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A. Anders (LBNL)
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HiPIMS

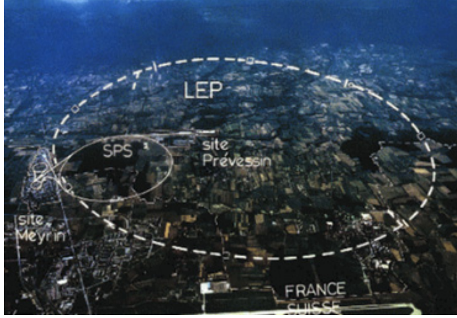
A New Generation of Films Deposition Techniques for SRF Applications

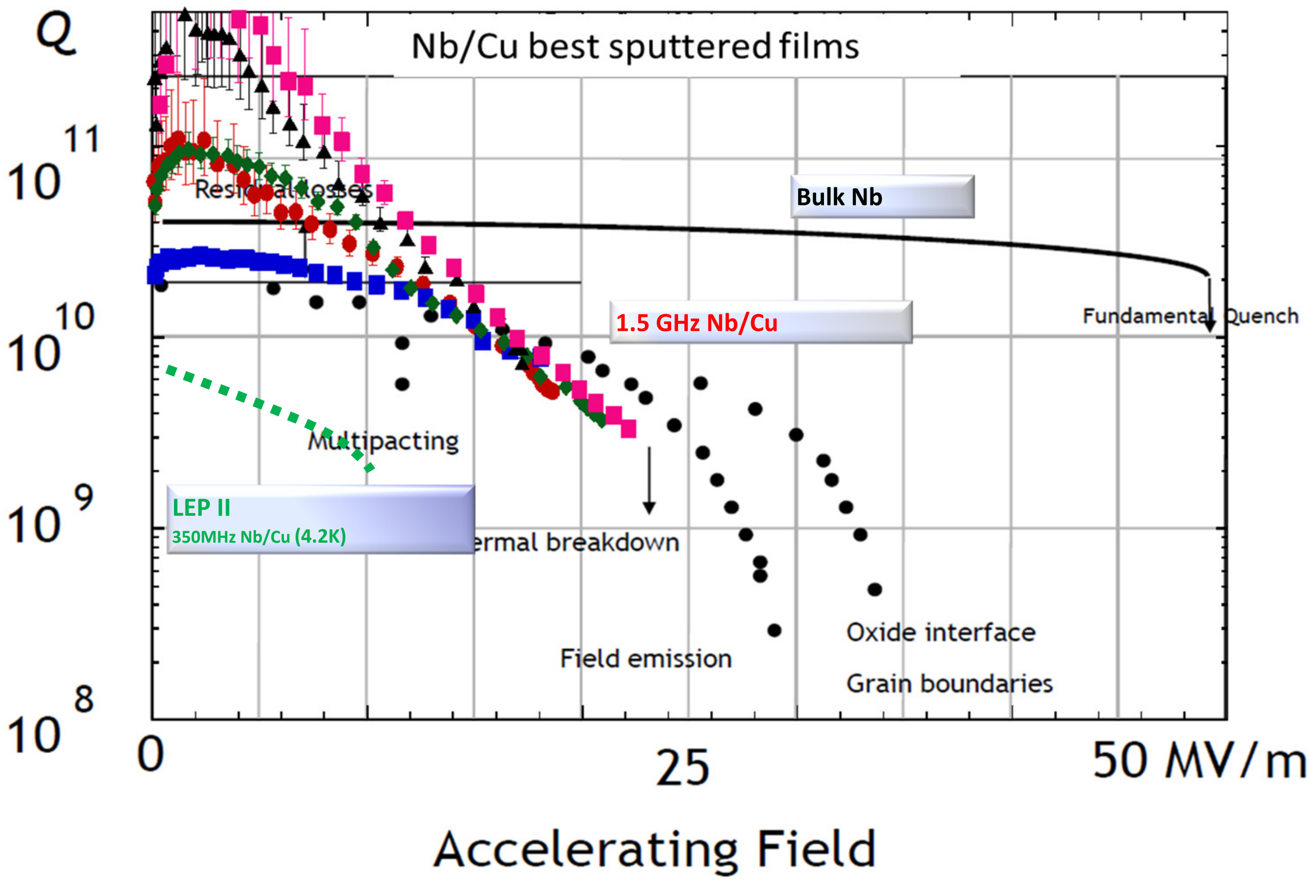
Outline

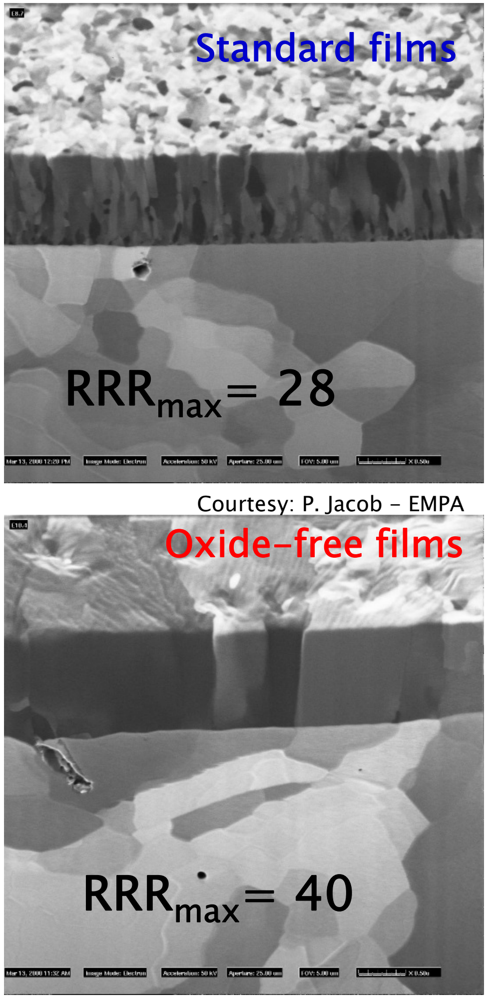
- SRF films- State of the Art
- Energetic Condensation
- Energetic Condensation Techniques
- HiPIMS Principle
- HIPIMS: an array of techniques
- Application to SRF Surfaces
- HiPIMS @ LBNL
- HiPIMS @ CERN
- HiPIMS @ JLAB
- Conclusions

Thin Films for SRF–state of the art

- •CERN LEP 2 — 272 x 353MHz Nb/Cu 4-cell cavities
- •INFN Legnaro — 52 x 160 MHZ Nb/Cu QWR



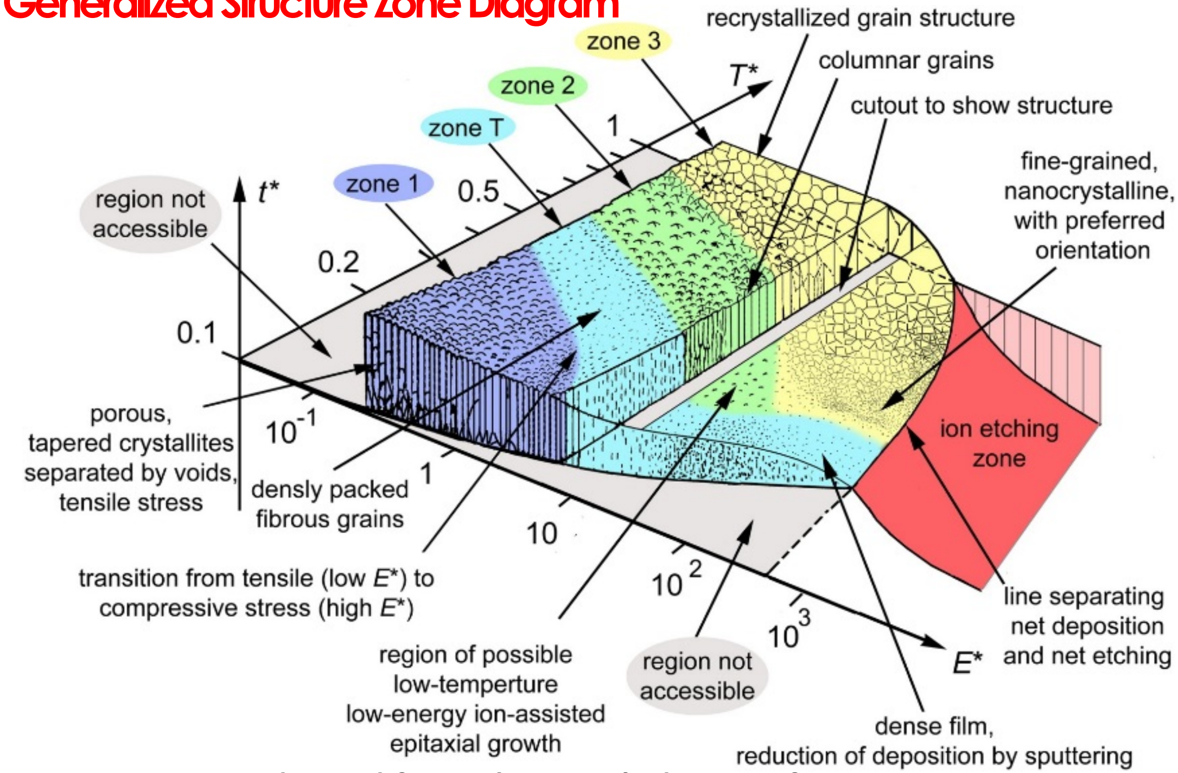




Energetic Condensation

Condensing (film-forming) species : hyper-thermal & low energies (>10 eV).

Generalized Structure Zone Diagram



derived from Thornton's diagram for sputtering (1974)

A. Anders, Thin Solid Films **518** (2010) 4087

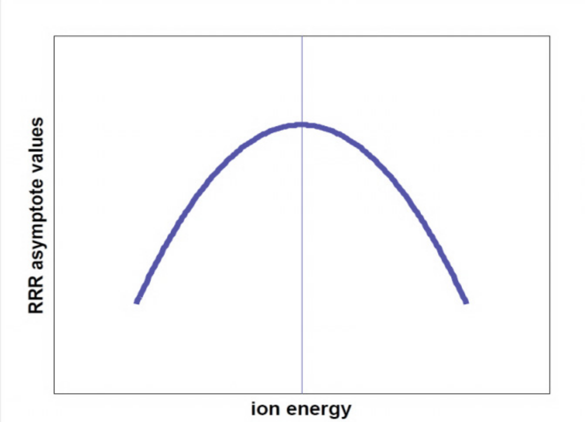
Additional energy provided by fast particles arriving at a surface ⇒ number of surface & sub-surface processes ⇒ changes in the film growth process:

- residual gases desorbed from the substrate surface
- chemical bonds may be broken and defects created thus affecting nucleation processes & film adhesion
- enhanced mobility of surface atoms
- stopping of arriving ions under the surface

⇒ Changes in
- morphology
- microstructure
- stress

As a result of these fundamental changes, energetic condensation allows the possibility of controlling the following film properties:

- Density of the film
- Film composition
- Crystal orientation may be controlled to give the possibility of low-temperature epitaxy



Energetic Condensation Techniques

A variety of techniques with distinct technologies

- High Impulse Power Magnetron sputtering (HiPIMS)
- Vacuum Arc Plasma
- Coaxial Energetic Deposition (CED)
- Electron cyclotron Resonance (ECR)

WEIOA02 Thin Film Growth by Energetic Condensation
Mahadevan Krishnan - Alameda Applied Sciences Corporation

Energetic Condensation Techniques

A variety of techniques with distinct technologies

- ❑ High Impulse Power Magnetron sputtering (HiPIMS)
- ❑ Vacuum Arc Plasma
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- ❑ Electron cyclotron Resonance (ECR)

Engineering for optimum RF performance

3 sequential phases for film growth

- ✓ **Film nucleation on the substrate**
- ✓ **Growth of an appropriate template for subsequent deposition of the final RF surface**
- ✓ **Deposition of the final surface optimized for minimum defect density.**

HIPIMS: A Form of "Ionized Sputtering"

One Approach to Energetic Deposition

"What distinguishes HIPIMS from the long-practiced pulsed sputtering?"

