Weapon Systems & Technology Directorate
US ARMY ARDEC and ARDEC- BENET Labs

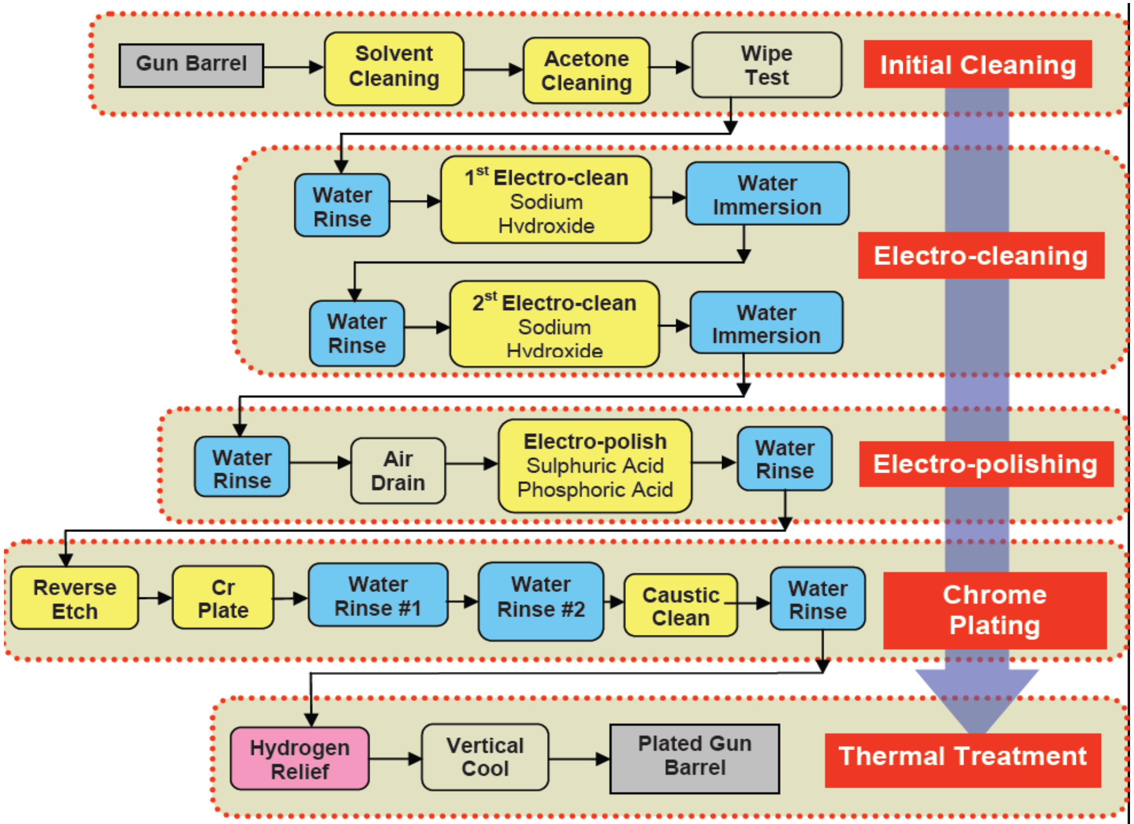
New Physical Vapor Deposition Processes for Durable Pollution-Free Ordnance

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

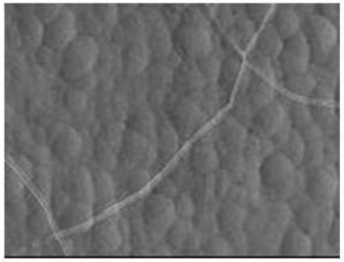
Dr. Sabrina Lee and Daniel Schmidt

2011 NDIA Gun and Missile Conference
Miami, Florida, Aug 29- Sept 1, 2011

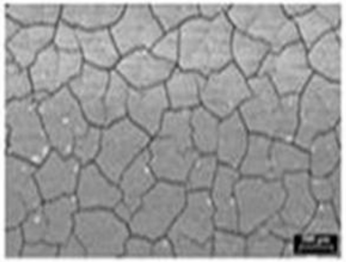
Production High Contraction Chrome (HC Cr) Process to Coat Ordnance



As-deposited Cr



After-fired Cr



From- M. Audino. DOD Metal Plating Workshop, May 22, 2006.

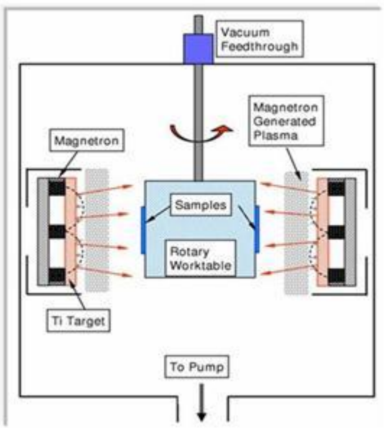
Physical Vapor Depostion Technology Alternative to HC Cr Process

- ***Arc Evaporation (filtered, steered, switched) Process.***
- ***Direct Current Magnetron Sputtering (DCMS) Process.***
- ***Plasma Enhanced Magnetron Sputtering (PEMS).***
- ***High Power Impulse Magnetron Sputtering (HIPIMS).***
- ***PEMS/HIPIMS Deposited CrN Coatings and Applications.***
- ***PEMS/HIPIMS Deposited Ta Coatings and Applications.***
- ***Conclusions.***

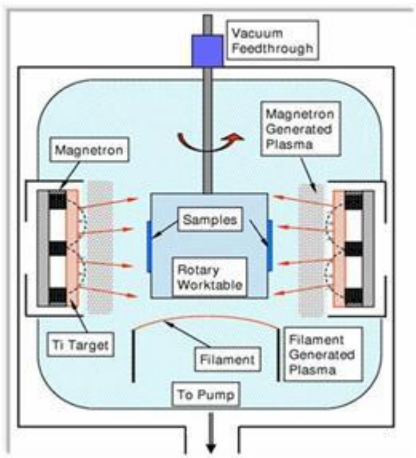
Plasma Enhanced Magnetron Sputtering (PEMS) Using Higher Plasma Intensity

Multiple Target PVD- DCMS and PEMS systems at SWRI

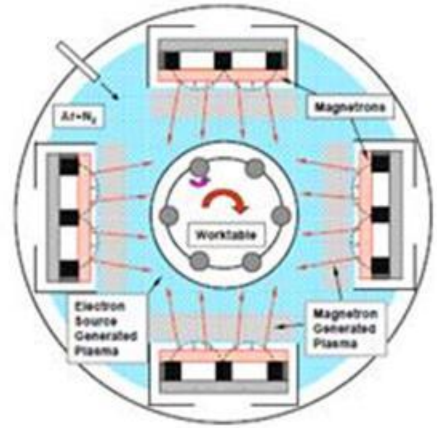
DCMS (0.2mA/cm^2)



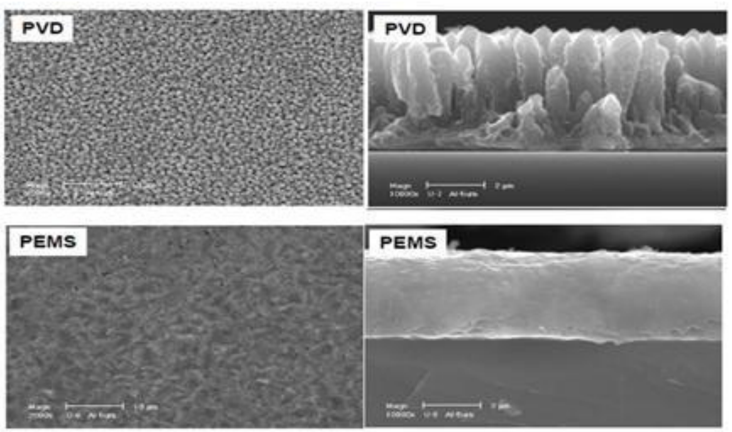
PEMS (4.9 mA/cm^2)



Multiple Target System



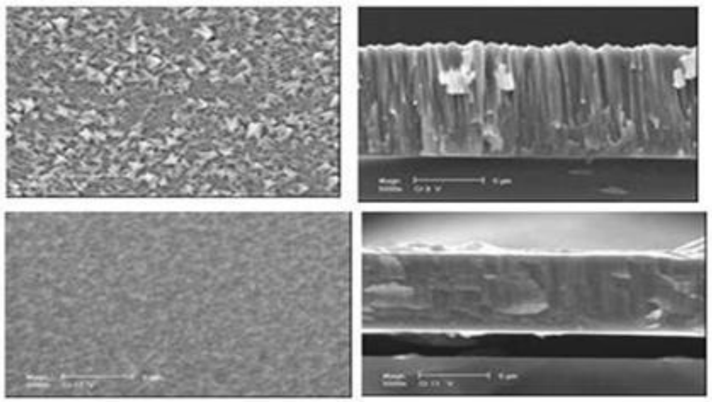
Aluminum



DCMS

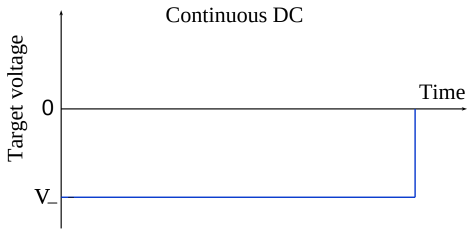
PEMS

Chromium



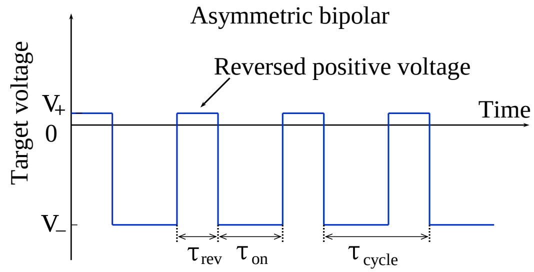
DCMS Versus HIPIMS-HPPMS-MPP
High Power Impulse Magnetron Sputtering

