



CRIAQ MANU419 project meeting

Nanocomposite Coatings Deposited by HiPIMS for Liquid Impingement Erosion (LIE) Resistance Application

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Important properties of coating for protection against LIE

Good adhesion to substrate

High resilience and toughness

High fatigue strength

Lower residual stress

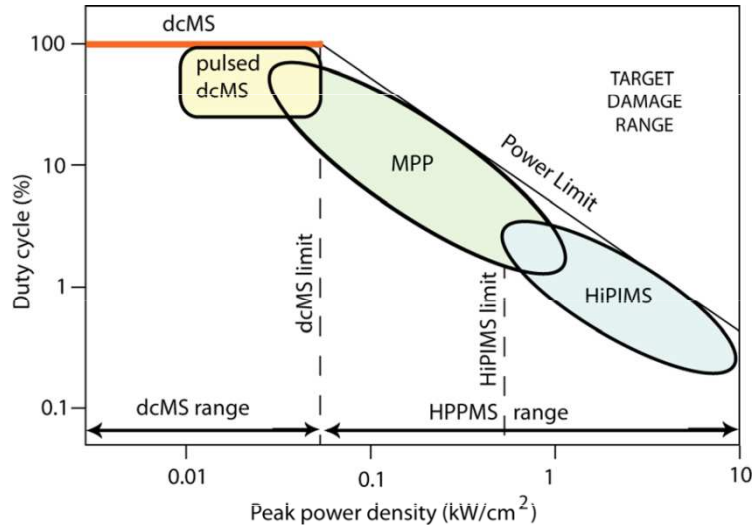
High thickness (5-25 μm) with dense microstructure

Functionally graded architectures with multifunctional properties

Is it possible to achieve above properties by using
HIPIMS/MPPMS ???

Yet to be EXPLORED !!!!

HIPIMS/MPPMS (Ionised PVD)



Plasma technique

Peak Power Density
 P_t (kW/cm²)

DCMS and Pulsed DC

< 0.05

MPPMS

$0.05 < P_t < 0.5$

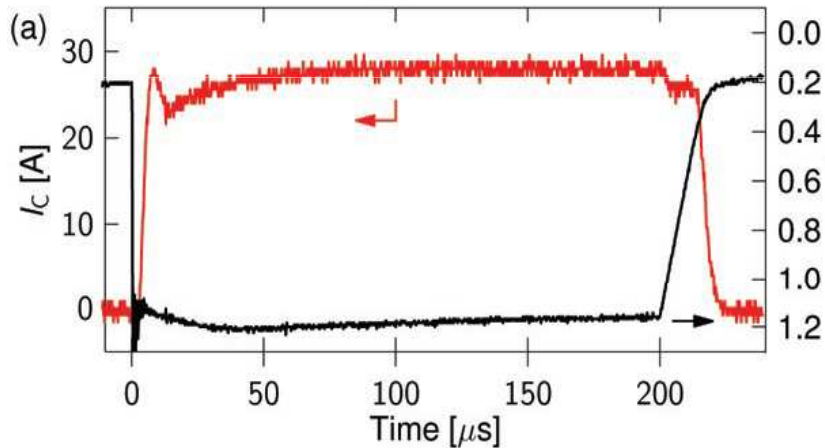
HiPIMS

$0.5 < P_t < 10$

Ref: Gudmundsson et.al, JVSTA, 30, 031801

High Power Impulse Magnetron Sputtering

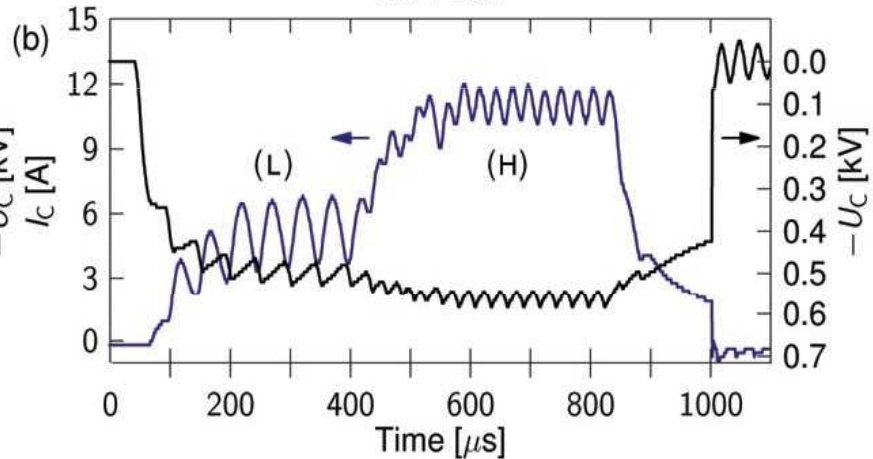
HiPIMS



Rectangular voltage pulse
Pulse duration 50-200 μ s
Duty cycle 1-10%

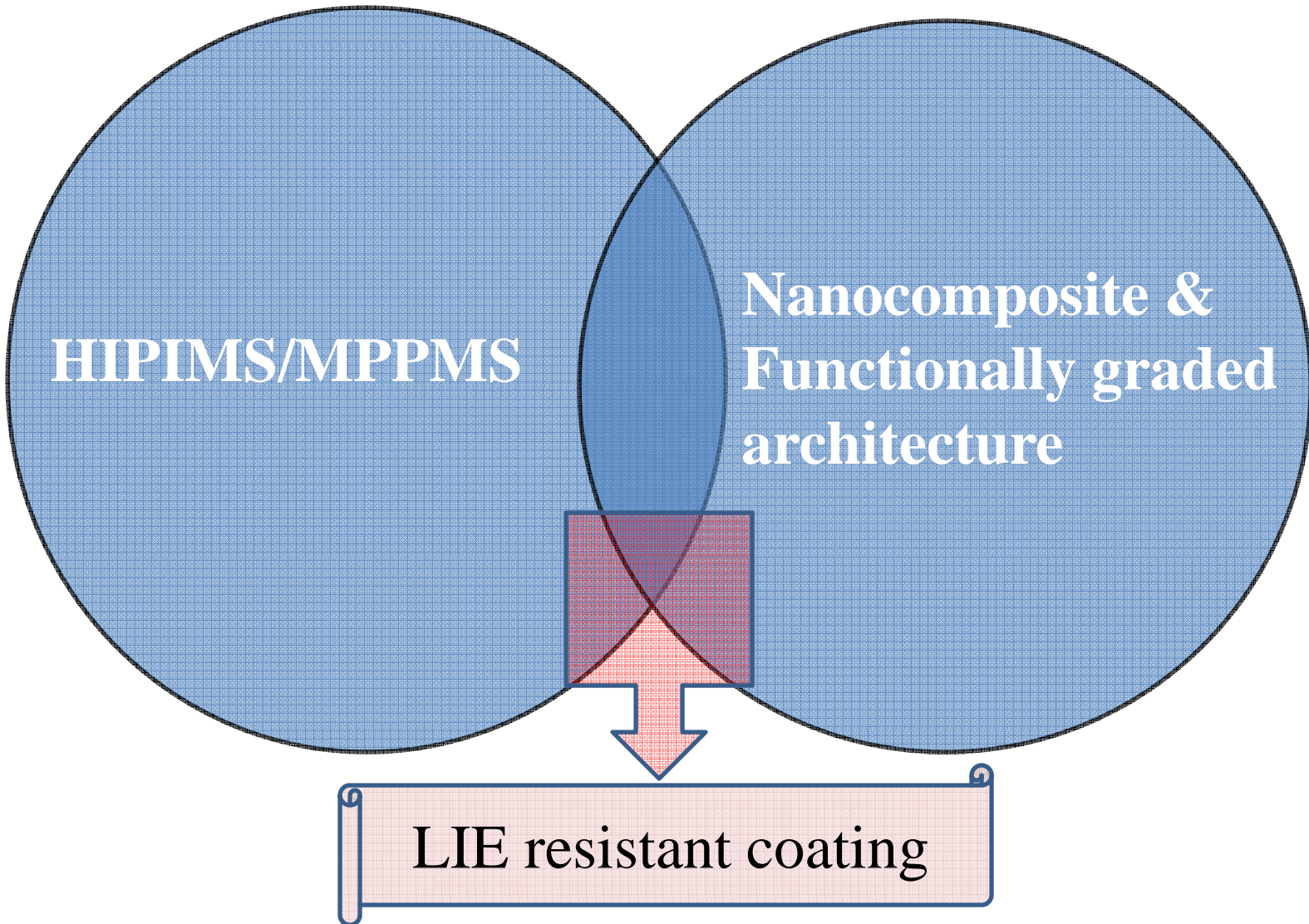
Modulated Pulse Power Magnetron Sputtering