Technical Definition:
HIPIMS is pulsed sputtering where the peak power exceeds the average power by typically two orders of magnitude.

(implies a long pause between pulses, hence the term "impulse")

Physical Definition:
HIPIMS is pulsed sputtering where a very significant fraction of the sputtered atoms becomes ionized.

(implies that self-sputtering occurs, which may or may not be sustained by target ions)

A. Anders, Surf. Coat. Technol. **205** (2011) S1.

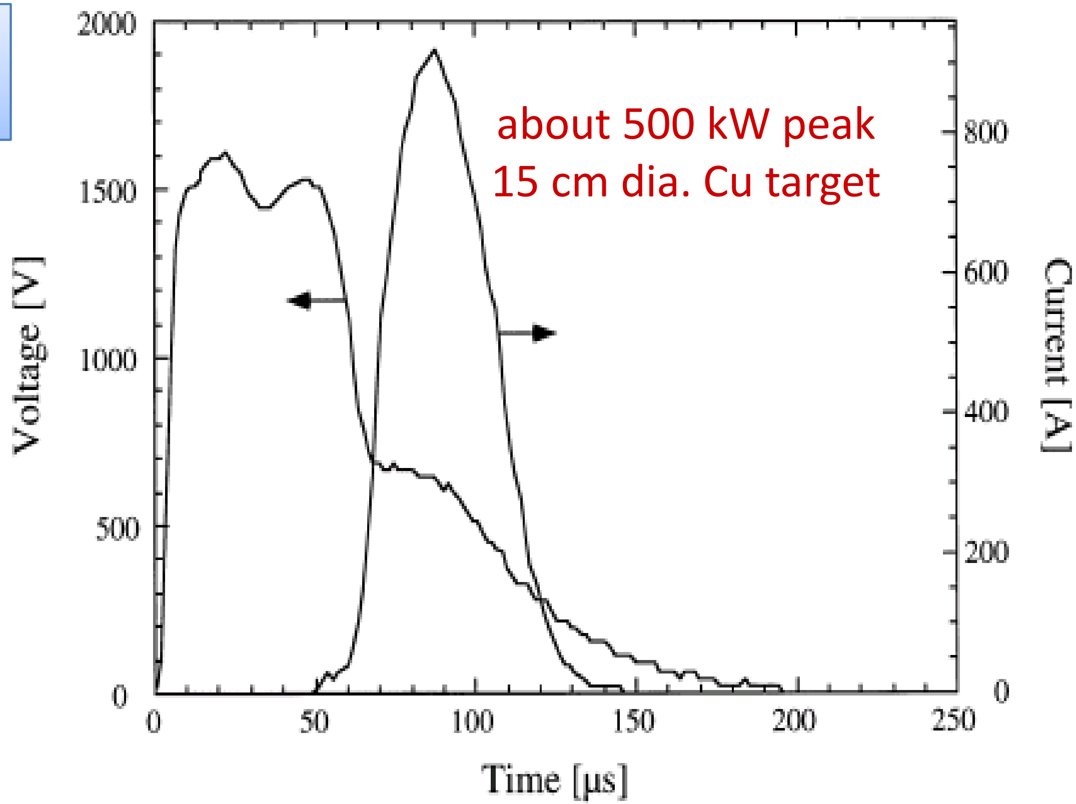
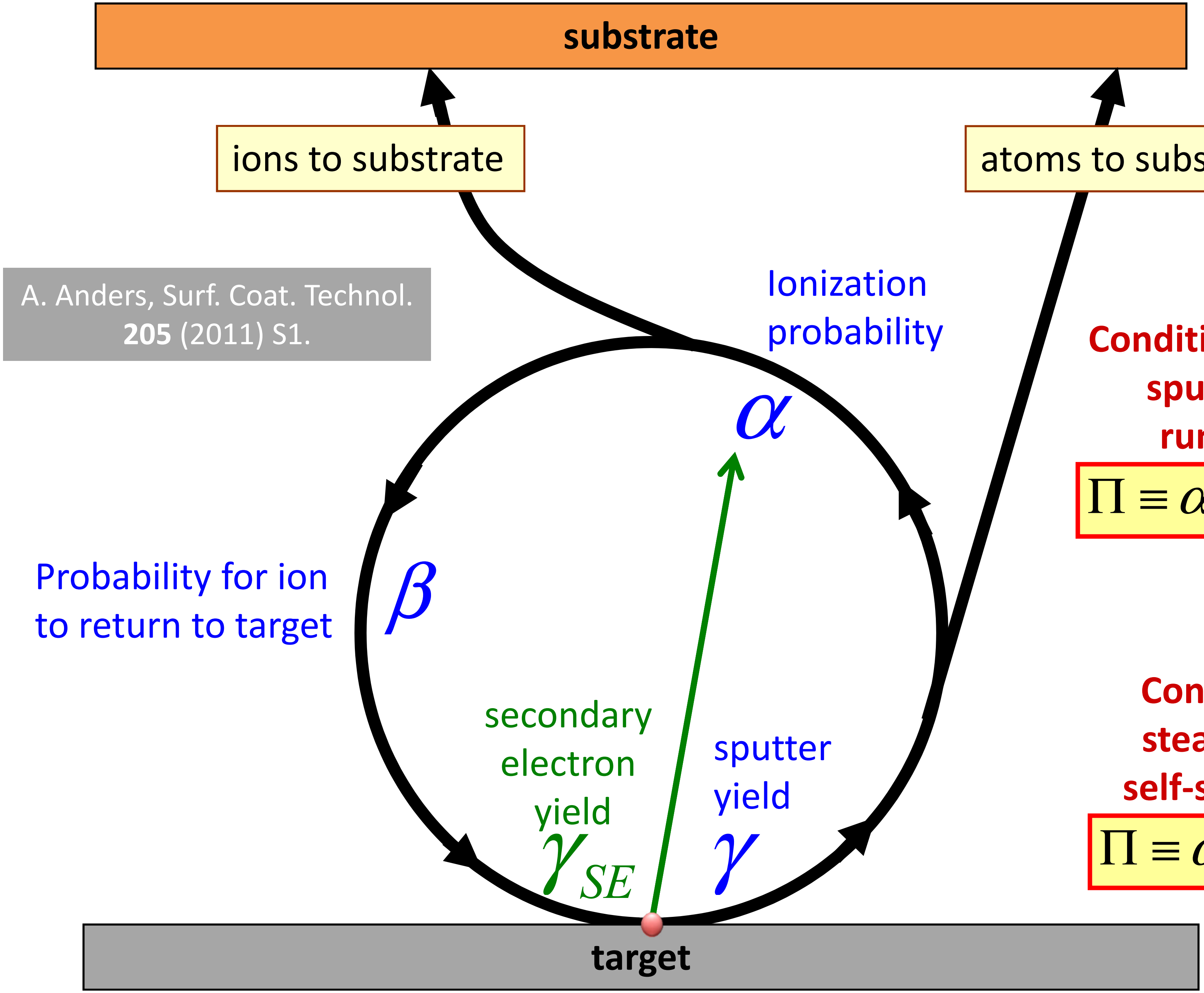


image from the seminal (but not first) paper:
V. Kouznetsov, *et al*., Surf. Coat. Technol. **122** (1999) 290

Why do we care? Because bias can be applied to affect film-forming ions (not atoms)!



Condition of self-sputtering runaway

$$\Pi \equiv \alpha \beta \gamma > 1$$

Condition of steady-state self-sputtering

$$\Pi \equiv \alpha \beta \gamma = 1$$

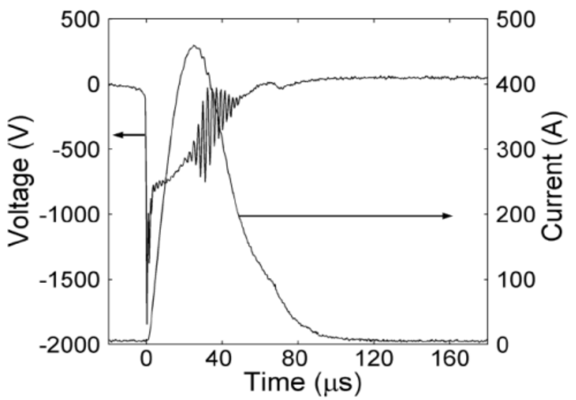
HiPIMS-High Power Impulse Magnetron Sputtering

High Impulse Power Magnetron Sputtering (HiPIMS)

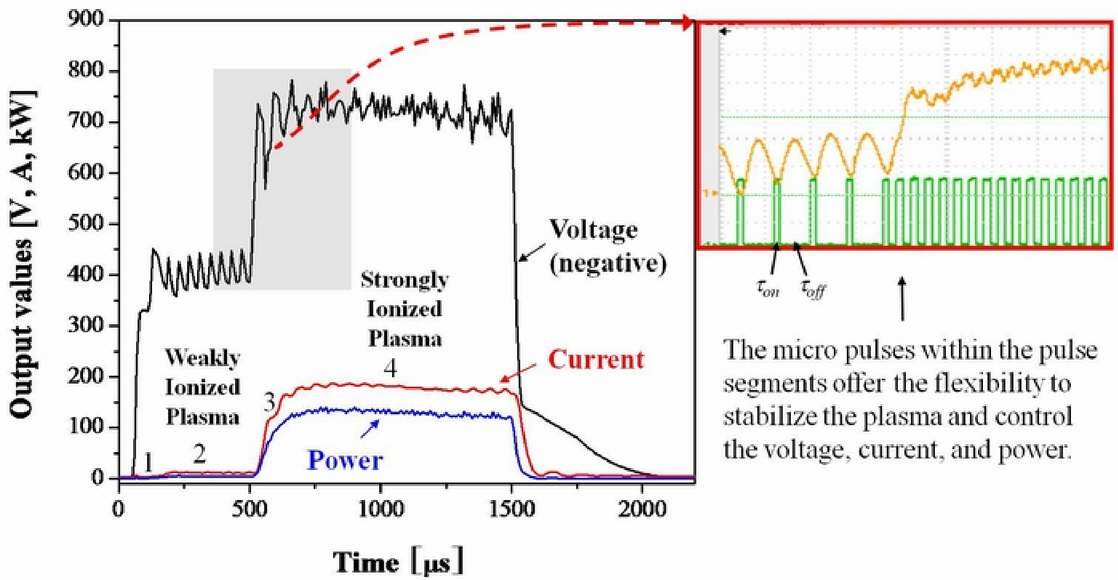
also known as HPPMS (High Power Pulse Magnetron sputtering)

Pulse with a **power density** at the target surface during the pulse exceeding the typical dc power density by about **two orders of magnitude**. This implies that the **off-time between pulses is long**, and the **duty cycle is only of order 1%.**

20 eV - 100 eV (vs. only about 2 -10 eV in conventional sputtering).

