Weapon Systems & Technology Directorate
US ARMY ARDEC- BENÉT LABS

TITLE: PLASMA ENHANCED MAGNETRON TECHNOLOGY FOR DURABLE POLLUTION-FREE COATINGS

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

Dr. Sabrina Lee and Dr. Rong Wei
NDIA Gun and Missile Conference, New Orleans, LA
April 21-24, 2008

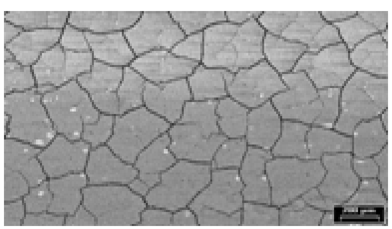
Outline

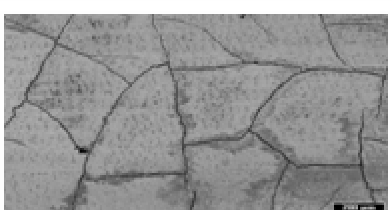
- Problems with HC Cr electroplating process.
- Alternative pollution-free coatings against high temperature wear and erosion.
- Plasma enhanced magnetron surface cleaning.
- Plasma enhanced magnetron deposition.
- Analytic characterization and adhesion testing.
- Conclusion

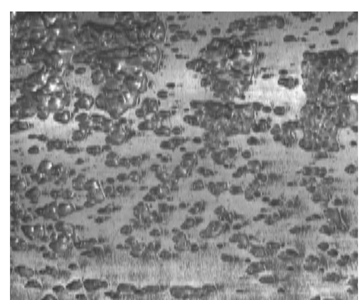
Problems with Electroplated Cr

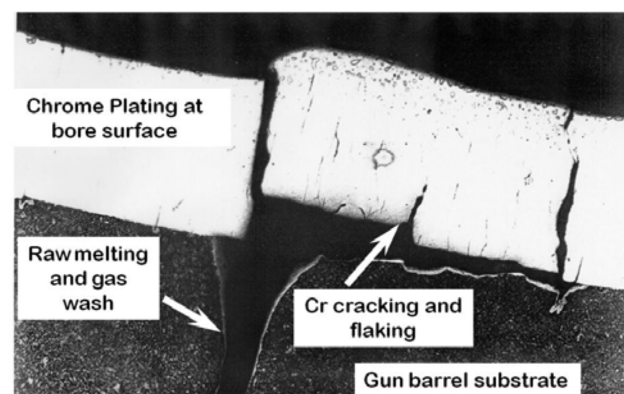
Environment Problem: Aqueous toxic Cr VI from Cr Electroplating Process

Performance Problem: Inadequate substrate protection, reduced service life for high temp wear & erosion applications



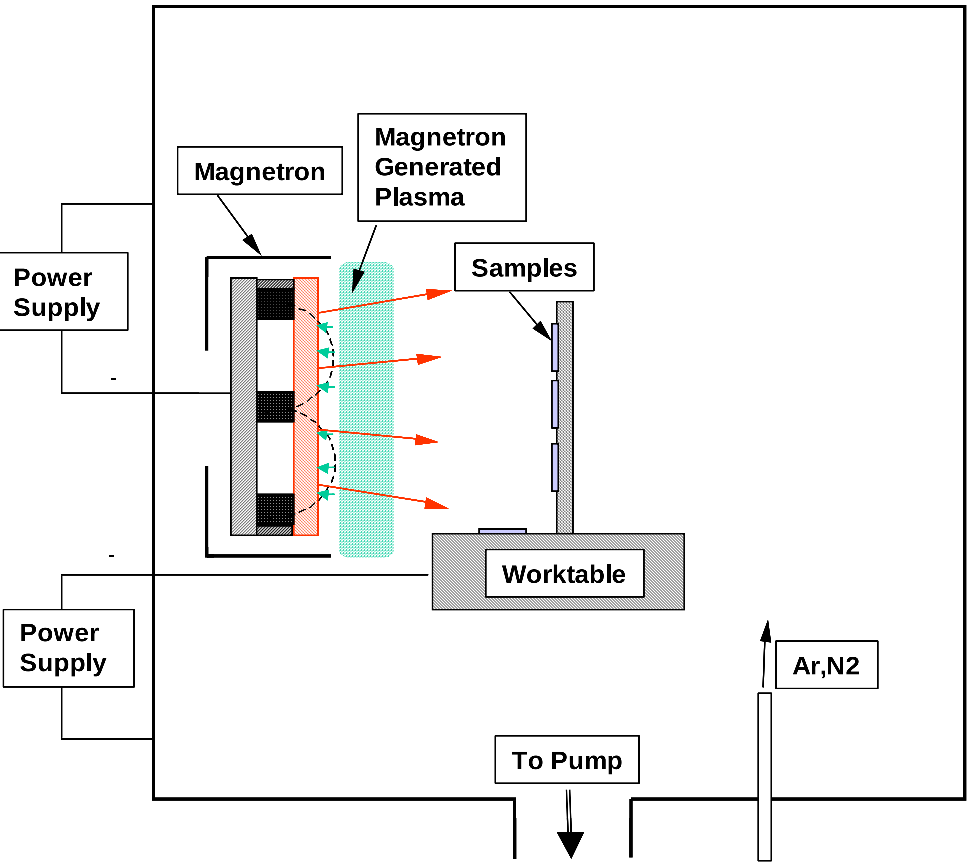




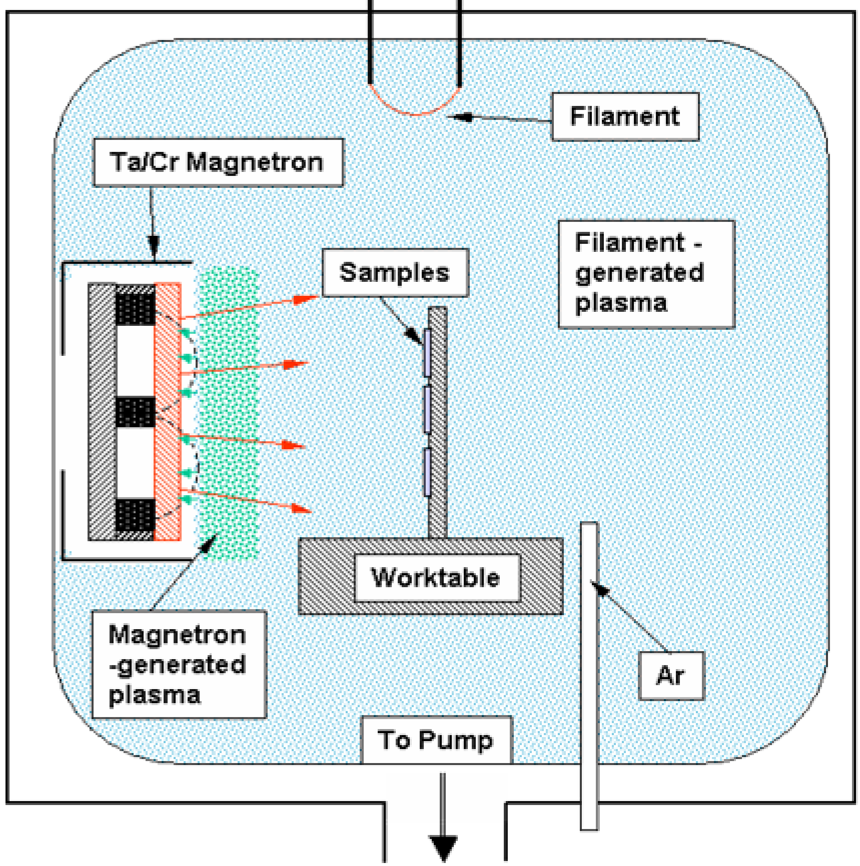


Plasma Enhanced Magnetron Sputtering

Conventional Physical Vapor Deposition via DC Magnetron Sputtering



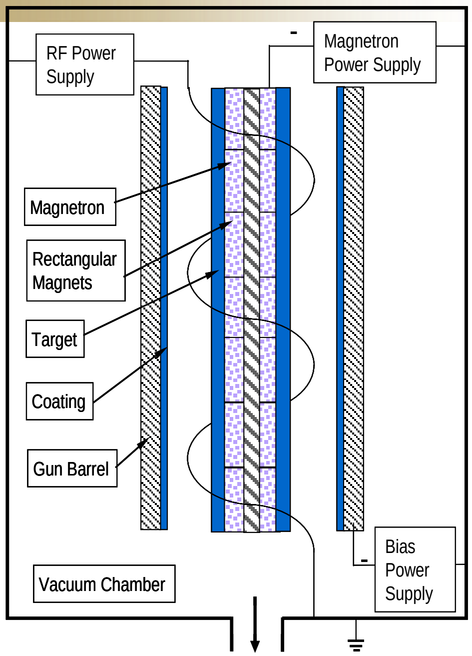
Plasma Enhanced Magnetron Sputtering with Filament and Biasing at SwRI