MPPMS

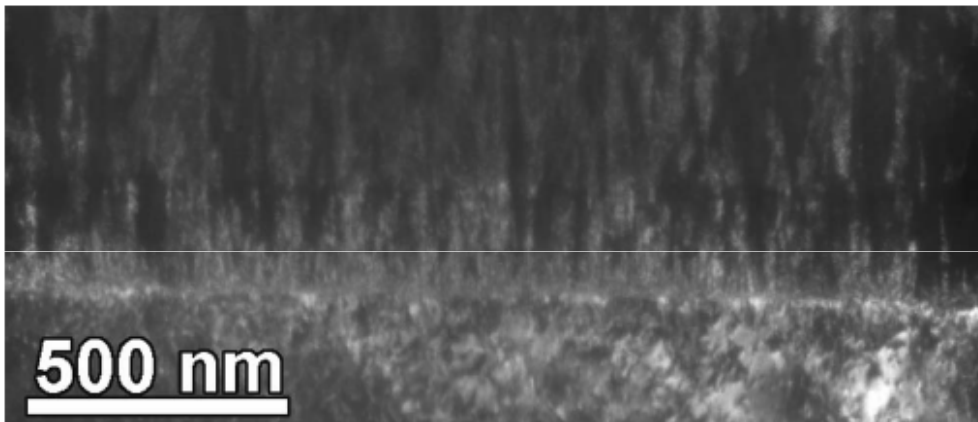
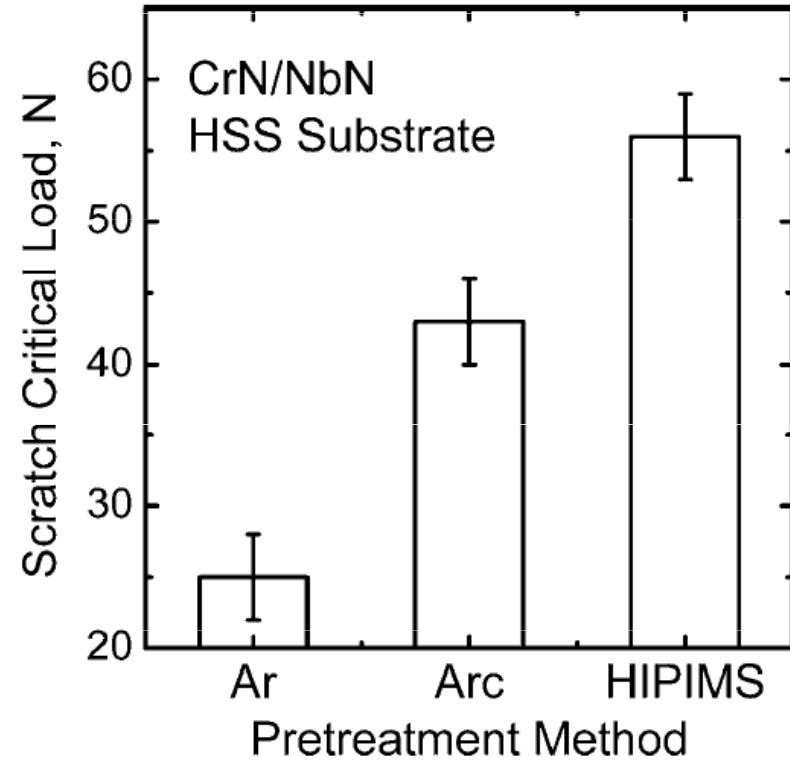
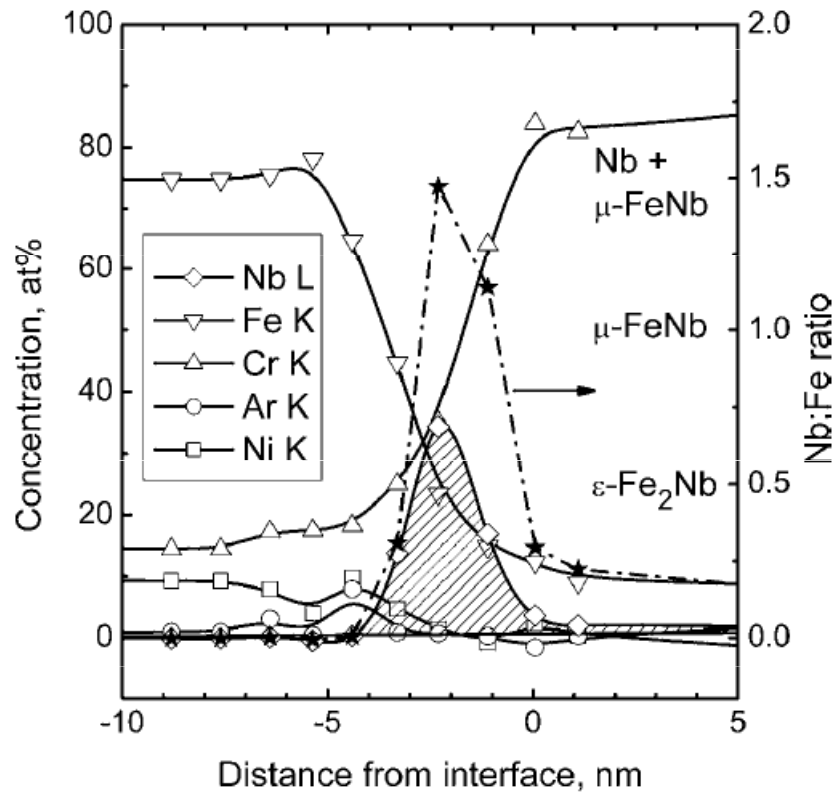