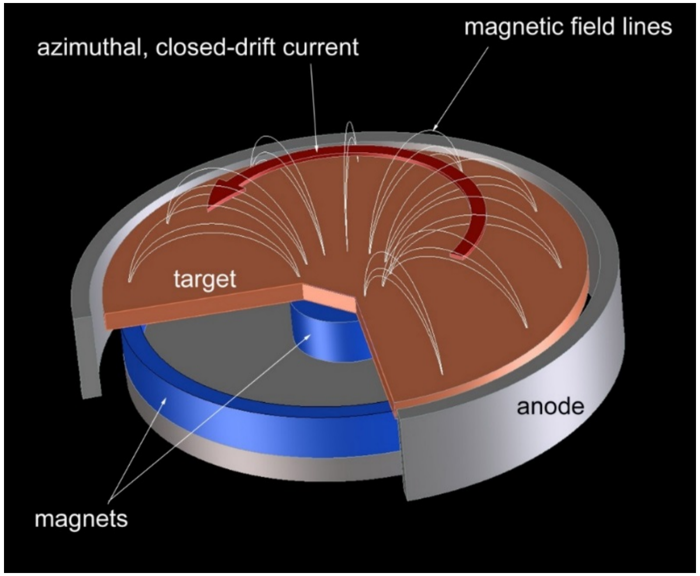


Variation: Burst HiPIMS or Modulated Pulse Power (MPP)



Increase High rate deposition

Magnetron as an Electron Trap



Ionization Zones

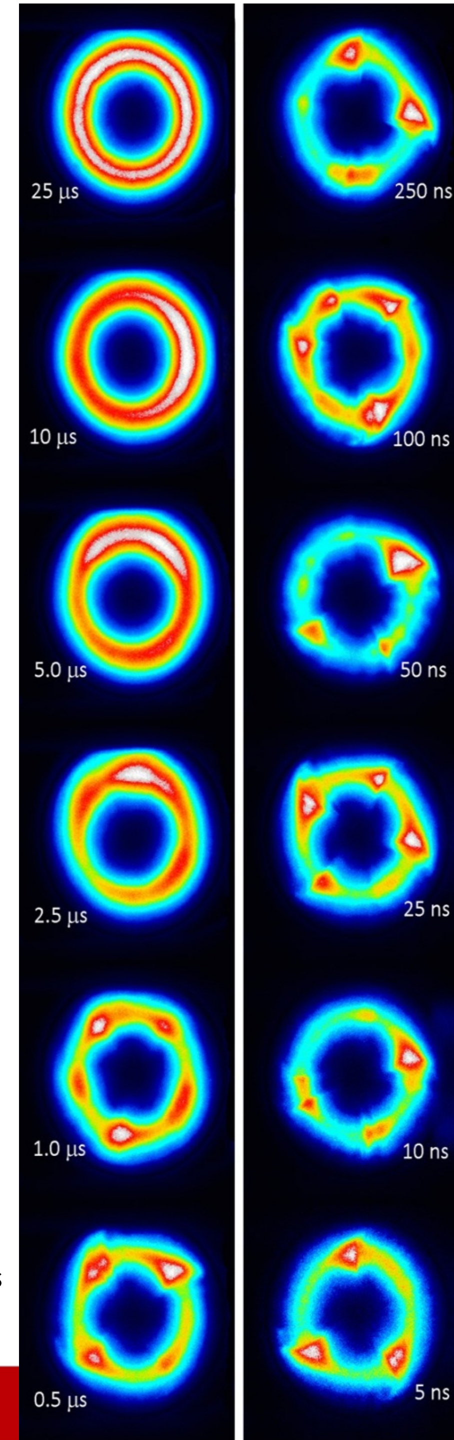
The HiPIMS plasma breaks down in isolated ionization zones (IZs) that rotate in the direction given by the ExB drift, with velocities around 10^4 ms^{-1} and frequencies in the range of about 100 kHz.

- Positive feedback loop between electron mean free path and ionization leads to “bunching” of plasma in **ionization zones**
- Ionization zones move in ExB direction because ions are “evacuated” from ionization zones by electric field, exposing new neutrals to ionization by drifting electrons
- Electrons drift according to the local E and B fields, perpendicular to both, and produce electron jets related to the azimuthal electric field of the plasma zone
- the physically relevant power density of HIPIMS is much higher than the typically reported average power density
- **Ionization zones explain why HIPIMS works as observed, and offer “energetic condensation” in the context of sputtering and SRF coatings.**

From the target point of view, a HiPIMS discharge represents therefore a situation of continuous temporal and spatial change in local sputtering conditions.

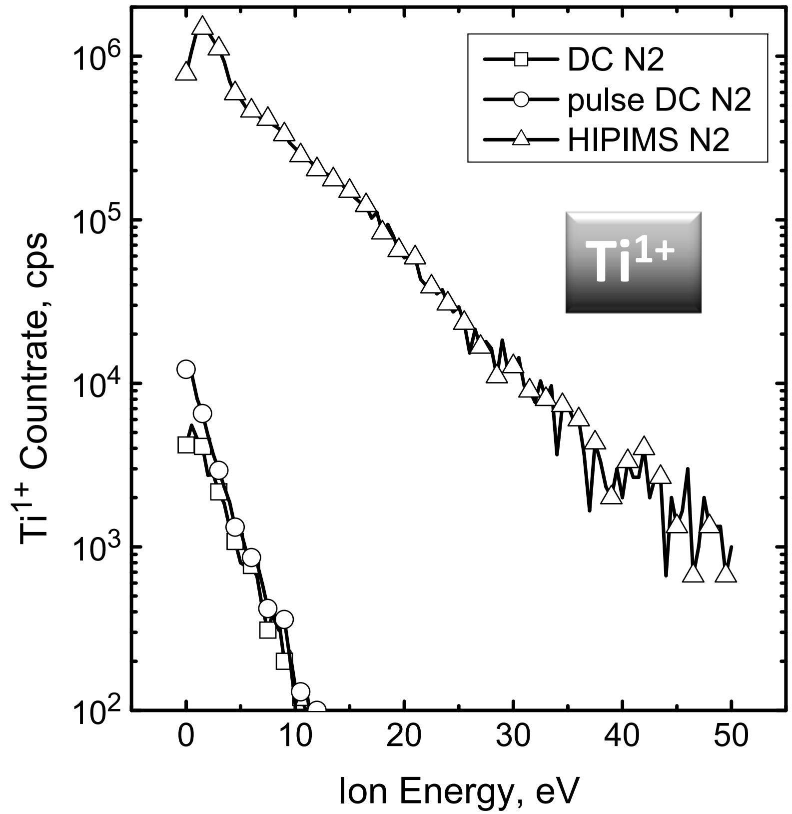
- 3'' Nb target, peak current ~ 200 A
- reduction of image exposure time gives immediate clues on rotational speed → ~ 10^4 m/s

A. Anders et al., J. Appl. Phys. ***111*** *(2012) 053304*



HiPIMS – Applications to SRF Surfaces

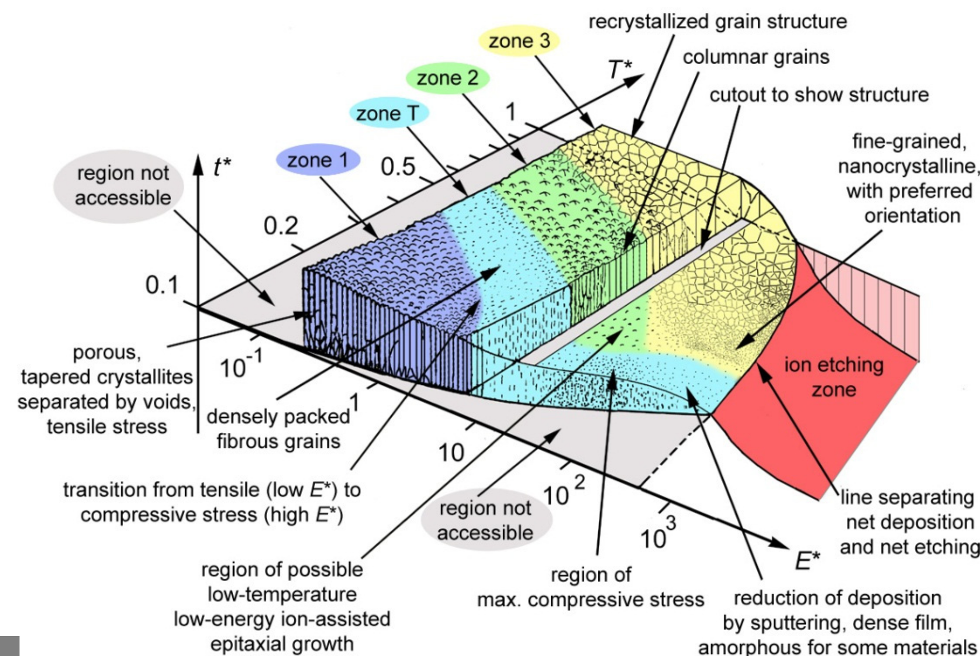
Ion Energy Distribution Function



A. P. Ehiasarian, *et al.*, Plasma Processes and Polymers **4** (2007) S309

Enhanced power density is the key to the desired ionization of sputtered material: films are denser, smoother, and some grain texture control is possible.