DCMS
Continuous DC



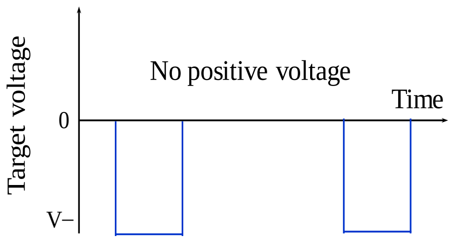
DCMS
Pulsed DC

Frequency: in kHz
Duty cycle: 50~90%



HIPIMS-HPPMS-MPP*

Frequency: in Hz
Duty cycle: 1-10%

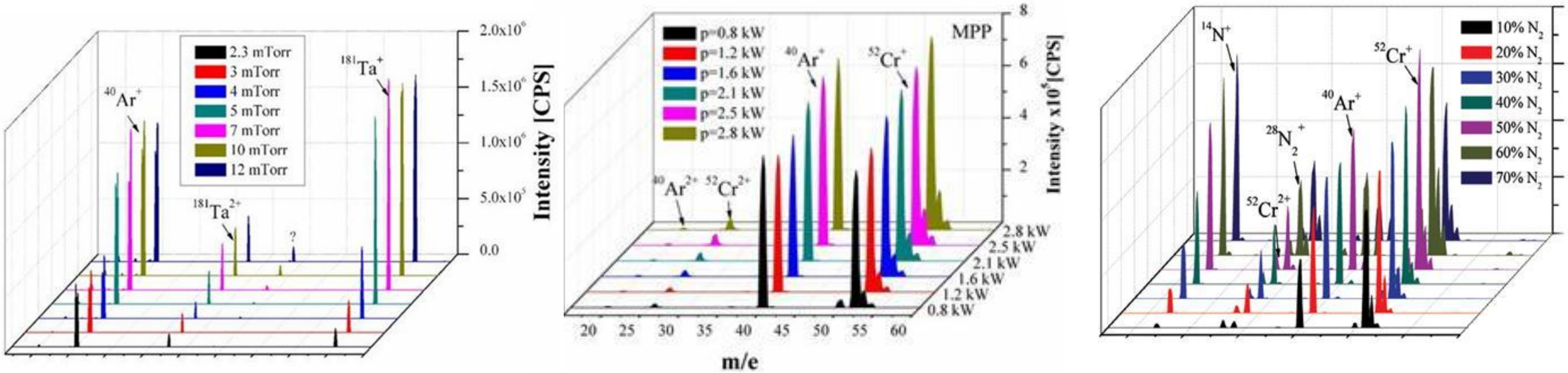


- **Introduced by Kouznetsov et al; HPPMS-MPP are slight variances of HIPIMS.*
- *Large number of target material ions and enhanced plasma density.*
- *High power pulses of short duration (100-150 μs); low duty cycle (1-10%).*
- *Peak value (1-3 kW/cm^2) typically 100 times greater than conventional DC magnetron sputtering (1-3 W/cm^2).*

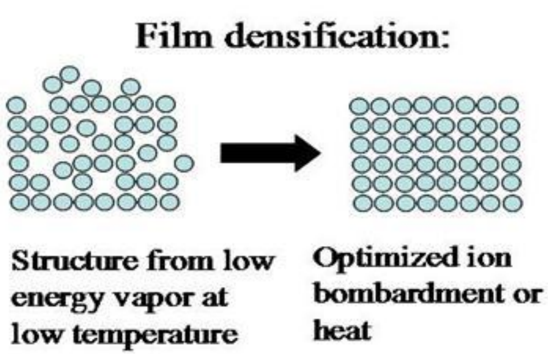
** From- V. Kouznetsov, K. Macák, J. M. Schneider, U. Helmersson, and I. Petrov, "A Novel Pulsed Magnetron Sputter Technique Utilizing Very High Target Power Densities," Surf. Coat. Technol. 122 (1999) 290.*

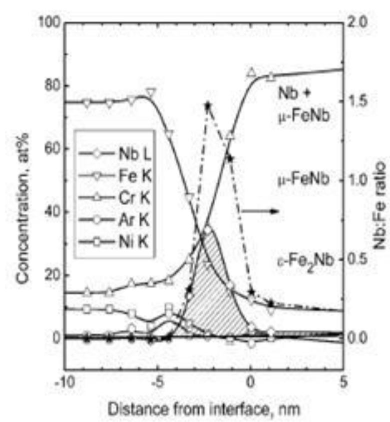
New High Power Impulse Magnetron (HIPIMS) Process Using Metal Plasma

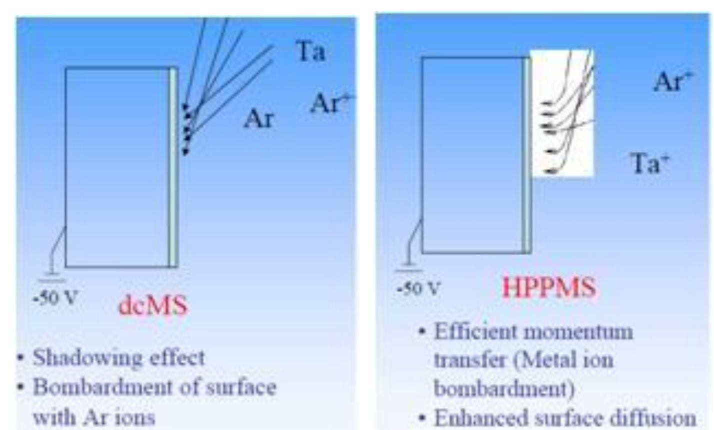
- ***High intensity target Metal Plasma Instead of Argon Plasma***
- ***Deposition of dense coatings at low temperature on complex shape.***



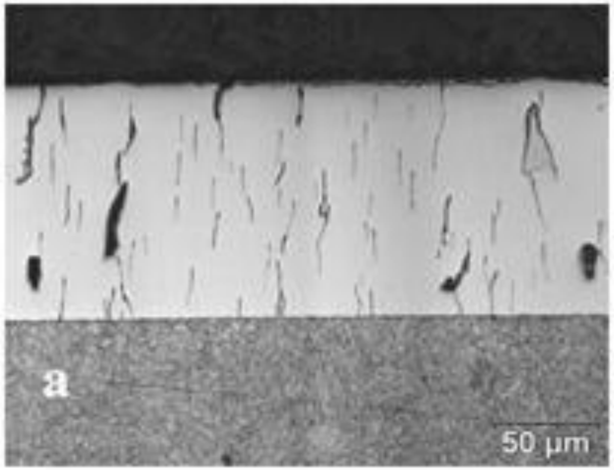
Plasma Mass-Ion Distribution using Tantalum and Chrome Targets measured using an electrostatic quadrupole plasma (EQP) mass spectrometer