Sequence of micropulses ($T_p \sim 10 \mu$ s)
Pulse duration 400-3000 μ s
Duty cycle – 10-28%

Strategy to reach the objective



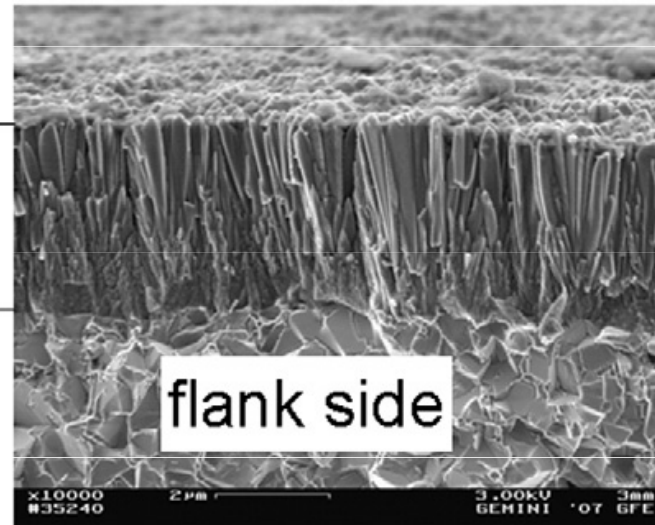
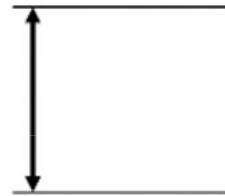
Advantage of HIPIMS 1: Interface engineering by HIPIMS



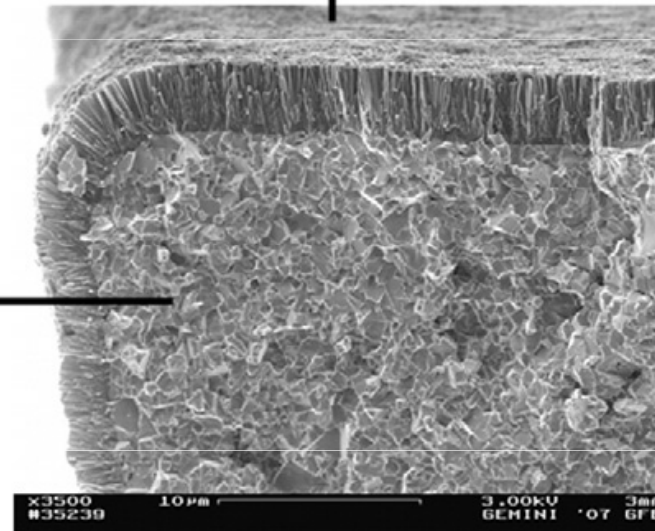
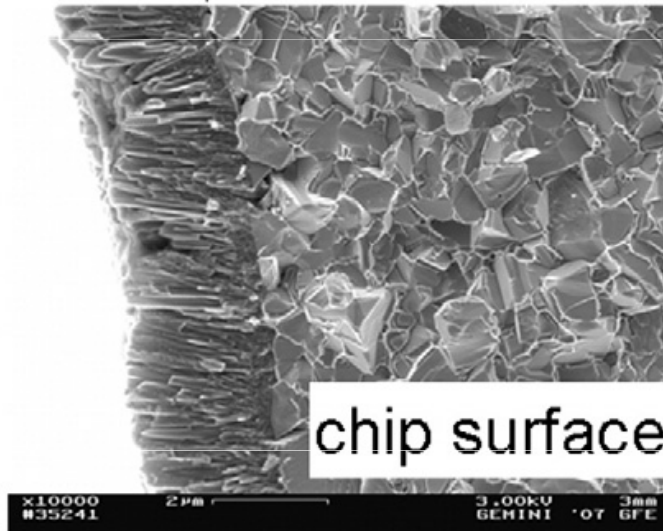
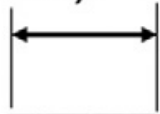
Ehiasarian et.al. J. Appl. Phys.
101, 054301 (2007)