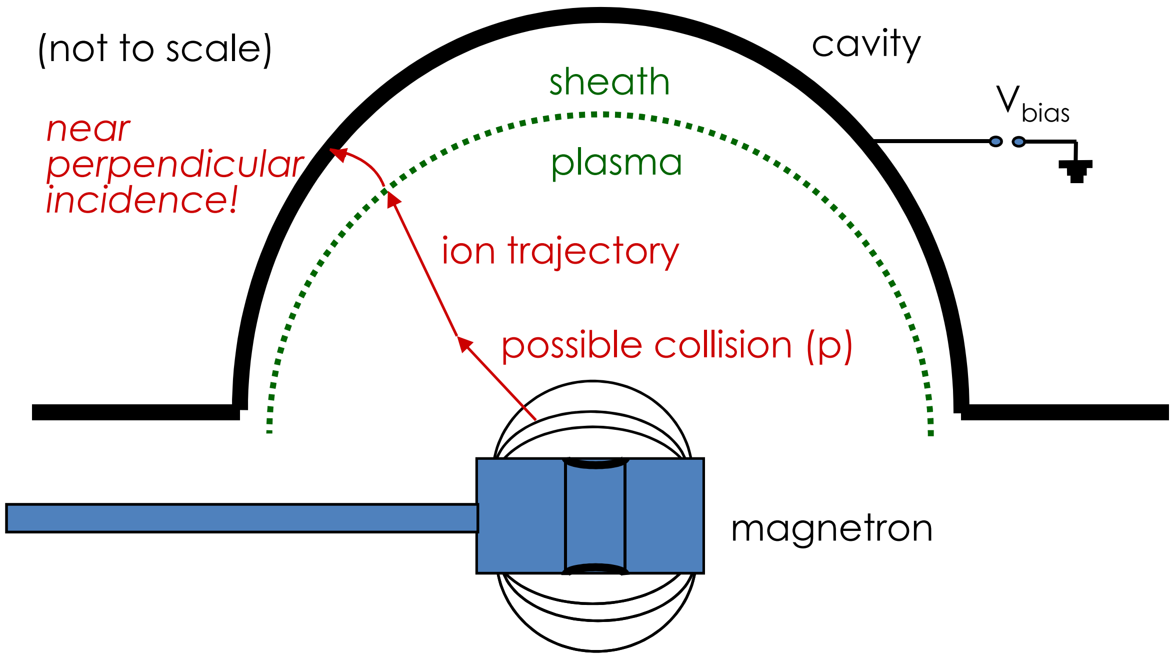
"Structure Zone Diagram"



A. Anders, Thin Solid Films 518 (2010) 4087.

HiPIMS – Applications to SRF Surfaces

Bias voltage:
Control ion trajectories & energy in collisionless sheath

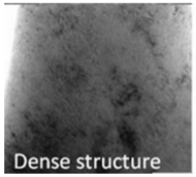


In-situ substrate pre-treatment:
Cleaning or oxide layer removal – typically with plasma etching (high voltage applied to the substrate)
Interface engineering - film forming species are implanted in the substrate, forming a gradient towards the surface

HiPIMS – Applications to SRF Surfaces

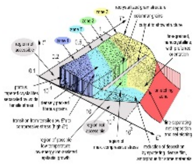
High level of ionization

High energy ions, tunable with bias voltages

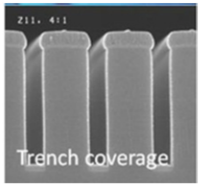


Film densification

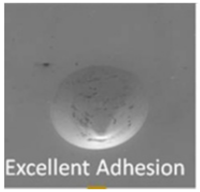
Film smoothness



Possibility to control the film structure



Good surface coverage (even for high aspect ratio objects)



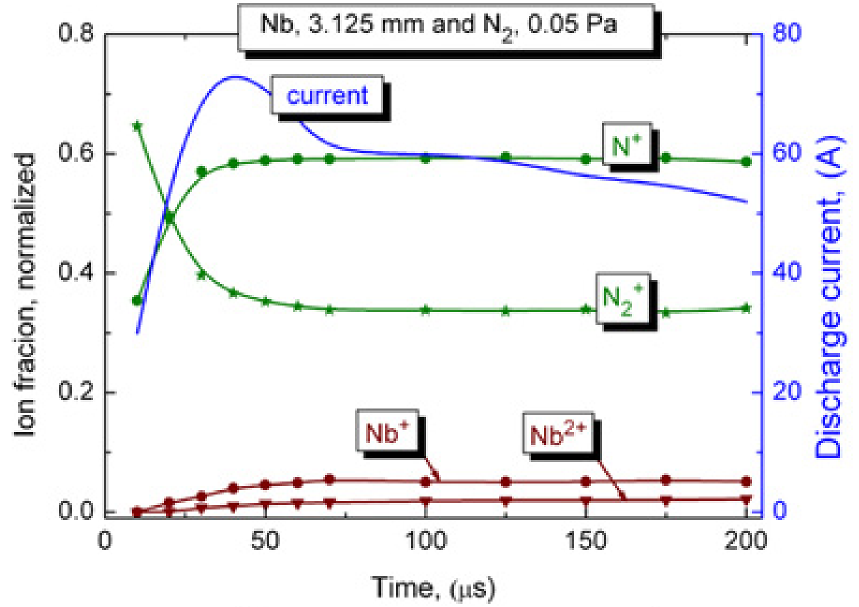
Enhanced adhesion to substrate

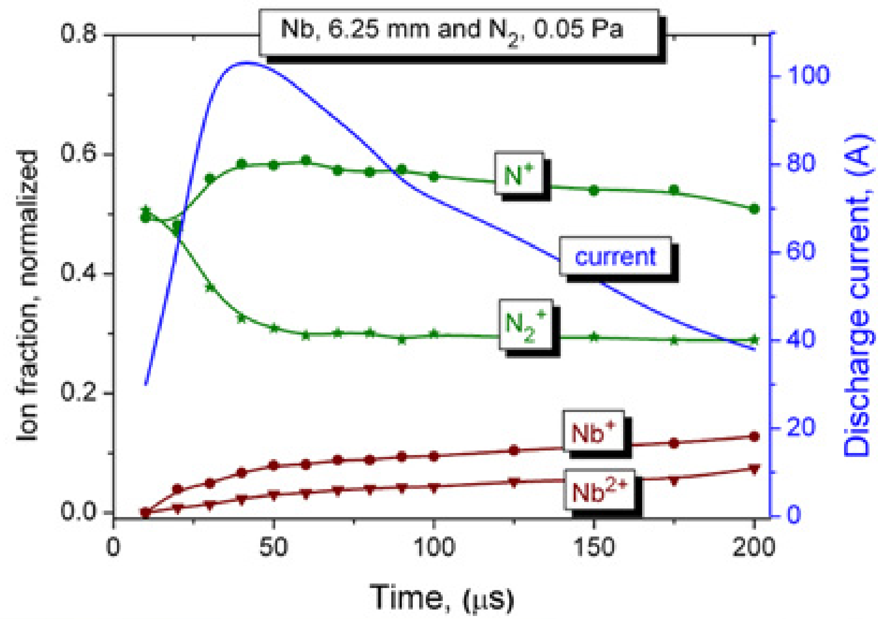
HiPIMS – Applications to SRF Surfaces

Easy extension to other materials

- Relatively straight forward to produce NbN, NbTiN & multilayers
- Possibility to use two different cathode materials with two asymmetrically operating magnetrons to produce Nb_3Sn, MgB_2, …

Preliminary TOF study of Nb HIPIMS with N_2 gas: the measured ion species promise the formation of dense, textured NbN

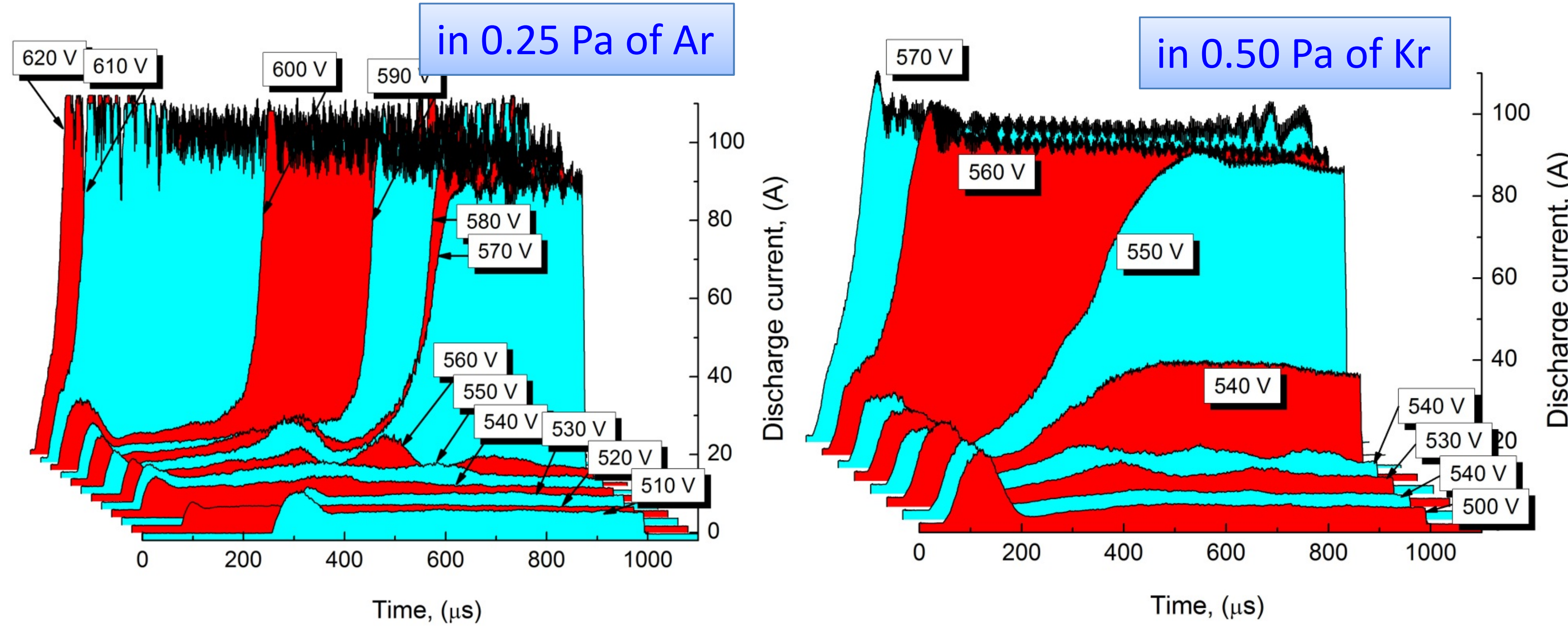




A. Anders and Yu. Yushkov, J. Appl. Phys. ***105*** *(2009) 073301*

HiPIMS – LBNL

***Plasma Studies** especially for Nb*



A. Anders, Surf. Coat. Technol. **205** (2011) S1.

HIPIMS and Self-Sputtering of Niobium

HIPIMS with Nb target , ∅ 5 cm

HiPIMS – LBNL

3 regimes in the HiPIMS discharge
Regime I: Low voltage (<550V), high impedance
Regime II: Mid voltage (550<V<650), low impedance
Regime III: High voltage (>650 V) high impedance

Plasma Studies *especially for Nb*

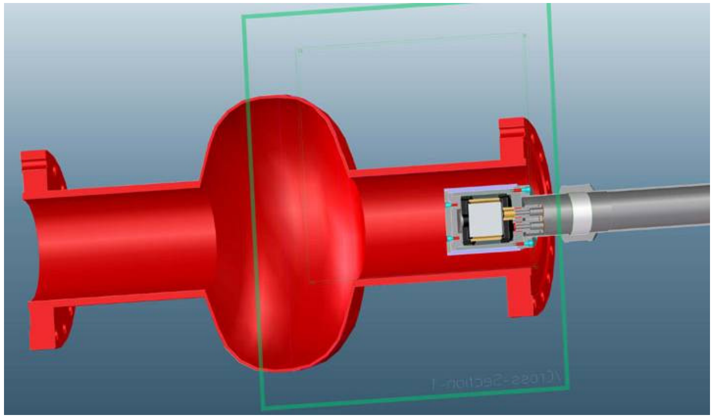
R. Mendlesberg, A. Anders

Superimpose a Mid Frequency (MF) discharge in between the HiPIMS pulses to lower frequency the HiPIMS pulses

