Ion bombardment energy effect on microstructure and J_c of Nb/Cu thin films.

C. P. A. Carlos, G. Rosaz, S. Leith, M. Bonura, C. Senatore, S. Pfeiffer, A.-T. Perez-Fontenla, A. Moros

Outline

Context

Experimental apparatus

Jc trends

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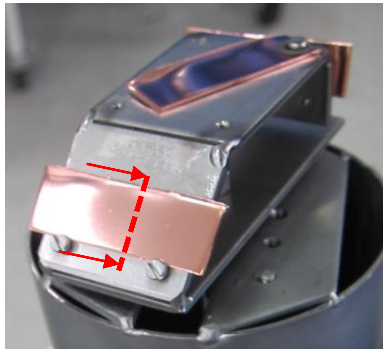
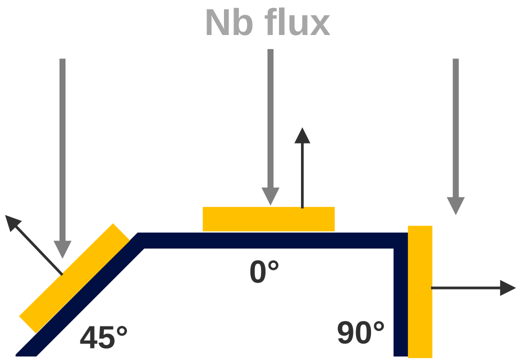
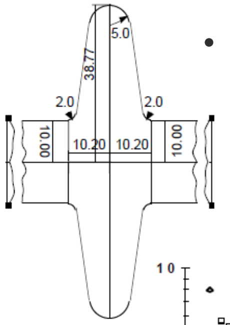
CERN strategy

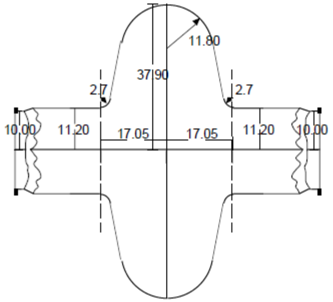
What do we know?

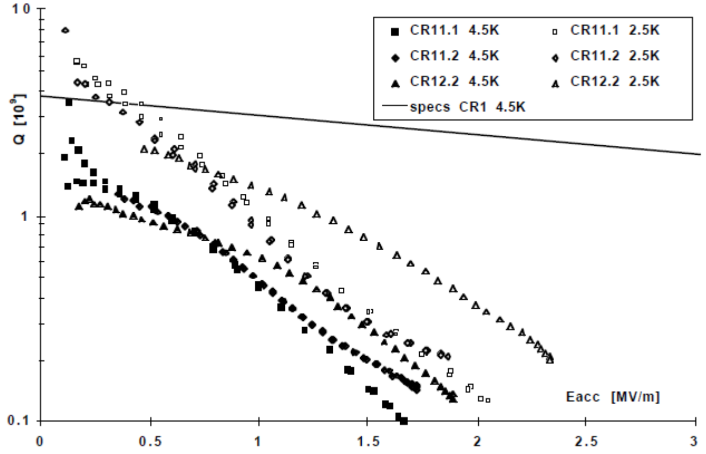
- **HiPIMS is a must (wrt DCMS)**
  - DCMS is excluded from our investigations
  - Cavities have complex shapes
    - atoms impinging angle has been proven to be responsible part of the Q-slope phenomenon