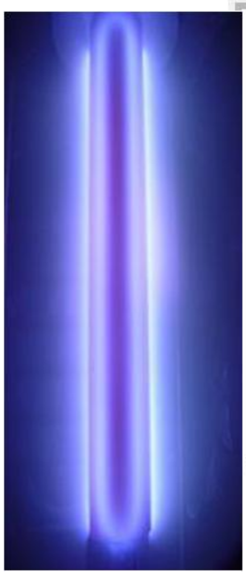


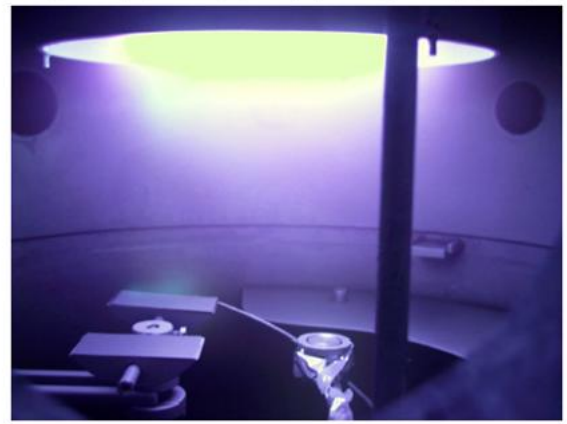
Filament Plasma + Magnetron Plasma ➡ Current Density ~ 4.9 mA/cm^2

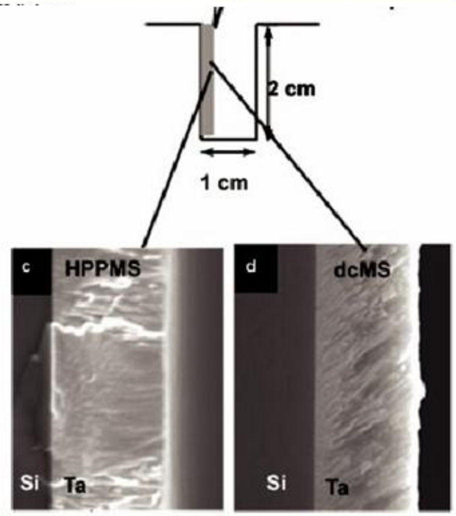
Magnetron Plasma without Filament Plasma ➡ Current Density ~ 0.2 mA/cm^2

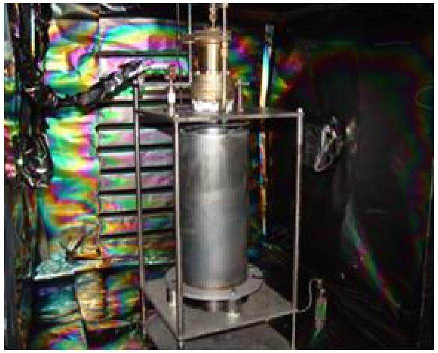
Planned Plasma Enhanced Cylindrical Magnetron Deposition





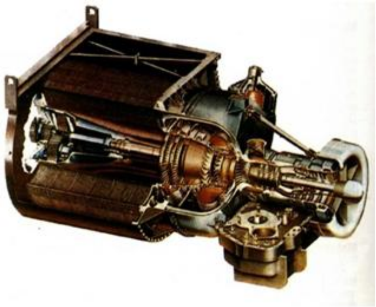






LEFT- RF-plasma enhanced cylindrical magnetron sputtering experiments at SWRI.
MIDDLE- HIPIMS (high power impulse magnetron sputtering) from- Dr. J. Bohlmark, Dr. J. Alami.
RIGHT- ARDEC-Benet Labs CMS platforms for coating full-length 120mm large cal bore.

Potential Applications of Technology



Properties of Ta, Cr, A723 Gun Steel

Properties	Steel	Cr	α-Ta
Melting Point Temperature [°C]	1535	1857	2996
Lattice Parameter [Angstroms]*	2.8665	2.8847	3.298
Lattice Mismatch with alpha Fe		-0.6%	-15.1%
Lattice Mismatch with alpha Ta	13.1%	12.5%	
*Thermal expansion at RT (K-1)***	1.35E-05	6.50E-06	6.50E-06
Difference in thermal expansion with Fe		51.9%	51.9%
*thermal conductivity, k [W/mK]****	45.1	98.5	57.7
*Young's Modulus, E (GPa)*****	207	248	173

*Cullity

**Smithell's 6th Ed.

***Underwood; Incorpera & DeWitt, Introduction to Heat Transfer

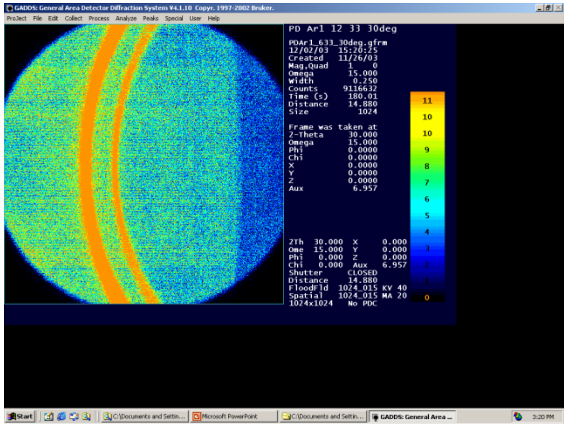
****Thornton & Colangelo, Fundamentals of Engineering Materials

Properties of bcc and tetragonal Ta

	α-Ta	β-Ta
Structure	BCC	Tetragonal
Lattice Parameters	*a*=*b*=*c*= 0.33058 nm	*a*=*b*=1.0194 nm *c*=0.5313 nm
Hardness	100-200 KHN	1000-1200 KHN
Resistivity	15-60 μΩ-cm	200 μΩ-cm
Thermal Stability	T_m = 2996°C	$T_{\beta \Rightarrow \alpha}$ ~ 750°C
Ductility	Ductile	Brittle

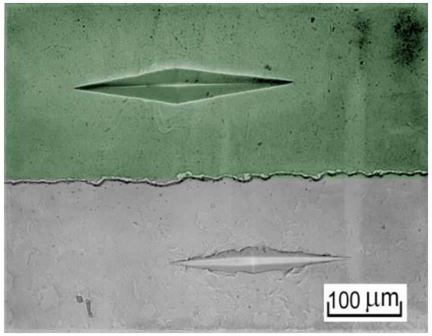
2-Theta

Chi

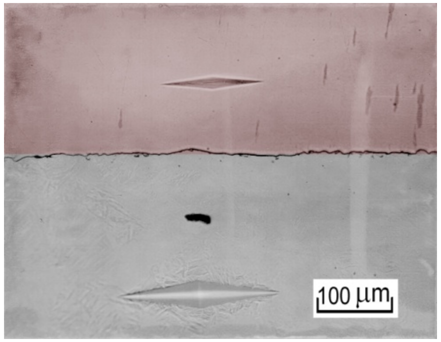


Random α Ta (110) (left ring)

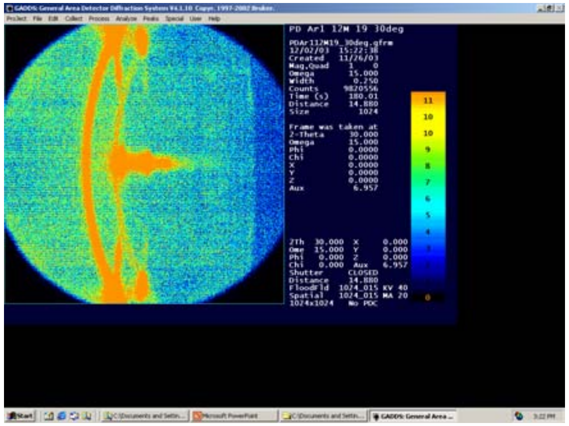
Random β Ta (002) (right ring)