Advantage of HIPIMS 2. Deposition on complex shaped substrates

Deposition rate
(flank): $1.45 \mu\text{m/h}$



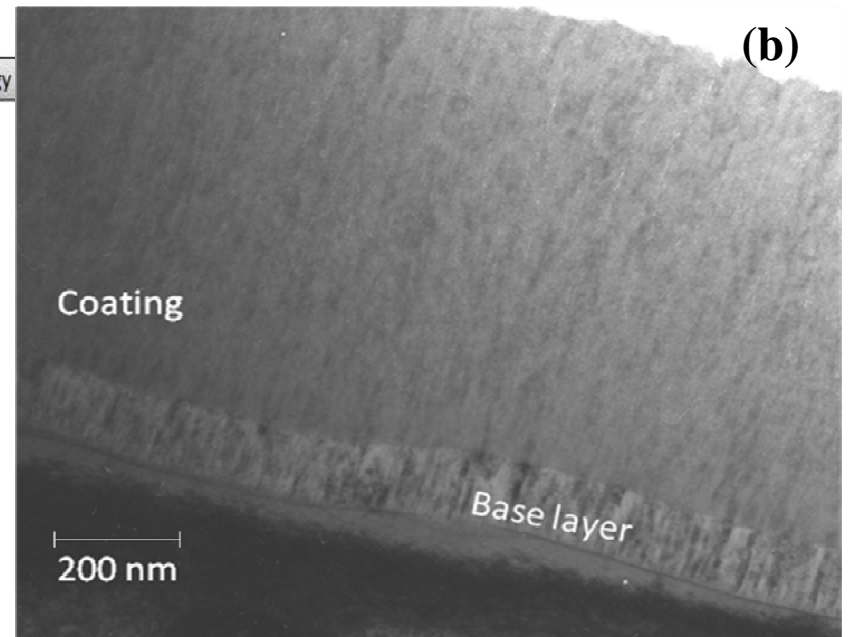
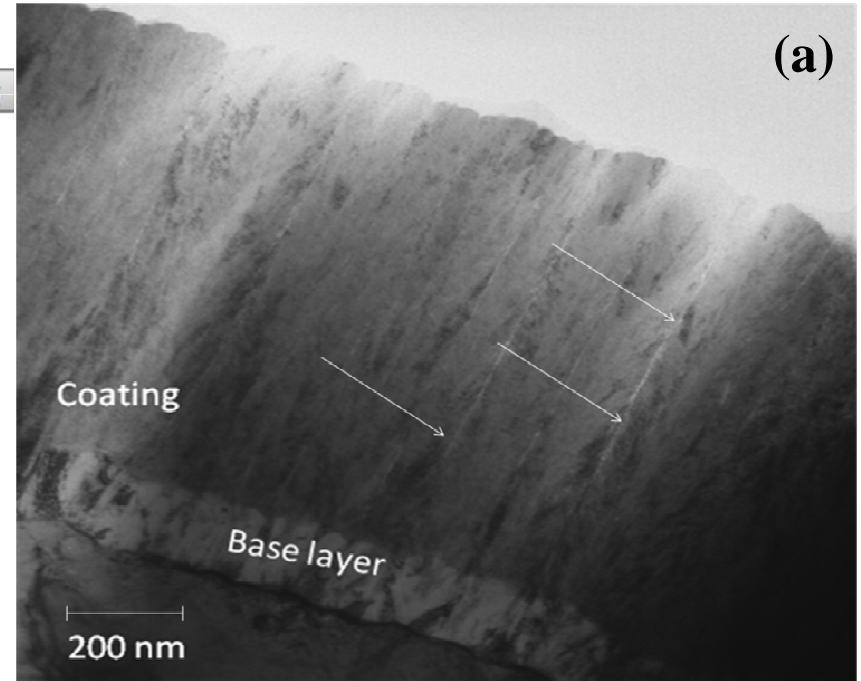
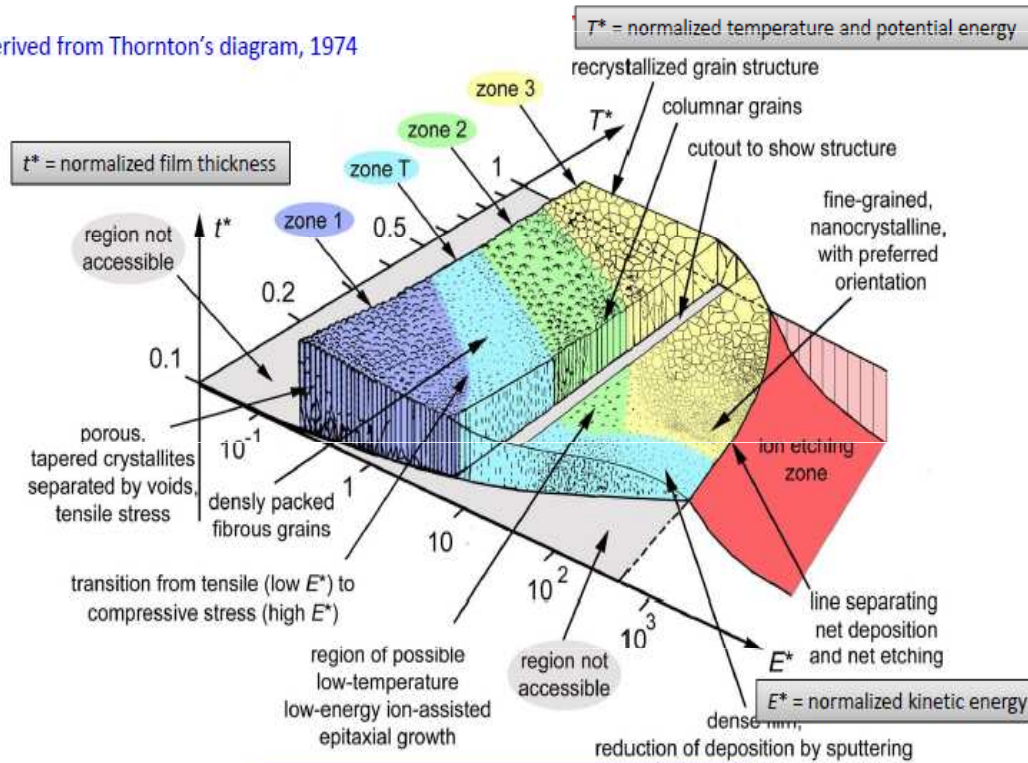
Deposition rate
(rake): $1.03 \mu\text{m/h}$



Ref: K. Bobzin et. al, J. Mater. Process. Technol. 209 (2008) 165.

