Residual Stress Measurements in Thin Coatings

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May 11, 2011

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Vehicle Technologies – Annual Review

Project ID
pm031

Sponsored by ***Propulsion Systems Materials***

Overview

Timeline

- Project start FY07
- Project end FY12
- 80% complete

Budget

- FY09 = $100 K (DOE)
- FY10 = $200 K (DOE)
- FY11 = $67.4 K* (DOE)

*Continuing resolution, funds received

Barriers

- Role of residual stresses in thin tribological coatings and its implications to the coating performance and reliability 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 vehicle applications

Relevance

- Minimizing friction and wear in vehicle drive trains and engine components that accounts for 20-30% of fuel consumption 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 *to extend component life and reduce life-cycle costs*

- 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 & correlate it to adhesion energy and tribological properties

Objectives

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

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

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

- Transfer technology to industry

FY 10 – Measure adhesion energies and evaluate tribological behavior of MoN coatings; correlate to residual stress measurements

Milestones

- FY10 (all completed)
  - Characterize mechanical properties of MoN and MoCuN coatings using nanoindentation
  - Apply scratch testing to evaluate adhesion energy for MoCuN coatings fabricated at different processing conditions
  - Correlate measured adhesion energies to residual stresses and processing conditions (MoCuN)
  - Initiate tribological properties/performance of the MoCuN coatings

- FY11
  - Characterize mechanical properties ZrN & TiC coatings fabricated under low and high deposition rates
  - Characterize ZrN & TiC coatings for adhesion energy and tribological properties
  - Correlate the residual stresses and and coating mechanical/tribological properties for coatings fabricated under different process conditions
  - Develop collaborations with industrial producers of coated vehicle parts

Approach

- Develop/refine high energy x-rays for profiling residual strains in thin coatings by measuring the change in the lattice parameter of the coating constituents
  - 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 & rate
  - composition
- Develop nanoindentation & 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

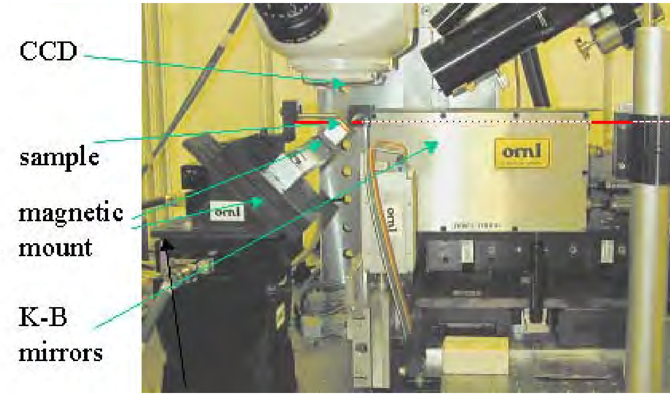
processing ⟷ **residual stresses** ⟷ **performance**

Accomplishments

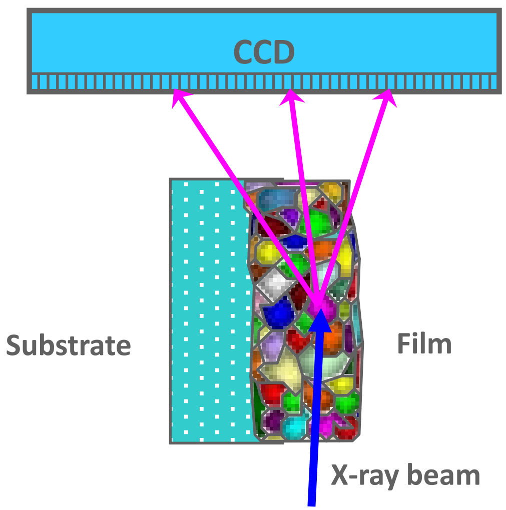
Strain measurement techniques

APS XOR/UNI
beamline 34-ID-E

Kirkpatrick-Baez mirrors
=> white or mono beam
~ 0.5 X 0.5 μm^2

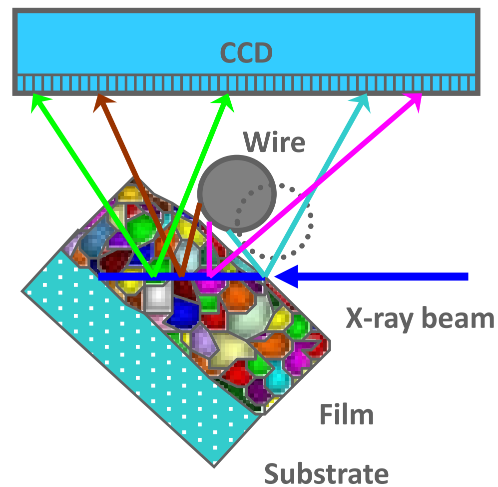


1. Cross-sectional microdiffraction



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

2. Differential aperture x-ray microscopy (DAXM)

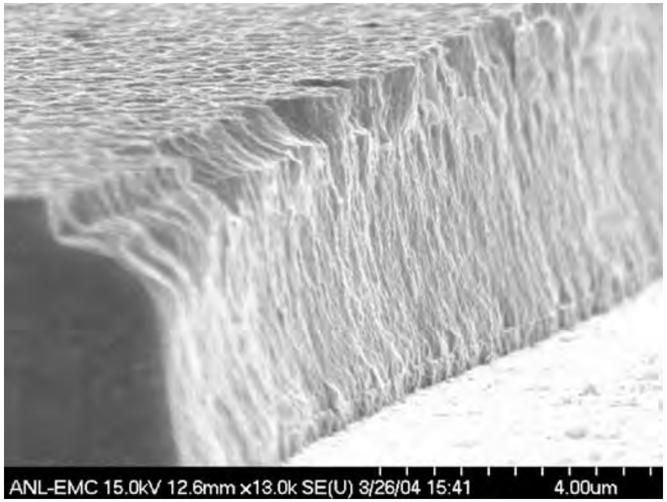


X-ray absorption wire acts as a differential aperture to separate information from different depths. (B.C. Larson et al. Nature **415**, 887 (2002)

