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Materials Engineering

Characterization of PVD Coatings in High Pressure Diesel Fuel Injection Systems

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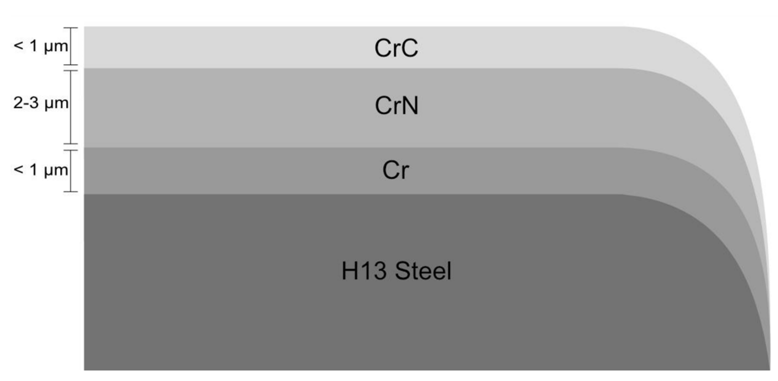
A characterization of a CrN multi-layer coating was completed on behalf of Cummins Fuel Systems. Three samples, each subjected to a different post-grind polishing method, were examined. The following properties were tested in each sample: residual stress, hardness, elastic modulus, surface roughness, and adhesion. Based on adhesive performance, sample 12 proved superior to samples 4 and 8, because less cracking and no delamination occurred. Greater variation in residual stresses present in the coatings are likely what resulted in more extensive cracking and delamination in samples 4 and 8. A large variation in hardness and elastic modulus values, similar to the variation for the residual stress, was seen in samples 4 and 8.

This work is sponsored by Cummins Inc., Columbus, IN

Project Background

- Cummins Fuel Systems employs a German coating supplier to coat hardware in their diesel fuel injection systems using physical vapor deposition (PVD).
- The mechanical properties and effects of coating processing are not well understood.
- Coated hardware is performing worse with respect to wear resistance than uncoated hardware.



Coatings present on each sample with their respective thicknesses.

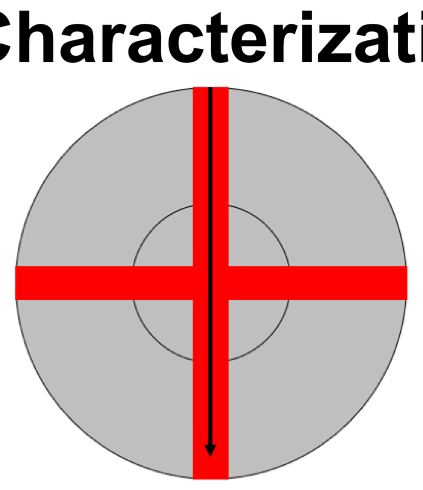
Objective: Perform a comprehensive characterization to identify why coated hardware is performing worse than uncoated hardware.

Materials

- All samples had the following characteristics:
  - H13 substrate
  - Vacuum core hardened with gas quench
  - Gas nitrided at 450°C for 10 hours
  - 50 μm of surface removed during grinding

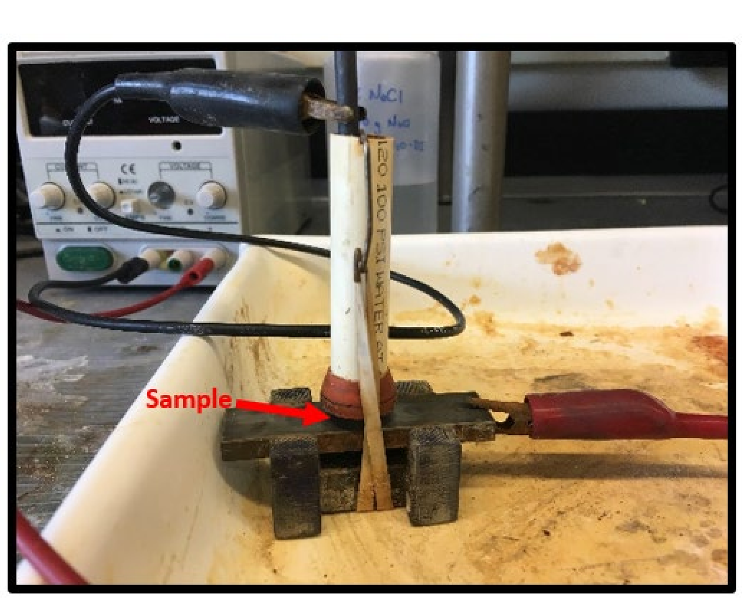
Sample	Post-grinding Polishing
4	None
8	Plasma Electrolytic Polishing
12	Tape Polishing

Characterization Methods



Regions (red) where residual stress was measured. The grinding direction is indicated by the black arrow.

