Residual Stress Measurements in Thin Coatings

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Purpose of Work

- Reduction of friction and wear in drive trains and engine components, and consequently, reduction in parasitic energy losses can result in ≈3-4% fuel efficiency

- Performance of low friction and wear resistant coatings is strongly dependent on the residual stress profiles in the coating and at the coating/substrate interface; thus, it is critical to understand the residual stresses in the coatings

- Currently, no reliable technique(s) available to profile residual stresses in thin coatings

Develop and measure depth-resolved residual stress in thin coatings

Objectives

- Develop and refine high-energy x-ray technique(s) at t Advanced Photon Source (APS) to measure residual strains/stresses for super-hard, low-friction coatings as of coating depth

- Correlate residual stresses in coatings systems to pro technique and variables, material properties, adhesion and tribological properties

- Compare experimentally measured residual stresses to calculated stresses from modeling

- Develop an coating processing protocol to produce reliable coatings for a specific coating system and engine system application and transfer technology to industry

Approach

- Develop/refine high energy x-rays for profiling residual strai coatings by measuring the change in the lattice parameter o constituents as a function of depth
  - tribological coatings ≈2-5 μm so they require meso-scale techniques with high depth resolutions (<1 μm)

- Deposit low friction high wear resistance superhard coatings on substrates under controlled processing conditions
  - deposition power & time
  - composition

- Develop indentation-based techniques to measure hardness, fracture toughness, and adhesion energy of thin coatings

- Relate residual stresses, mechanical & tribological properties, and processing to coating durability

processing ⟷ residual stresses ⟷ performance

Performance Measures –
Progress in Meeting Objectives

- Using the high-energy X-ray source measured stresses as a function of depth in 3-4 µm thick coatings of nanocrystalline MoN and MoCuN deposited on silicon and steel substrates

- Profiled residual stresses in coatings as a function of:
  - coating deposition conditions (composition, power, time)
  - annealing

- Correlated the measured residual stresses to coating processing and annealing treatments

- Completed preliminary coating/substrate adhesion energies using an indentation technique

