Residual Stress Measurements in Thin Coatings

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May 21, 2009

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Project ID # pmp_17_singh

Vehicle Technologies – Annual Review – May 18-22, 2009

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Overview

Timeline

- Project start FY07
- Project end FY12
- 50% complete

Budget

- Total project – $600 K
- FY08 = $125 K (DOE)
- FY09 = $100 K (DOE)

Barriers

- Role of residual stresses in thin tribological coatings and its implications to the coating performance is not well characterized or understood

⇒ accurate stress/strain profiling not available
⇒ analysis and interpretation of data
⇒ adhesion energy measurements are not trivial
⇒ correlations of coating processing, stresses and performance is quite complex

Partners

- Borg Warner
- Galleon International
- Hauzer Techno Coatings, Inc.

This project complements the overall effort in the area of development of low friction high wear resistant coatings for heavy vehicle applications

Why Measure Residual Stresses in Tribological Coatings?

- Minimizing friction and wear in vehicle drive trains and engine components can significantly reduce parasitic energy losses, and consequently, will result in petroleum displacement

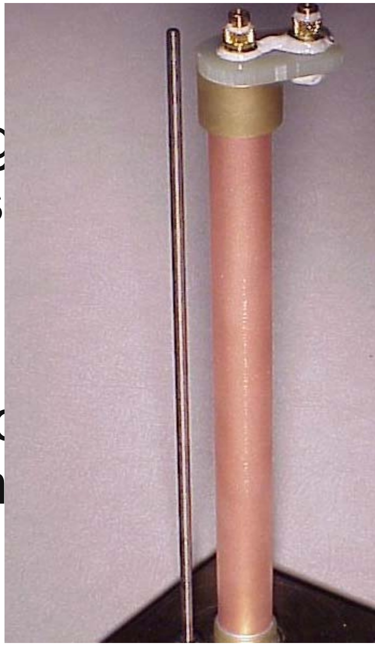
- Performance (low friction and wear) of tribological coatings and its long-term durability is strongly dependent on the residual stress profiles in the coating and at the coating/substrate interface; thus, it is critical to understand residual stresses in the coatings

- No reliable technique(s) available to profile residual stresses in thin coatings

- Inter-relationships between processing, residual stresses, and coating performance not available

Develop and measure depth-resolved residual stress in thin coatings

Objectives



- Develop and refine high-energy x-ray and/or scanning microscopic techniques to profile residual strains/stres hard, low-friction coatings for vehicle applications

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

- Compare experimentally measured residual stresses to calculated stresses from modeling

- Develop a coating processing protocol to produce reliable coatings with engineered residual stresses for a specific coating system applicable for vehicles and transfer technology to industry

Approach

- Develop/refine high energy x-rays for profiling residual strains in thin coatings by (a) measuring the change in the lattice parameter of the coating constituents and/or (b) measuring shift in specific location of the coating surface (stress relief) as focused ion beam (FIB) channels are created in coating nearby regions
  - tribological coatings ≈2-5 μm so they require meso-scale techniques with high depth resolutions (<1 μm)

- Deposit low friction high wear resistance coatings and profile residual stresses
  - deposition power & time
  - composition

- Develop indentation or scratch-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

Milestones

- FY08 *(all completed)*
  - Produce superhard, nanocrystalline coatings (MoCuN)
  - Procure commercial coatings(ZrN, TiC) with varying processing conditions: high-rate reactive sputtering (HRRS) & activated reactive evaporation (ARE)
  - Develop high-energy x-ray techniques
  - Conduct preliminary stress measurements (MoCuN)

- FY09
  - Analyze the stress measurement data (ZrN, TiC)
  - Correlate stresses and the processing conditions (MoCuN)
  - Explore measuring film mechanical behavior and adhesion energy using indentation techniques
  - Procure a scratch tester for adhesion energy measurements
  - Develop collaborations with industry