Accomplishments

Coating systems 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

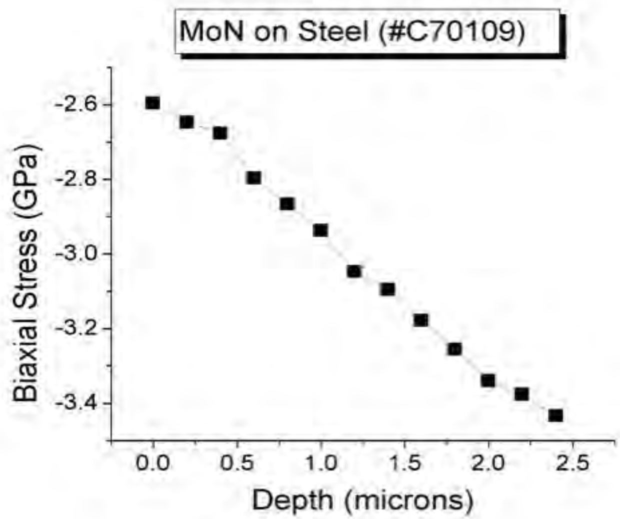
(B) TiC deposited on steel – high and low deposition rates

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

Accomplishments

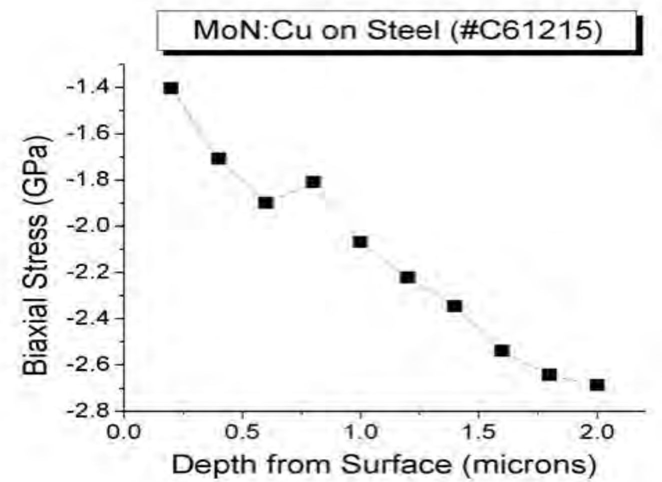
Depth-resolved stresses in MoNCu films

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

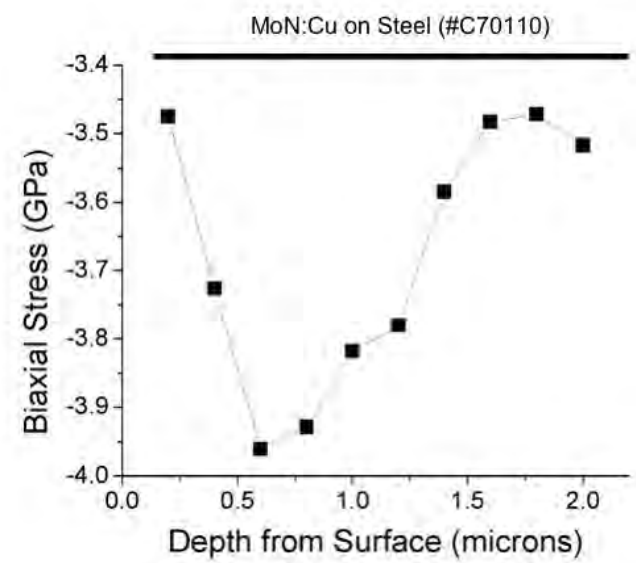


In-plane biaxial compressive stresses as a function of coating depth in the MoNCu coatings deposited on steel for various processing conditions

