
2. E. Voce, "The Relationship Between Stress and Strain for Homogeneous Deformation," *J. Inst. Metals* **74** (1948) 537-62.
3. D.A. Hills, D. Nowell, and A. Sackfield, *Mechanics of Elastic Contacts*, Oxford 1993.

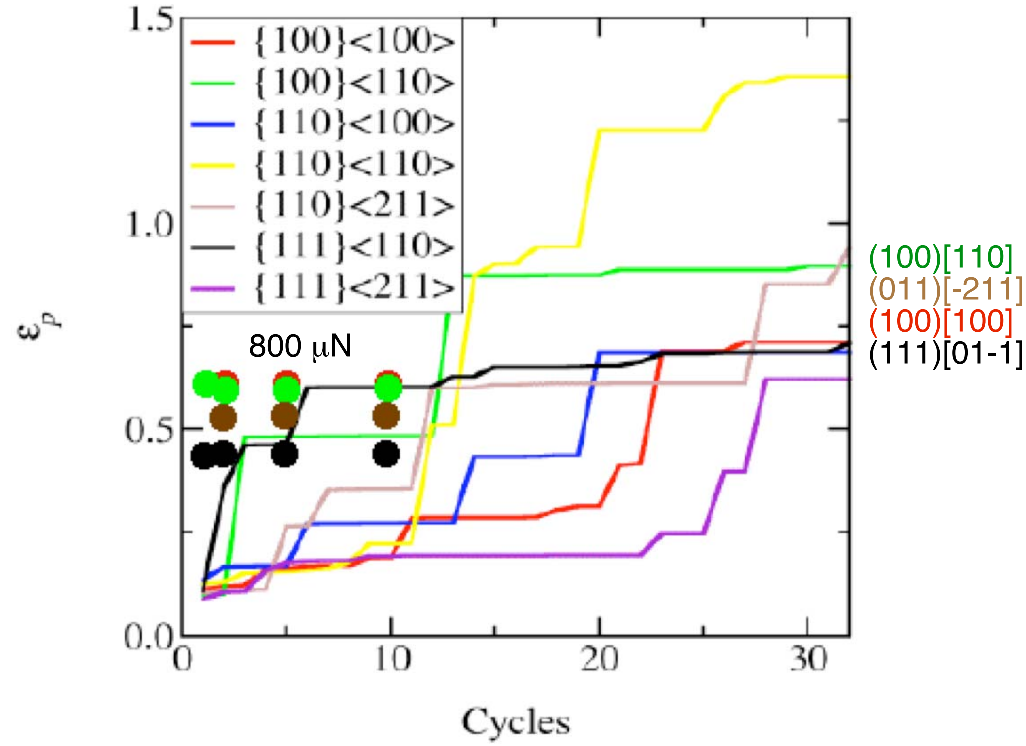
The asperity contact model predicts much higher near surface plastic strains than the ideal contact model





A strong dependence on scratch orientation occurs at a high number of cycles for the {110} and {111} crystals but not for the {100} crystal.

Predicted plastic strains under asperity contact are close to estimated subsurface nanowear values



Including asperity contact shows significant promise in accurately simulating material behavior under sliding contacts.

Summary

In single crystal nickel, hardness varies significantly only with applied load.

Estimates for stress and strain show a strong hardening response under nano indentation that follows the evolution of microtensile test results.

The origin of behavior correlates to dislocation emission and slip band formation and suggests that multiple slip systems create a near surface nanocrystalline-like structure.

Simulation of plastic strains and their effect on microstructure from ideal contact do not agree with experiments. Simulation of plastic strains from asperity contact do agree.

Including asperity contact shows significant promise in accurately simulating material behavior under sliding contacts.

Acknowledgments

The support of Sandia National Laboratories is gratefully acknowledged.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000