Accomplishments

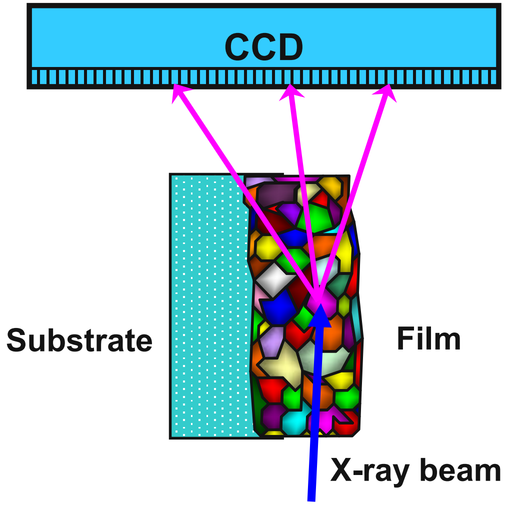
Strain measurement techniques

APS XOR/UNI
beamline 34-ID-E

Kirkpatrick-Baez mirrors
=> white or mono beam

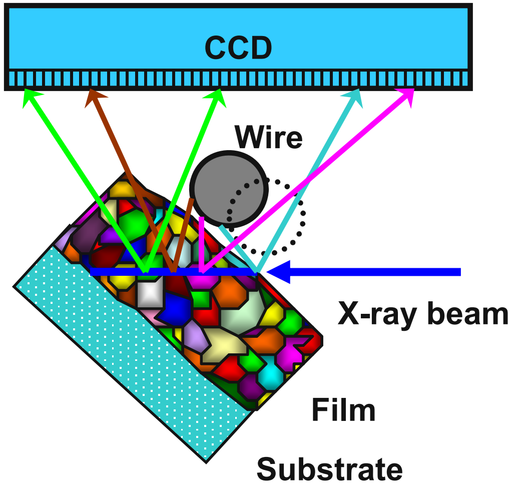
~ 0.5 X 0.5 μm^2

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)

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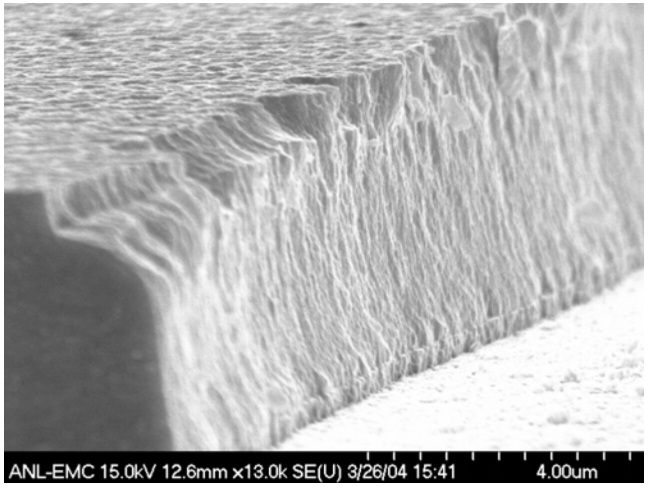
Accomplishments

Coating systems investigated:

(A)



- MoCuN on H13 steel
- Fabricated in-house using PVD
- Mo bond coat
- Cu concentration
- deposition power & time varied

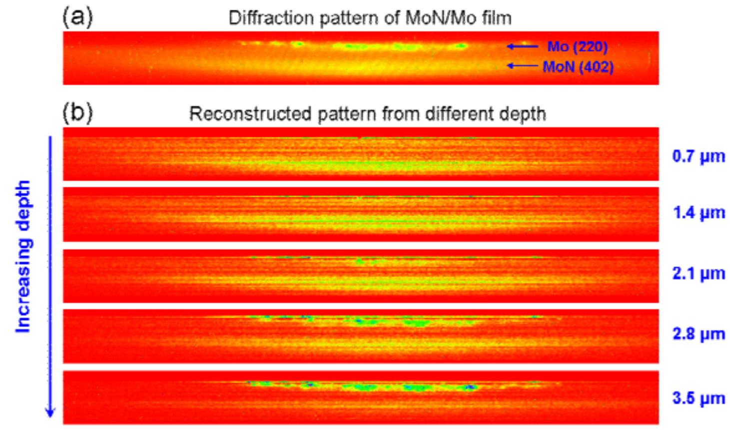
Sample ID	Mo & Cu Deposition Power (kW)	Deposition Time (s)
C70109	Mo:8; Cu: 0	7200
C70110	Mo: 8; Cu: 0.5	6600
C61215	Mo: 8; Cu: 0.8	7200

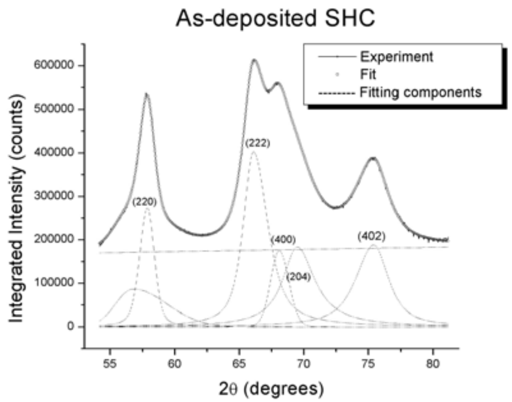
(B) TiC deposited on steel – high and low deposition rates