“softer” bcc Ta on Steel



“harder” tetragonal Ta on steel

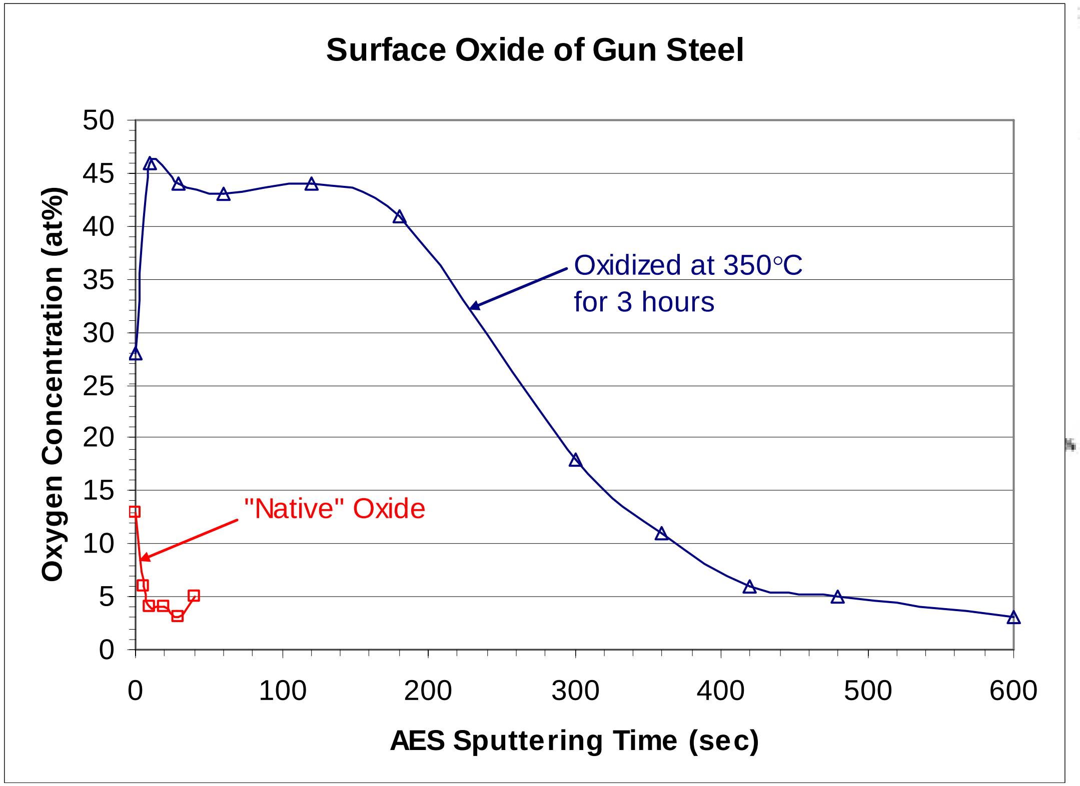


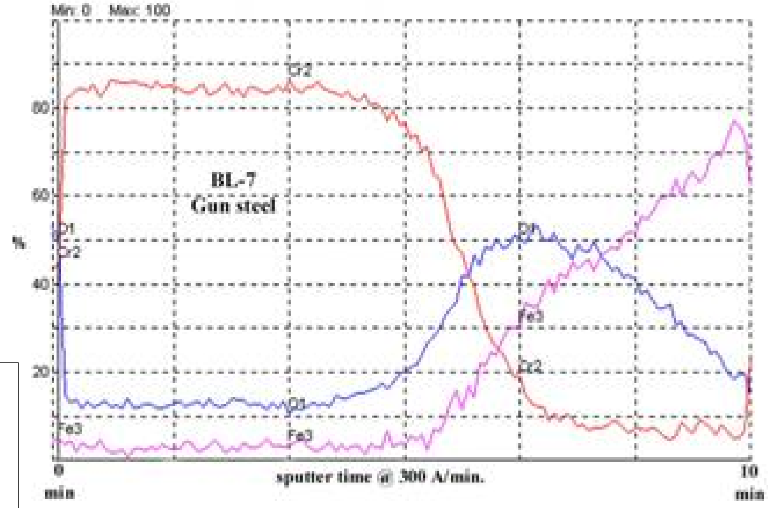
Random α Ta (110) (left ring)

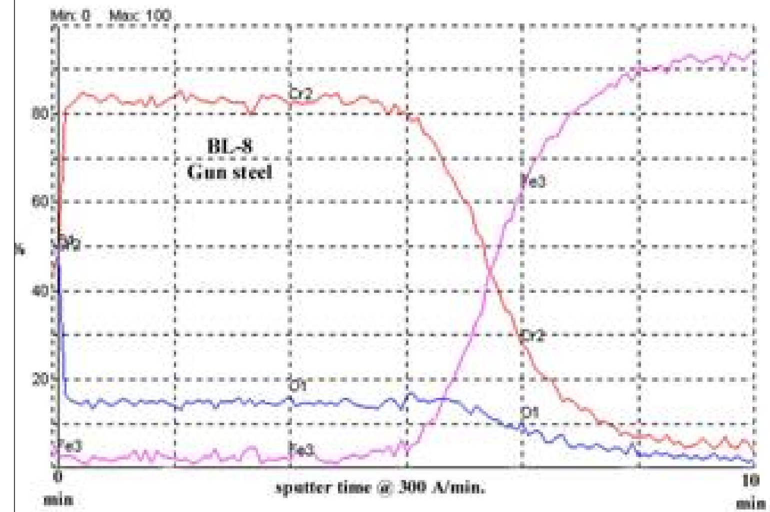
Textured β Ta (002) (right ring)

Sputter Cleaning of Gun Steel

- Native oxide on gun steel surface is ~10-20 nm
- Heat-Generated (350ºC for 3 hrs in air) oxide is ~300-350 nm.
- AES showed ion sputter cleaning for 60 min removed all native and heat-induced oxides.