Advantage of HIPIMS 3: thin Film Microstructure

derived from Thornton's diagram, 1974

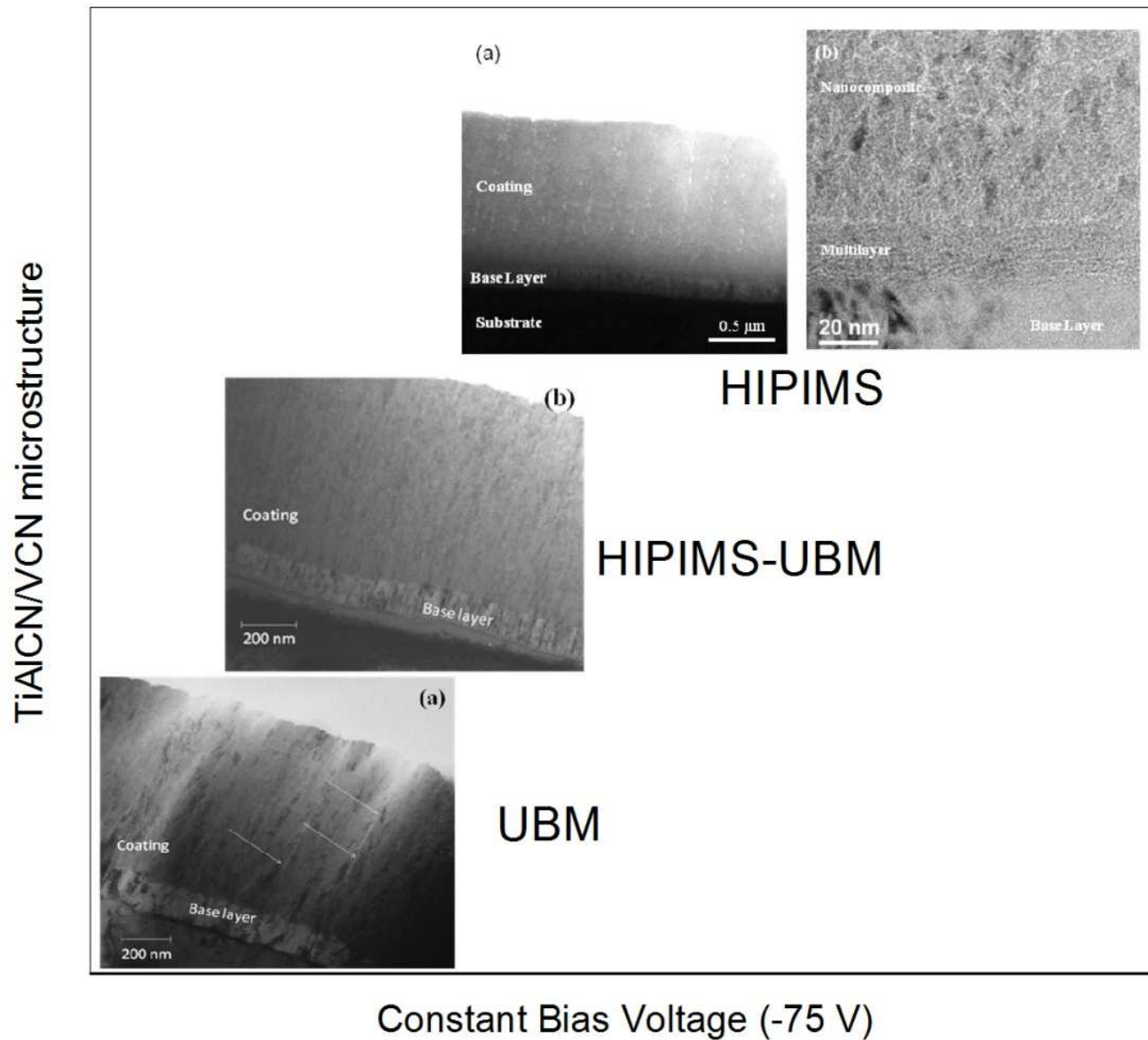


Biassing is to control the energy of incoming ions.

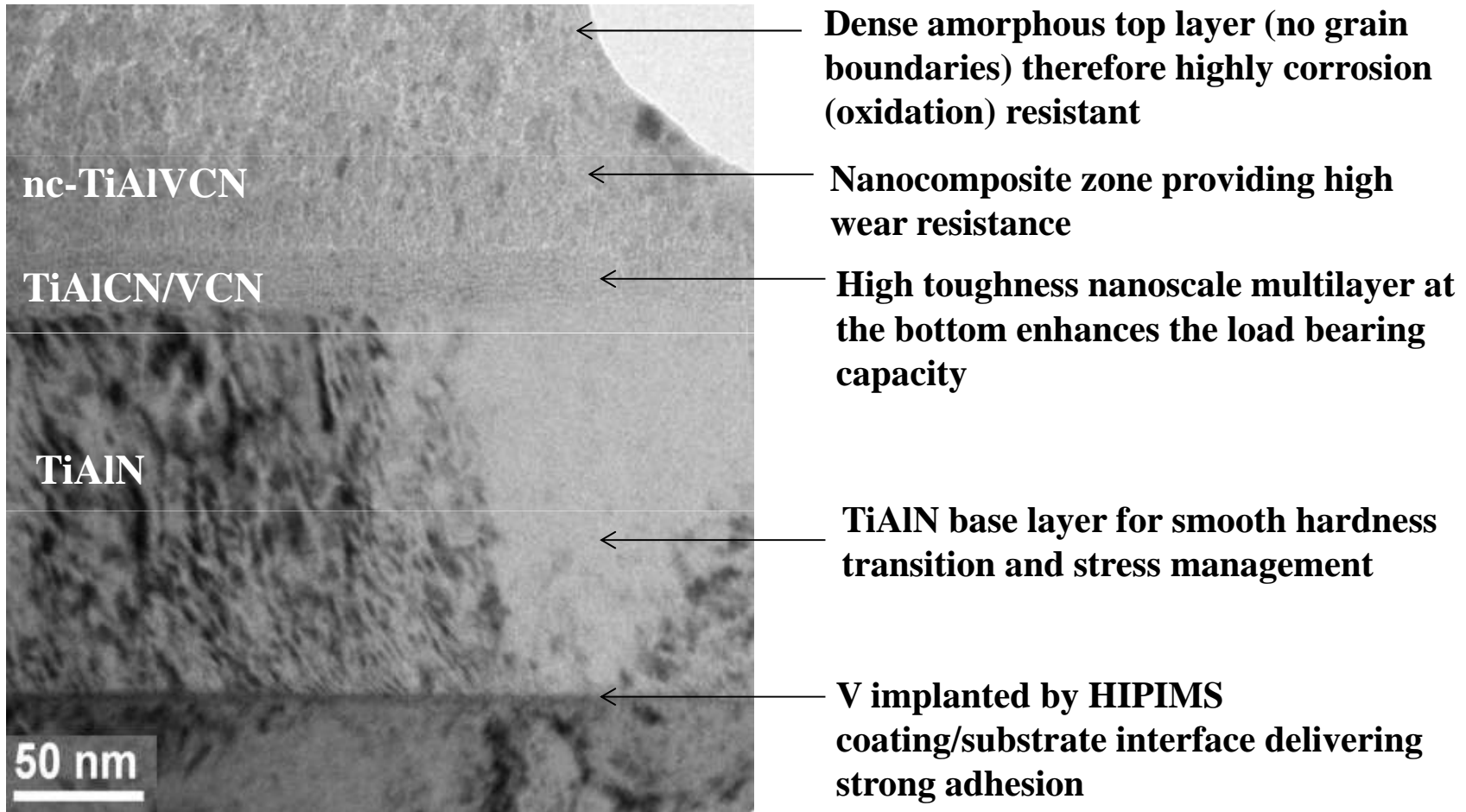
Low kinetic energy (10 eV – 100 eV) of arrival ions during ion assisted growth.