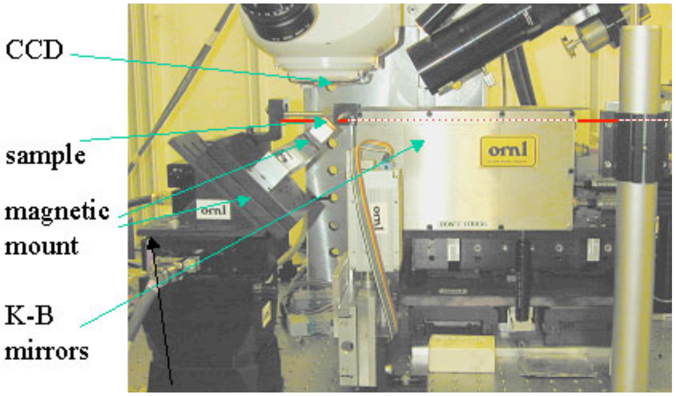
Accomplishments

Strain measurement techniques

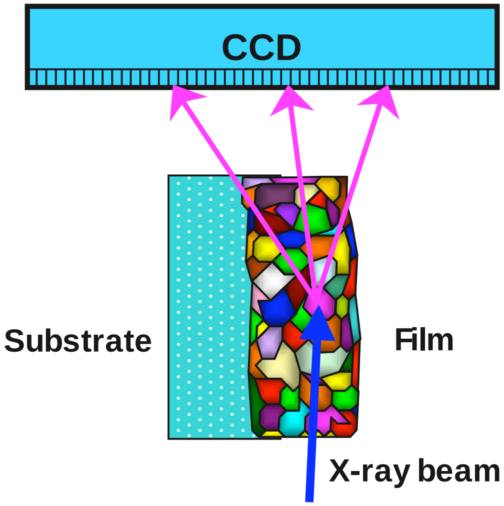
APS XOR/UNI beamline 34-ID-E

Kirkpatrick-Baez mirrors => white or mono beam

~ 0.5 X 0.5 μm^2

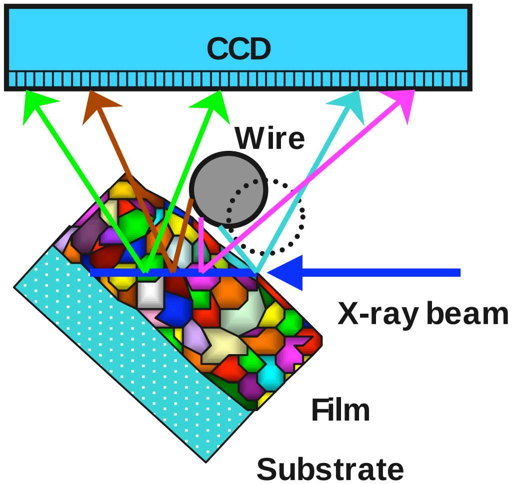


1. Cross-sectional microdiffraction



Scanning the cross section of a film using submicron mono x-ray beam.

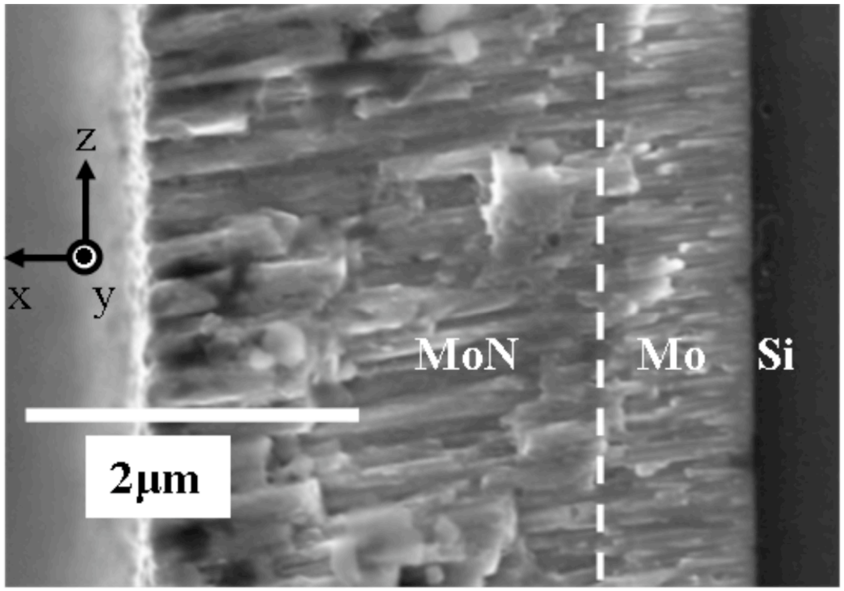
2. Differential aperture x-ray microscopy (DAXM)

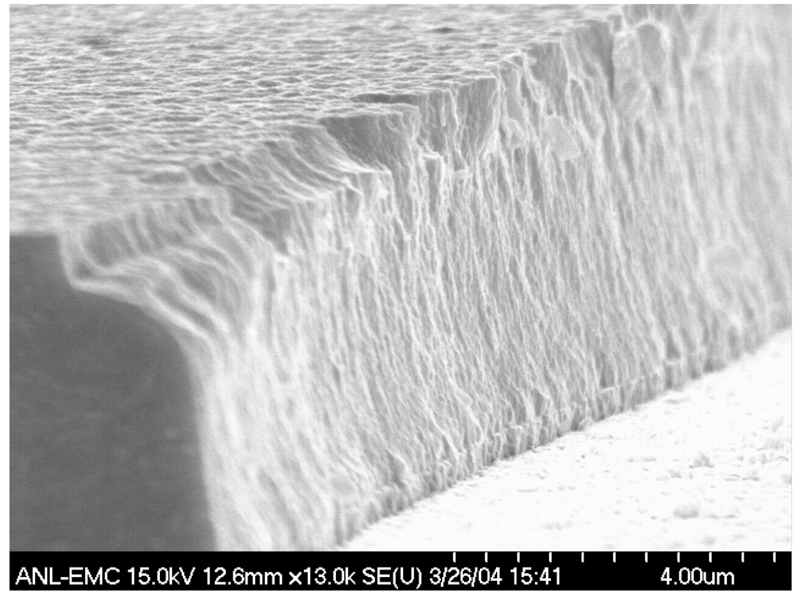


X-ray absorption wire acts as a differential aperture to separate information from different depths. (B.C. Larson et al. Nature **415**, 887 (2002)