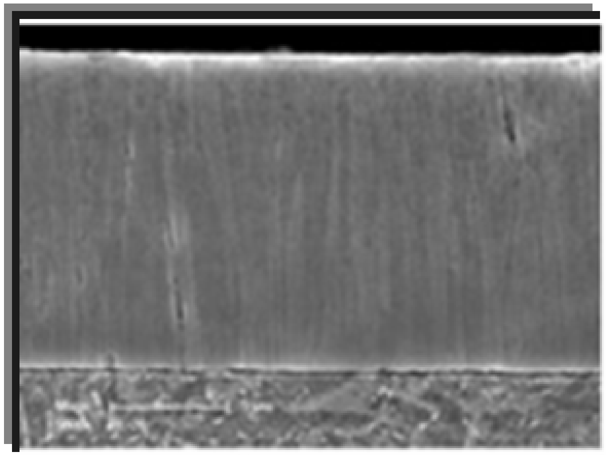




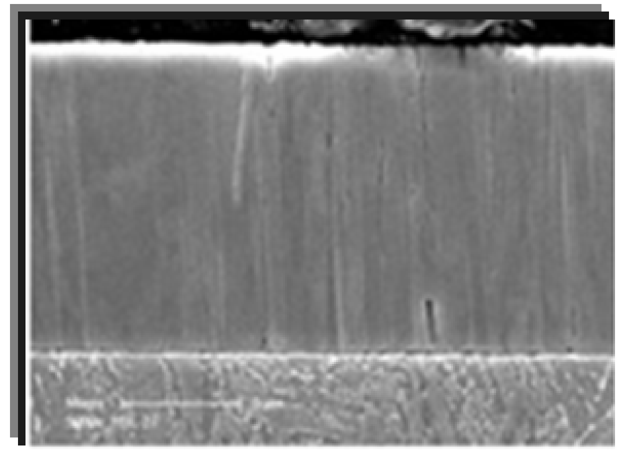


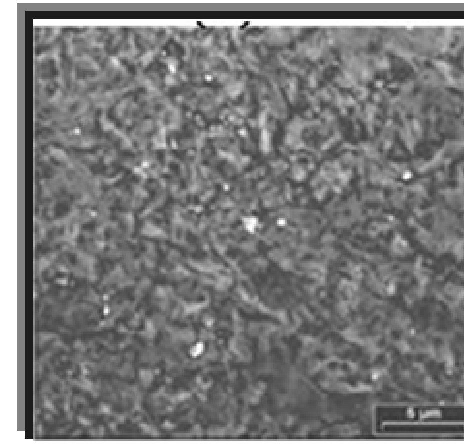
Sputtered Cr on A723 Gun Steel

Higher discharge current increases hardness, density, improves microstructure

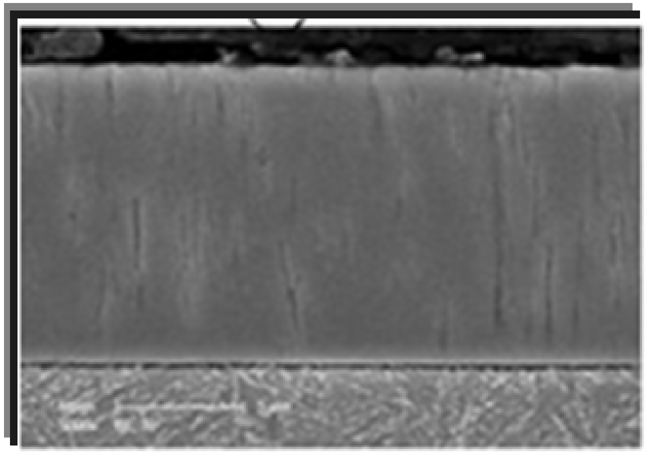


0A, 14 µm, 85 min, HK_{10} 399

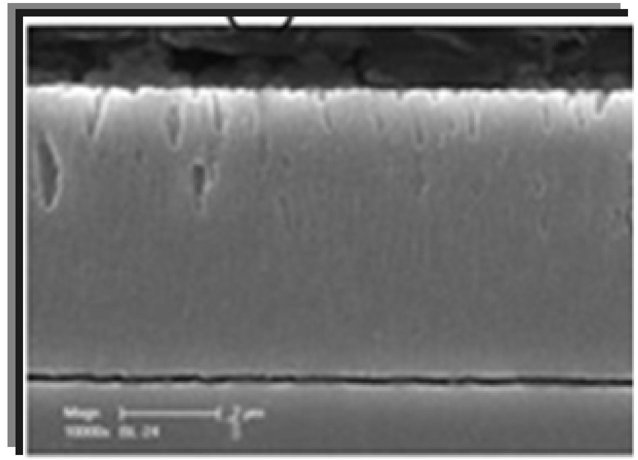


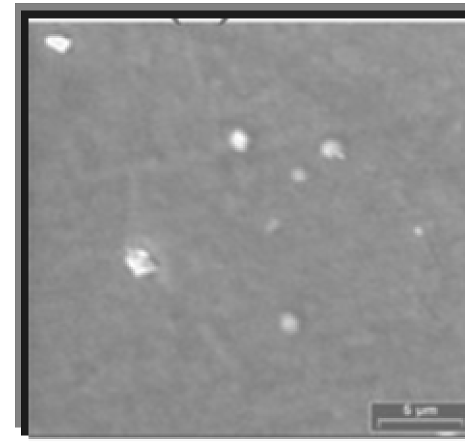


5A, 13 µm, 85 min, HK_{10}633



10A, 13 µm, 85 min HK_{10}1226





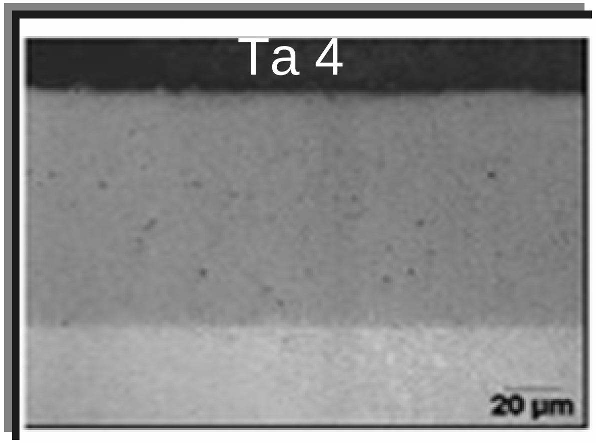
20A, 6 µm, 90 min, HK_{10}2144

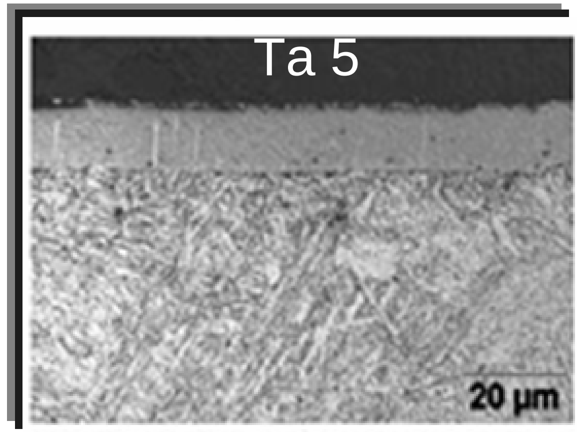
RDECOM

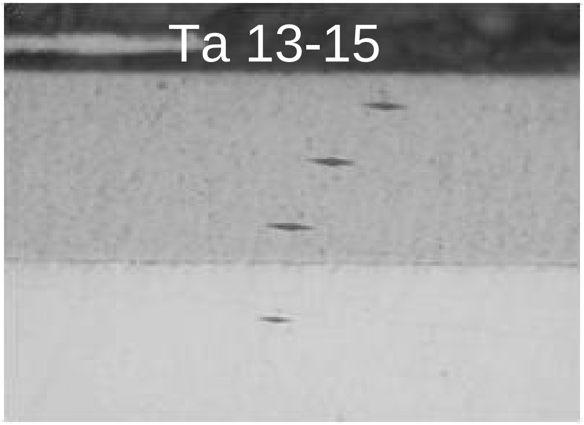
Sputtered Ta on A723 Gun Steel

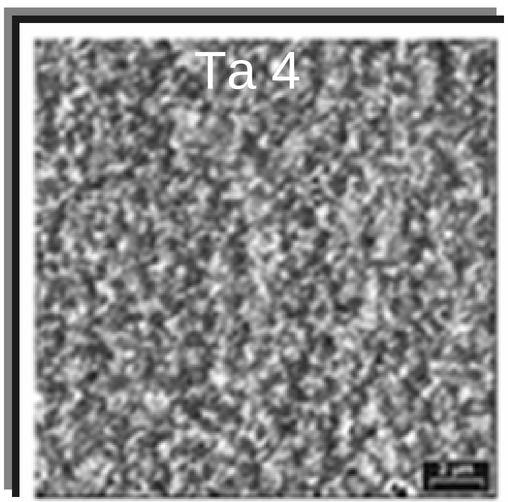
BENÉT LABS

Higher discharge current increases hardness, density, residual stresses, α-Ta formation

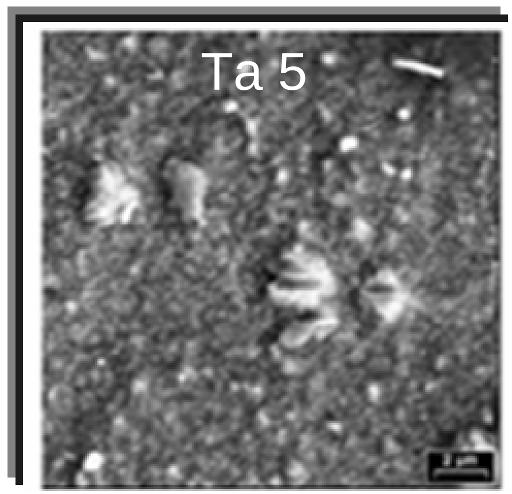




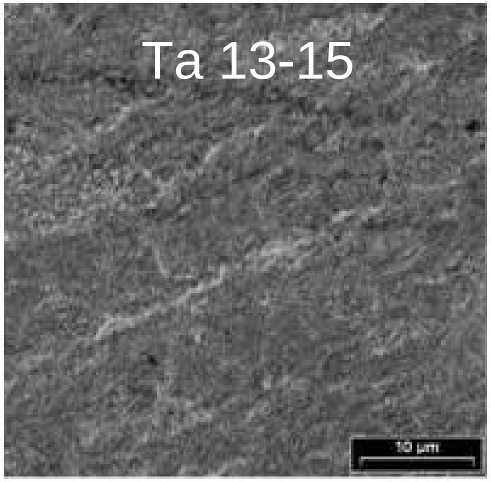




10A, 90µm, 600 min HK_{10}602, α-Ta



0A, 10µm, 60 min HK_{10} 552, (α+β)-Ta

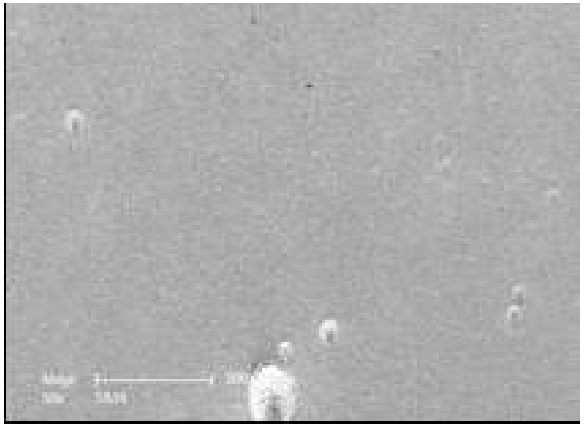


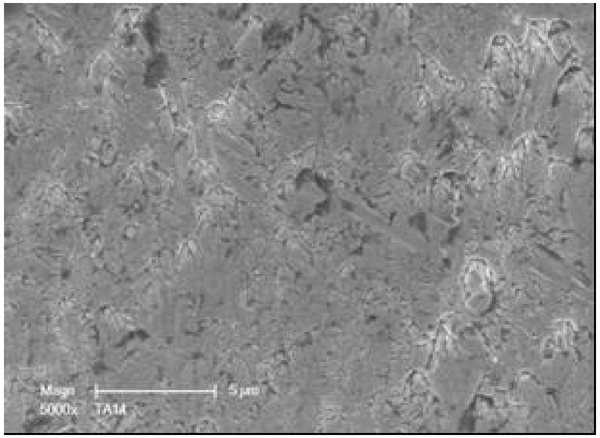
10A, 148µm, 900 min, HK_{10} 337, α-Ta

Sputtered Ta on Rifled 155mm

Plasma Enhanced DC Magnetron Coating Topography

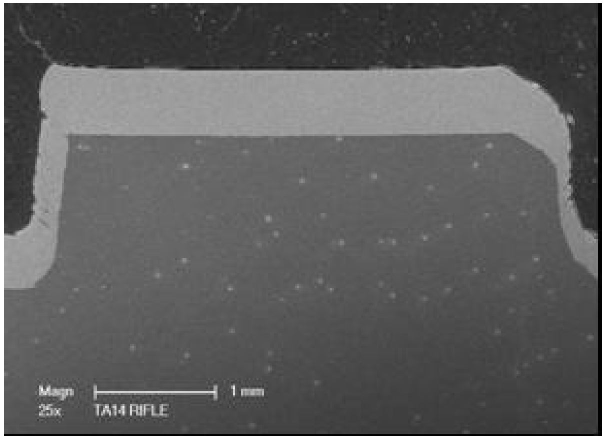
Land-Groove, 450 micron Ta Walls- 250 micron Ta