(C) ZrN deposited on steel – high and low deposition rates

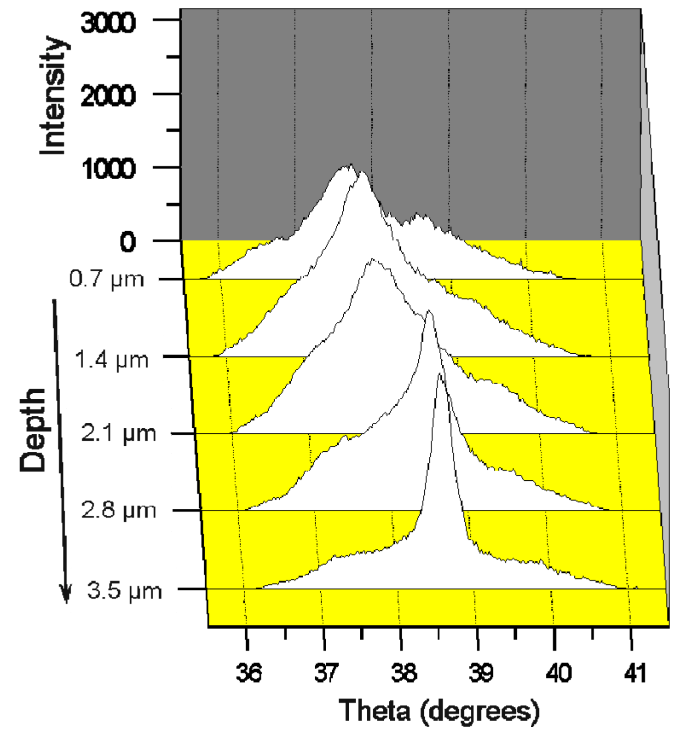
Accomplishments

Microdiffraction





Diffraction pattern of MoN/Mo coating

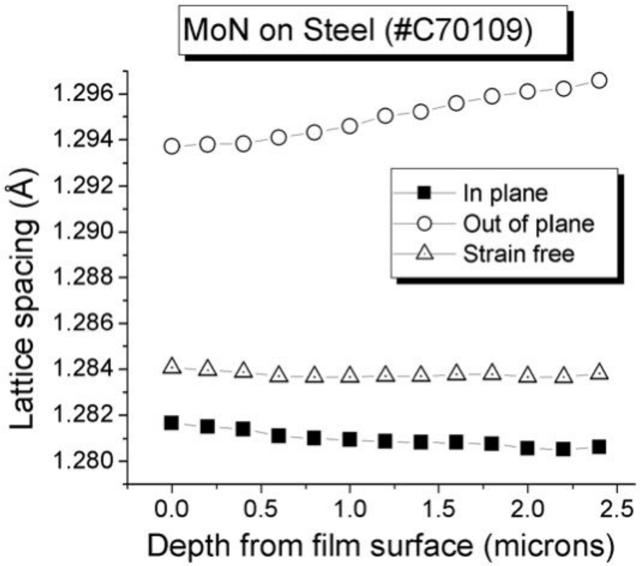


Shift in the diffraction peaks (MoN & Mo) as a function of depth indicative of variation of residual stresses as a function of depth