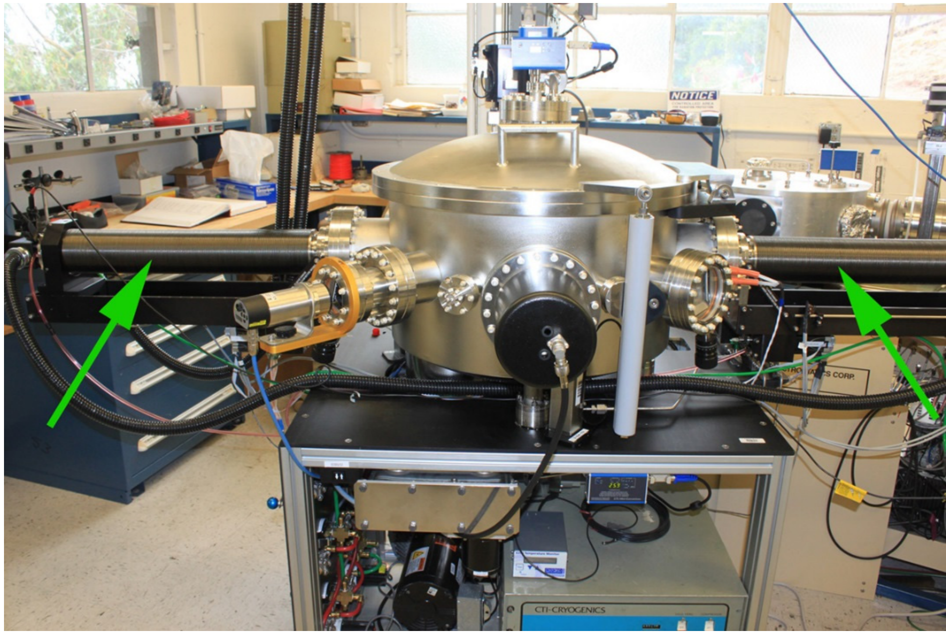
- **HiPIMS pulses do not depend on MF pulse pattern**
- **As long as the MF discharge is there, the HiPIMS pulses can be spaced as far (or close) in time as desired.**
- **HiPIMS pulses must be longer than 50 µs**
- **Relative power in HiPIMS and MF components is tunable**
- **Nb+ emission lines dominate the emission spectrum starting at moderate voltages**
- **Intensity of all lines increases with increasing discharge voltage**
- **OES peak ratios give some information on changes in relative density of each ionic species**
- **For film growth, applied voltage should be about 650V**
- discharge right in the low voltage end of Regime III for pulse widths greater than 100 µs
- Large population of Nb+ at this voltage, Creation of Nb^{2+}, which doesn't aid film growth any more than Nb+, is minimized
- Moderate voltage will reduce tendency for arcing

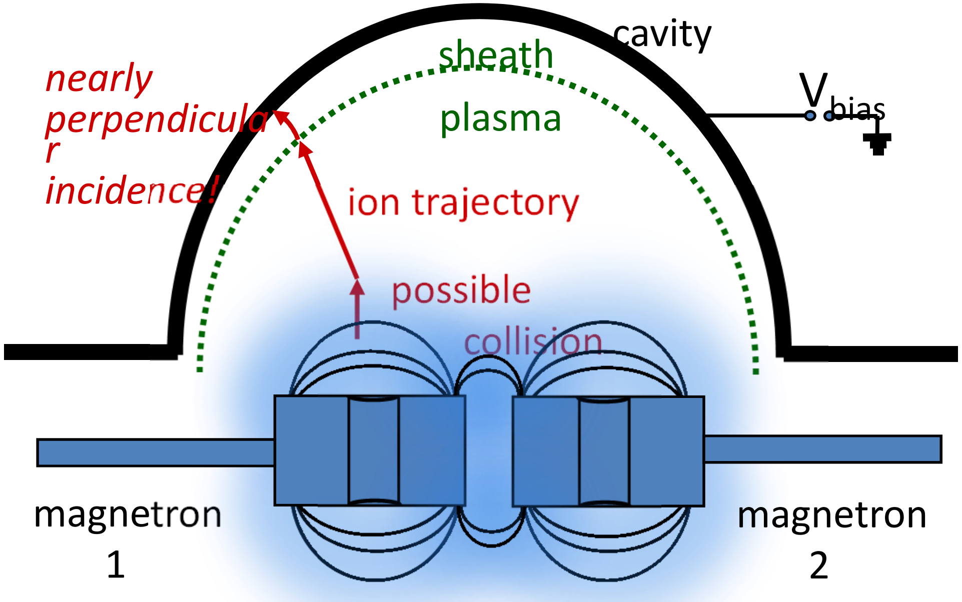
HiPIMS – LBNL

(collaboration with ANL, JLab)



Dedicated Nb-HIPIMS Chamber

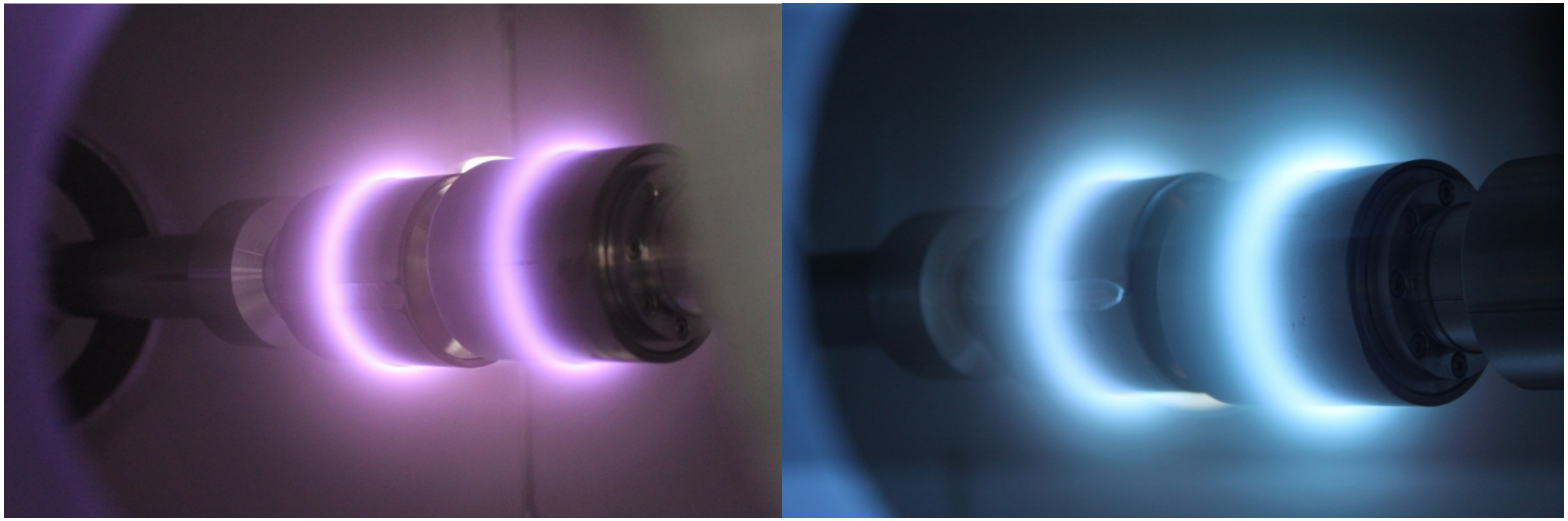




Dual Magnetron: Most effective for a Biasing & influencing Ion Energies and Trajectories

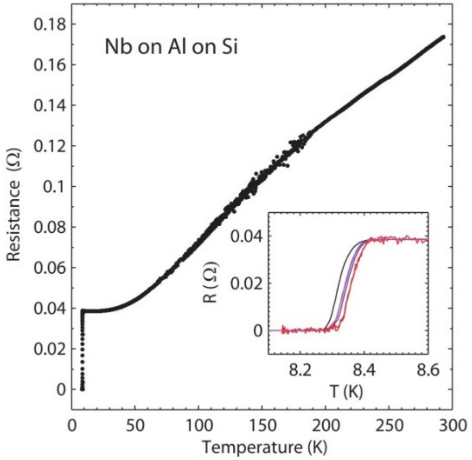
- chamber for 1.3 GHz SRF cavities
- base pressure in the low 10^{-8} range
- residual gas analyzer
- 2 small cylindrical, movable magnetrons
- decoupled substrate heating and biasing
- pyrometer 100-600 °C
- 2 SIPP pulsers for dual-HIPIMS and bias

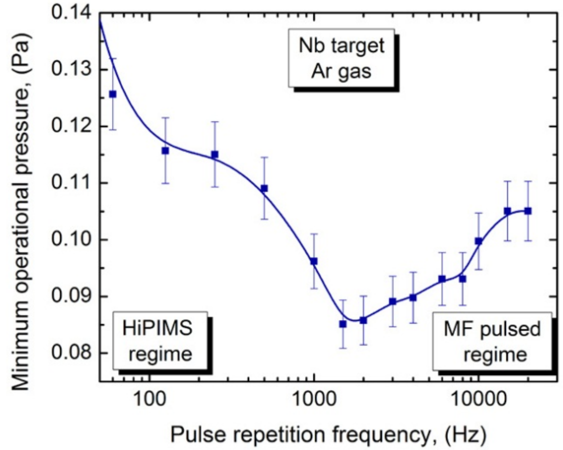
HIPIMS -LBNL



- dual cylindrical magnetron in at relatively low power sputtering mode
- Dominated by argon emission

- dual cylindrical magnetron in high power mode (above runaway threshold)
- Dominated by niobium emission