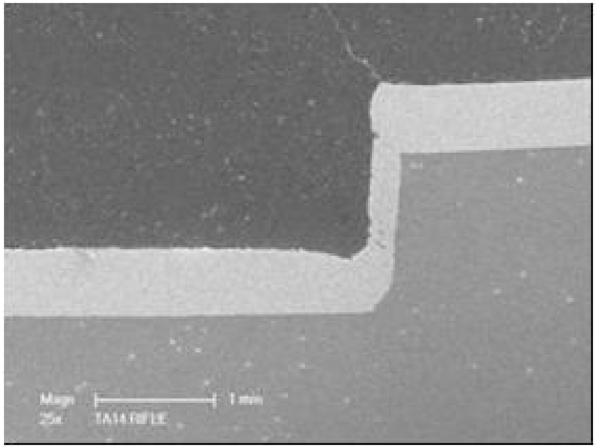




Plasma Enhanced DC Magnetron Coating Microstructure

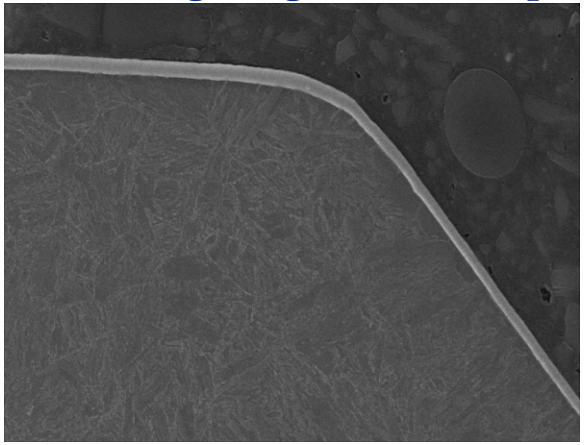
Land-Groove, 450 micron Ta Walls- 250 micron Ta

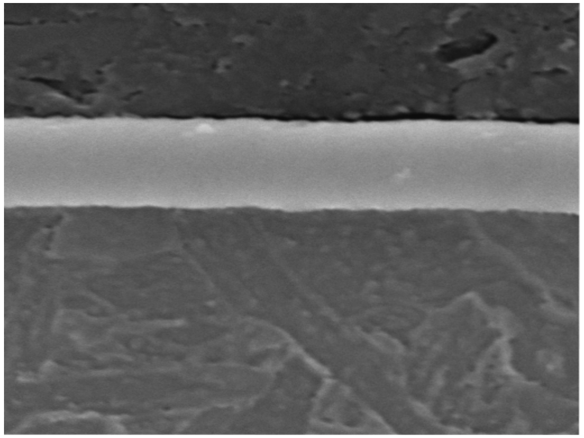




HIPIMS (High Intensity Impulse Magnetron Sputtering) (Helmersson/Rhode/Lee)

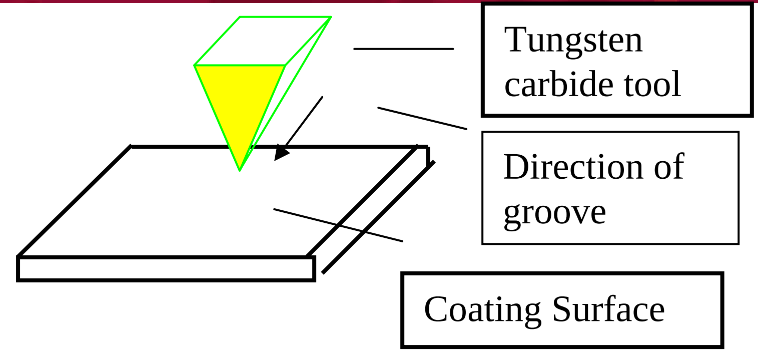
Uniform 2 micron bcc Ta on slanted edge of gun steel sample



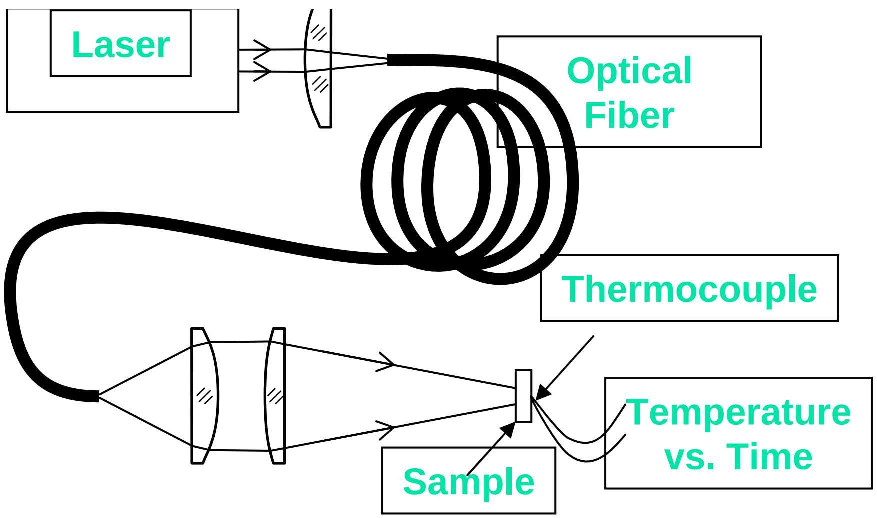


Coatings Adhesion Testing

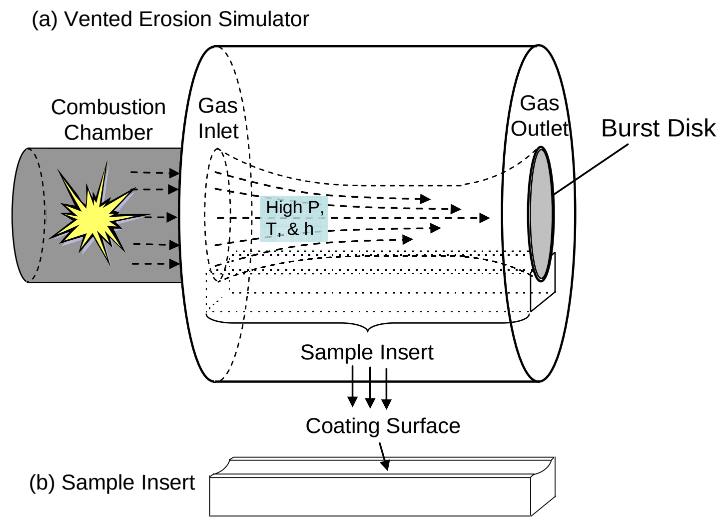
- Microscratch Test of Thin Coatings
- Groove Test (ASTM B571-91)
- Pulsed Laser Heat Test
- Vented Erosion Simulator Test (In-Door Firing Range)



Groove Test for Coatings Adhesion Strength



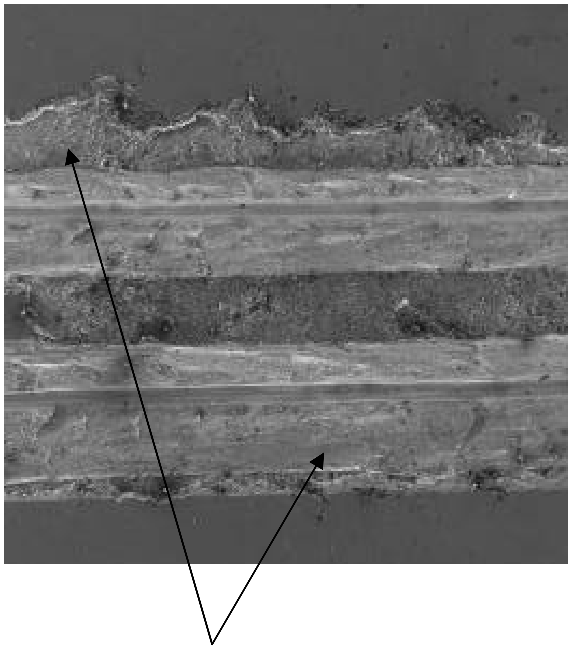
PLH (Pulsed Laser Heating) Test of Thermal Properties of Coatings



VES (Vented Erosion Simulator) Test of Thermal-Mechanical-Chemical Properties of Coatings

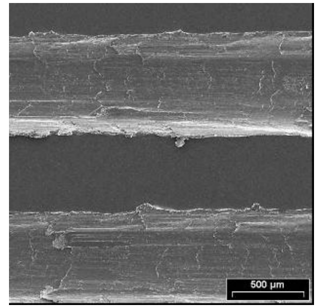
Groove Adhesion Testing
(ASTM B571-91)