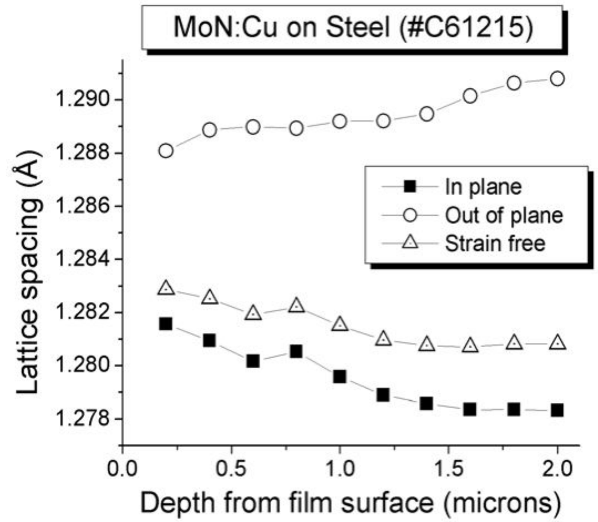
Accomplishments

Lattice Parameters of MoNCu Coatings

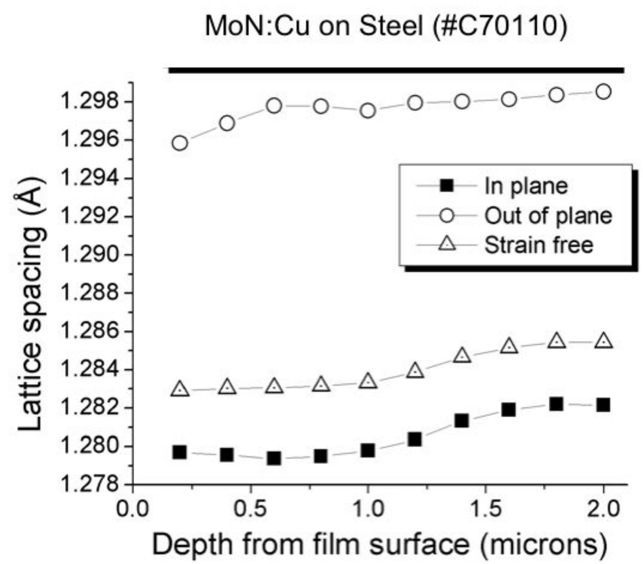
Mo: 8 kW, Cu: 0 kW, t = 7200 s



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



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

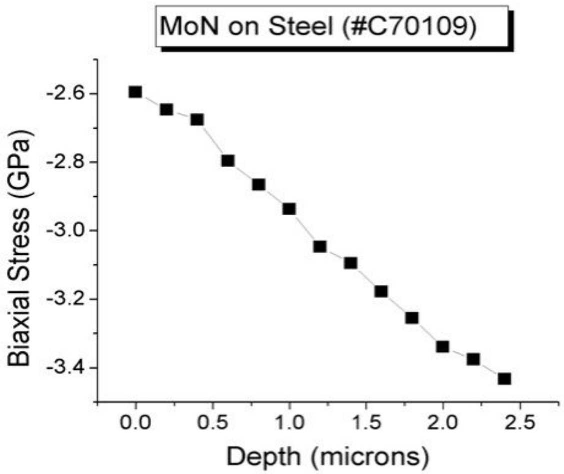


- Lattice parameters of MoN 222 reflections for various stress-states as a function of coating depth
- Strain free lattice parameters determined from $\sin^2\phi$ approach