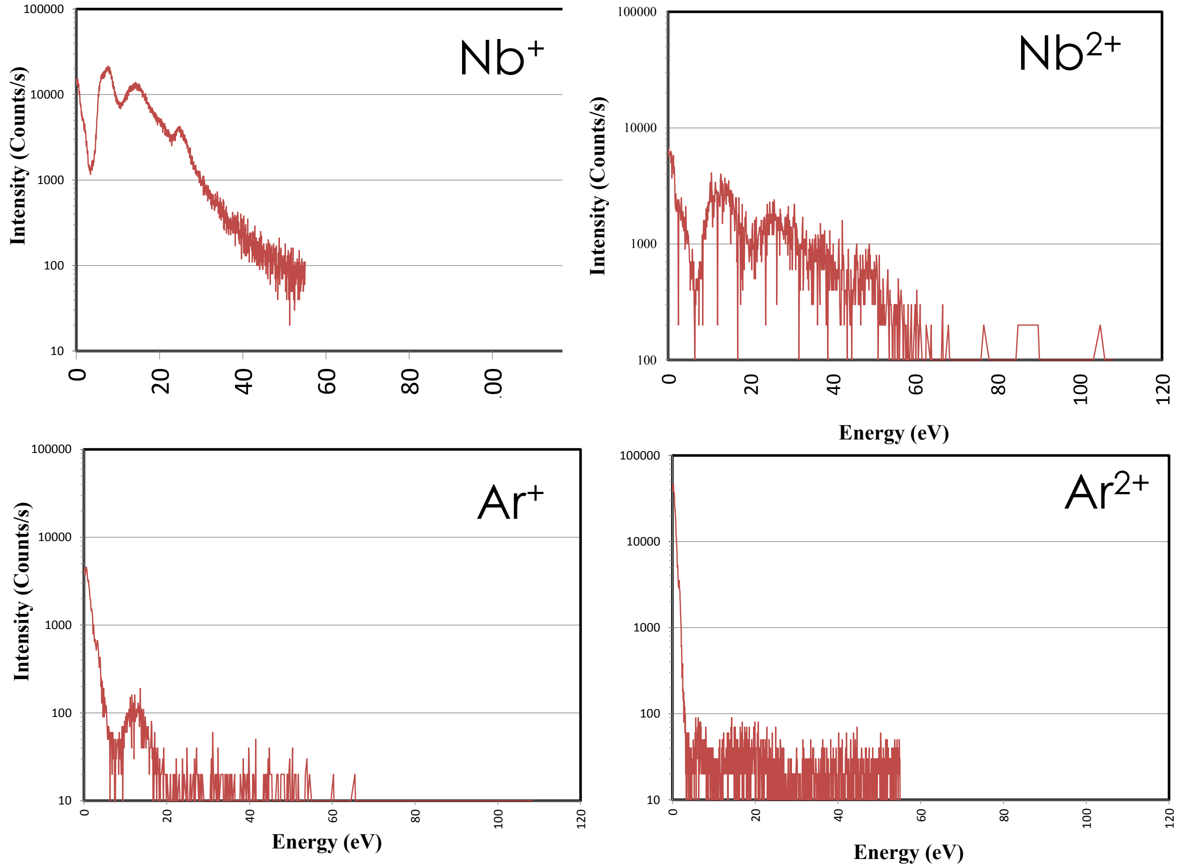




HiPIMS – CERN
Collaboration with Sheffield Hallam University

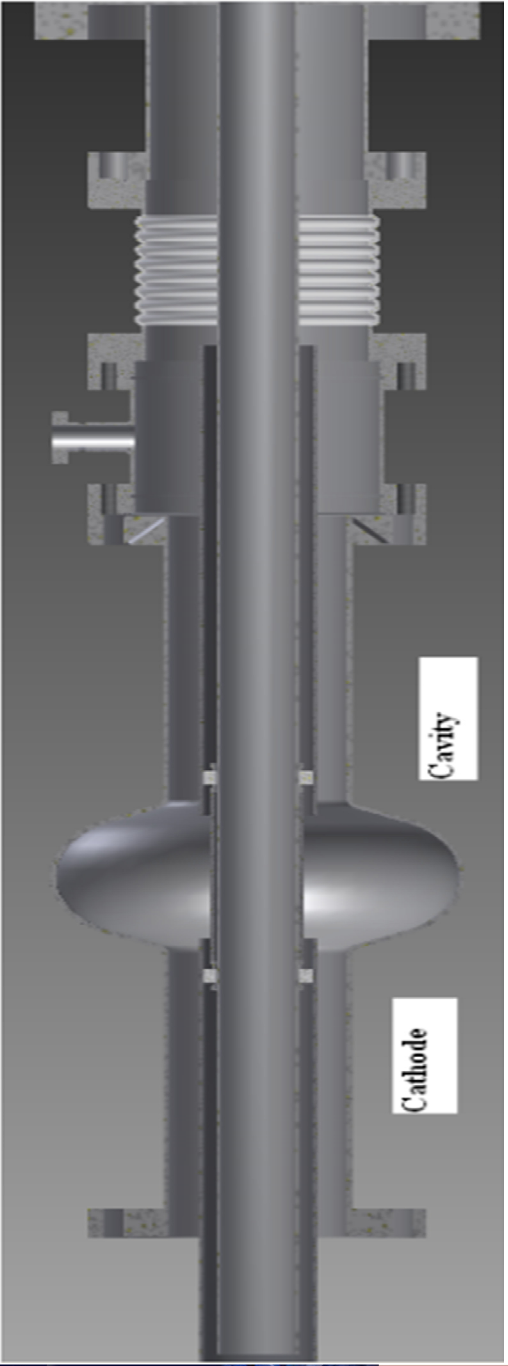
Sheffield Hallam University: Plasma Studies
CERN: 1.3-1.5 GHz cavity deposition

Ion Energy Dispersion Function (IEDF)

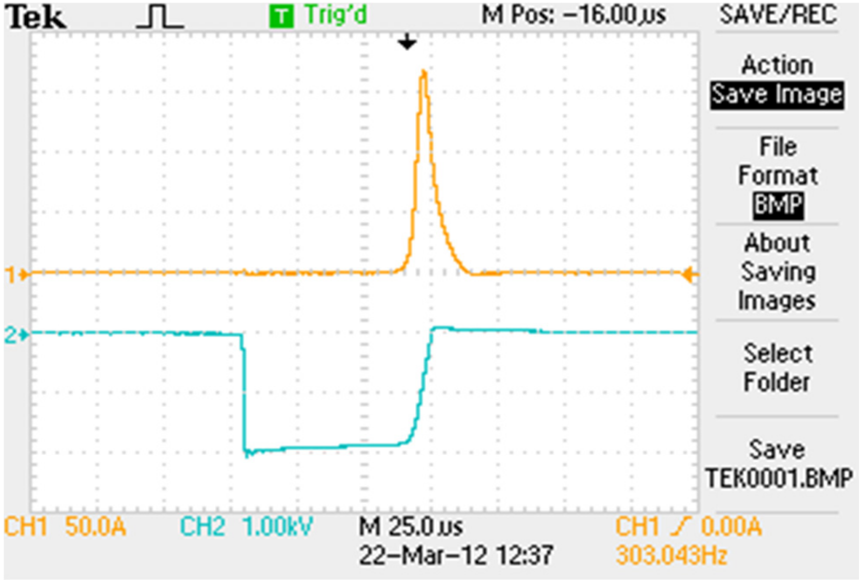


G. TERENZIANI, S. CALATRONI, A.P. EHIASARIAN, NB COATINGS FOR SUPERCONDUCTING RF APPLICATIONS BY HIPIMS, 2013-09-11

HiPIMS – CERN



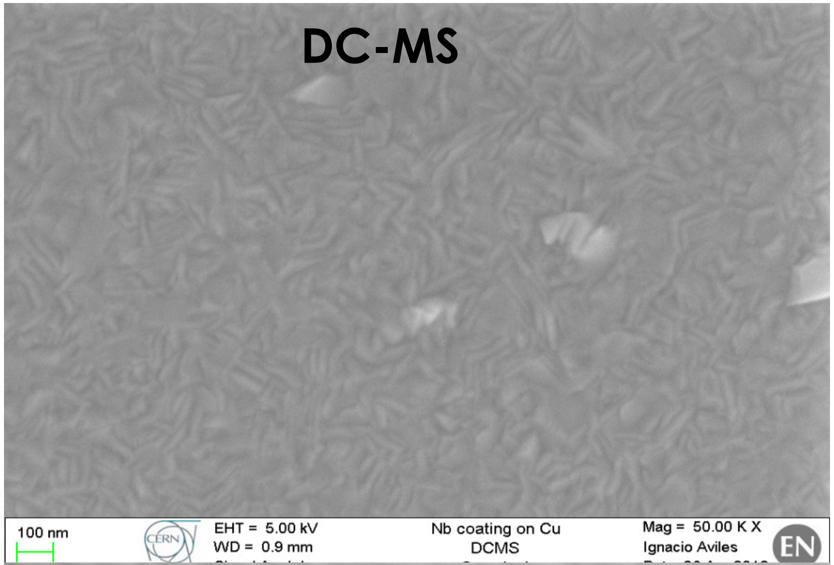
Vertical cylindrical magnetron
Base pressure $10^{-10} – 10^{-11}$ mbar
Movable SmCo magnet



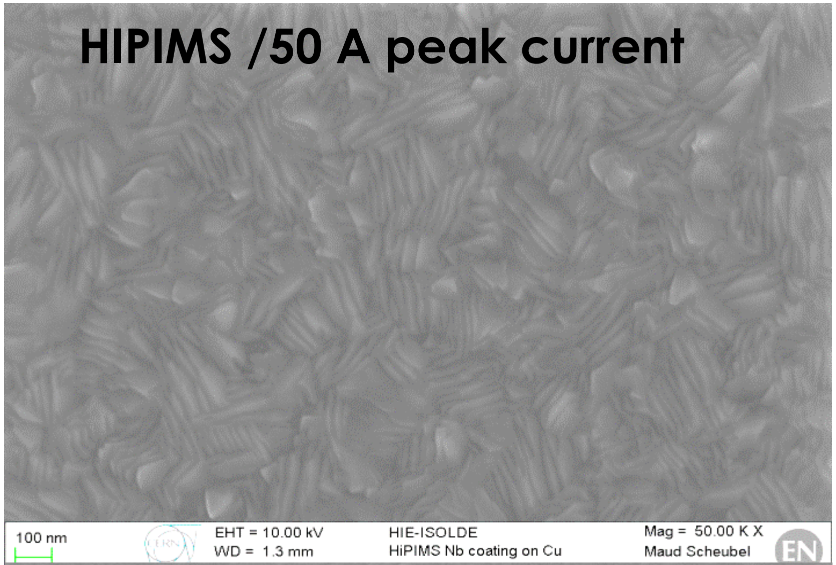
Typical HiPIMS current and voltage behavior

Hüttinger Electronics TruPlasma Highpulse DC Unit:
10 kW max average power delivered
voltage up to 2 kV
pulse width up to 200 us
frequency up to 500 Hz.