Electroplated Cr

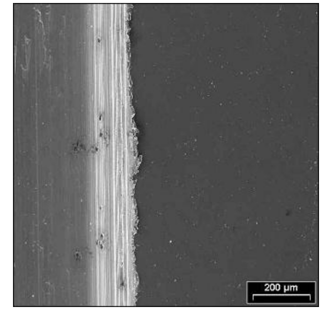


Cohesive failures

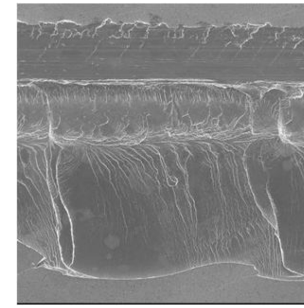
Cr16



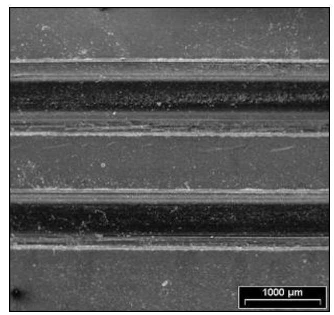
Cr24



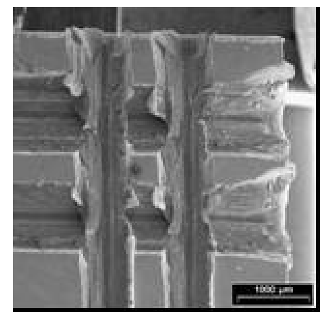
Cr 15



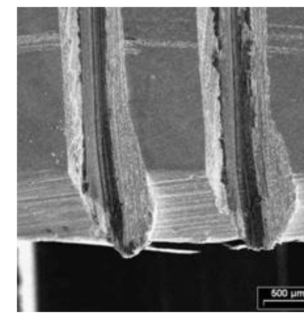
Ta3



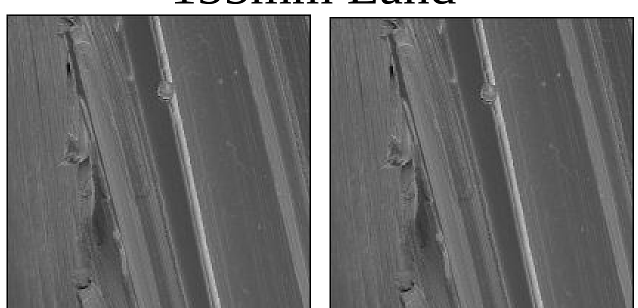
Ta12-2



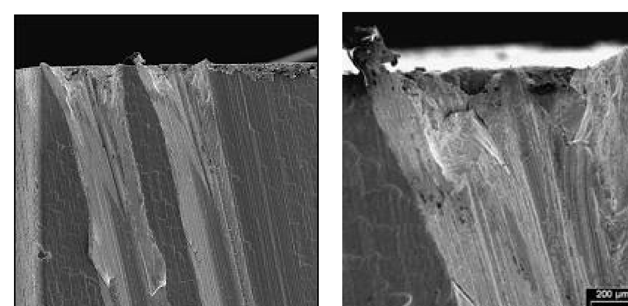
Ta13-15



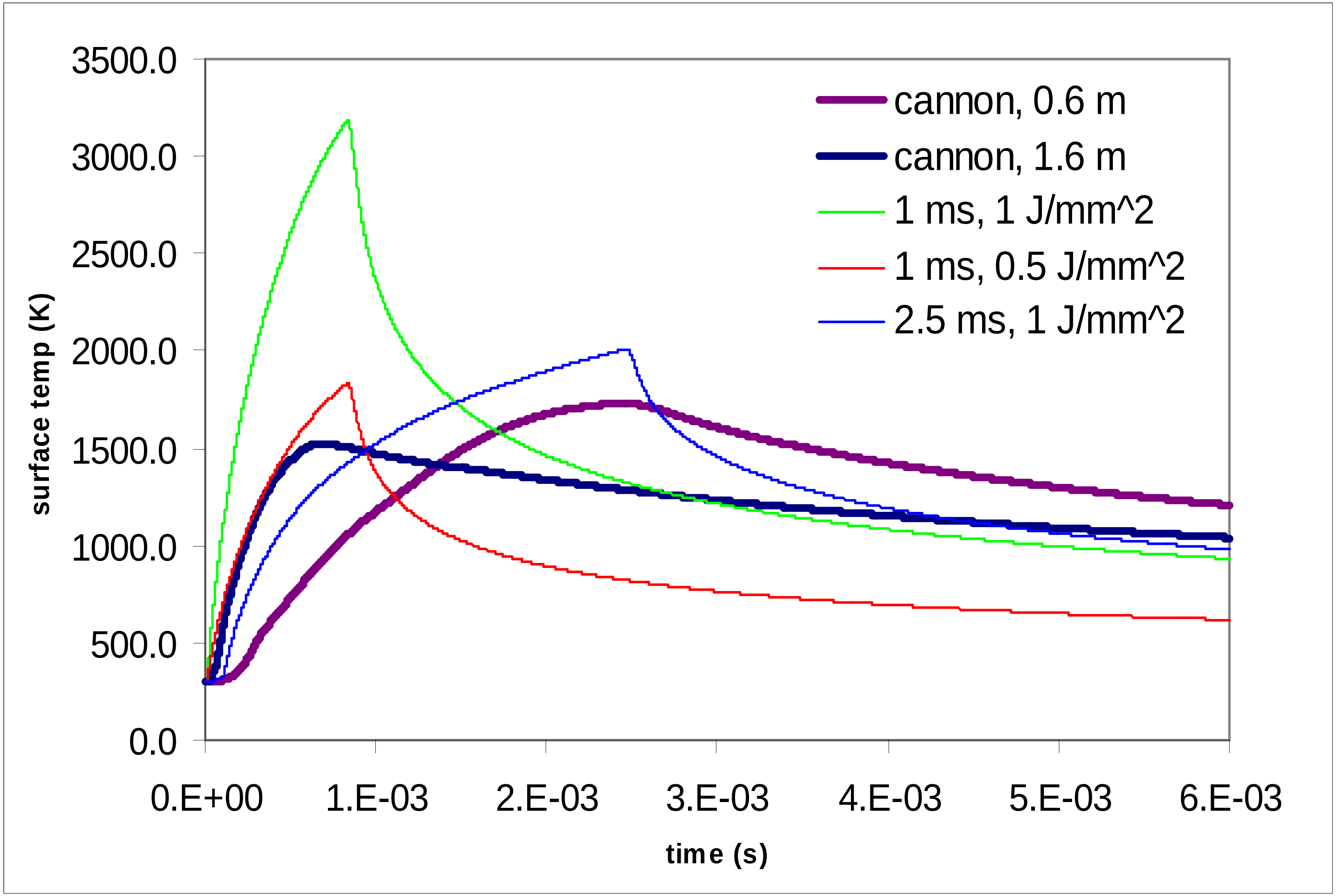
155mm Land



155mm Groove

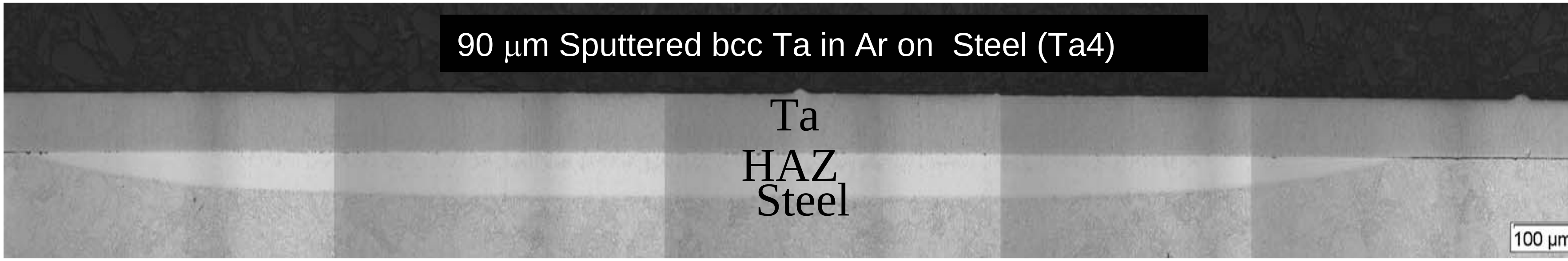


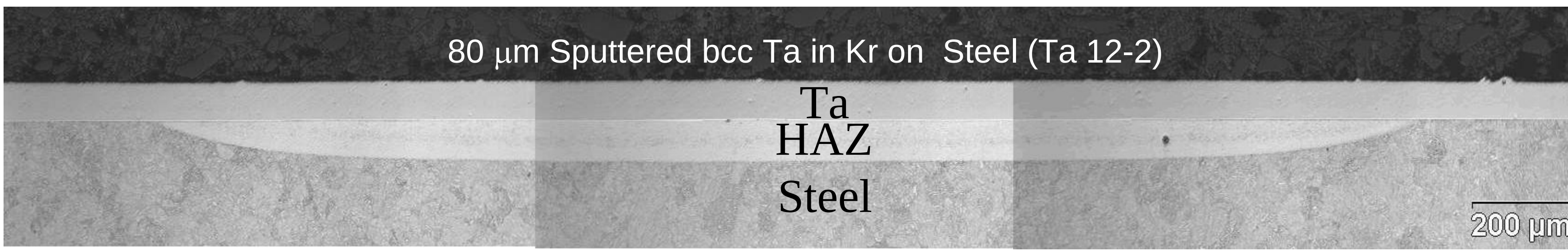
Internal Ballistic and Transient Thermal Simulation
120mm Cannon Firing and Pulsed Laser Heating