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

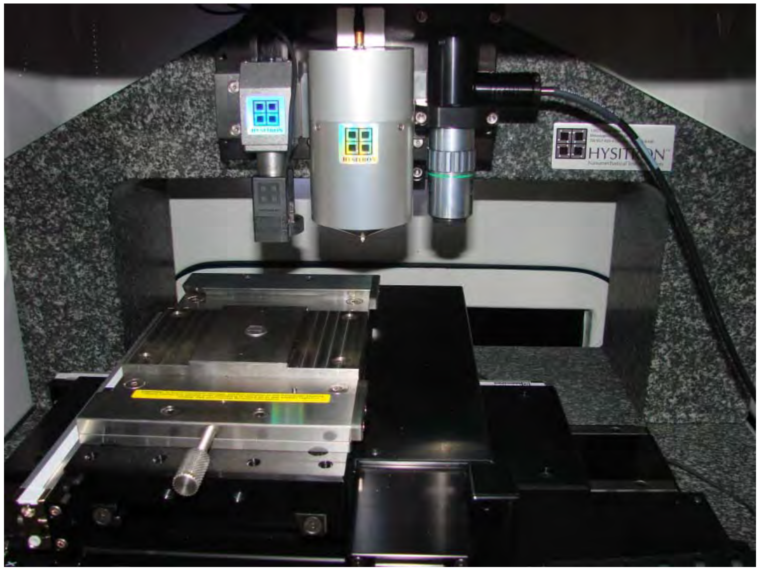


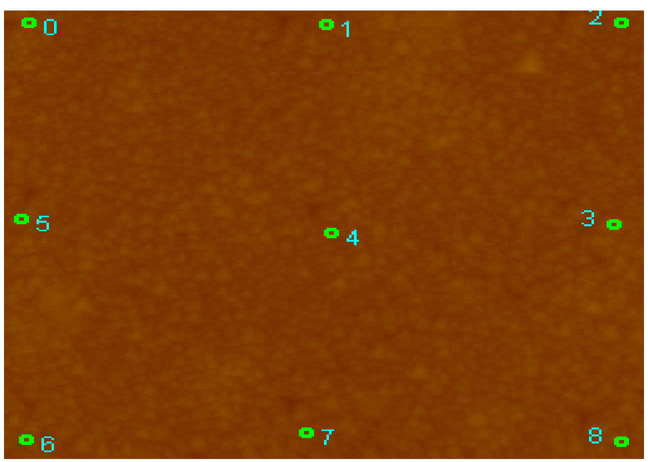
Mo: 8 kW, Cu: 0.5 kW, t = 6600 s

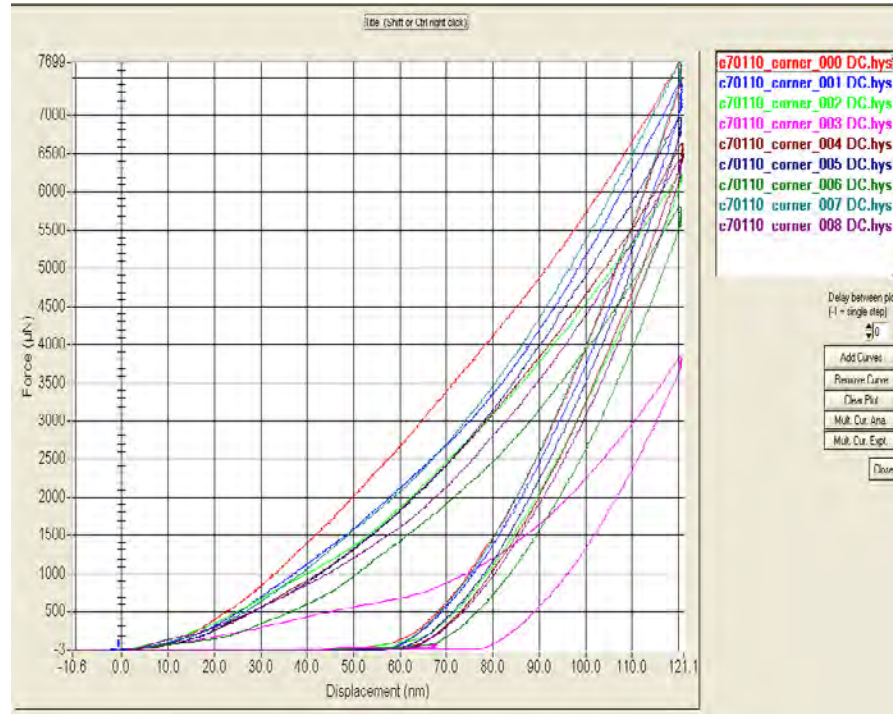


Accomplishments

Mechanical behavior of MoNCu coatings



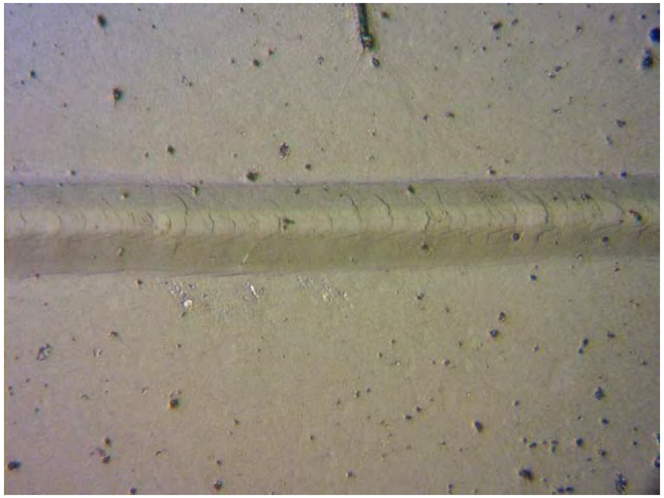




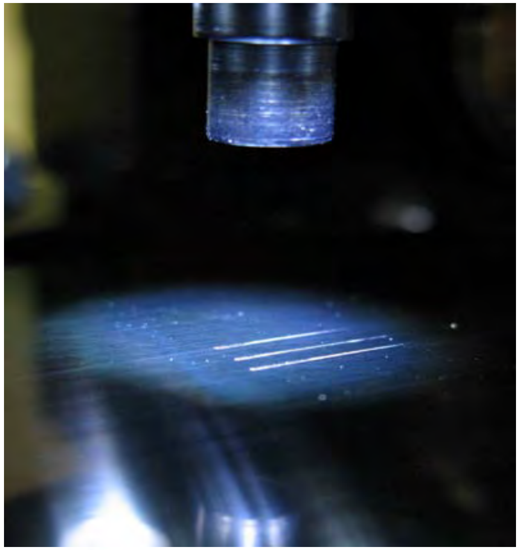
Coating	Hardness (GPa)	Modulus (GPa)
0Cu	22.4 ± 4.3	292.1 ± 28.2
5Cu	25.9 ± 5.5	317.8 ± 35.3
8Cu	23.0 ± 4.0	282.6 ± 16.0