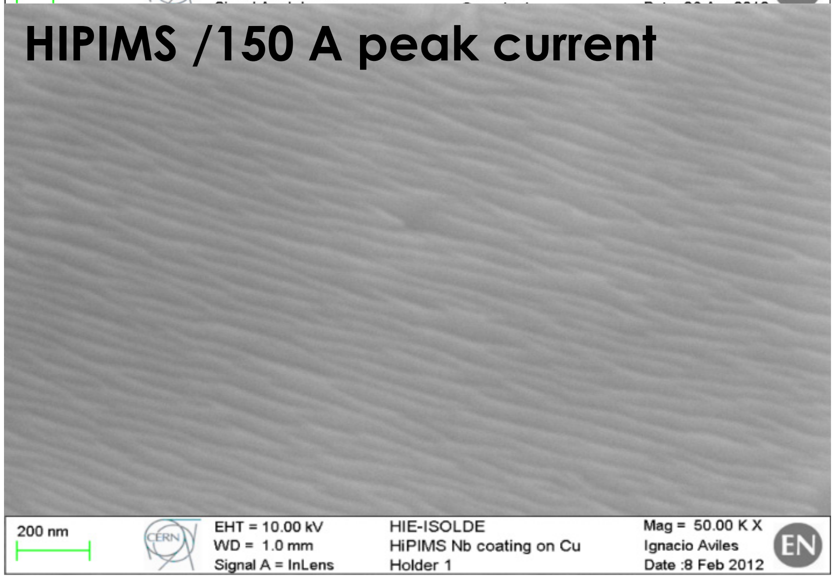
HiPIMS – CERN



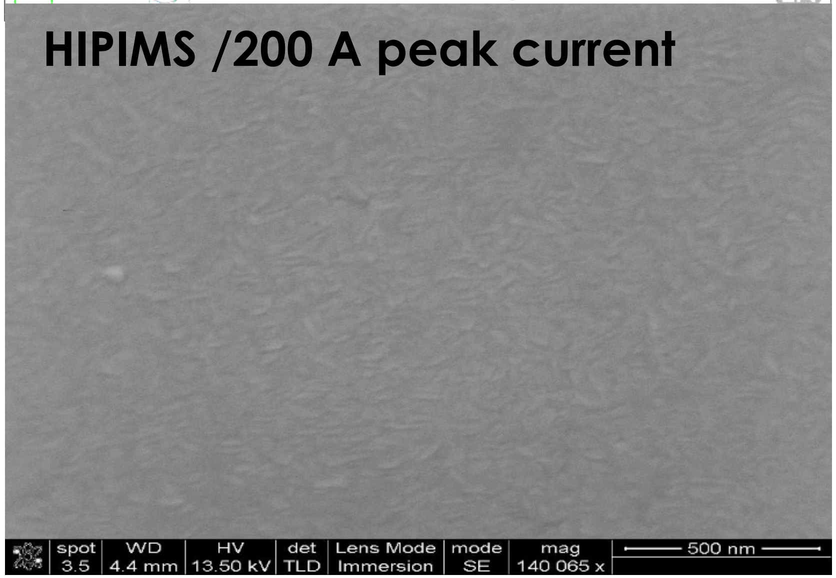
DC-MS
100 nm
EHT = 5.00 kV
WD = 0.9 mm
Nb coating on Cu
DCMS
Mag = 50.00 K X
Ignacio Aviles
EN



HIPIMS /50 A peak current
100 nm
EHT = 10.00 kV
WD = 1.3 mm
HIE-ISOLDE
HiPIMS Nb coating on Cu
Mag = 50.00 K X
Maud Scheubel
EN



HIPIMS /150 A peak current
200 nm
EHT = 10.00 kV
WD = 1.0 mm
Signal A = InLens
HIE-ISOLDE
HiPIMS Nb coating on Cu
Holder 1
Mag = 50.00 K X
Ignacio Aviles
Date :8 Feb 2012
EN

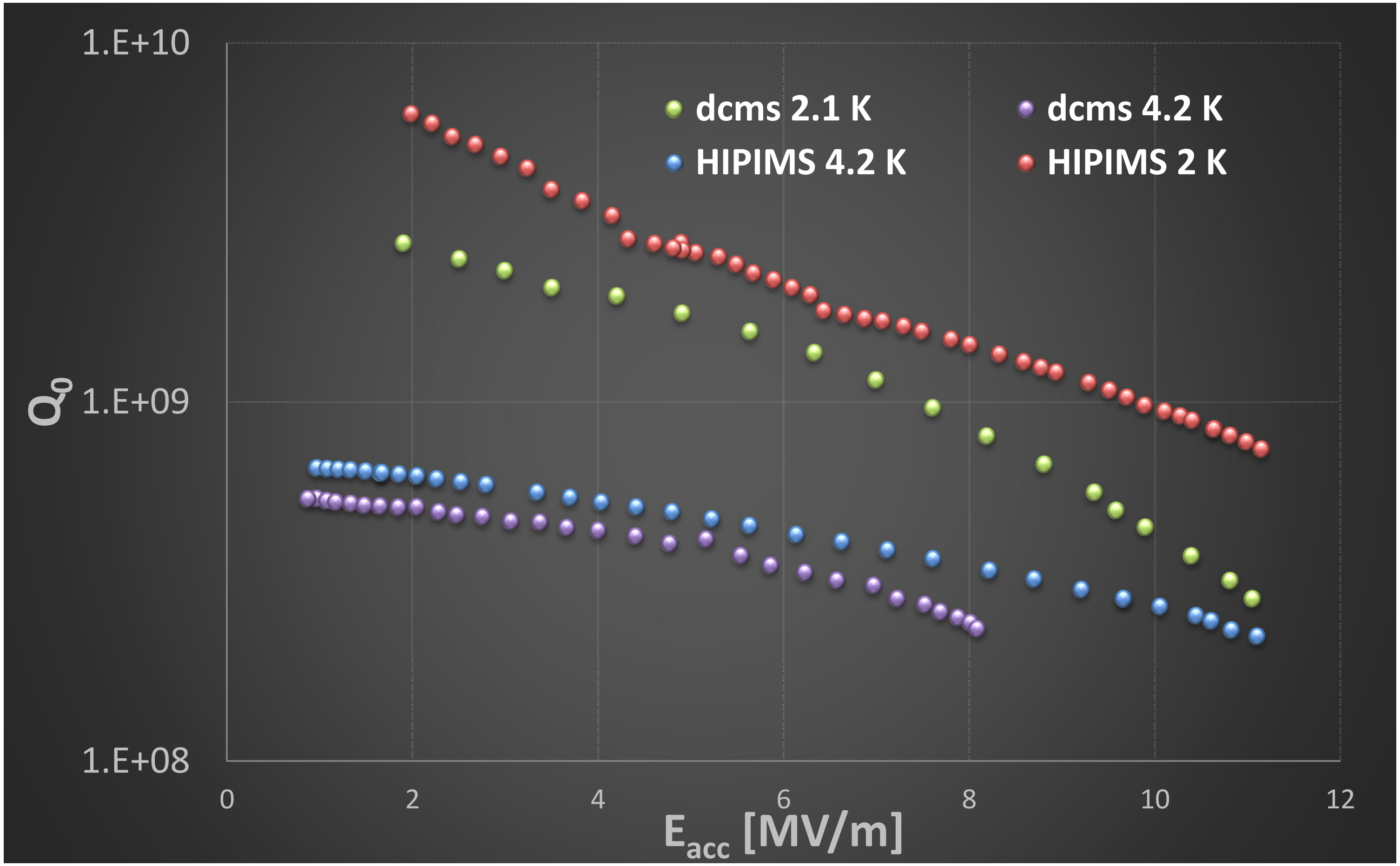


HIPIMS /200 A peak current
spot 3.5
WD 4.4 mm
HV 13.50 kV
det TLD
Lens Mode Immersion
mode SE
mag 140 065 x
500 nm

HiPIMS – CERN

Nb/Cu cavities have been produced both by DC cylindrical magnetron sputtering and cylindrical HiPIMS with Kr

peak current 200 A ($2 A/cm^2$)
$\Delta V = 570 \pm 10$ V
Average current 2.6 A
Pulse width 200 µs
frequency 106 Hz.



***Note: -** Substrate preparation is SUBU (by opposition to electropolishing for the best 1.5GHz DCMS Nb/Cu cavities to date)*

- Measurement at higher fields than 11MV/m prevented by interlock system due to radiation

HiPIMS – CERN

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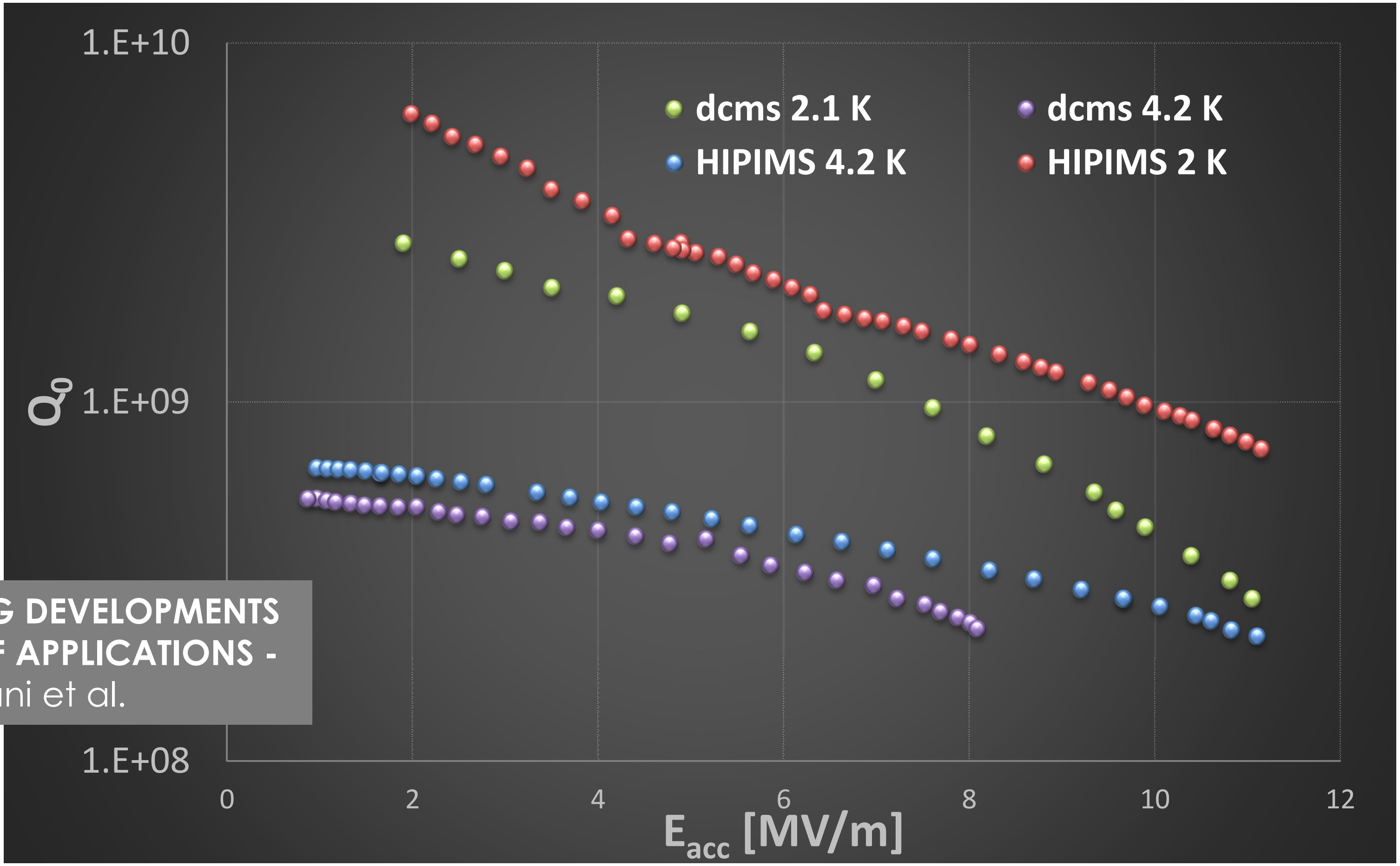

TUP078 NB COATING DEVELOPMENTS WITH HIPIMS FOR SRF APPLICATIONS - G. Terenziani et al.