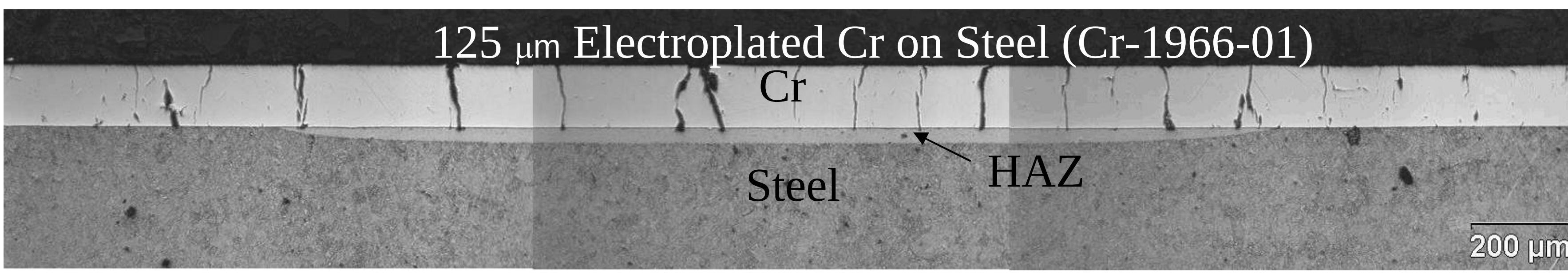


Comparative Pulsed Laser Heating Tests

PLH parameters (2.5 msec, 1.0 J/mm2, 20 cycles, simulating ~1480°C temperature)



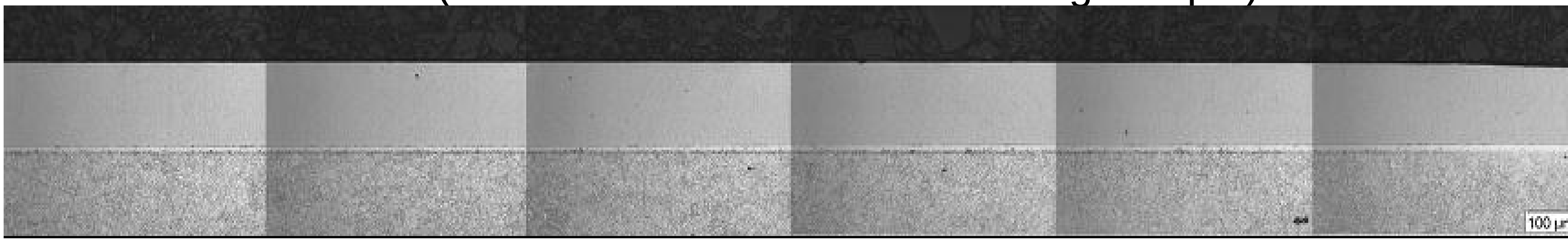




- **HAZ (Heat affected zone) in steel is due to tempered to untempered martensite transformation.**
- **During heating-cooling cycle, martensite transforms to austenite, then back to martensite**

148µm Ta on Gun Steel after 25 VES Firing High Erosive RDS

Axial (1.0 inch from end of 1.6 inch long sample)

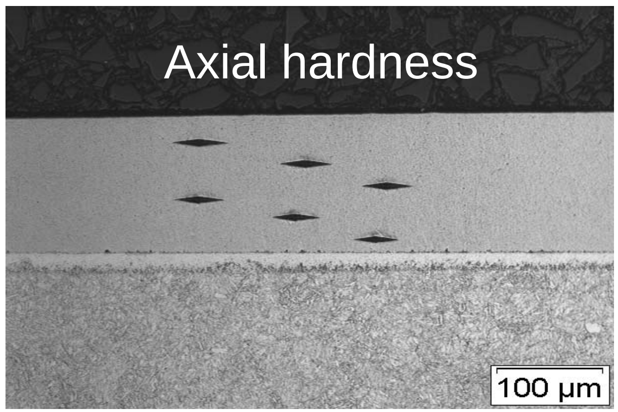


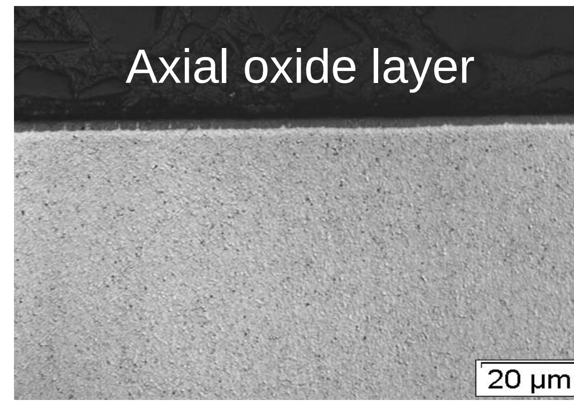
After 25 rounds

DACP-Ta-15hr	
Indents (Top Down)	
Knoop (Hk50)	Depth of Diamond (µ)
216	28.87
254	52.99
267	77.02
278	91.91
298	110.90
322	135.60
422	steel

As deposited

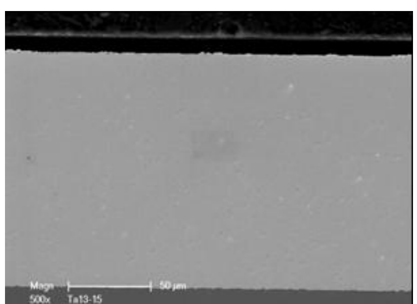
Ta-13_15	
Indents (Top Down)	
Knoop (Hk50)	Depth of Diamond (µ)
323	24.62
340	73.14
349	128.90
523	208.90

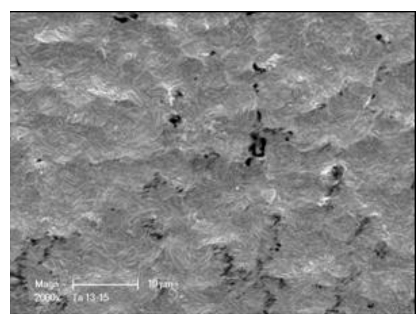


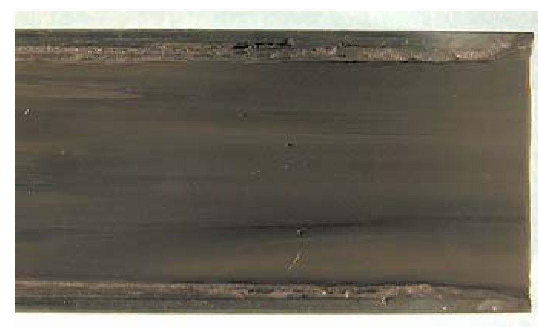


As-deposited topography - microstructure

After fired surface

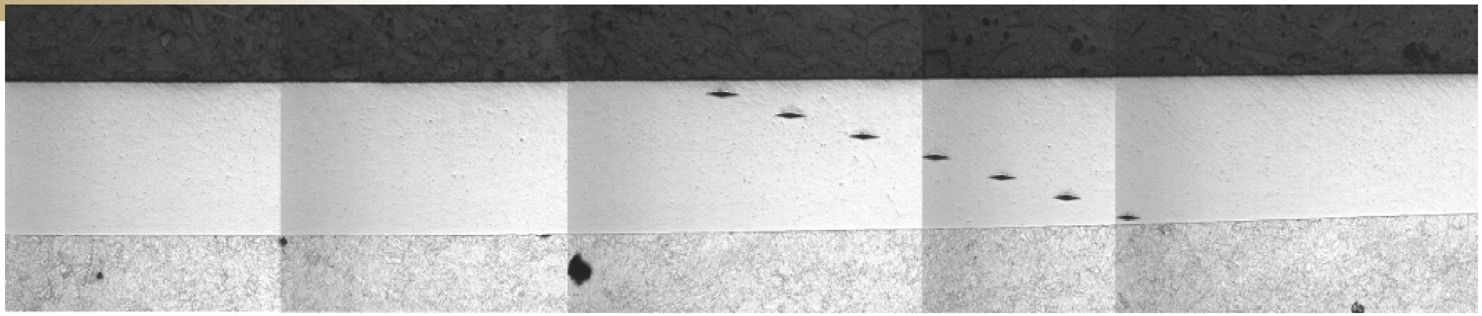






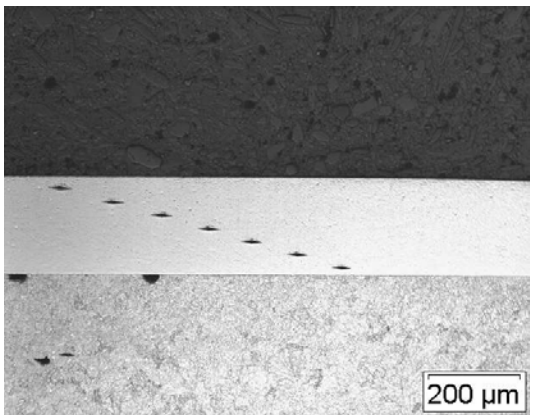
Minimal softening, good adhesion, no cracking after 25 VES rds!