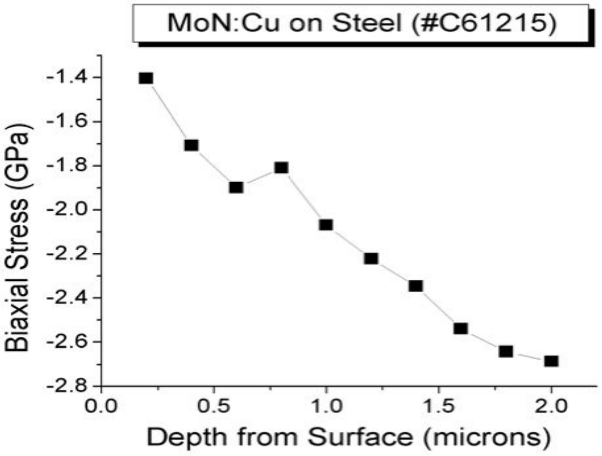
Accomplishments

Depth-resolved stresses in MoNCu films

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



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



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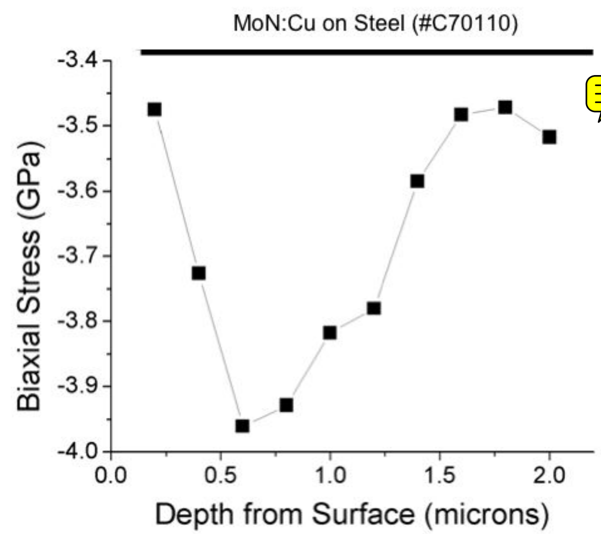
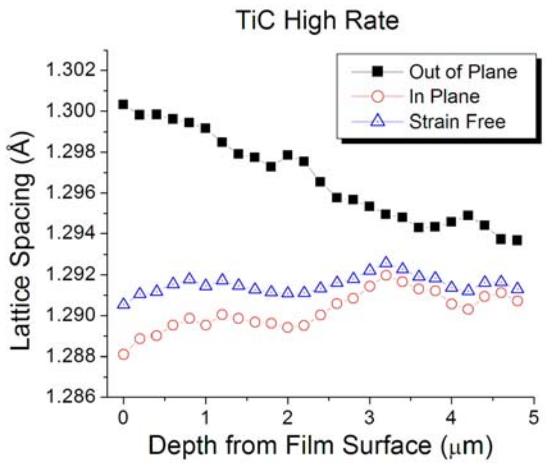


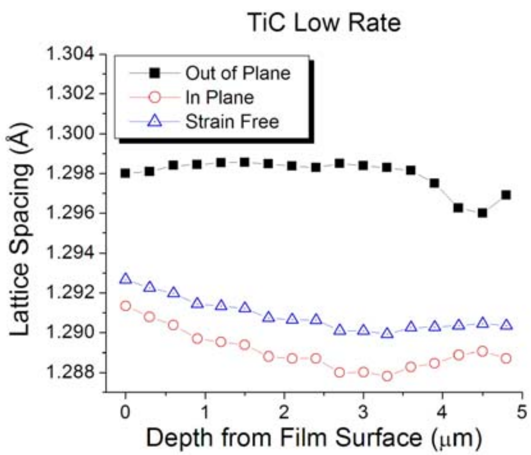
In-plane biaxial compressive stresses as a function of coating depth in the MoNCu coatings deposited on steel for various processing conditions