Accomplishments

Adhesion energy measurements of MoNCu coatings



Scratch Test Track Prior to Spall

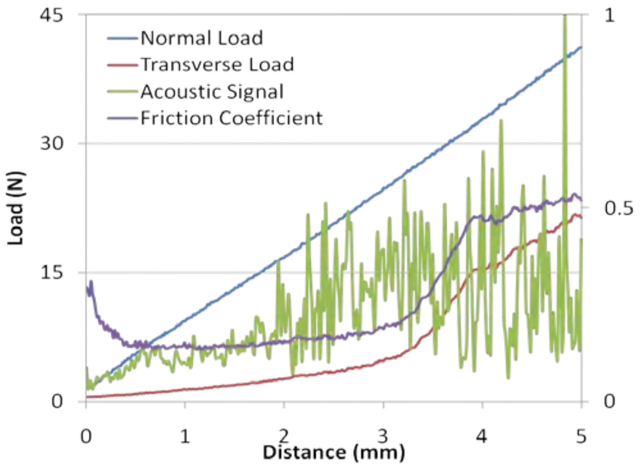


Rockwell C 125 µm stylus

Loading rate: 10 mm/min

Test length: 5 mm

Max. Load: 50 N

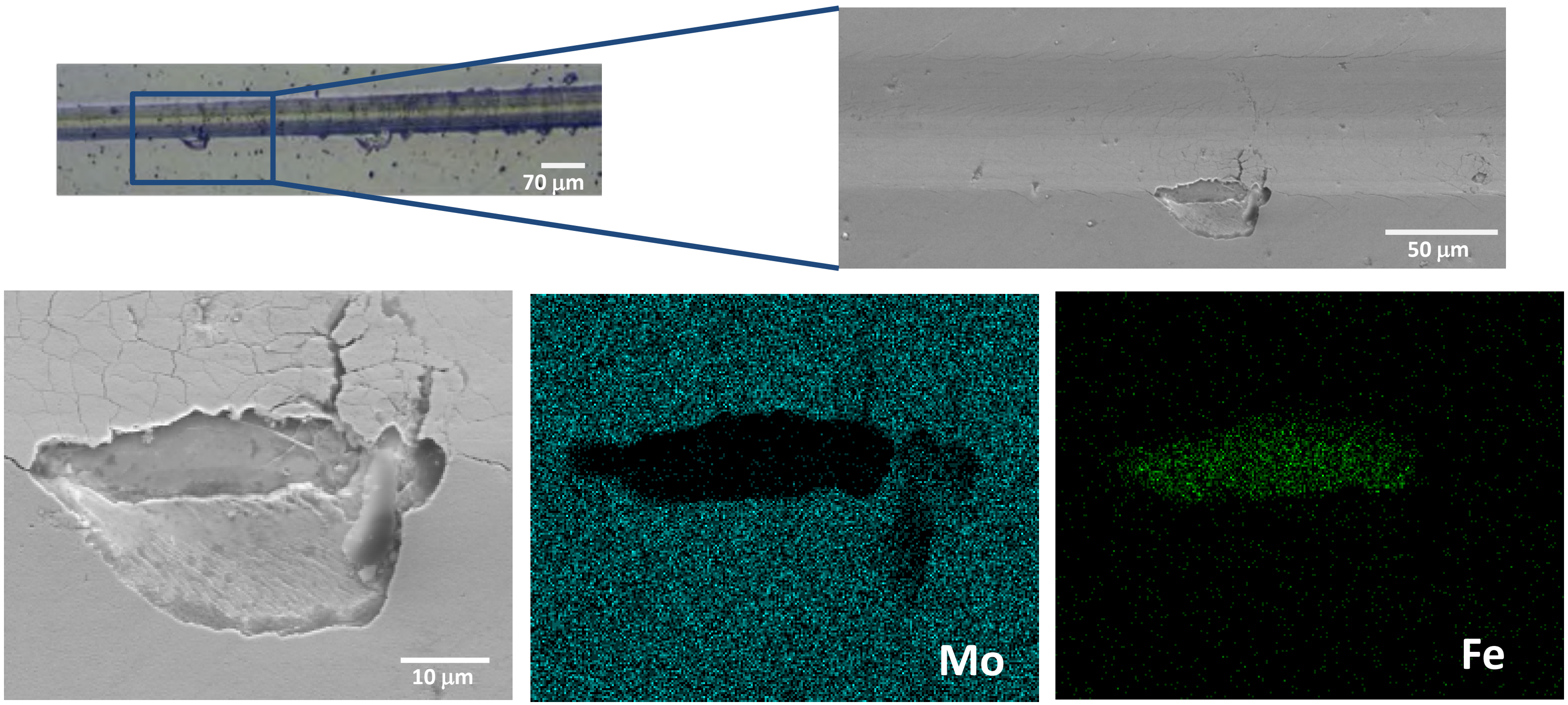


Various Data Recorded During Scratch Test

Accomplishments

Adhesion Energy Measurements

- Microscope images of first feature on 0Cu scratch track
- EDS mappings confirm coating removal- first spallation
- Correlate position on scratch track to the **critical load**



Accomplishments

Adhesion energy calculations

Xie and Hawthorne, *Surface and Coatings Technology* **141**: 15-25, 2001.