286 μm Ta on Steel After 129 VES Firing High Erosive RDS



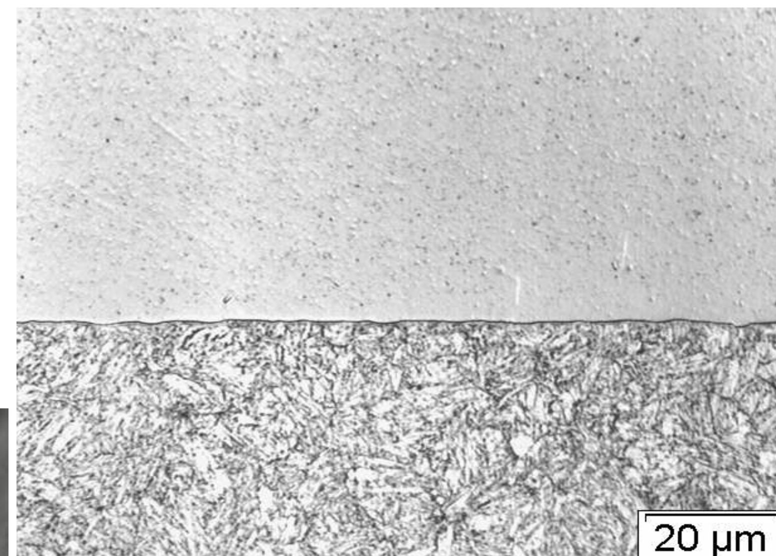
DACP-Ta-13-20hr	
Indents (Top Down)	
noop (Hk50)	Depth of Diamond (µ)
216	19.32
247	49.35
249	80.25
273	108.00
303	137.00
325	165.70
420	195.30

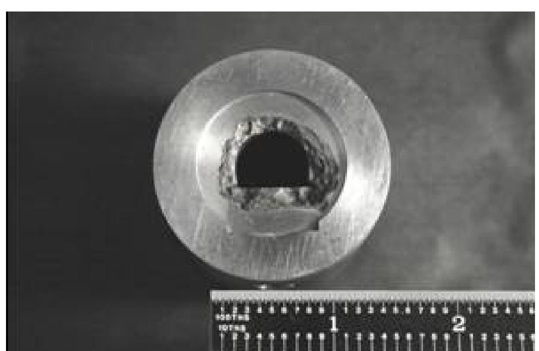
Longitudinal Ta Thickness = 204 µm



DACP-Ta-13-20hr	
Indents (Top Down)	
Knoop (Hk50)	Depth of Diamond (µ)
240	21.64
267	50.84
289	78.45
299	108.30
328	135.60
344	162.30
371	190.10

Transverse Ta Thickness = 203 µms

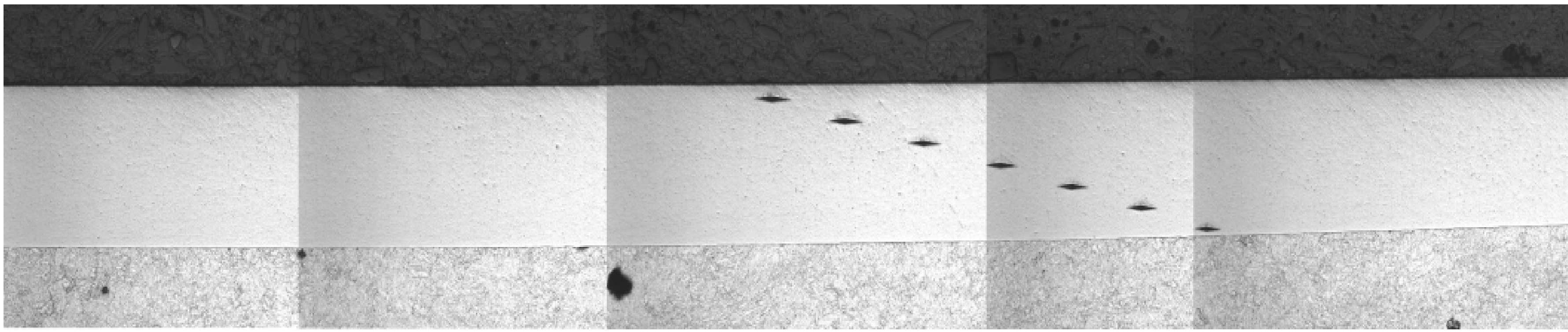




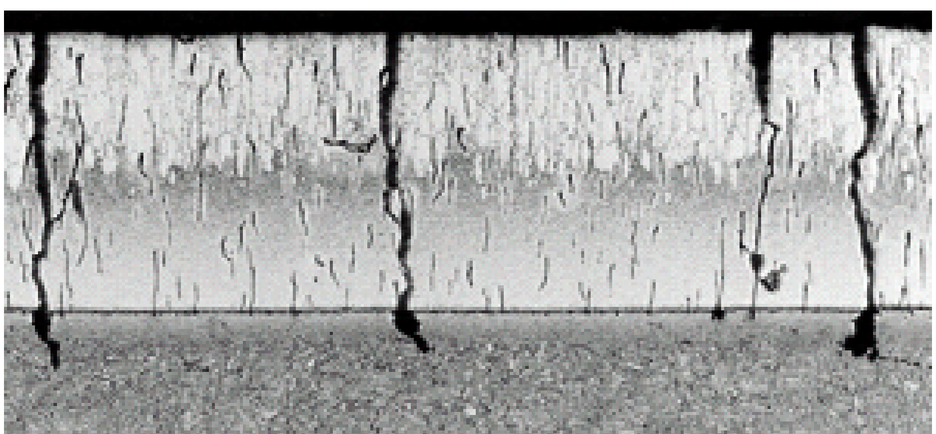
Minimal softening, good adhesion, no cracking after 129 VES rds!

VES Firing Comparison: Electroplated Cr vs. Plasma Enhanced Sputtered Ta

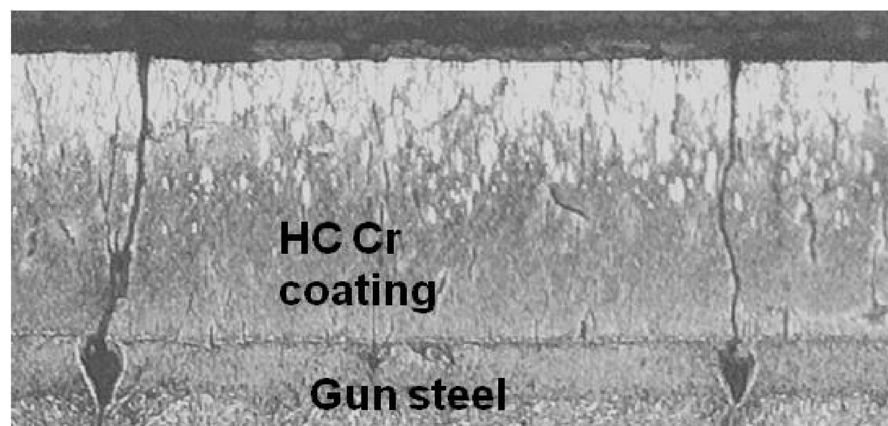
Plasma Enhanced bcc Ta (286µm) on steel after 129 cycles VES high erosive rds
Showing excellent structure, adhesion, and crack-resistance properties



Electroplated HC Cr (120µm) on Steel
~ 100 cycles of VES under same conditions



Electroplated HC Cr (120µm) on Steel
~100 cycles of actual firing of high erosive rds



Conclusions

1) Plasma enhanced magnetron technology using higher ion bombardment can deposit hard, dense, adhesive, pollution-free coatings on steel with improved microstructure.

2) Plasma enhanced sputtered Ta on 120mm and 155mm test samples demonstrated excellent structure, ductility, adhesion, and resistance against thermal shock cracking and high temperature erosion.

3) Plasma enhanced sputtered Ta has potential to coat 120mm and 155mm barrels and other armament components, with expected improved cycle life due to the high melting point temperature and absence of cracks.