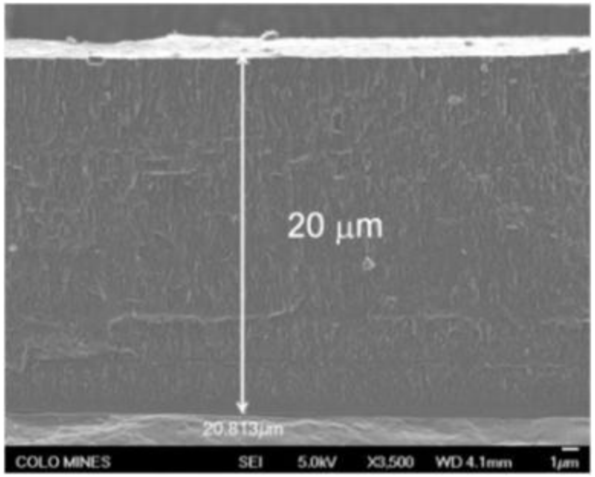


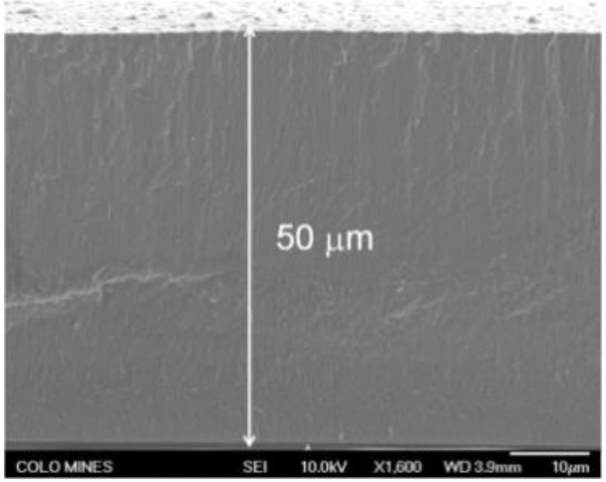


Improved Microstructure in HIPIMS-MPP Deposited Thick Ta and CrN Coatings

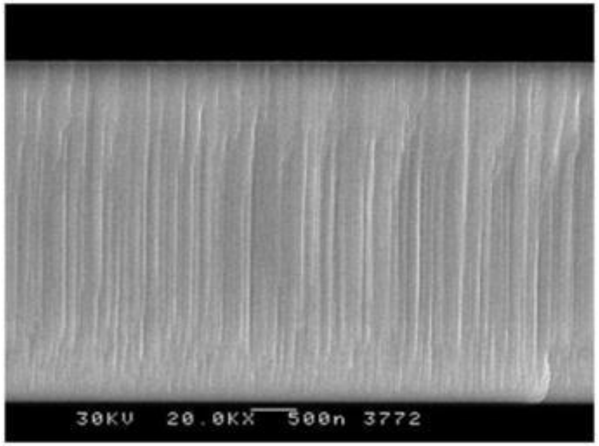


Production Electroplated Cr

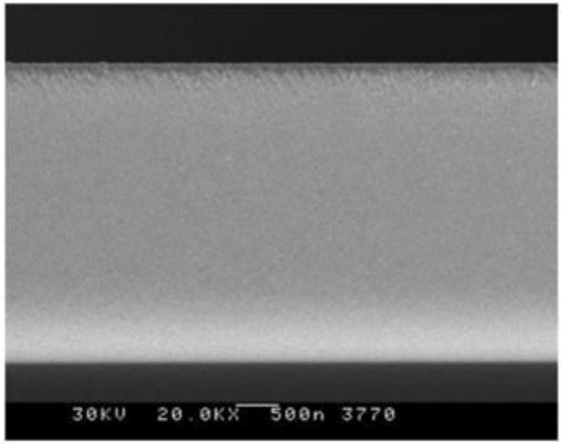




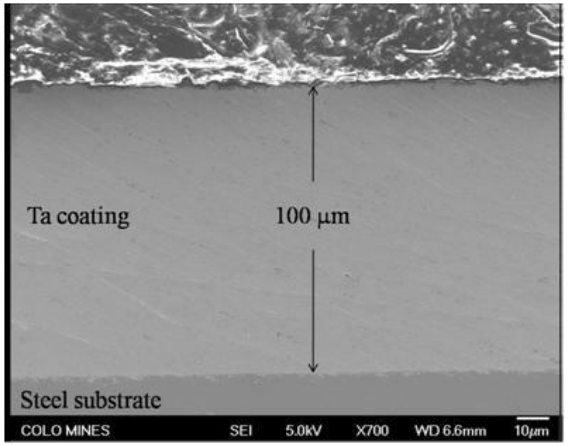
HIPIMS-MPP Deposited Thick CrN



DCMS Ta (10µm)

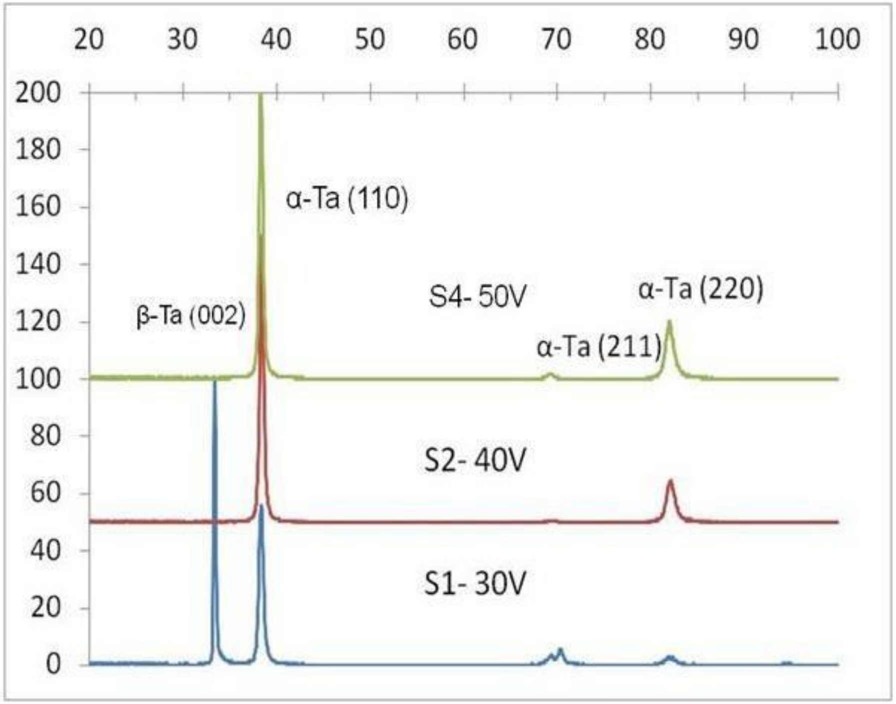


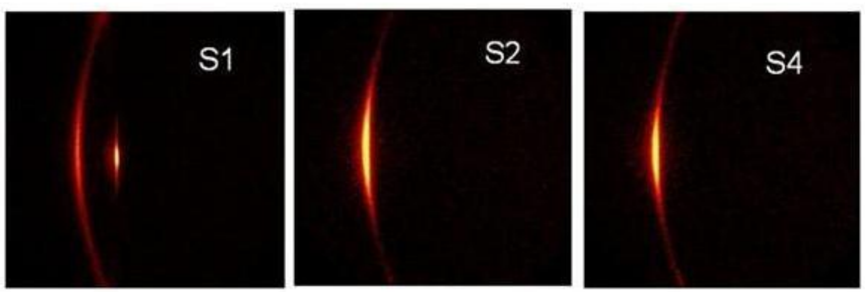
HIPIMS Ta (10µm)



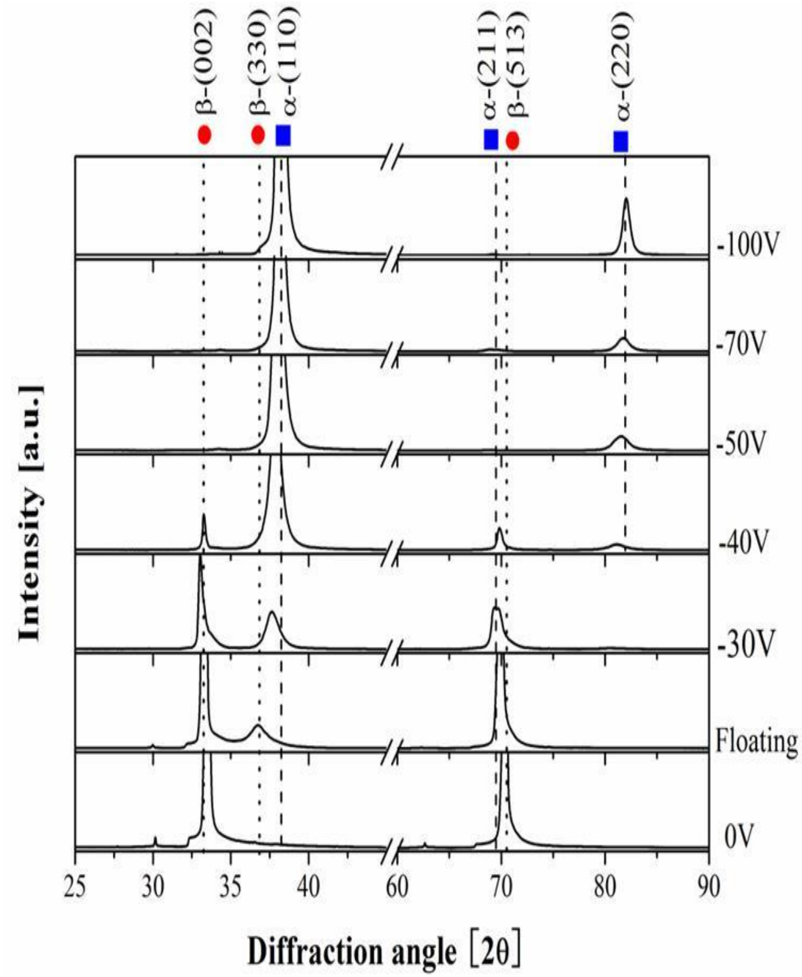
HIPIMS Ta (100µm)

HIPIMS Tantalum Phase Dependence on Deposition Parameter- Bias Voltage





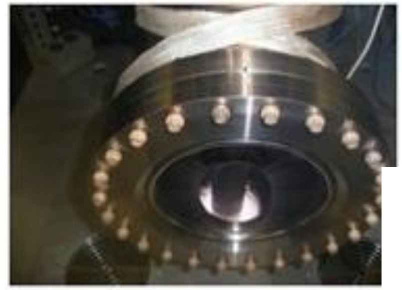
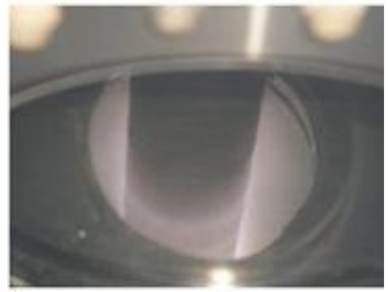
** From- S.L. Lee, M. Cipollo, F. Yee, R. Chistyakov, B. Abraham, SVC 52nd Tech Conf. Proc.,(2009) 44-49.*