Ref: A. Anders, Thin Solid Films 518 (2010) 4087–4090

Advantage of HIPIMS 4. Tailoring Phase composition and graded architectures



Example of earlier work on HIPIMS



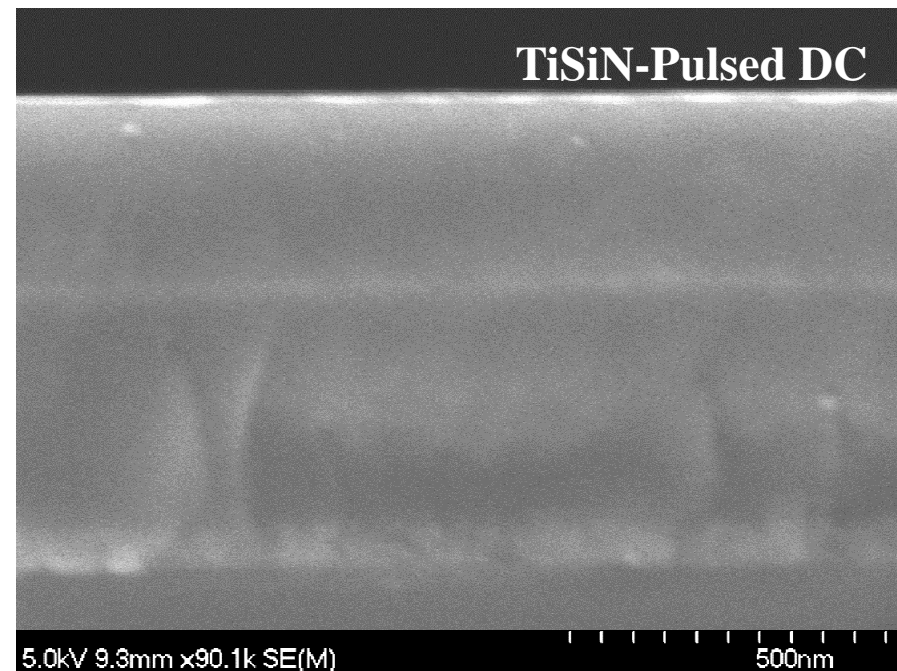
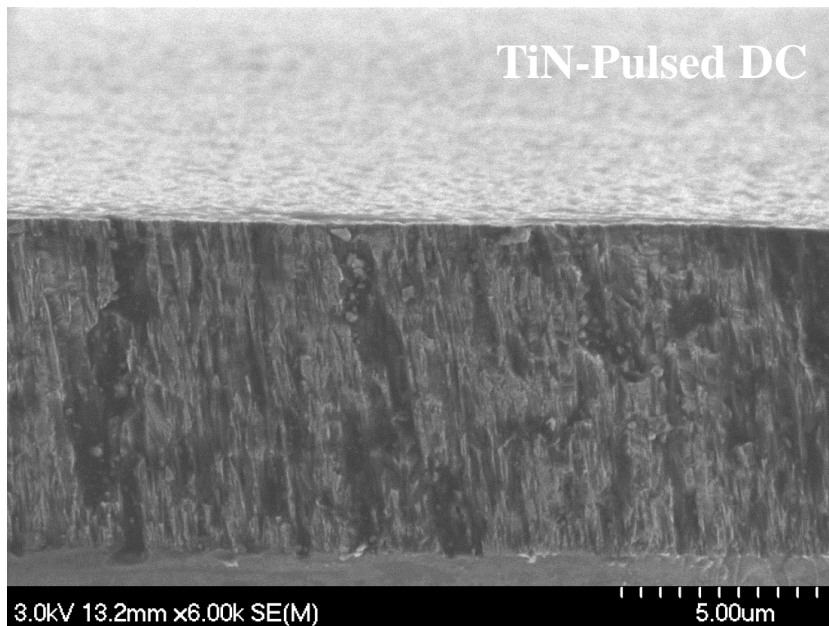
TEM cross section of nanostructured TiAlCN/VCN coating

Ref: G. Kamath, P. Hovsepian and A. Ehiasarian, SVC 2011

What has been done earlier ?

Optimization of TiN and TiSiN by Pulsed DC

Coating	Technique	Thickness (μm)	Hardness (GPa)	Young's modulus (Gpa)	Ar:N ₂	Power	
						Ti	Si
TiN	PDC	8	25	~ 301	3:1	0	0
TiSiN	PDC	1	32	~ 300	3:1	400	50
TiSiN	PDC	1	37	~ 300	3:1	400	100

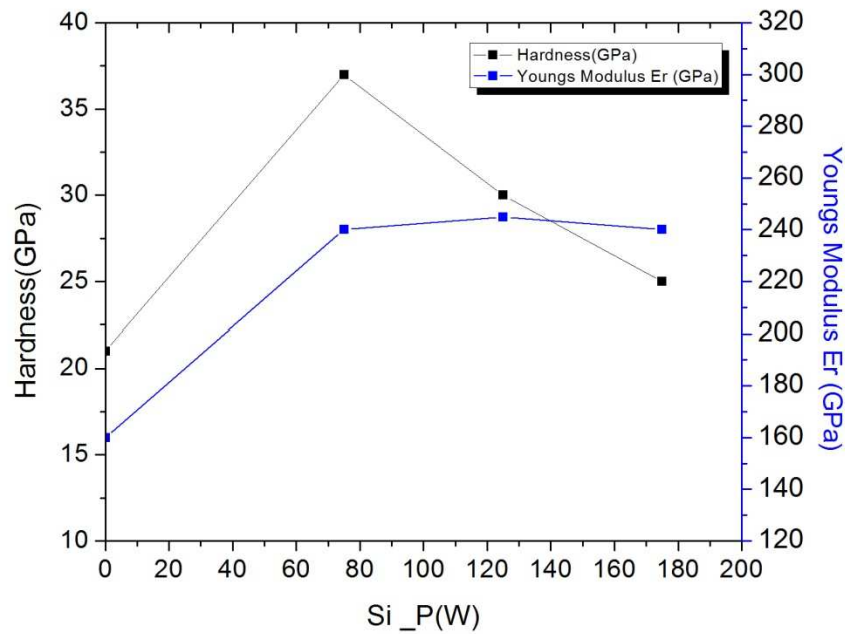


Deposition parameters for TiSiN and TiN by MPPMS and Pulsed DC

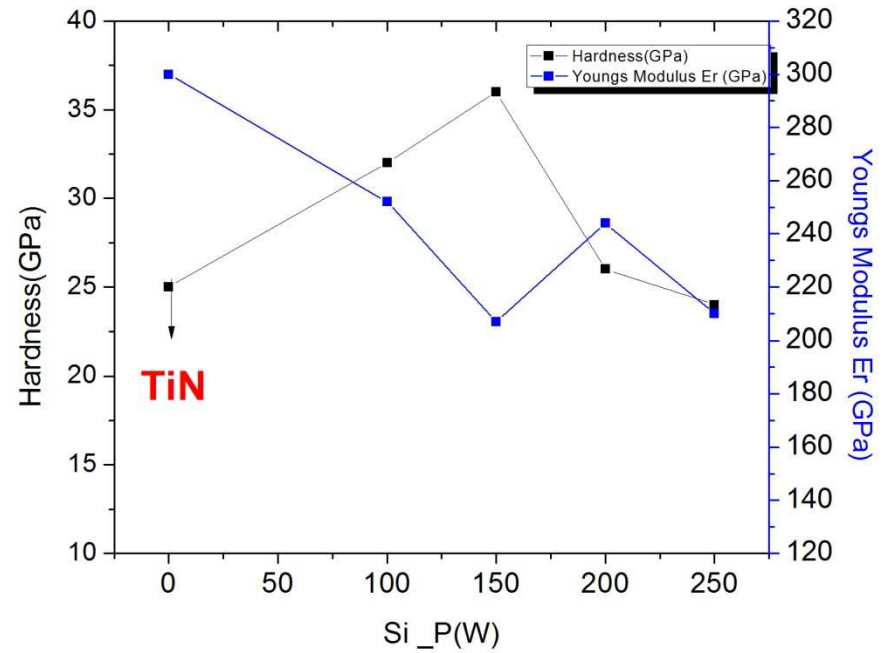
		TiN		TiSiN	
		Pinnacle	MPPMS	PULSED DC	MPPMS
Power supply					
Pressure		5.2 mTorr	4.5 mTorr	5.2 mTorr	4.6 mTorr
Ar : N ₂ flow ratio		3: 1 sccm	16:1 sccm	3: 1 sccm	16 : 1 sccm
Applied power	Ti	400 W	300 W	400 W	300 W
	Si	0	0	150	75
Cathode voltage		-350 V average	-600 V average	-350 V average	-600 V average
Bias voltage		- 100 V	-75 V	-100 V	-70 V
Pulse length		3 μs	1300 μs	3 μs	1300 μs
Frequency		300 kHz	75 Hz	300 kHz	75 Hz
Dep. rate		0.13 nm/s	0.15 nm/s	0.18 nm/s	0.20 nm/s