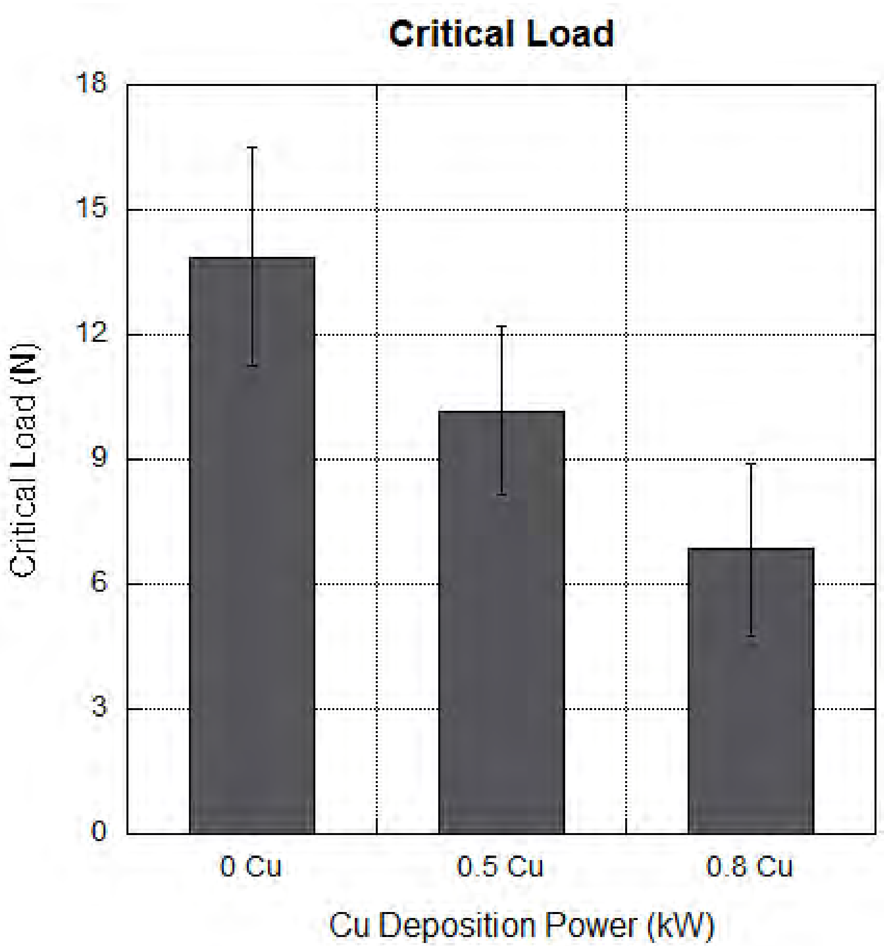
$$\sigma_S = \frac{0.15}{R}\left(\frac{PH_f}{H}\right)^{0.5} E_f^{0.3} E^{0.2}$$

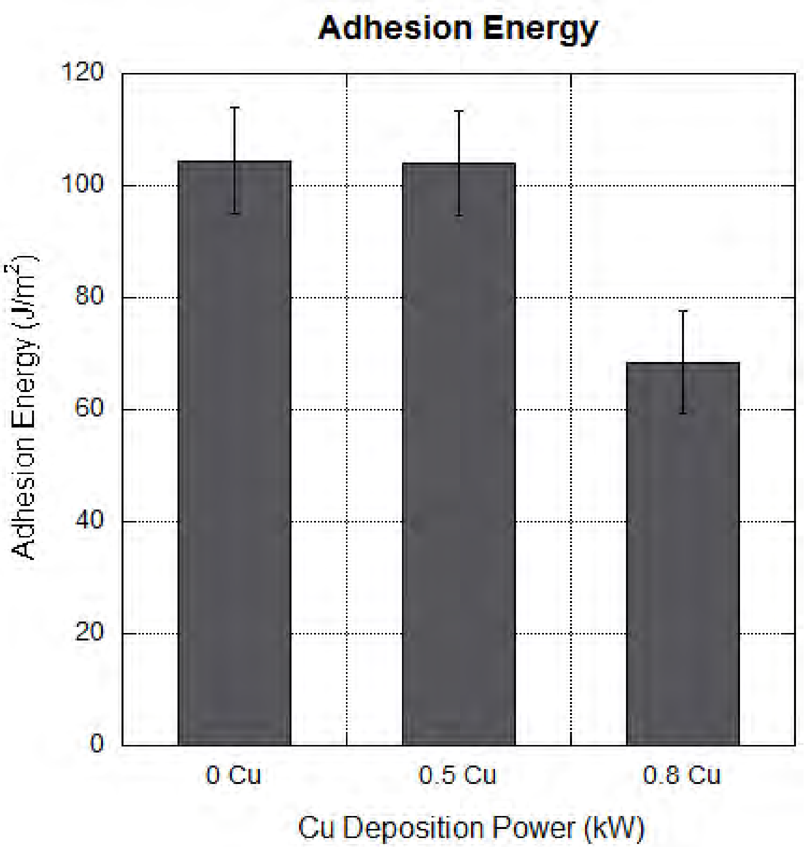
$$W = K_2(\sigma_s + \sigma_R)^2 t \frac{1-\nu_f^2}{E_f}$$

σ_S = scratch test stress
R = indenter radius
P = critical load
H_f = coating hardness
H = substrate hardness
E_f = coating modulus
E = substrate modulus
W = Adhesion energy
K_2 = constant (for spallation = .343)
$\sigma = \sigma_R + \sigma_S$ = total stress
σ_R = residual stress
t = coating thickness
ν_f = coating Poisson's ratio