Accomplishments

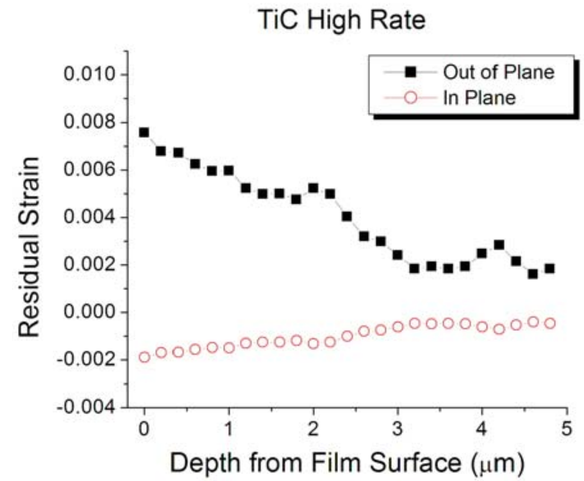
TiC Coating on Steel Results

Lattice Spacing

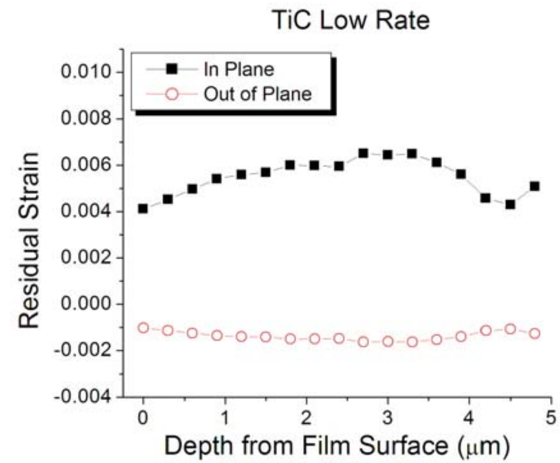




Residual Strain



HRRS process



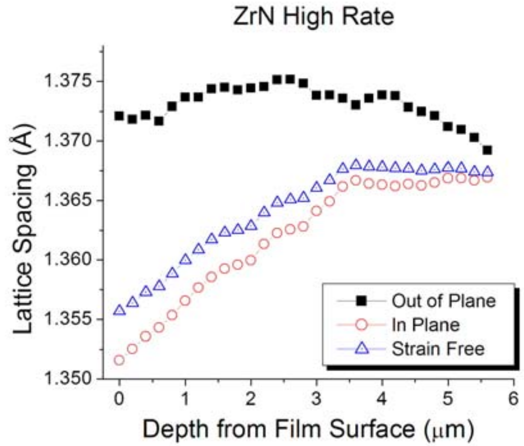
ARE process

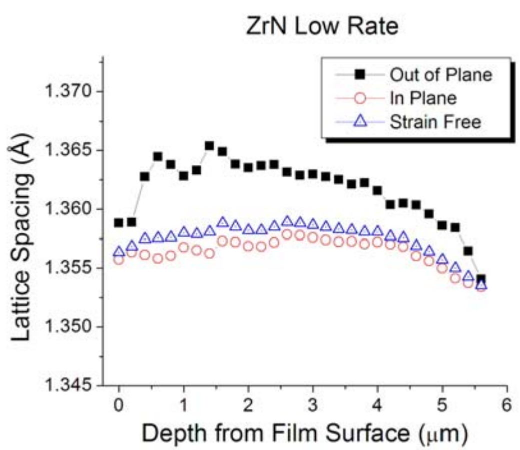
At high deposition rate, in-plane stresses are compressive as desired

Accomplishments

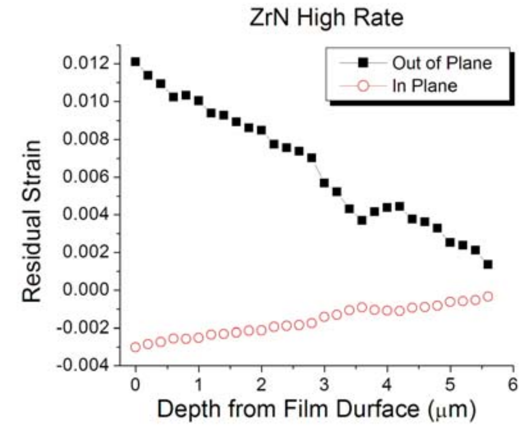
ZrN Coating on Steel Results

Lattice Spacing

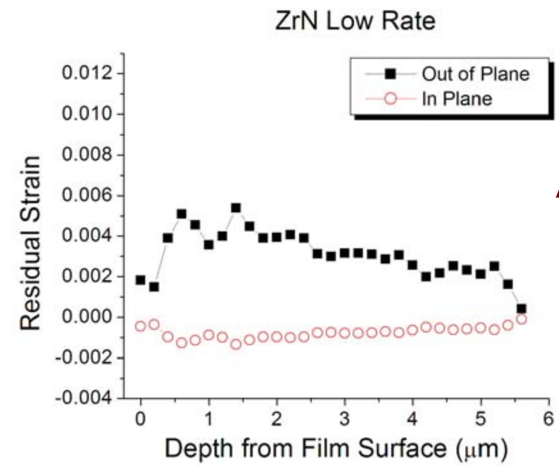




Residual Strain



HRRS process



ARE process

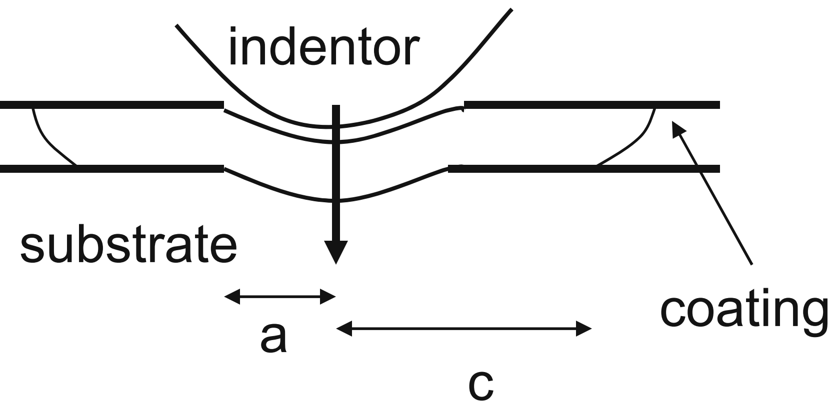
At high deposition rate, in-plane stresses are more compressive