* From- J. *Lin, J. Moore, W.D. Sproul, S.L. Lee, J. Wang, IEEE Transactions on Plasma Science, Vol 38, No. 11, (2010) 3071-3078.*

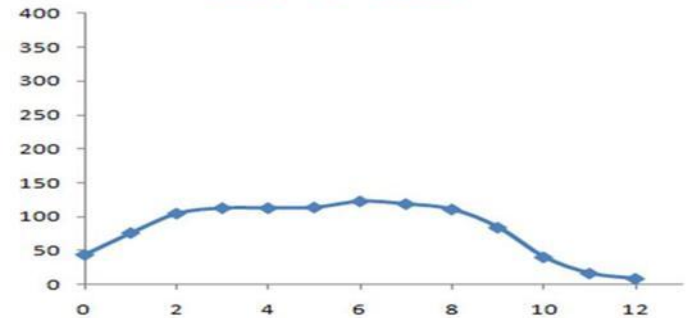
PEMS-HIPIMS-MPP Cylindrical Magnetron Systems for Ta Depositions on 120mm Bore



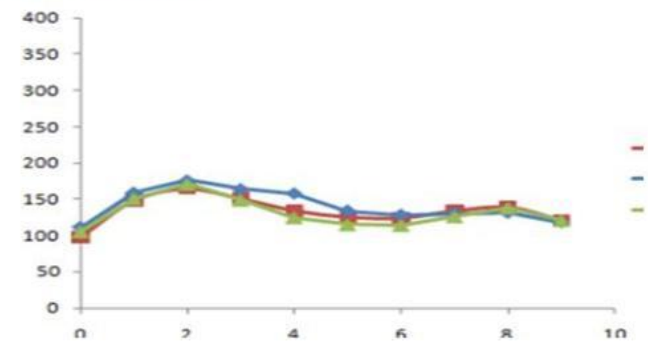
Adding biasing and HIPIMS-MPP to Benet DOE Cylindrical System

Benet DOE, HIPIMS-MPP Barrel
8 hrs,100-120µm thick
blue- 6 o'clock



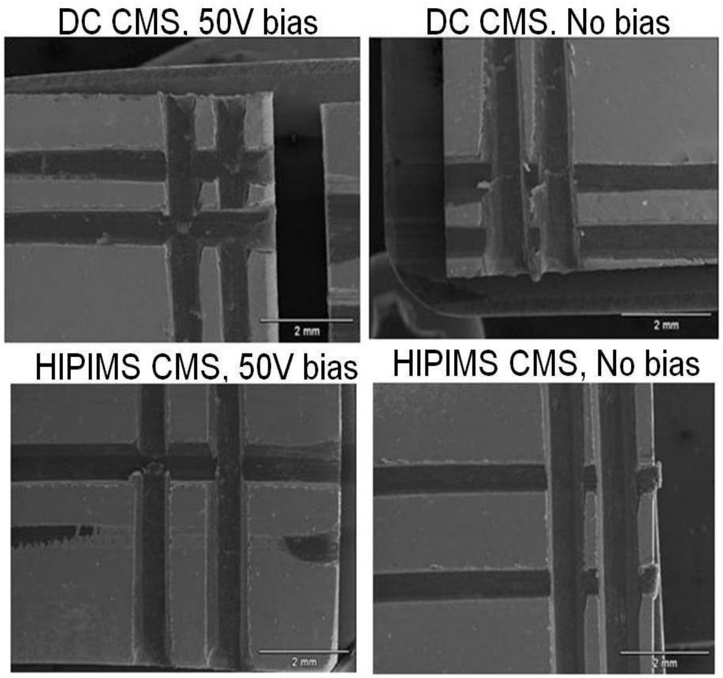


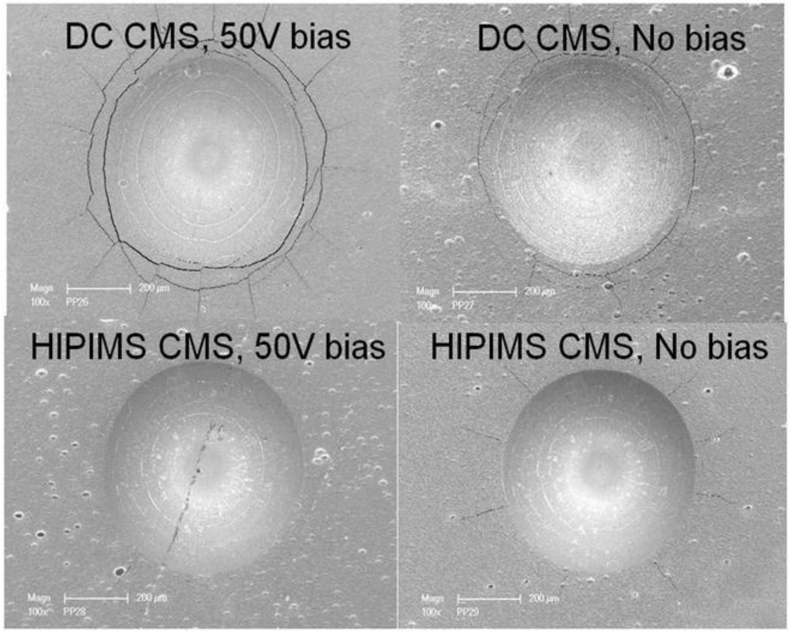
Tube PP36 on Carbon Tube; Biased DC, 15 hrs, **100-150 µm thick**; red- 6, blue- 9, green 12 o'clock



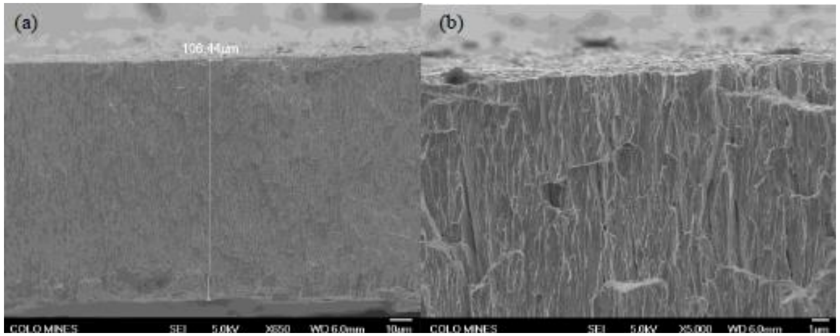
From- S.L. Lee, R. Wei, F. Yee, M. Cipollo, W. Sproul, J. Lin, presentation at ICMCTF, San Diego, CA, April 26-30, 2010.

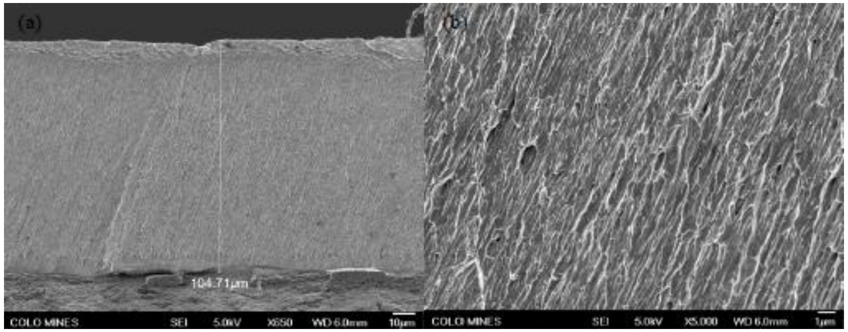
PEMS-HIPIMS Cylindrical Magnetron Deposited Ta on 120mm Steel Cylinder Bore





Fractured surface of 104-106 μm PEMS Ta on 1-ft long steel cylinder



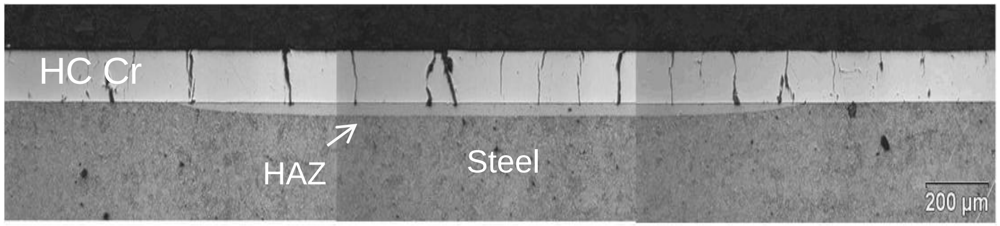


From- S.L. Lee, M. Todaro, S. Smith, R. Wei, K. Coulter, SVC 52nd Tech Conf. Proc., (2009) 558-563.