Accomplishments

Coating systems (sputtered) investigated



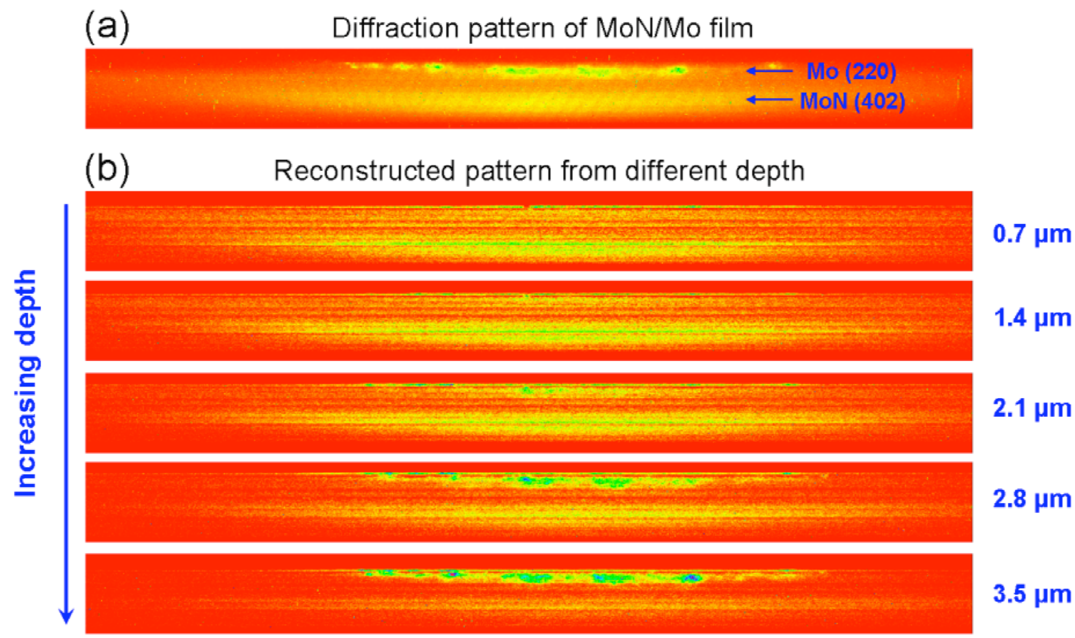


- MoN on Si wafer
- Mo bond layer
- model system used for developing x-ray measurement techniques

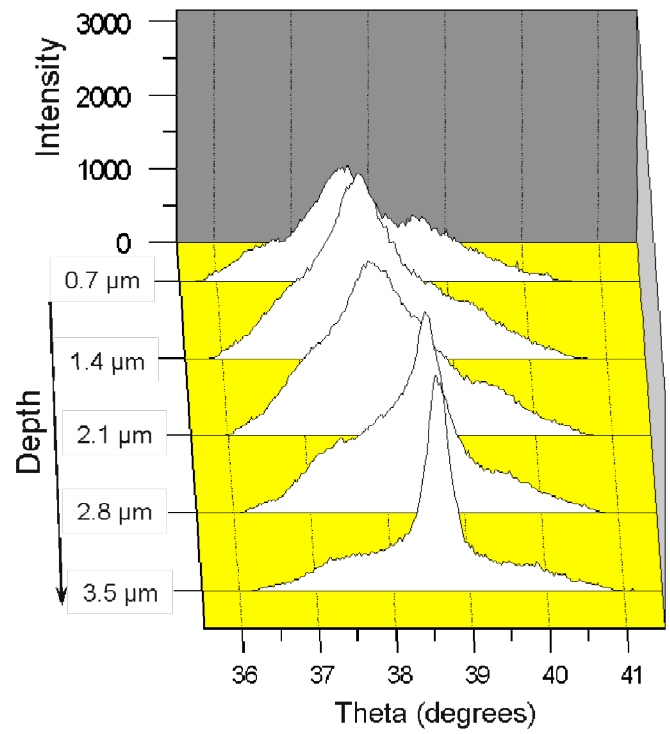
- MoCuN on H13 steel
- Cu concentration varied
- deposition power & time varied