- **Residual Stress** was measured using the Pulstec μ-X360, with a beam angle set to 30°, measuring perpendicular to the grinding direction.
- Residual stress gradients for samples 4 and 12 were measured after the substrate was incrementally removed using electropolishing.



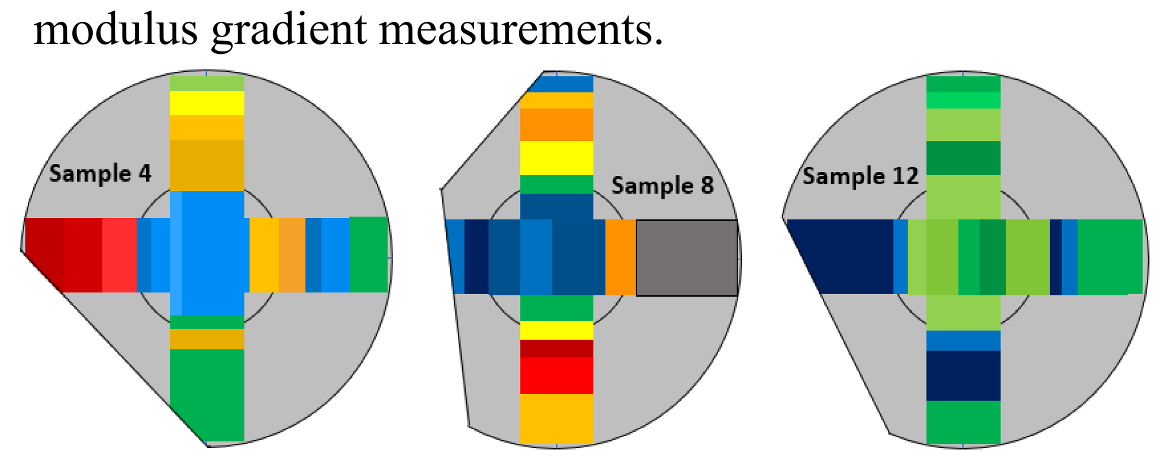
Electropolishing setup used to remove material from center of samples 4 and 12

- **Hardness/Elastic Modulus** measurements were made by nanoindentation on a Hysitron TriboIndenter with a Berkovich tip using loads of 5mN to 30 mN.
- **Adhesion** was qualitatively assessed via an indentation test. Optical images were captured to measure indent radius and crack length using ImageJ.
- **Surface Roughness** R_a and R_z were measured from 10, 20, 50, and 100μm scans using a Bruker Multi-mode AFM and Gwyddion.

Results

RESIDUAL STRESS

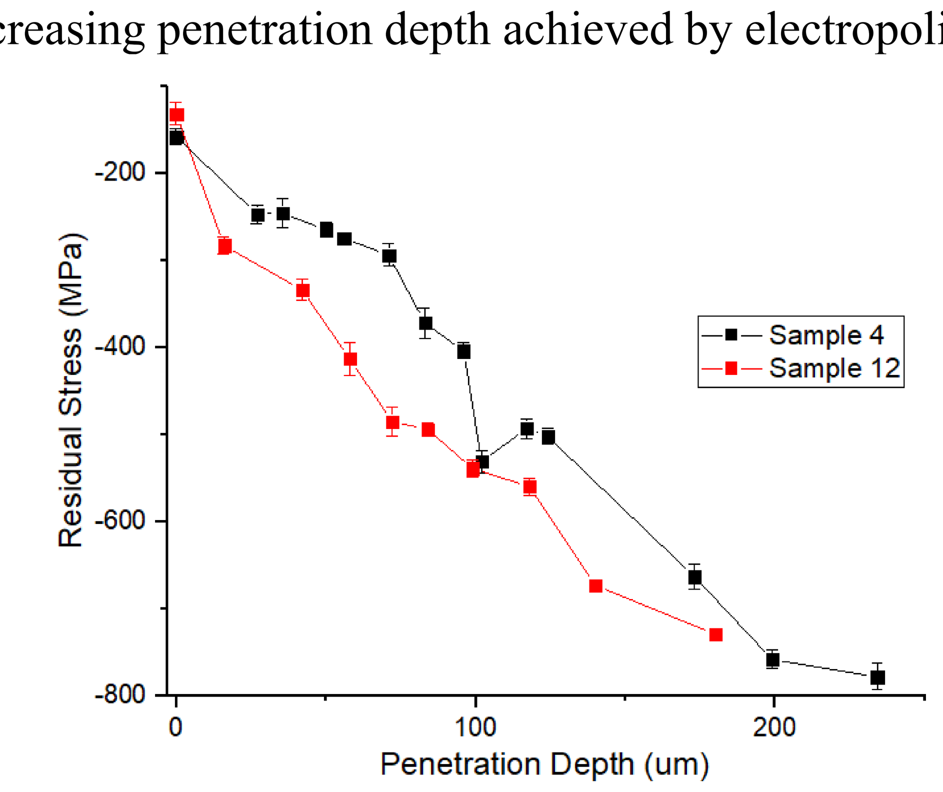
The surface measurements shown indicate the level of internal stresses in the steel substrate just below the multi-layer coating. Missing portions of each circle correspond to areas that were removed for hardness and modulus gradient measurements.