Accomplishments

Critical load & adhesion energy for various Cu deposition powers

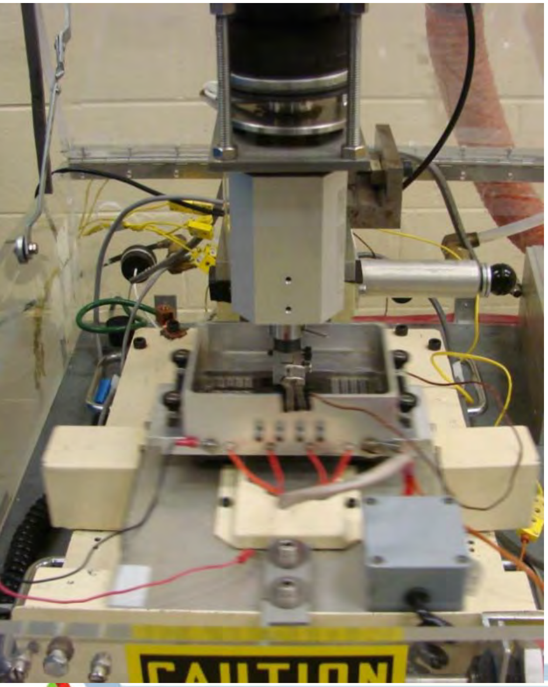


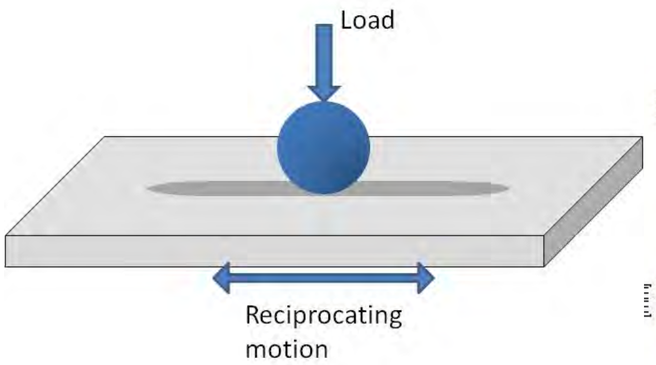


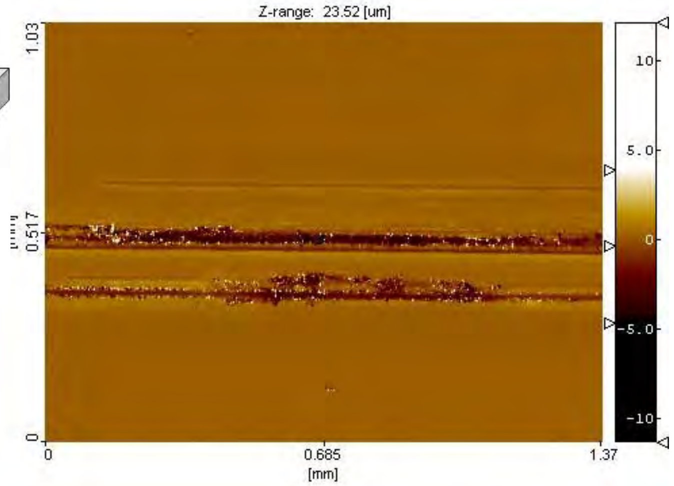
Accomplishments

Wear rate

- Friction and wear resistance determined using ball-on-flat configuration
  - Used silicon nitride ball, no lubricant, 30 minutes at 30 rpm
  - Tested each coating at 25N & 50 N load (F)
  - Total volume material removal (V) determined using profilometer
  - Calculate wear rate (WR) from V, F and total distance of wear test (l)
- Friction coefficient decreases slightly with increasing copper content