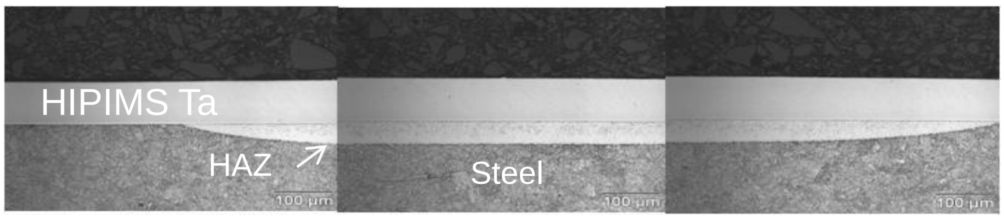
Properties of HIPIMS-MPP Deposited Thick Ta Coatings on A723 Steel

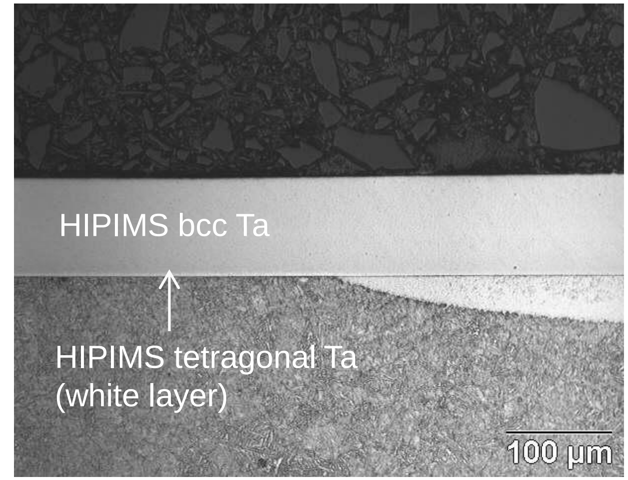
Pulse Laser Heating (2.5 msec, 1.0 J/mm2, 20 cycles, simulating ~1400°C temperature)

125 µm HC Cr on 120mm Diameter Steel Cylinder

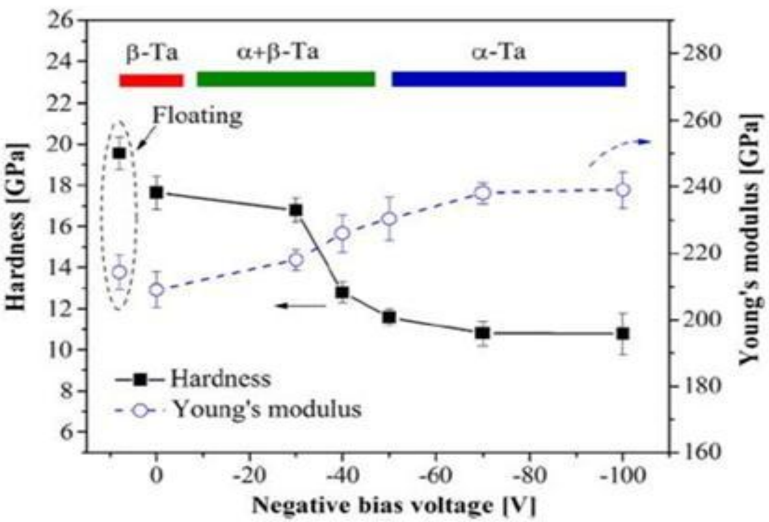


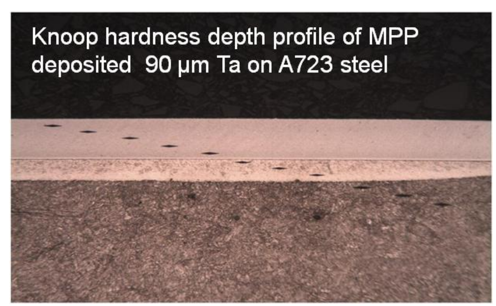
90µm HIPIMS-MPP Ta on Steel





HAZ (Heat affected zone) in steel is due to tempered to untempered martensite transformation.



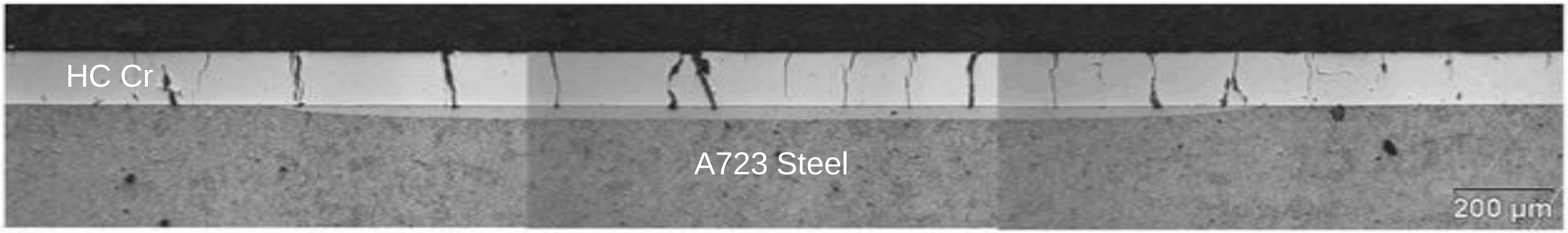


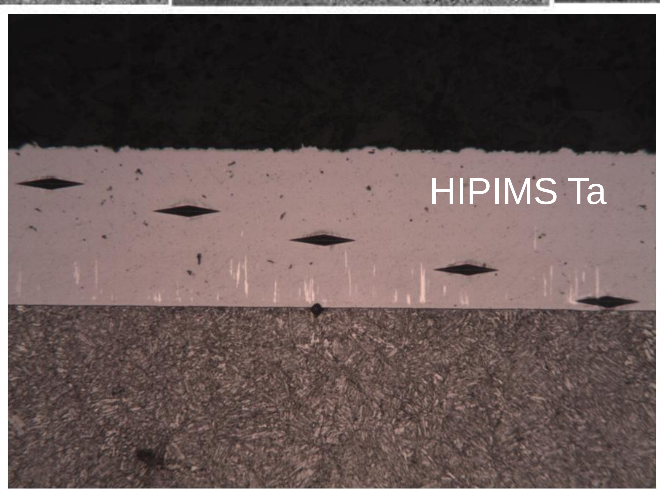
PL-Ta Mines 2-01

Diamond Length (µ)	Knoop (Hk_{50})	Depth of Diamond (µ)	
36.64	530	-98.35	-Steel
37.57	505	-79.34	
39.68	451	-62.38	
29.54	815	-31.43	- HAZ
30.75	752	-16.72	
29.84	823	-6.63	
35.33	569	12.15	- Ta
34.52	598	28.53	
33.10	649	46.51	
33.10	649	61.02	
33.82	623	75.43	

HIPIMS-MPP Cylindrical Magnetron Deposited Ta on 120mm Cylinder Bore

* Pulse Laser Heating (2.5 msec, 1.0 J/mm2, 20 cycles, simulating ~1400°C temperature)
* DOE-Z2-2 (MPP, substrate ground, 106 µm, no HAZ, no cracking, no delamination)

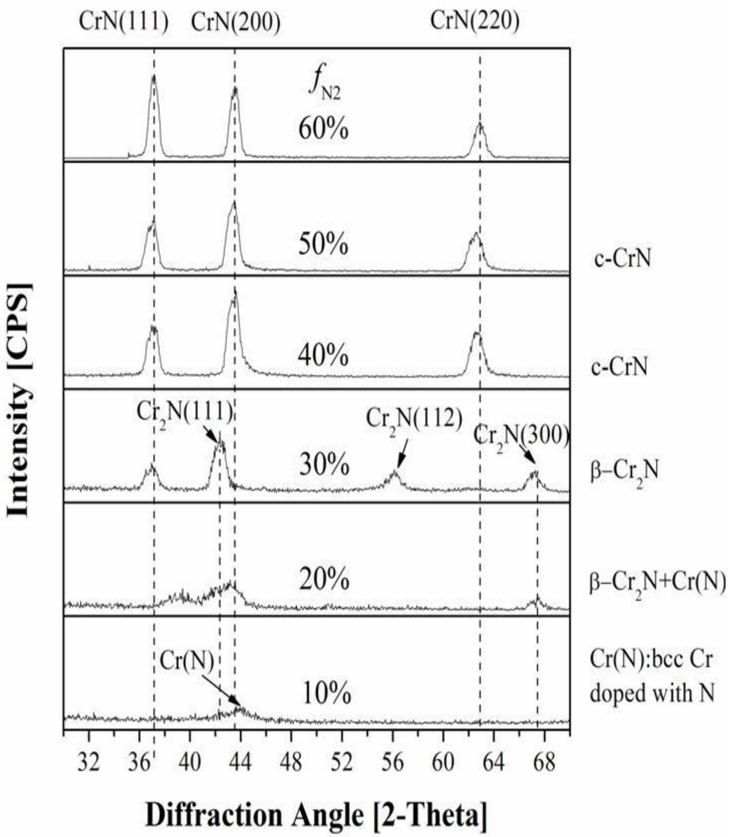




Z2-2		
Diamond Length (µ)	Knoop (Hk_{50})	Depth of Diamond (µ)
46.86	326	5.67
47.18	319	25.78
47.51	315	45.53
47.32	318	64.51
48.96	297	82.74

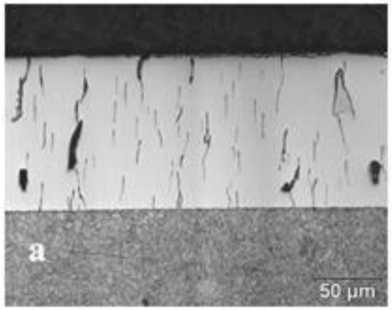
HIPIMS-MPP CrN Coatings For Weapons Coatings and Remediation

Phase formation of CrN coatings depends on the % N_2 in Ar gas.