Tension (MPa)	Compression (MPa)
0 to 50	0 to (-50)
50 to 150	(-50) to (-150)
150 to 300	(-150) to (-300)
300 to 500	(-300) to (-500)

Residual stress mapping across surfaces of samples 4, 12, 8.

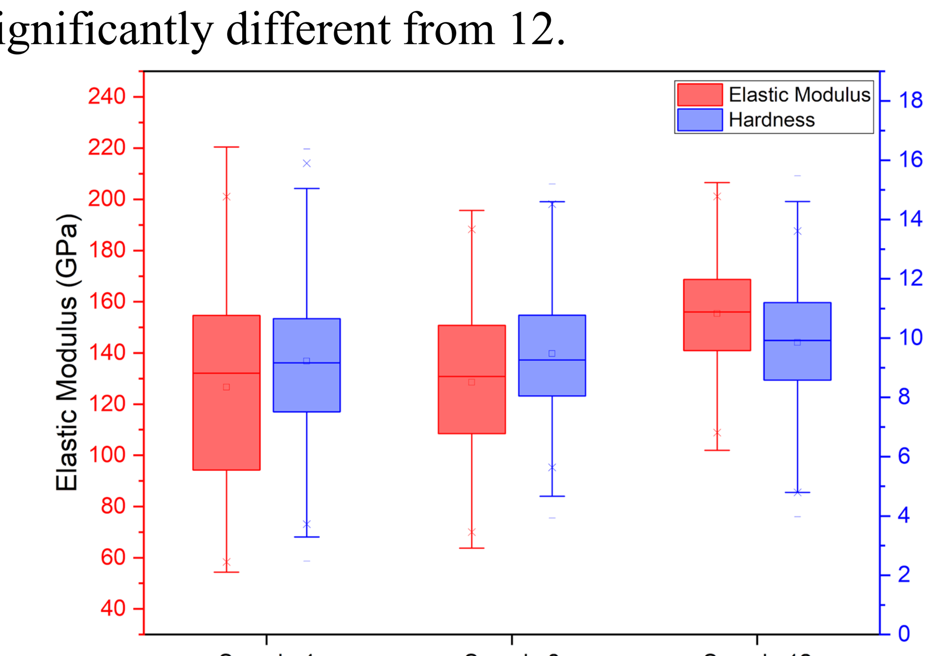
The residual stress in the substrate increased with increasing penetration depth achieved by electropolishing



Residual stress change with respect to penetration depth in the sample substrate.

HARDNESS and ELASTIC MODULUS

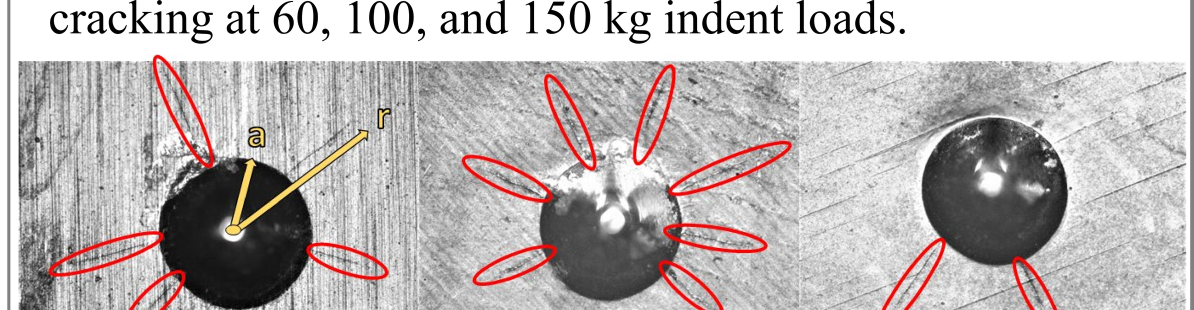
For a 95% confidence interval, samples 4 and 8 are not significantly different from each other, but they are both significantly different from 12.



A comparison of the elastic modulus and the hardness obtained from nanoindentation of the coating for each sample type.

ADHESION

Samples 4 and 8 exhibit cracking and delamination at 60, 100, and 150 kg indent loads. Sample 12 exhibits cracking at 60, 100, and 150 kg indent loads.