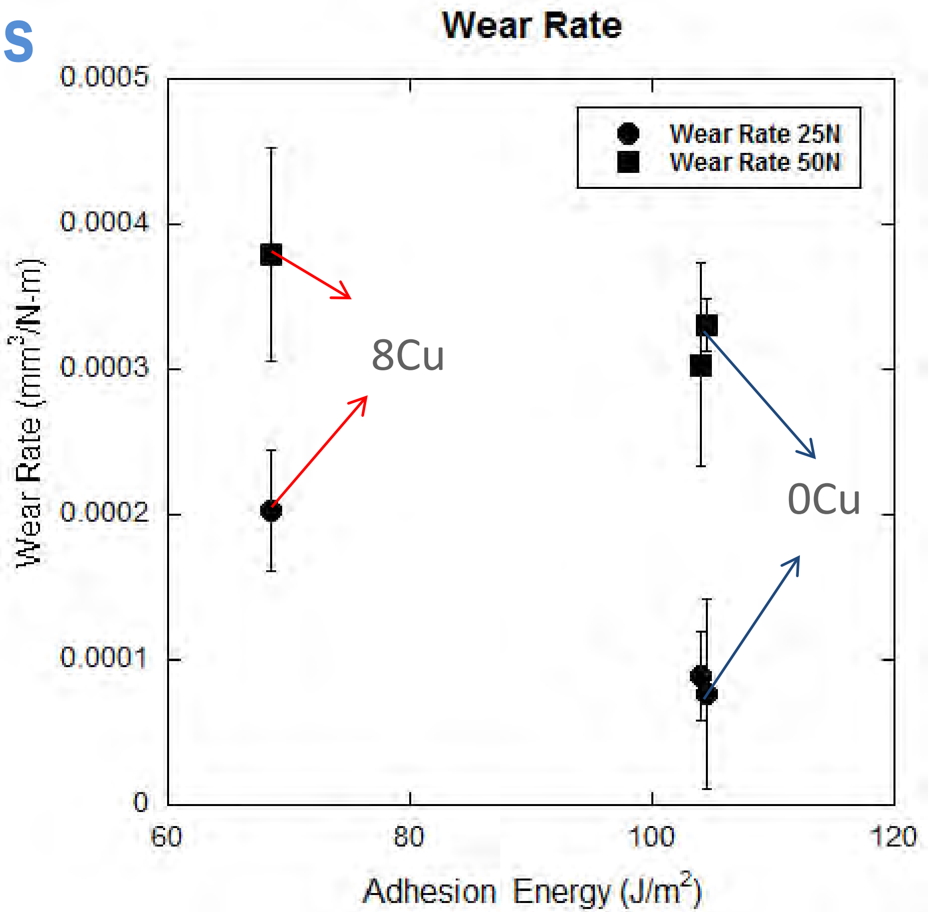






Accomplishments

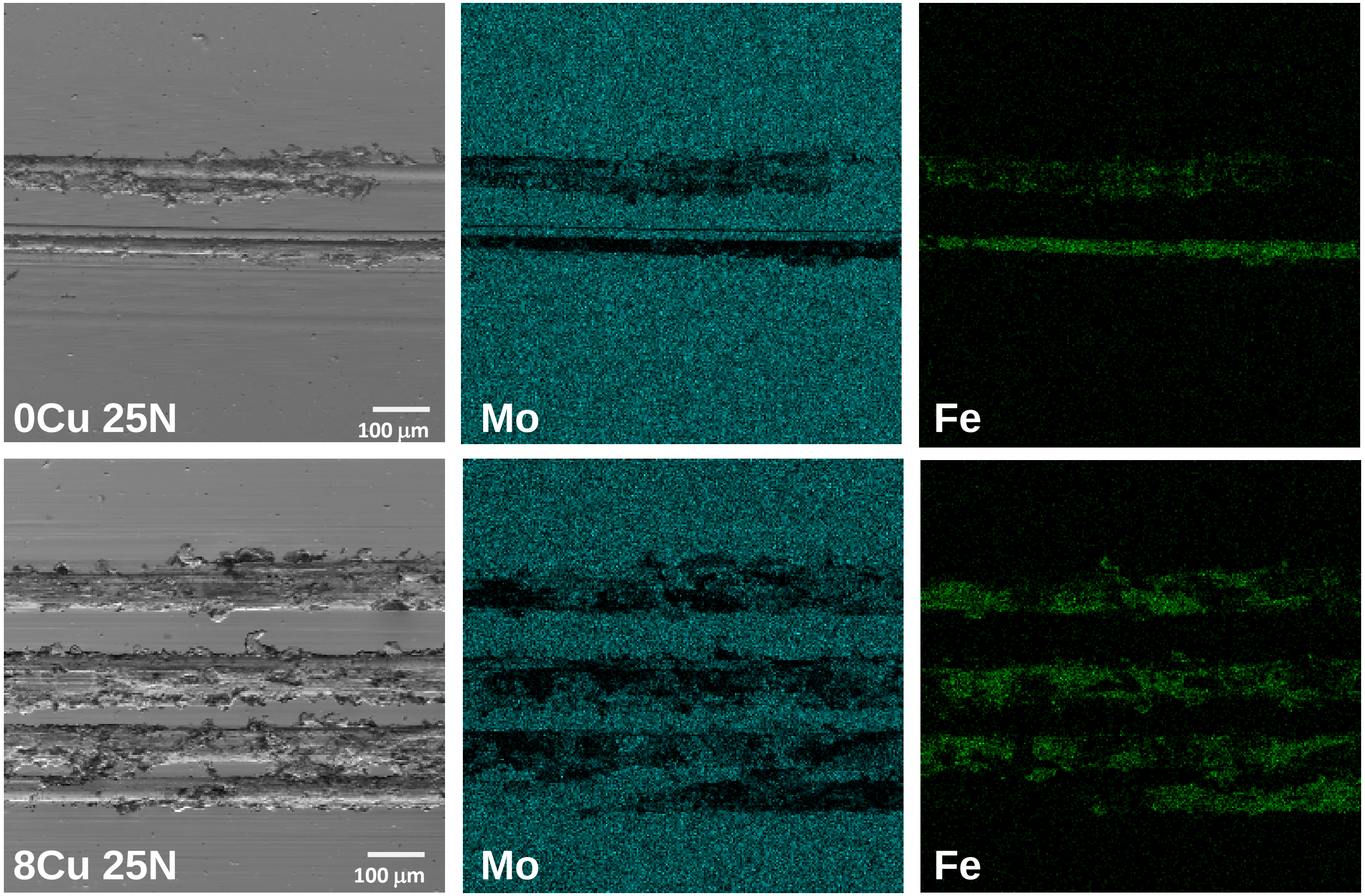
Wear rate



- Wear rate highest for 8Cu, lower and similar for 0Cu and 5Cu
  - Wear rate decreased with higher adhesion energy
  - Wear rate increased with increasing Cu content