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

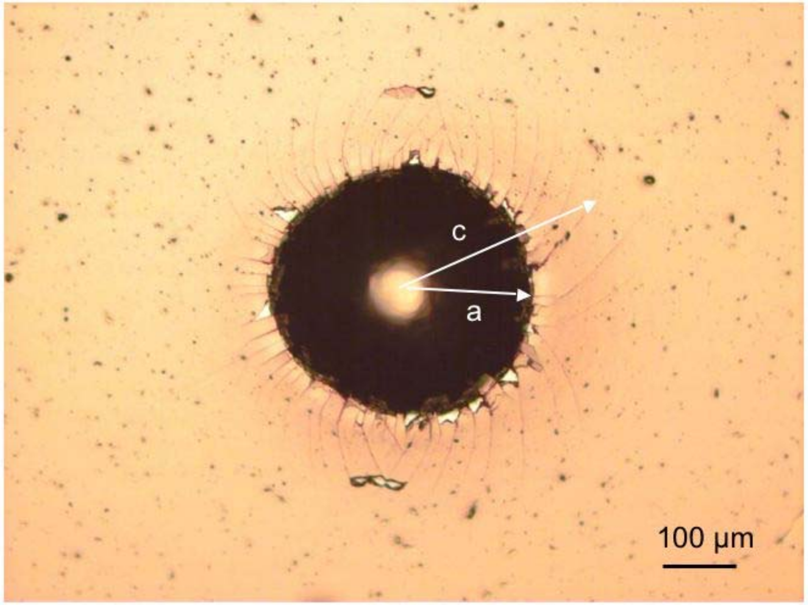


$$G_c = \left(\kappa \frac{dP}{dc} + \lambda\right) t \Big/ \left(c - a \frac{da}{dP}\frac{dP}{dc}\right)$$

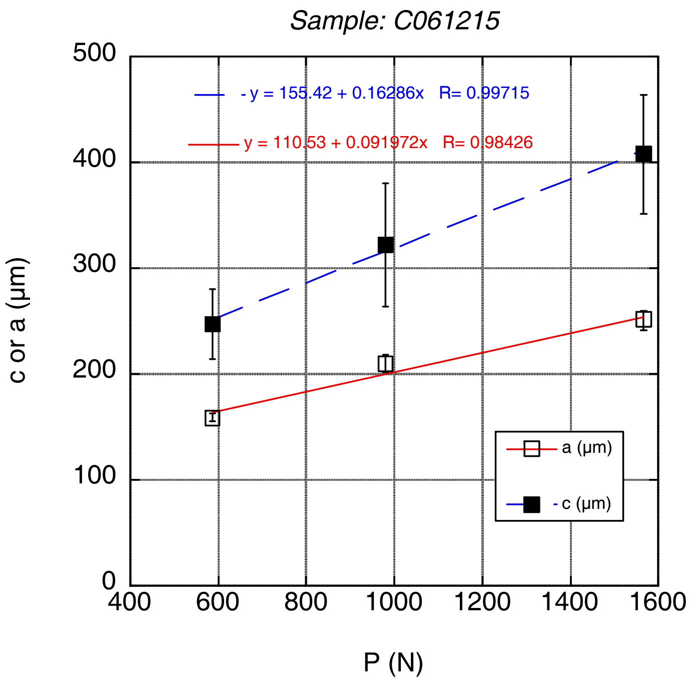
P = indentation load
t = film thickness
a = indentation radius
c = delamination radius
κ, λ = constants based on material properties, residual stresses