Accomplishments

Microdiffraction



Diffraction pattern of MoN/Mo coating

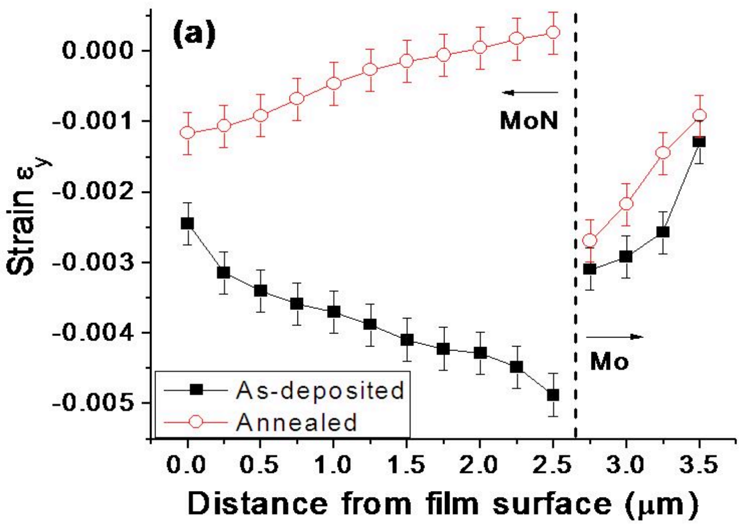


Shift in the diffraction peaks (MoN & Mo) as a function of depth indicative of variation of residual stresses as a function of depth

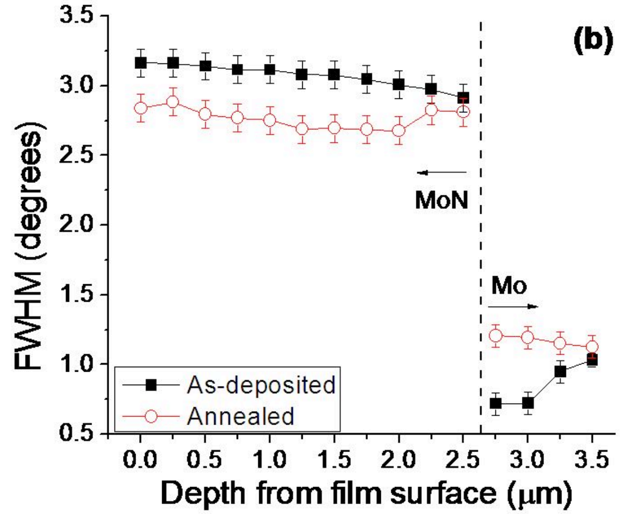
Accomplishments

Depth-resolved strain/structure in MoN films

Depth-resolved residual strain



Depth-resolved diffraction peak width



Film was annealed at 500 °C for 1 hr

Compressive strains with large gradients were observed in the MoN and Mo layers.

Annealing significantly changes strain profile in the MoN layer, but only slightly relieves strain in the Mo layer.

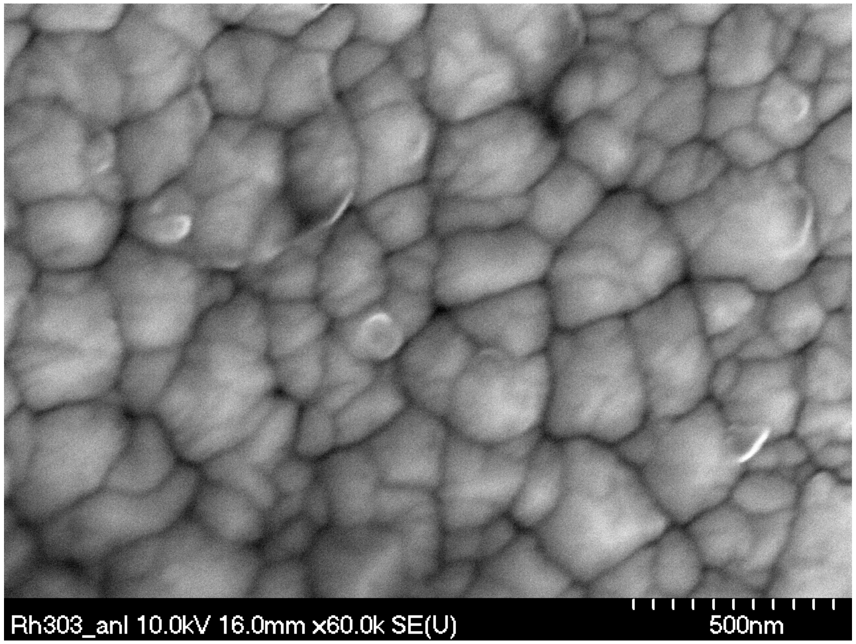
MoN layer has an average grain size of 5 nm, and that of the Mo layer is 20 nm.