Accomplishments

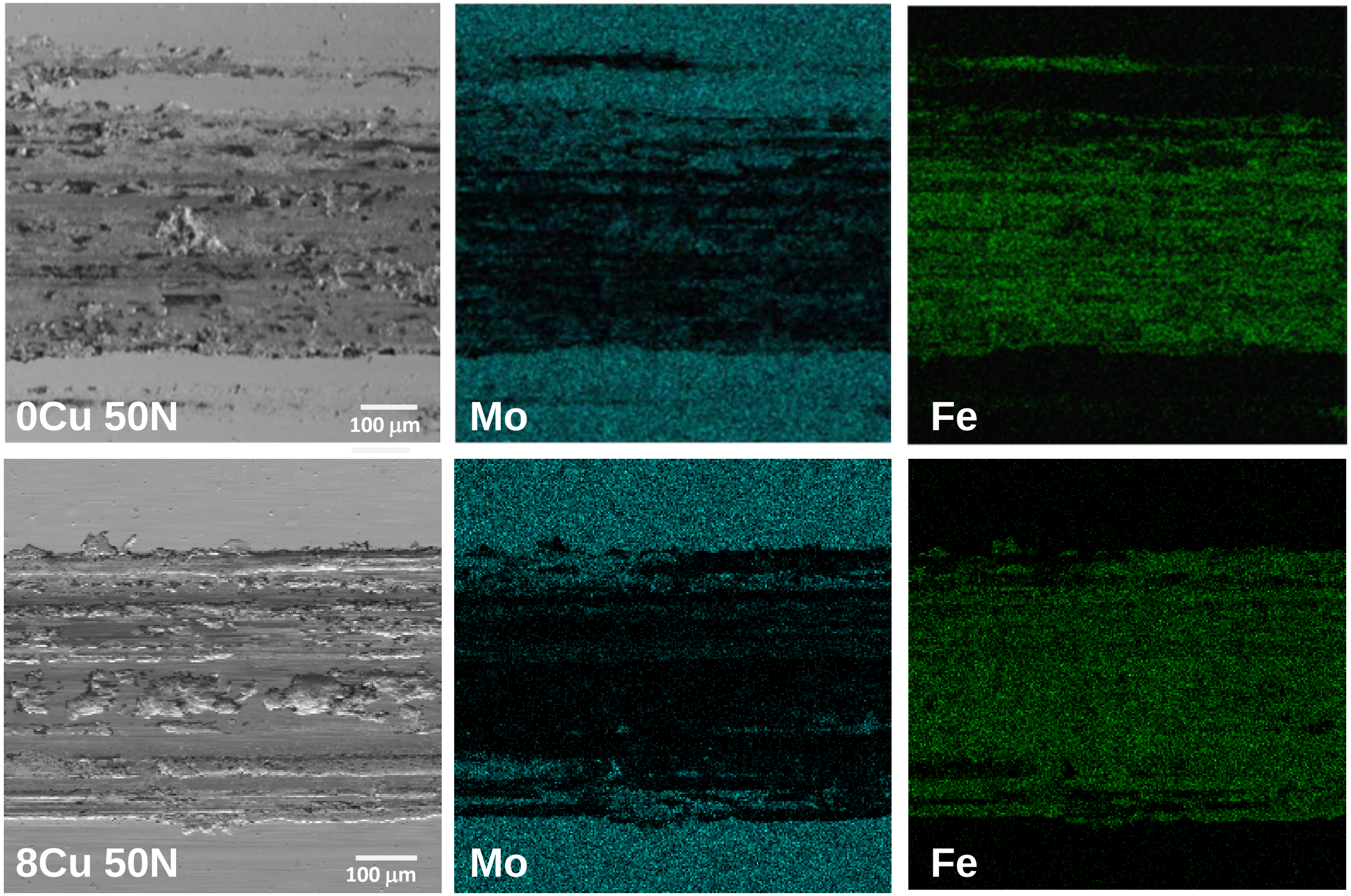
Wear tracks: 25N



- SEM and EDS mappings correlate well with wear rate values
- More material removed for 8Cu than 0Cu and 5Cu

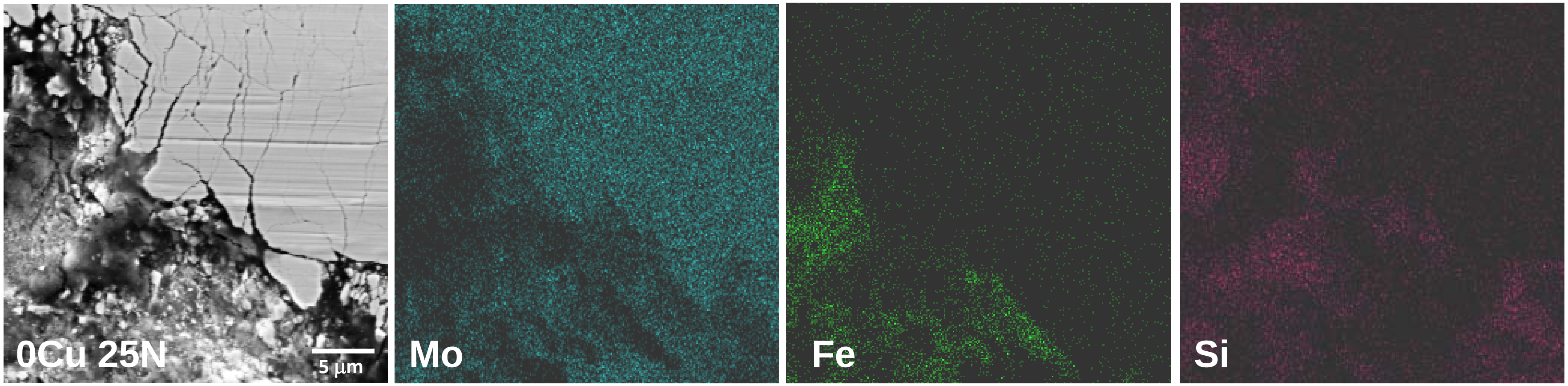
Accomplishments

Wear tracks: 50N

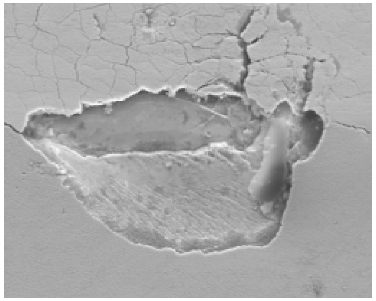


- At 50N, much more material removed compared to 25N
- 0Cu and 8Cu have similar degrees of coating removal

Wear Damage Mechanisms



- Wear damage mechanism investigated using SEM/EDS
  - At 25N, coating mostly still present, though removed in many spots
  - Many damage mechanisms present simultaneously
    - Abrasion and polishing: lines in coating track
    - Chipping: cracks in coating
    - Delamination: removal of coating from the substrate
    - Reaction with Si_3N_4 ball: Si presence



Spallation observed during scratch test

Damage mechanism during scratch and wear tests are similar

Collaborations

- Since we have now completed correlations between residual stresses, adhesion energy, and tribological properties of in-house produced coating system, we will now actively work with one of our industrial partners

- Initiated discussions on technology with industrial partners to obtain test samples

- U of Ohio, Athens, OH – residual stress analysis

Path Forward

- Complete adhesion energy evaluations for TiC and ZrN
- Complete mechanical properties of TiC and ZrN coated samples for varying processing conditions
- Measure tribological performance for TiC, and ZrN coated samples
- Correlate the measured residual stresses in ZrN, TiC coatings to tribological properties and processing
- Initiate discussions with coating manufacturers for collaboration

Conclusions

- Cross-sectional microdiffraction have been used for studying strain gradient in nanocrystalline MoNCu films deposited on silicon and steel substrates. As-deposited MoN film is under in-plane compression.

- Effects of Cu additions on coating adhesion and tribological performance was investigated.
  - does not affect coating hardness or modulus
  - reduces the residual stresses
  - decreases the adhesion of coating to steel substrate
  - decreases wear resistance of the coating
  - delamination mechanism during scratch and wear tests appear to be similar

- Coating processing variables and resulting properties are correlated to their structure and can be used to optimized for enhanced tribological performance by optimizing adhesion energy.