Accomplishments

- Adhesion Energy Measurements Using Indentation



MoNCu on Steel

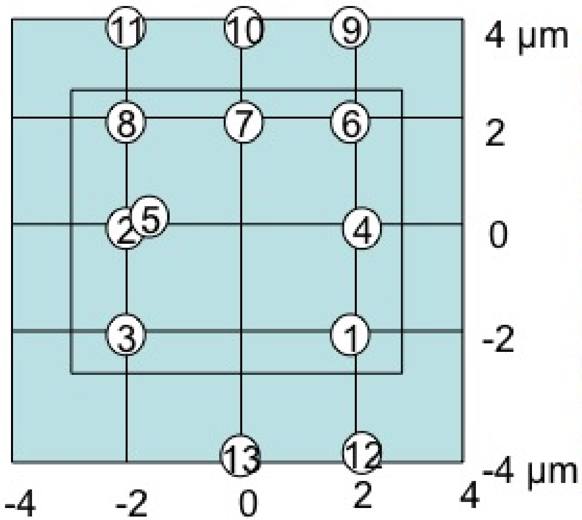


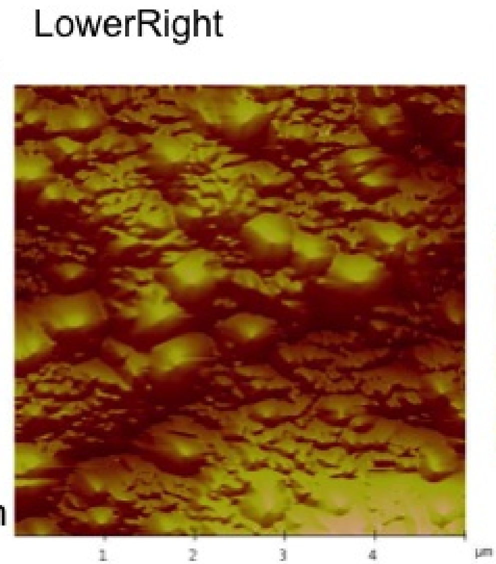
Linear variation of indentation and delamination radii with applied load

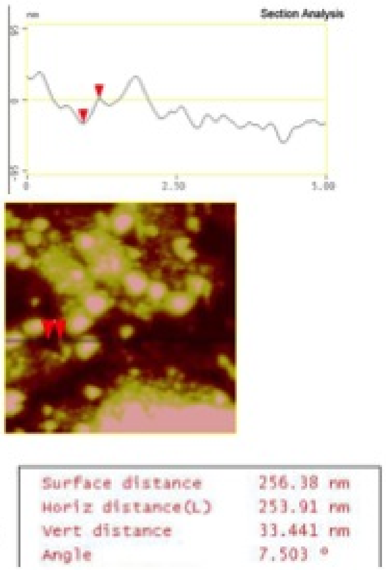
- Adhesion energy ≈ 600 J/m^2
- Similar to adhesion energies reported in literature for diamond like carbon coatings on steel
- No significant effect of the processing conditions on adhesion energy

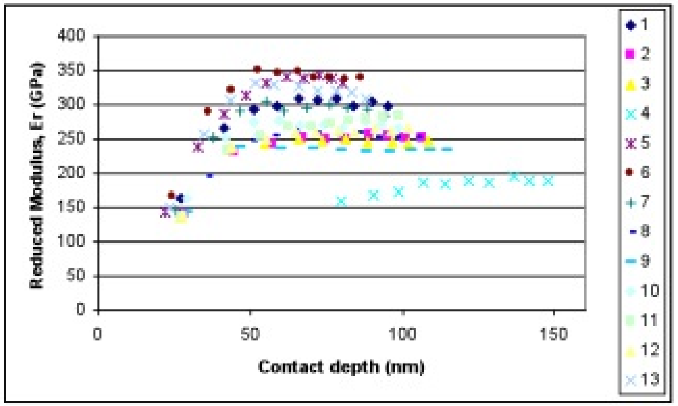
Accomplishments

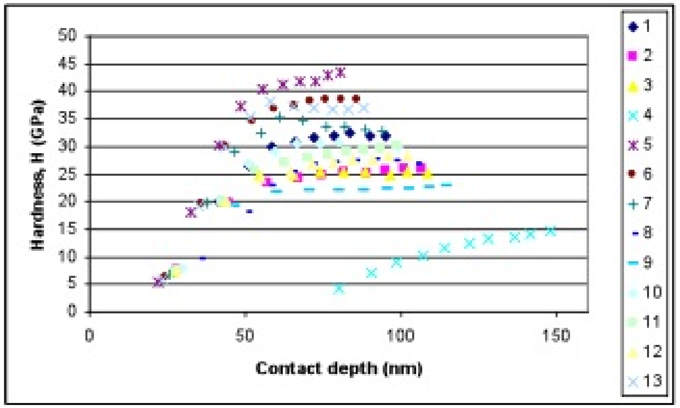
Mechanical Behavior of ZrN Coatings Characterized Using Nanoindentation











Variability of coating properties indicative of processing and potential failure locations

Path Forward

- Continue to refine and improve resolution of x-ray technique
- Explore Focused Ion Beam technique for residual stress measurements and compare with x-ray measurements
- Develop indentation technique to:
  - evaluate mechanical properties such as hardness, toughness of the coatings, surface adhesion energy
  - investigate edge-effects on adhesion of coatings
- Procure and install scratch test system for adhesion property characterizations
- Correlate the measured residual stresses in MoCuN, ZrN, TiC coatings to tribological properties and processing
- Initiate discussions with coating manufacturers for collaboration

Conclusions

- Two x-ray based techniques developed and applied for residual stress measurements
- Stresses correlated to processing conditions (deposition rates)
- Progress made in determination of coating adhesion energy
- Nano-indentation technique has been demonstrated for characterization of coating