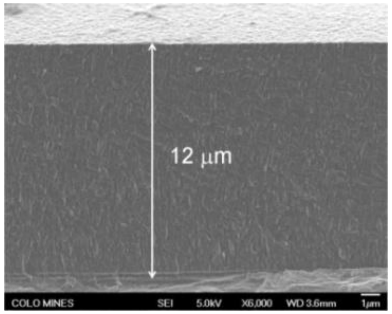


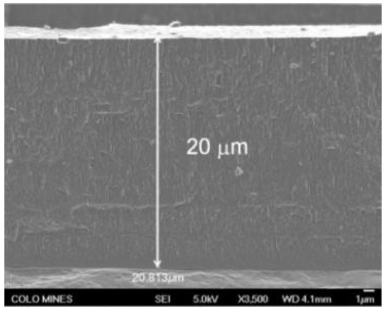
From- *Jianliang Lin, William Sproul, John Moore, Sabrina Lee, S. Myers, Surf Coat Tech 205 (2010), 3226-3234.*

HC Cr

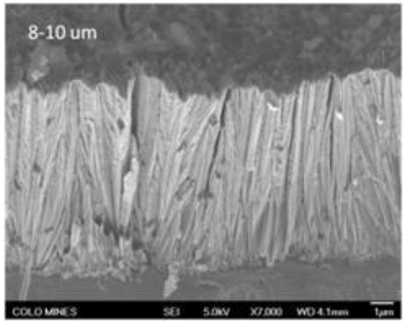


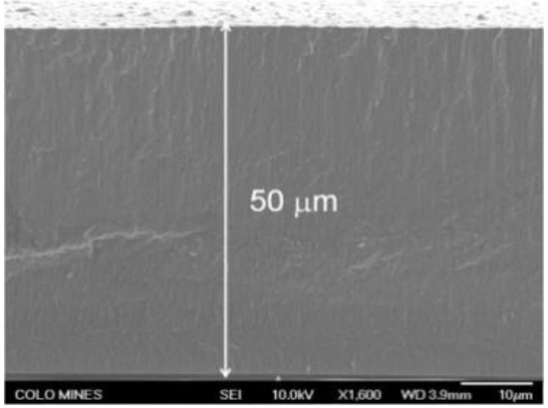
HIPIMS-MPP CrN

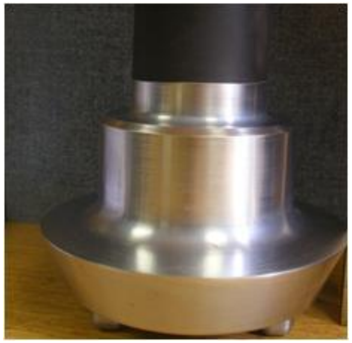




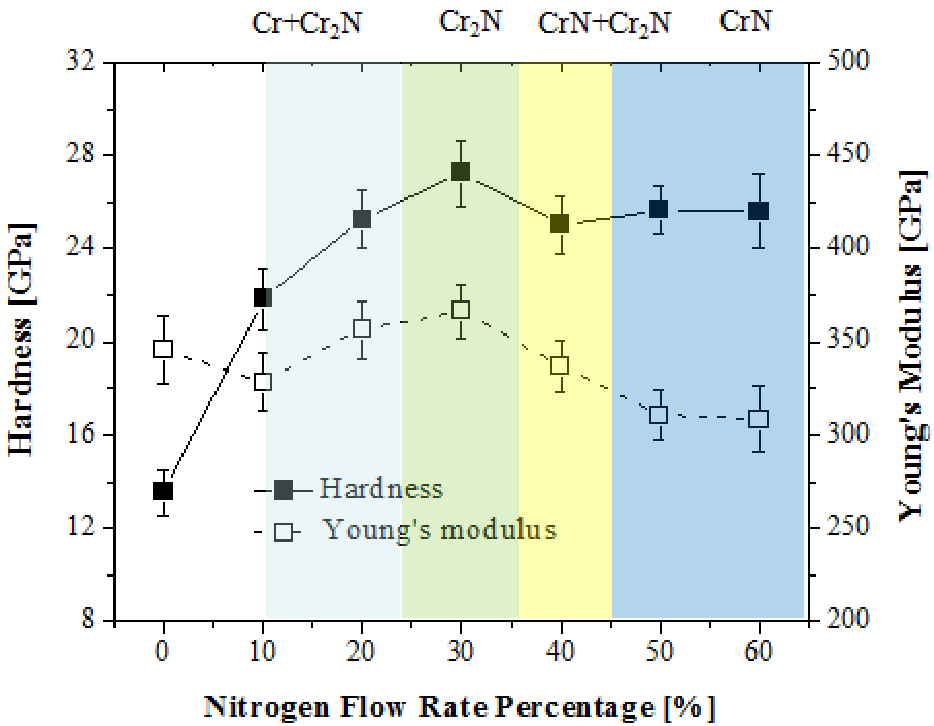
DCMS CrN



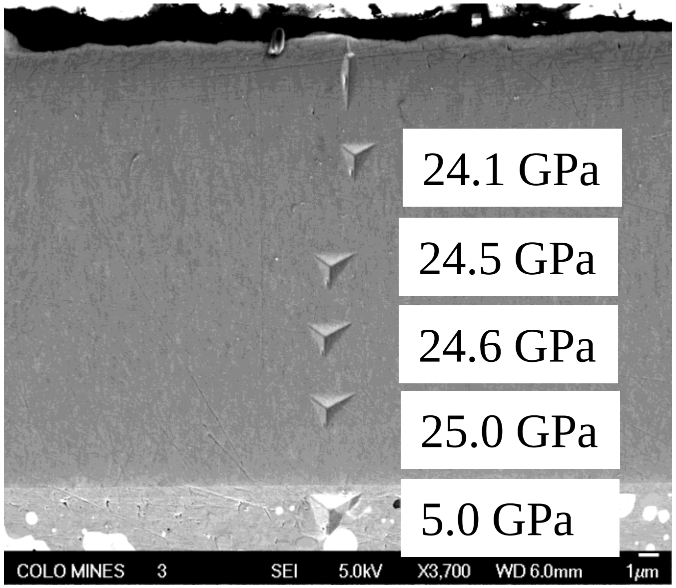


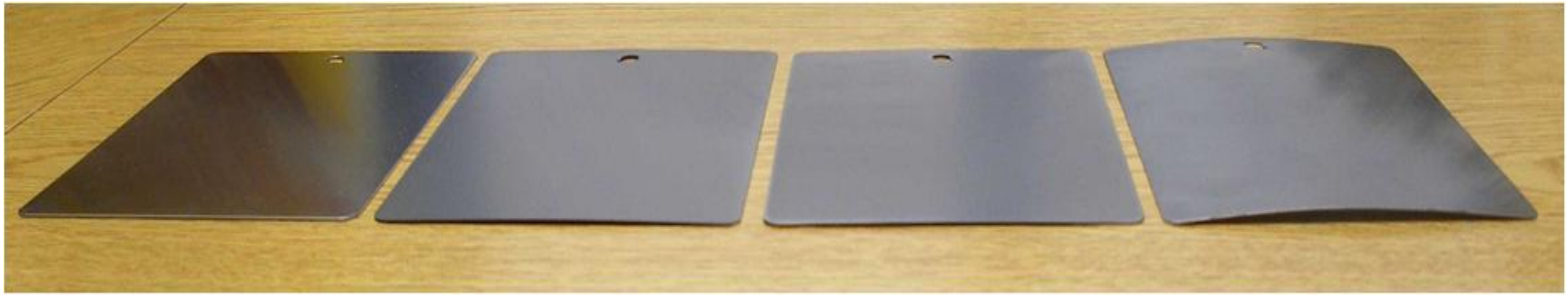


HIPIMS-MPP CrN Coatings Hardness and Young's Modulus



20 μm CrN coating

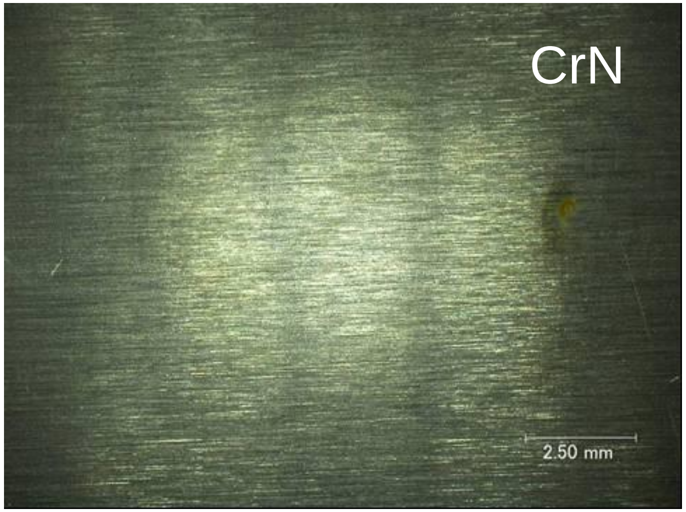


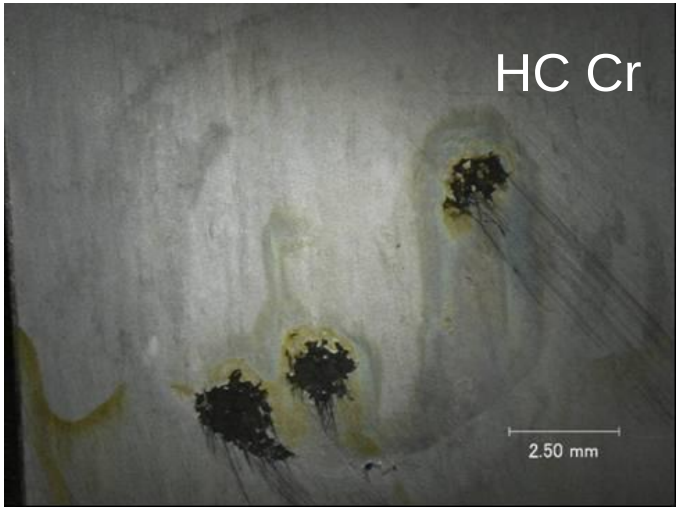