Annealing reduces defects concentration in the MoN layer and increases that in the Mo layer.

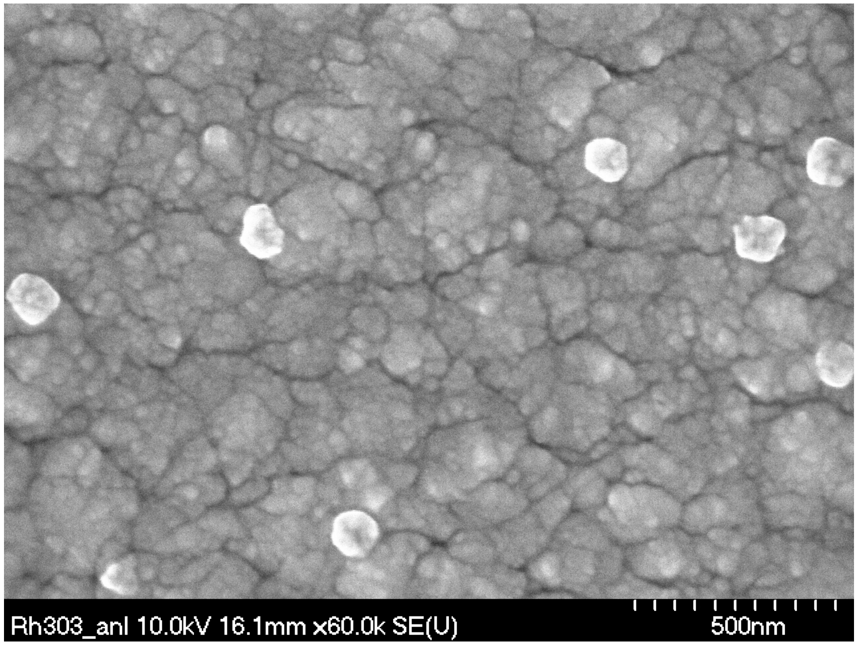
Accomplishments

MoN film: effect of annealing

As-deposited



Annealed

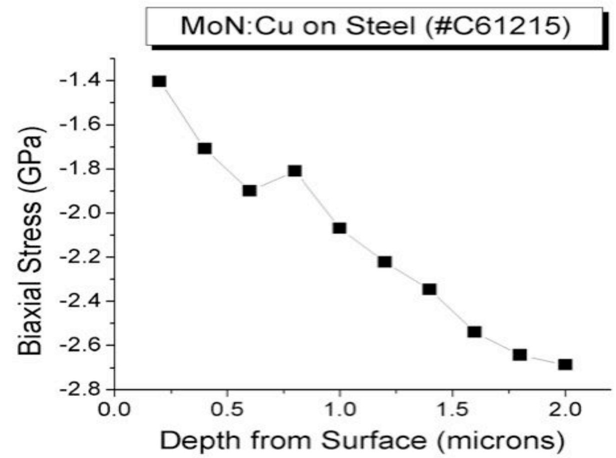


Annealing causes microstructural changes in the MoN coating
Boundary between clusters less defined