Mechanical properties of TiSiN by MPPMS and Pulsed DC

TiSiN by MPPMS



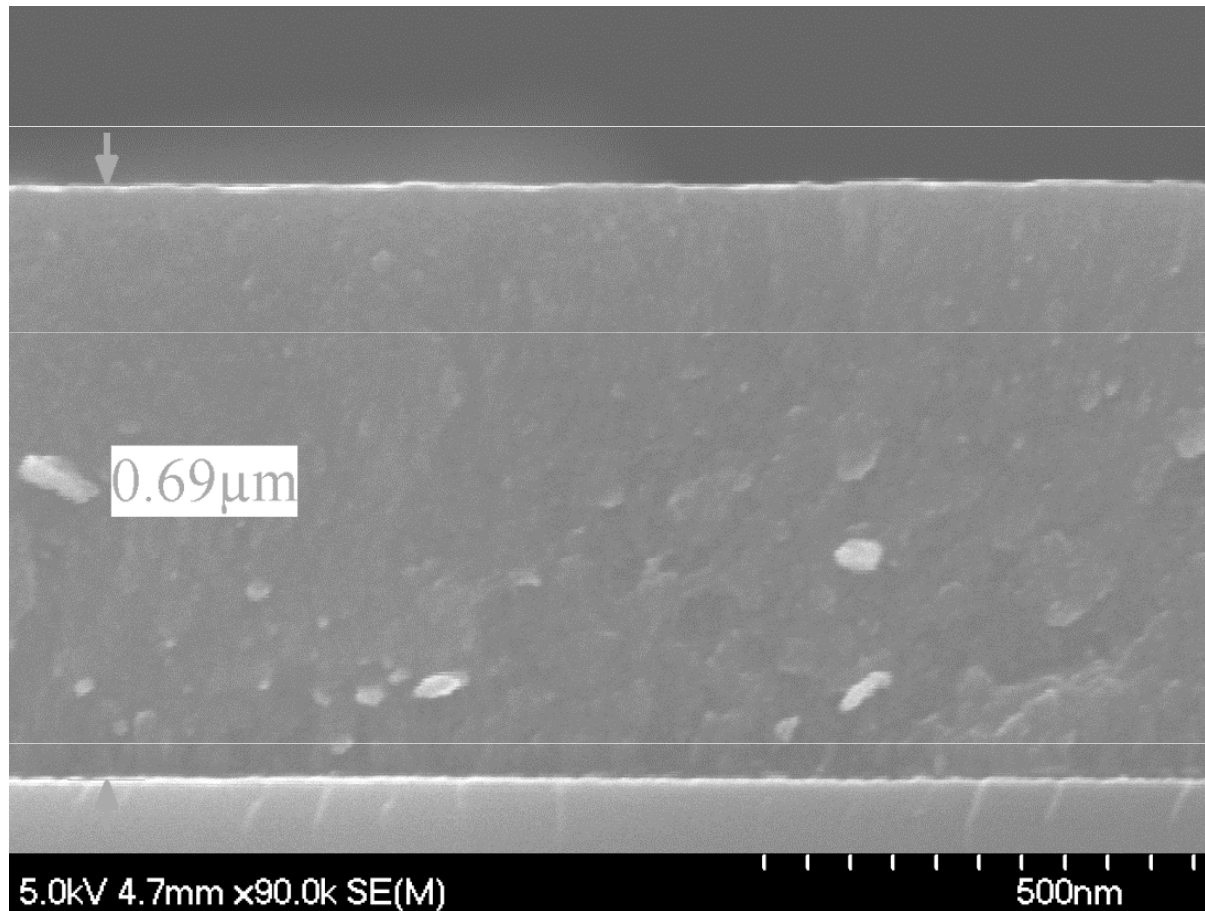
TiSiN by Pulsed DC



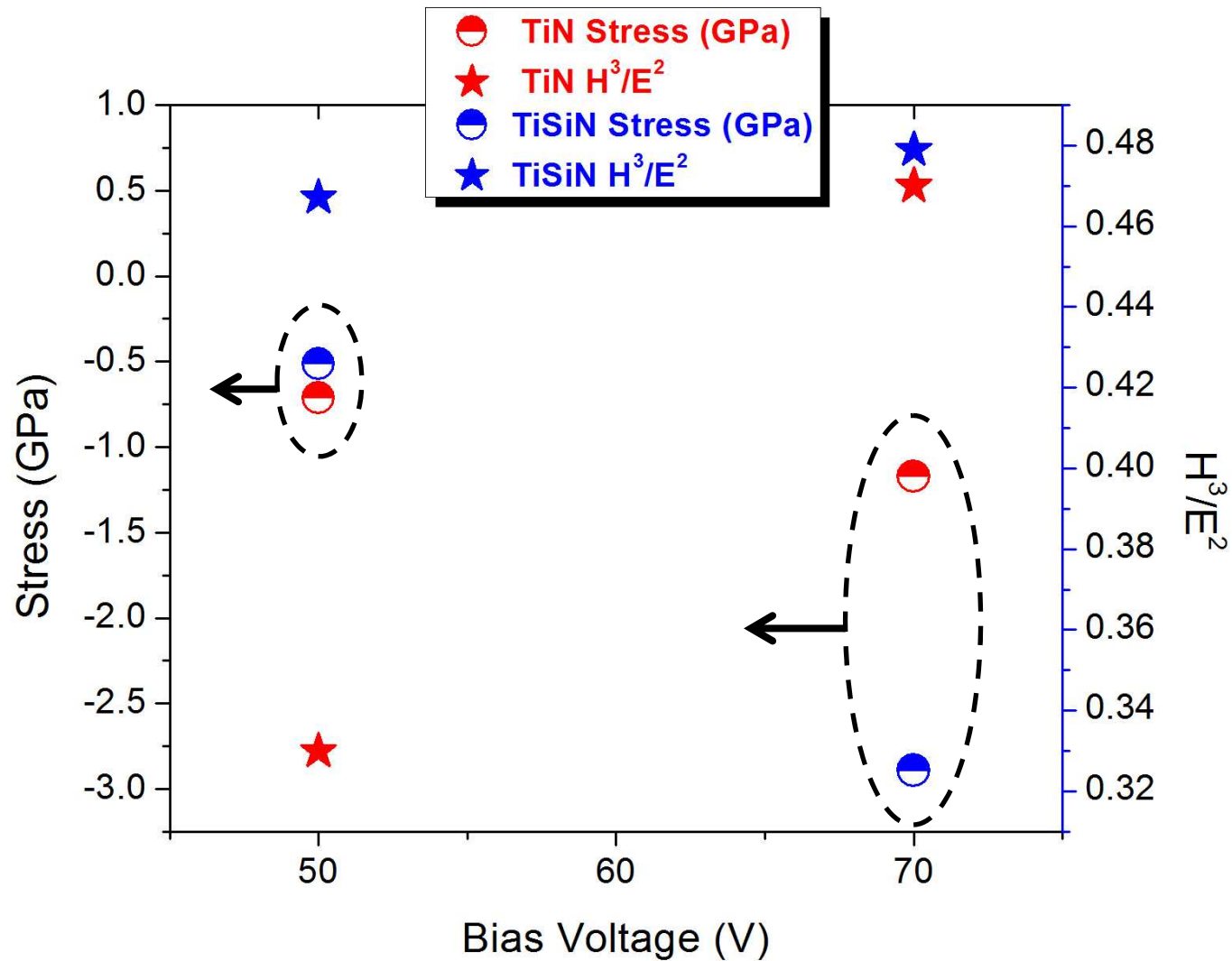
SEM microstructure of TiSiN deposited by HIPIMS