From left to right at 100x magnification: samples 4, 8, and 12 with indents made at 150kg. Cracks are highlighted by red ovals. On sample 4, 'a' and 'r' denote indent radius and indent radius plus crack length, respectively.

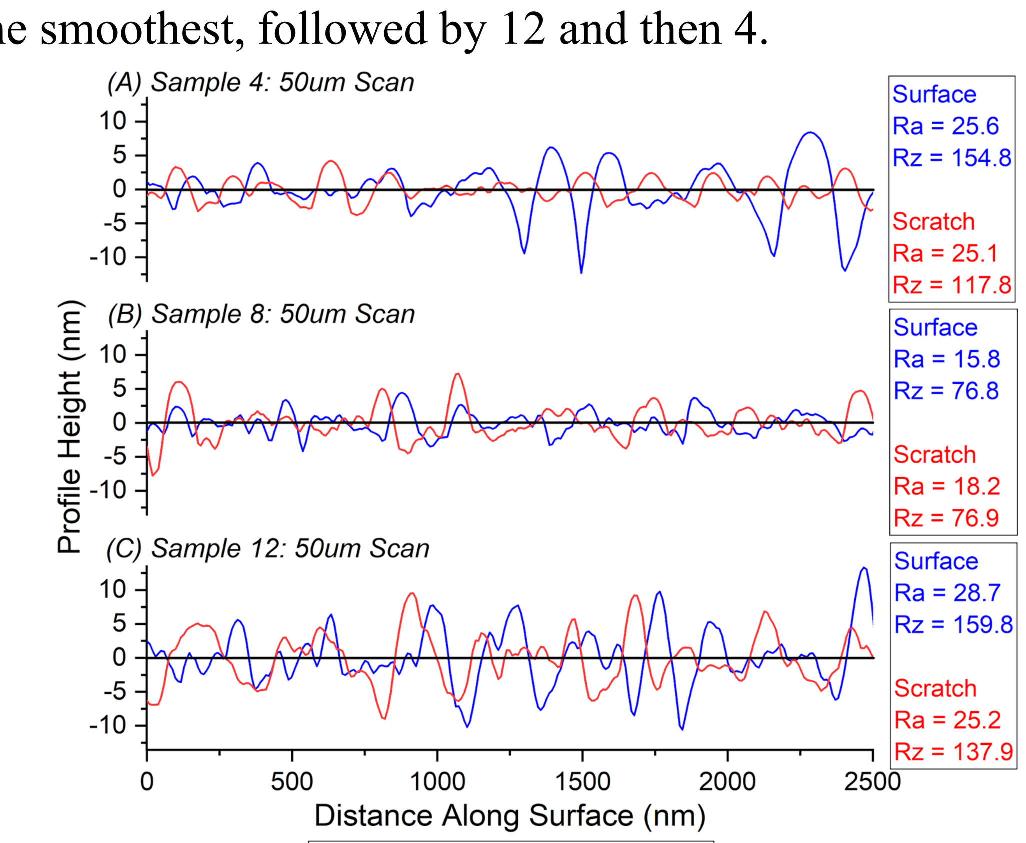
Average crack length, average indent radius, and crack ratio for samples 4, 8, and 12 at 60kg, 100kg, 150kg indent loads.

Sample	Load [kg]	Average Crack Length [μm]	Average Indent Radius [μm], a	Crack Ratio, r/a
4	60	14.128	24.174	1.58
	100	5.895	16.677	1.35
	150	19.187	20.622	1.93
8	60	6.547	12.201	1.53
	100	6.570	8.266	1.79
	150	11.097	10.607	2.10
12	60	5.557	6.304	1.88
	100	7.334	8.118	1.90
	150	9.448	10.270	1.92

SURFACE ROUGHNESS

For a 95% confidence interval, microscale (<50μm) roughness measured in visible scratches was lower than values taken outside of scratches (on the surface), which is indicative of differences in initial film growth. Macroscale (≥50μm) scratch and surface roughness is indistinguishable in all samples.

Microscale roughness for 4 and 8 were comparable, while 12 was larger than both. At the macroscale, 8 was the smoothest, followed by 12 and then 4.



Roughness profiles at 50μm parallel to scratch direction

Discussion

- Sample 12 exhibited the least variation and greatest uniformity in property measurements. A single conclusion or average value for each property was more easily converged upon for sample 12 than for samples 4 and 8.
- Residual stress in samples 4 and 8 have regions of both high tension and high stress. Such variation effects adhesion and crack behavior across the surface. Areas of the coating in tension are more susceptible to film fissure and areas in compression are more likely to experience delamination.

Recommendations

- Tape polishing correlates with uniform property measurements, and should be used as the preferred post-grind polishing method
- Residual stress mapping should be used to predict coating fracture behavior