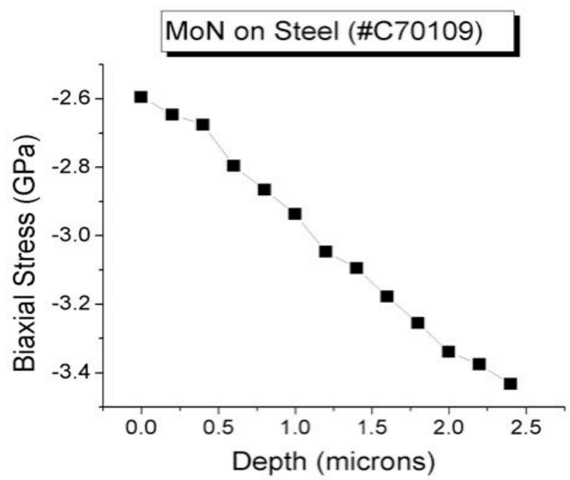
Accomplishments

Depth-resolved stresses in MoNCu filn

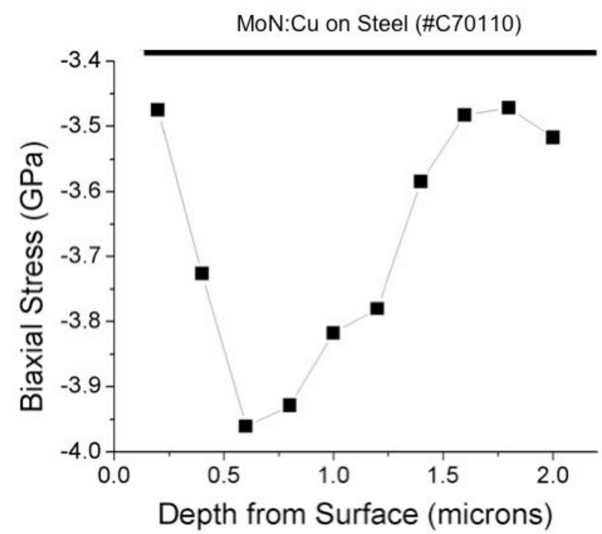
Mo: 8 kW, Cu: 0.8 kW, t = 7200 s



Mo: 8 kW, Cu: 0 kW, t = 7200 s



Mo: 8 kW, Cu: 0.5 kW, t = 6600 s



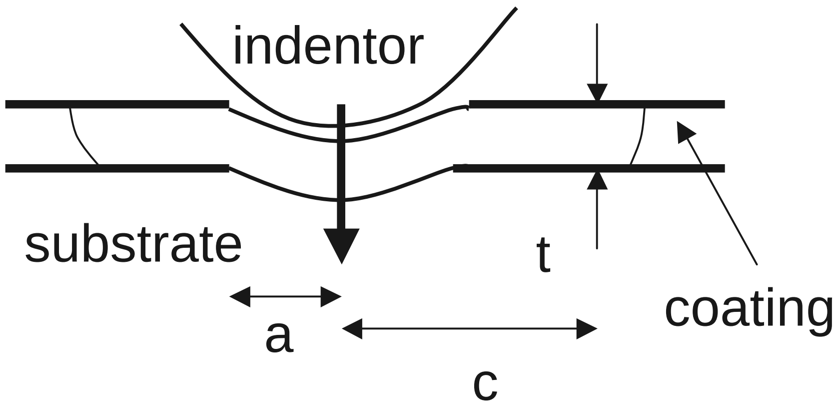
In-plane biaxial stresses as a function of coating depth in the various MoNCu coatings deposited on steel

Accomplishments

Adhesion Energy Measurements Using Indentation

(Kim et al., Thin Solid Films 441 (2003) 172-179)

- Hard brittle coating on relatively ductile substrate using Brale C
- Assumes that sub-surface crack is concentrated at the interface
- At a critical indentation load, the stress concentration at the interface becomes greater than the adhesion energy; causing delamination