TiSiN by HIPIMS

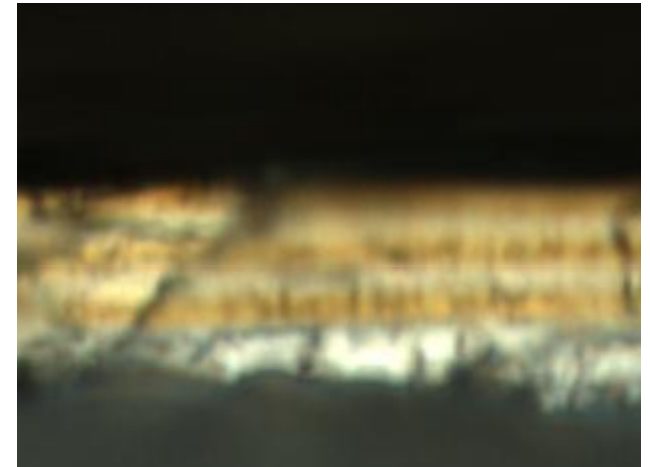
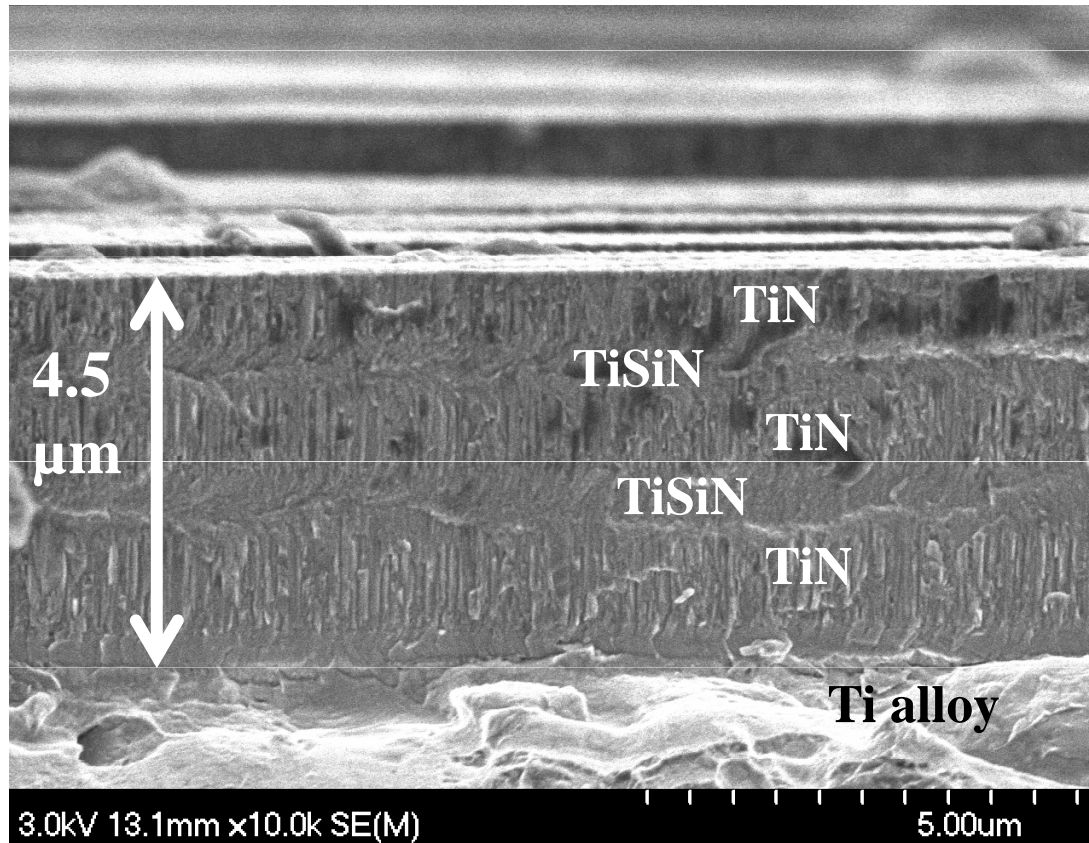
T (μm)	H (GP)	E_r (GPa)	Power (W)	
			Ti	Si
0.7	37	240	500	75



Mechanical Properties and Stress of TiN and TiSiN deposited by HIPIMS



TiN/TiSiN multilayer deposited by HIPIMS



TiN/TiSiN by HIPIMS

T (μm)	H (GP)	E _r (GPa)	Power (W)	
			Ti	Si
4.5	22	200	500	75

Conclusion :

- TiN and TiSiN is successfully optimized by HIPIMS for optimal mechanical properties.
- About 5 μm thick TiN/TiSiN multilayer coatings were deposited by HIPIMS on Ti-alloys substrates.

Near future work include :

- Deposition of thick ($> 10 \mu\text{m}$) TiN/TiSiN multilayer on various substrates and characterization of microstructure, mechanical and tribological properties .
- Incorporate *carbon* during the growth of TiN/TiSiN multilayer in order to improve the coating's elasticity (and LIE resistance?).