From- *Jianliang Lin, William Sproul, John Moore, Sabrina Lee, S. Myers, Surf Coat Tech 205 (2010), 3226-3234.*

ASTM Standard Corrosion Test Using a Potentiodynamic Tester & Sea Water

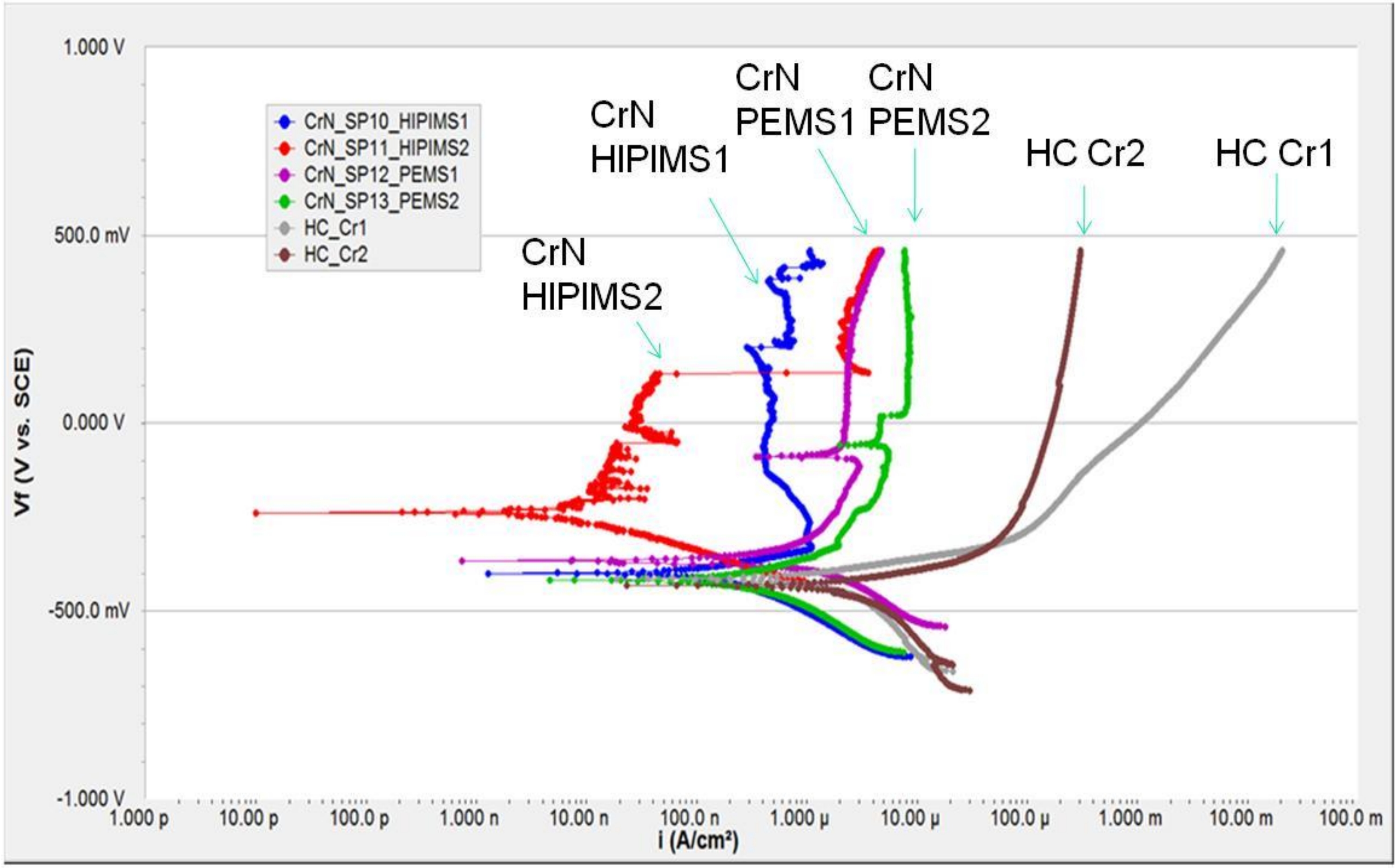
- ASTM D1141-98 “Standard Practice for the Preparation of Substitute Ocean Water”
- ASTM G3-89 “Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing”
- ASTM G5-94 “Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements”





ASTM Standard Corrosion Test
Potentiodynamic Polarization Test

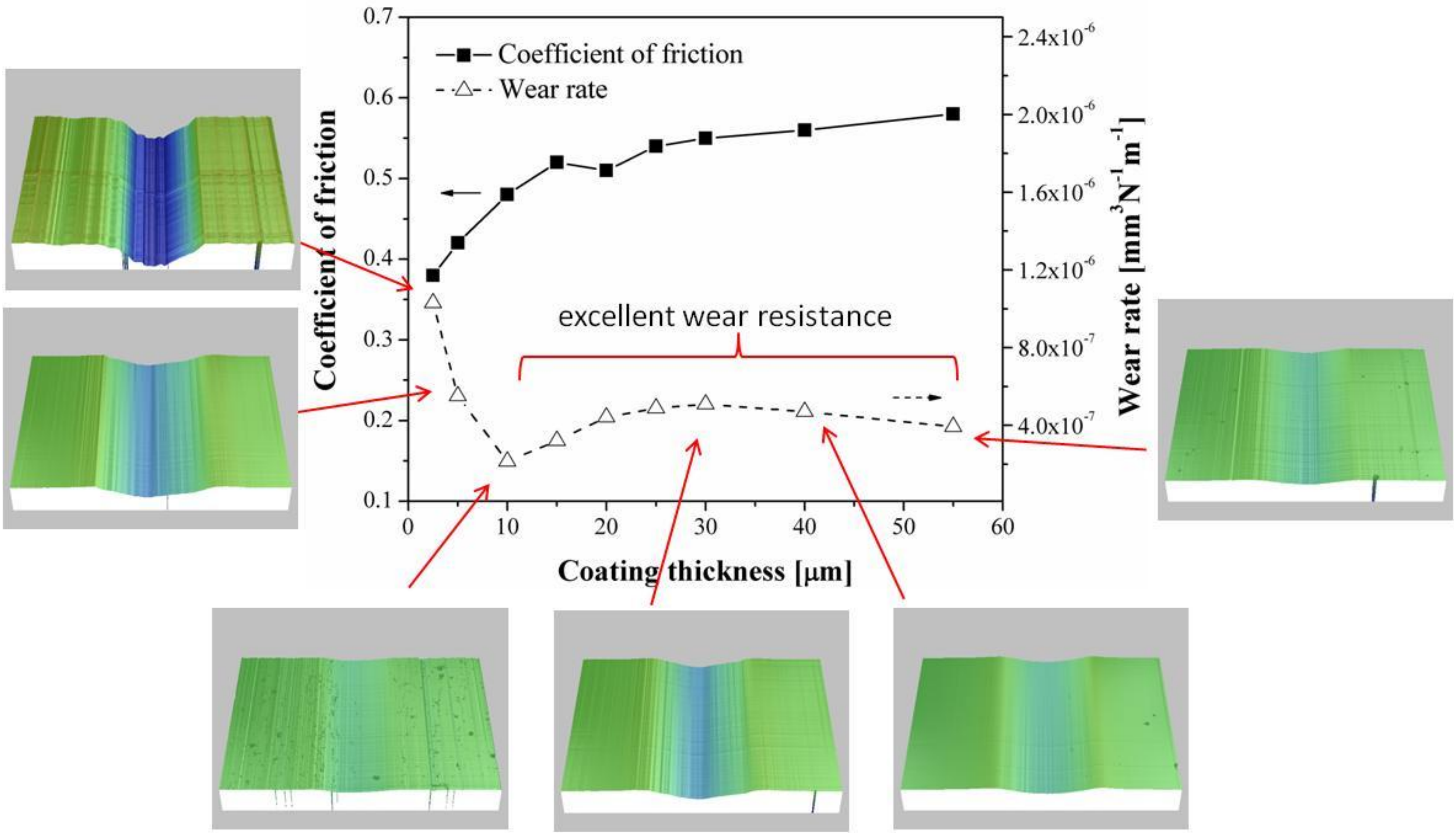
PEMS and HIPIMS CrN coatings showed 2 orders of magnitude improved corrosion resistance compared to electroplated HC Cr