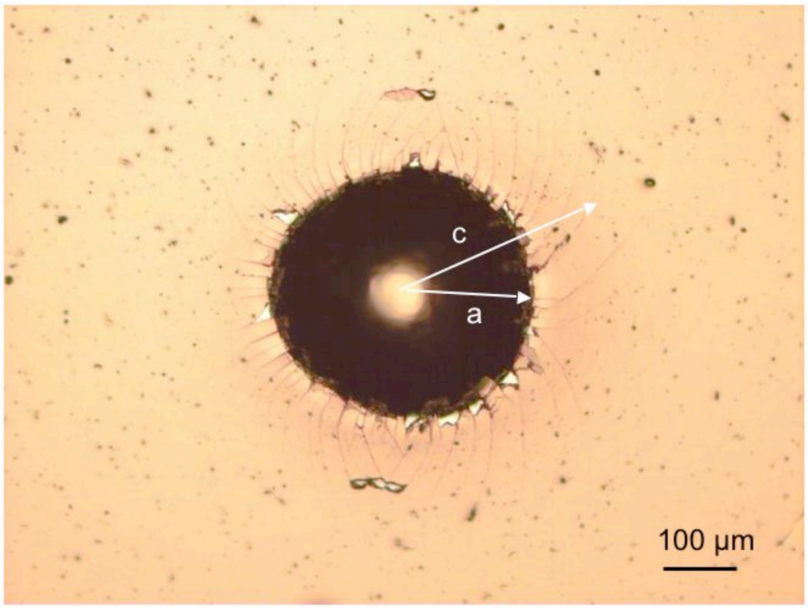


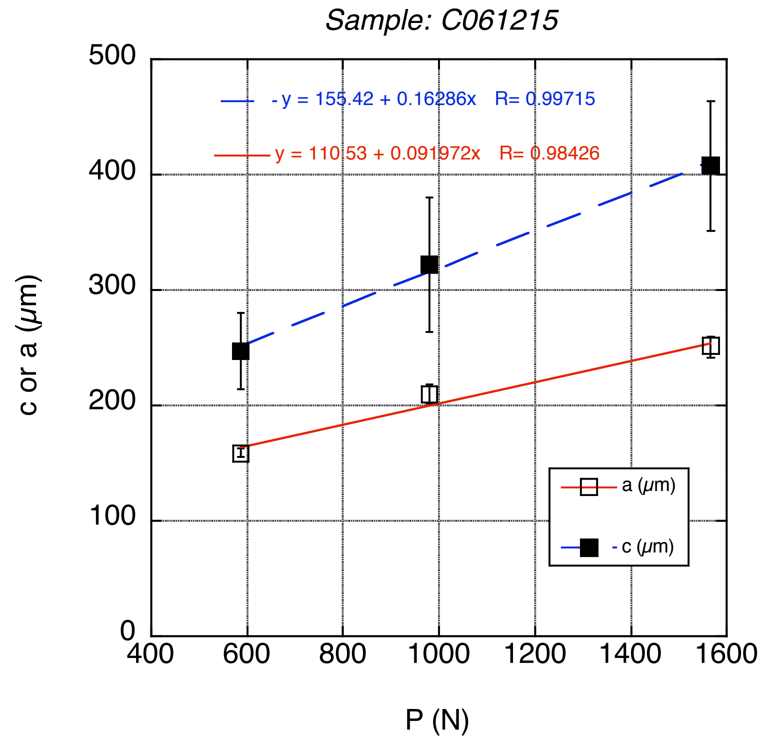
$$G_c = \left(\kappa \frac{dP}{dc} + \lambda\right) t \Big/ \left(c - a \frac{da}{dP} \frac{dP}{dc}\right)$$

P = indentation load
t = film thickness
a = indentation radius
c = delamination radius
κ, λ = constants based on material properties, residual stresses

Accomplishments

- Adhesion Energy Measurements Using Indentation





MoNCu on Steel

- Adhesion energy ≈ 600 J/m^2
- Similar to adhesion energies reported in literature for diamond like carbon coatings on steel
- No significant effect of the processing conditions investigated on the adhesion energy

Planned Activities for Next Fiscal Year

- Continue to refine and improve resolution of x-ray technique
- Develop indentation technique to measure film adhesion
  - evaluate mechanical properties such as hardness, toughness of the coatings
  - debond lengths estimations
  - investigate edge-effects on adhesion of coatings
- Correlate the measured residual stresses in MoCuN coatings to tribological properties
- Investigate other commercial coatings systems such as ZrN on steel

Summary

- Potential for petroleum displacement
  - 500 million gallons/yr reduction in diesel fuel consumption
- Approach to research
  - Use x-ray micro-diffraction to study depth-resolved residual strains/stresses in thin tribological coatings
- Technical Accomplishments
  - Two x-ray based techniques developed and applied for residual stress measurements
  - Stresses correlated to processing conditions (deposition conditions, annealing)
  - Progress made in determination of coating adhesion energy
- Technology Transfer
  - Enabling technology
- Plans for Next Fiscal Year in support of Residual Stresses in Coatings
  - Residual stress measurements will be extended to commercial coatings
  - Further refine adhesion energy measurement technique
  - Residual stresses/processing will be correlated to coating tribological properties
  - Identify an industrial partner for collaboration

Publications/Presentations (for reviewers only)

- G. Chen, D. Singh, O. Eryilmaz, and J.L. Routbort, “Depth-Resolved Residual Strain in MoN/Mo Nanocrystalline Films,” Appl. Phys. Lett., **89,** 172104 (2006).

- Invention report filed for measuring residual stresses using cross-sectional technique.