Note:* - *Substrate preparation is SUBU (by opposition to electropolishing for the best 1.5GHz DCMS Nb/Cu cavities to date)

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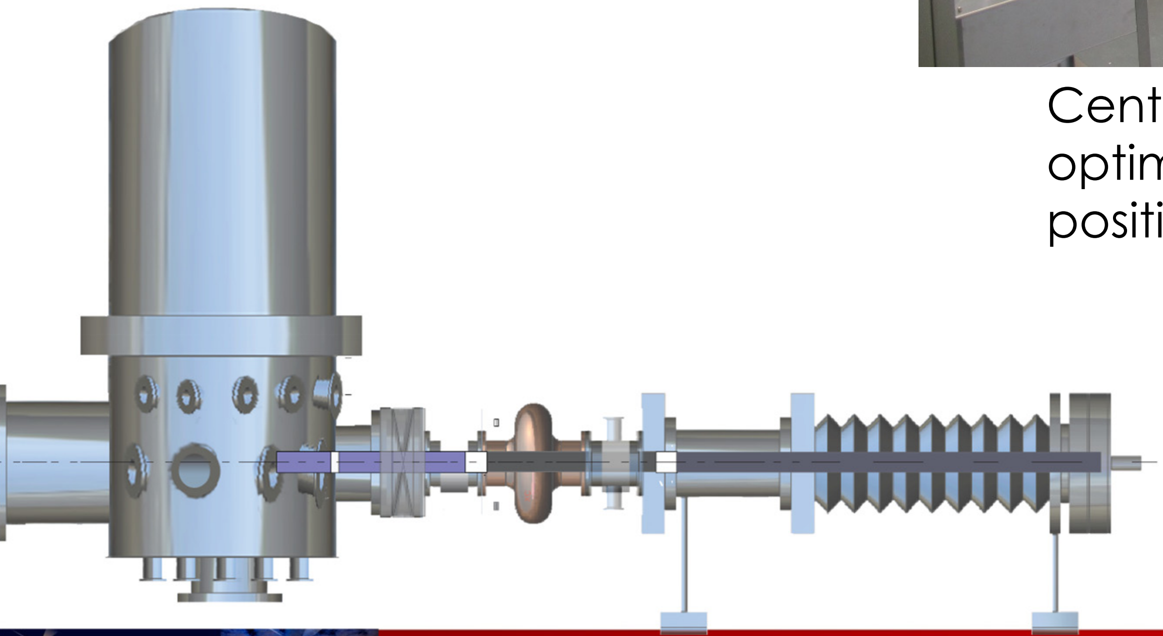
HiPIMS – JLab
Collaboration with LBNL, College William & Mary

Study on samples & cavities

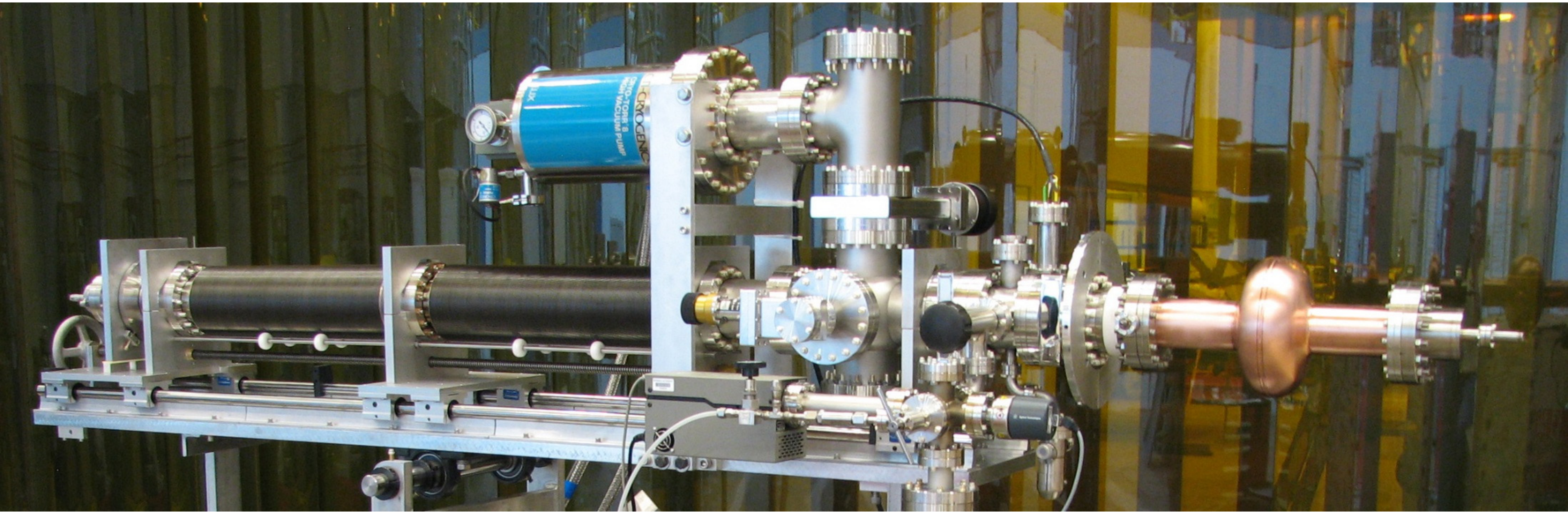
2 coating systems:
- ❑ **UHV Multi-technique Deposition system**
- ❑ **Cylindrical HiPIMS cavity system**

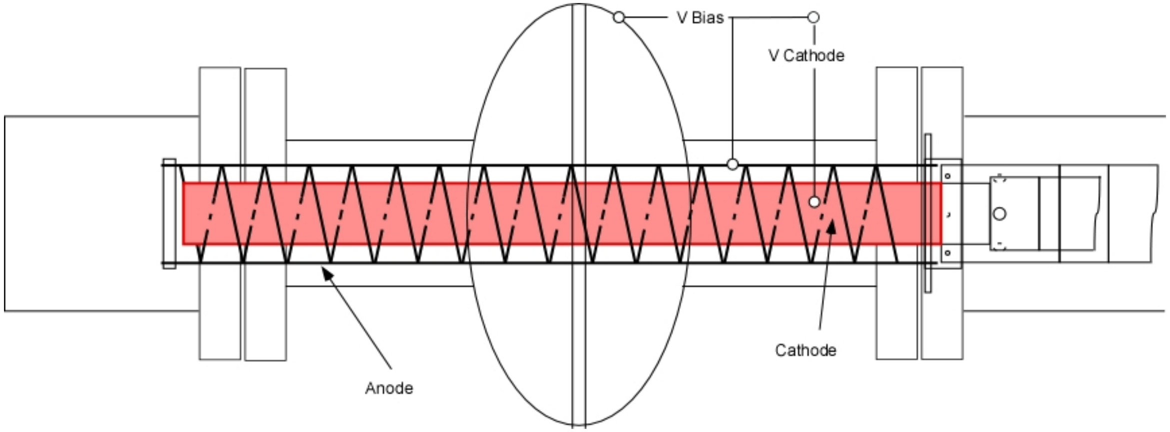
Center stage in development to optimize the sample-target relative positions for Nb and Multilayer films



Bridge studies with energetic condensation Nb films produced by ECR (Nb^+ ions in UHV)

HiPIMS – JLab

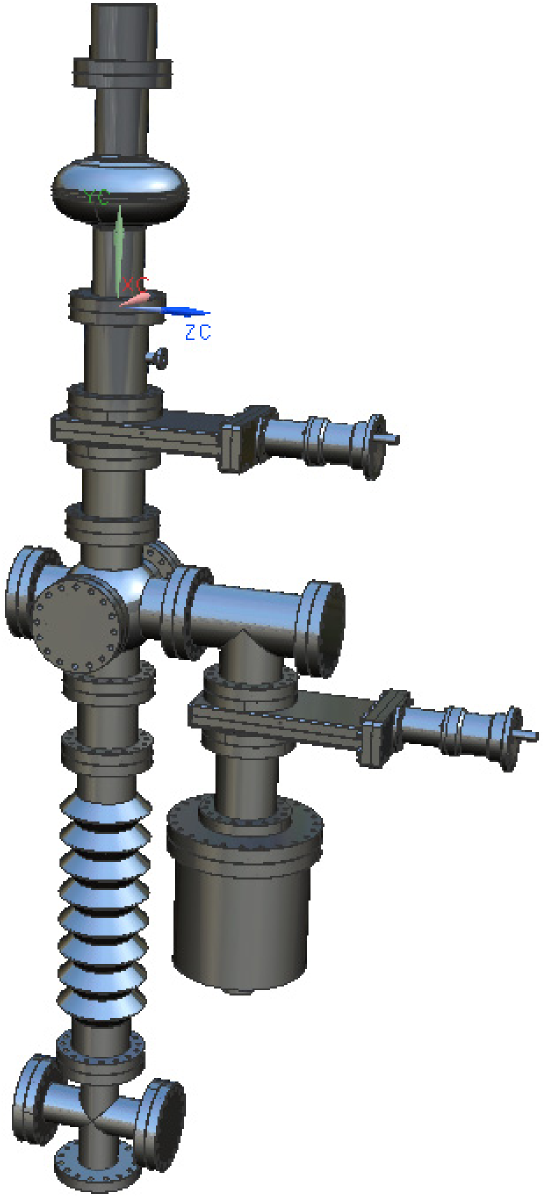




- Niobium cathode
- Open grid-like Nb anode
- internal rare earth magnets with field extending entire cathode & cavity length
- Penetrating cathode every 1.7 cm producing a peak B field of 0.2 T at a 4 mm distance from the cathode surface with the same longitudinal periodicity.
- Water cooled

HiPIMS – JLab

- Ion flux characterization under various pulse conditions
- Ion energy spectrum & Nb ions to neutral ratio. (several anode designs)
- System parameter space & film properties (initially T_c & *RRR*)



Cavity coating procedure:

- Substrate preparation: CBP + Electropolishing
- Assembly in clean room
- Pump down and bake-out to adequate temperature
- Coating parameters derived form work done with ECR plasma
- Coating with Kr: less trapped in the niobium film, tends to damp plasma instabilities than argon

Conclusions

- HiPIMS is an emerging array of energetic condensation techniques with extensive studies in plasma physics and materials
- HIPIMS has the advantage of not generating macroparticles (assuming that arcing is prevented)
- Nb has a relatively low self-sputtering yield→ “gasless” self-sputtering in vacuum could not be demonstrated
- low pressure operation works well with optimized pulse frequency.
- HIPIMS cavity systems are ready in the Laboratories involved with HiPIMS.
- Nb/Cu cavities have already been coated at CERN with encouraging results.
- Technique directly applicable to material systems beyond Nb.