Current Density in μA/cm^2, Log Scale

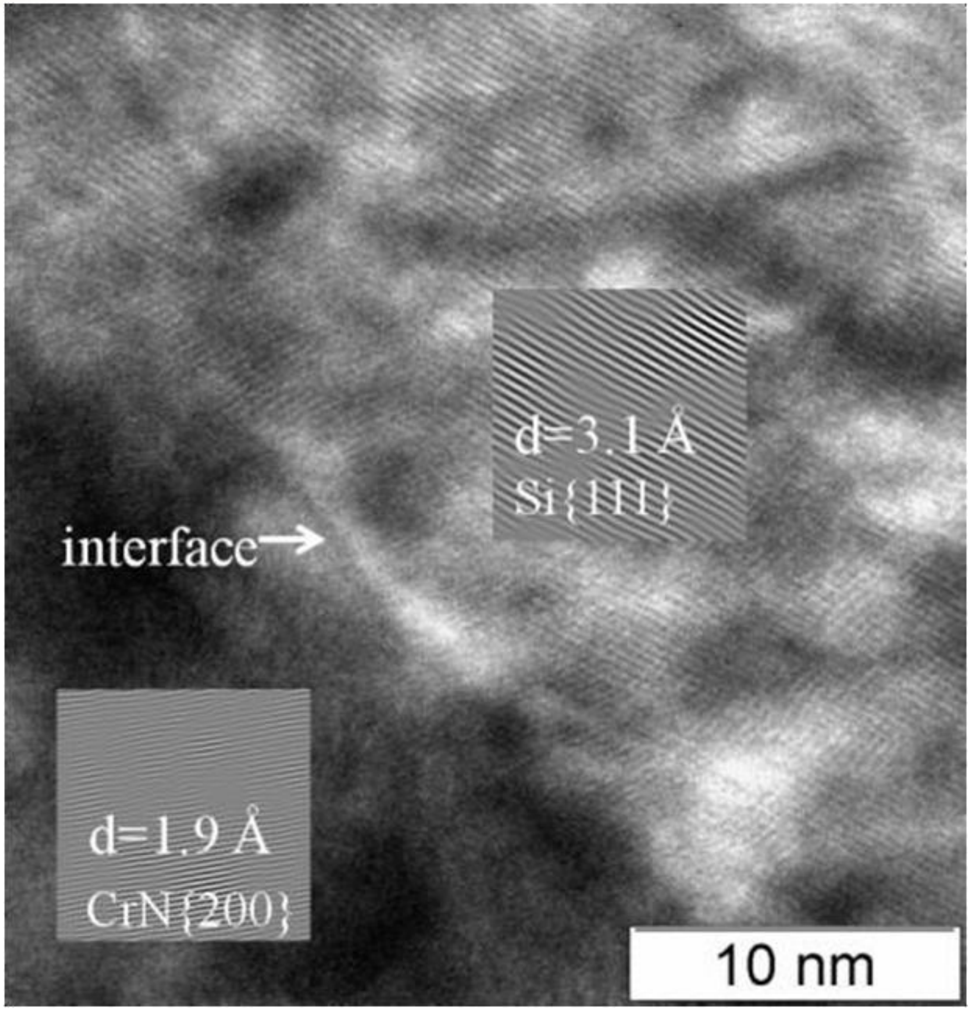
HIPIMS-MPP CrN Coatings Coefficient of Friction and Wear Rate

Test conditions: 10 N normal load, 200 rpm, 5hr, sliding against a 5 mm Al_2O_3 ball



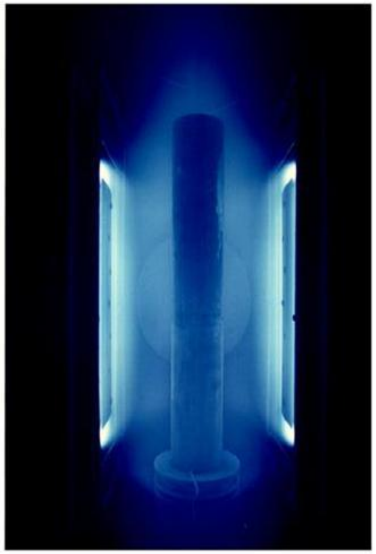
* Jianliang Lin, William Sproul, John Moore, Sabrina Lee, S. Myers, Surf Coat Tech 205 (2010), 3226-3234.

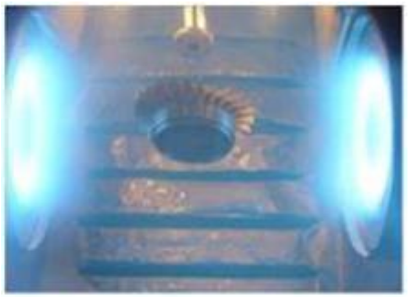
Clean and Dense Interface Microstructure of MPP CrN Film on Si

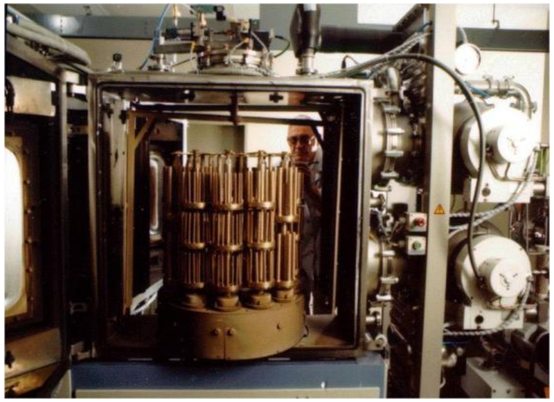


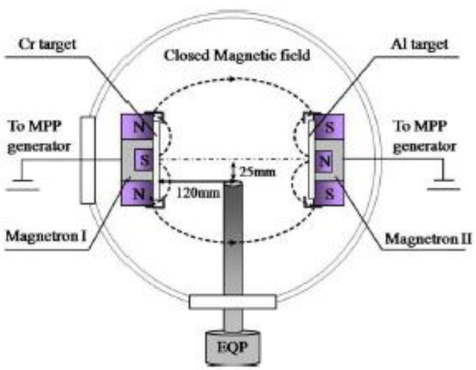
From- J. Lin, W.D. Sproul, J. Moore, S. L. Lee, R. Chistyakov, 'Recent advances in Modulated Pulsed Power Magnetron Sputtering for Surface Engineering', JOM, June (2011) 48-58.

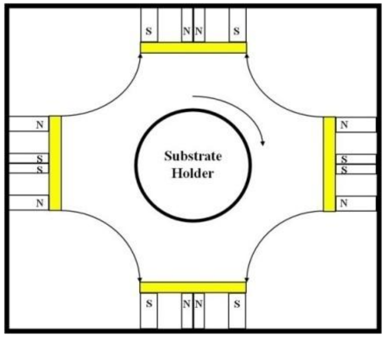
State-of-Art PVD Deposition Systems

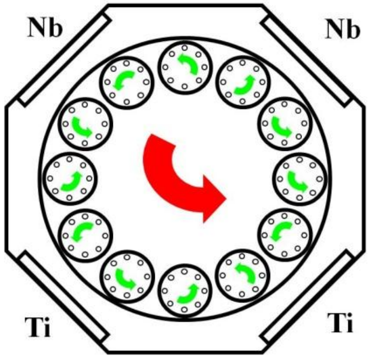


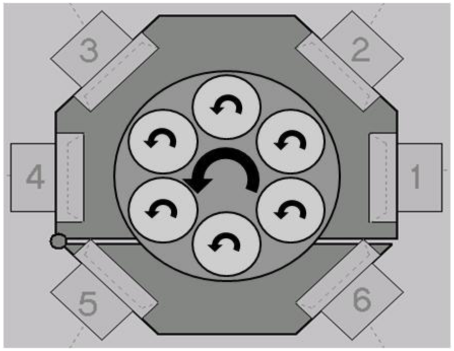












Conclusions

- *New PVD technology PEM with higher plasma density, and HIPIMS-MPP processes generating high intensity metal plasma for deposition of dense quality coatings with less columnar microstructure.*

- *New technology successfully deposited Ta 100-150 µm on 120mm diameter cylinder bore; Ta phase is sensitive to deposition parameters.*

- *New technology successfully deposited 10-55 μm fcc CrN coatings on steel; formation of CrN and Cr_2N phases depends on N_2 concentration.*

- *New thick PVD Ta demonstrated dense bcc Ta coatings with excellent ductility, microstructure, and high temperature properties.*

- *New thick PVD CrN demonstrated dense coatings, good microstructure, high hardness, good modulus, superior corrosion resistance, superior wear resistance properties.*

- *New technology can deposit environmental-friendly coatings, Ta & CrN, for potential replacement of production HC Cr coatings for ordnance.*