Residual Stress Measurements in Thin Coatings

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Overview

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

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

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 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

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))

APS XOR/UNI beamline 34-ID-E
Kirkpatrick-Baez mirrors => white or mono beam
~ 0.5 X 0.5 μm²
Accomplishments

Coating systems investigated:

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

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

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

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

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Mo &amp; Cu Deposition Power (kW)</th>
<th>Deposition Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C70109</td>
<td>Mo:8; Cu:0</td>
<td>7200</td>
</tr>
<tr>
<td>C70110</td>
<td>Mo:8; Cu:0.5</td>
<td>6600</td>
</tr>
<tr>
<td>C61215</td>
<td>Mo:8; Cu:0.8</td>
<td>7200</td>
</tr>
</tbody>
</table>
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
Accomplishments
Mechanical behavior of MoNCu coatings

<table>
<thead>
<tr>
<th>Coating</th>
<th>Hardness (GPa)</th>
<th>Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0Cu</td>
<td>22.4 ± 4.3</td>
<td>292.1 ± 28.2</td>
</tr>
<tr>
<td>5Cu</td>
<td>25.9 ± 5.5</td>
<td>317.8 ± 35.3</td>
</tr>
<tr>
<td>8Cu</td>
<td>23.0 ± 4.0</td>
<td>282.6 ± 16.0</td>
</tr>
</tbody>
</table>
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


\[
\sigma_s = \frac{0.15}{R} \left( \frac{PH_f}{H} \right)^{0.5} E_f^{0.3} E^{0.2}
\]

\[
W = K_2 \left( \sigma_s + \sigma_R \right)^2 t \frac{1 - \nu_f^2}{E_f}
\]

- \(\sigma_s\) = 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
- \(\sigma_R\) = residual stress
- \(t\) = coating thickness
- \(\nu_f\) = 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 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$_3$N$_4$ ball: Si presence

**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.