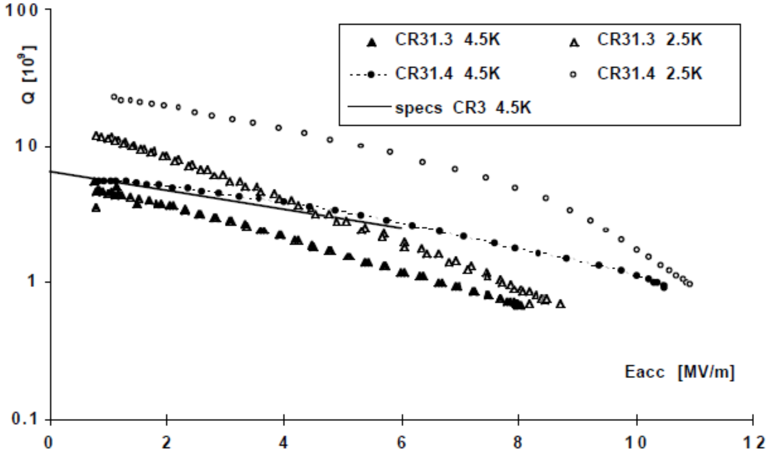












C. Benvenuti et al, Production and test of 352 MHz Niobium Sputtered Reduced Beta cavities, 1997, SRF97D25

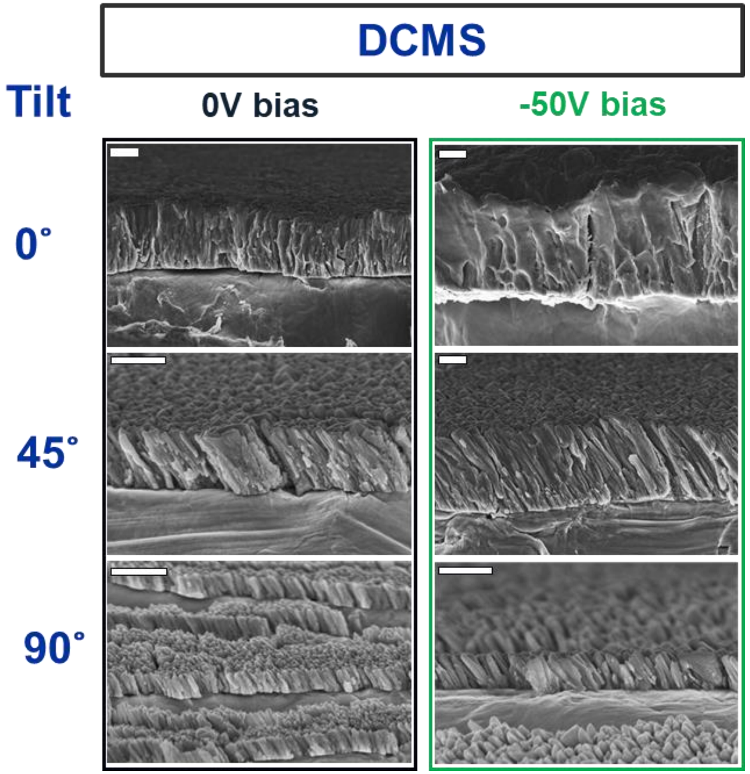


Fig.1 SEM images of Nb/Cu films cross-sections elaborated by DCMS at various impinging angles and substrate biases.

CERN strategy

What do we know?

- **HiPIMS is a must (wrt DCMS)**
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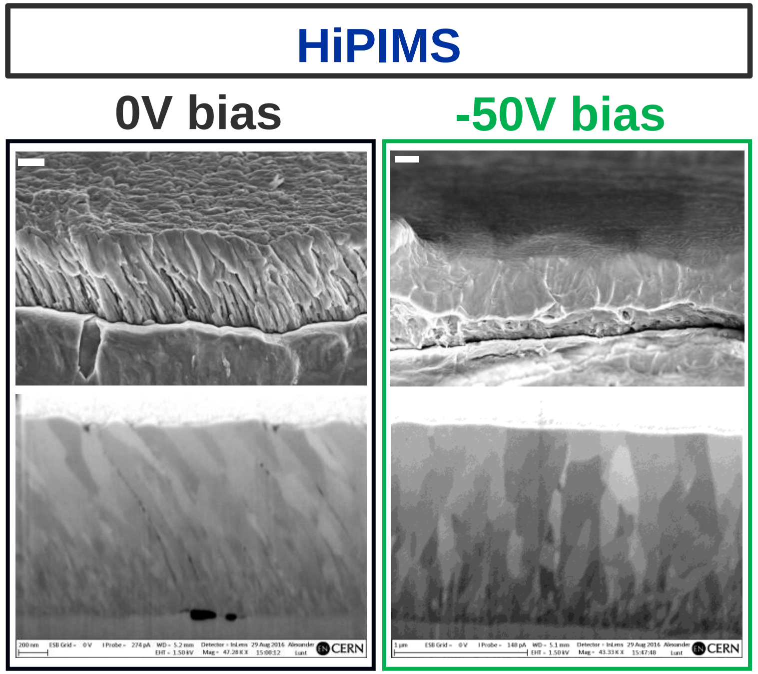
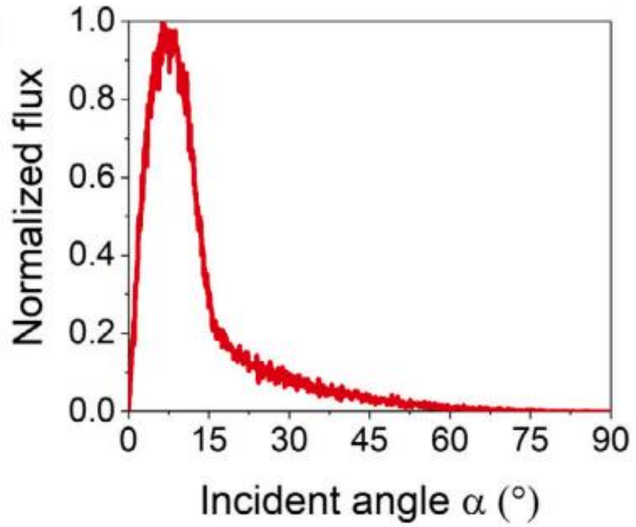
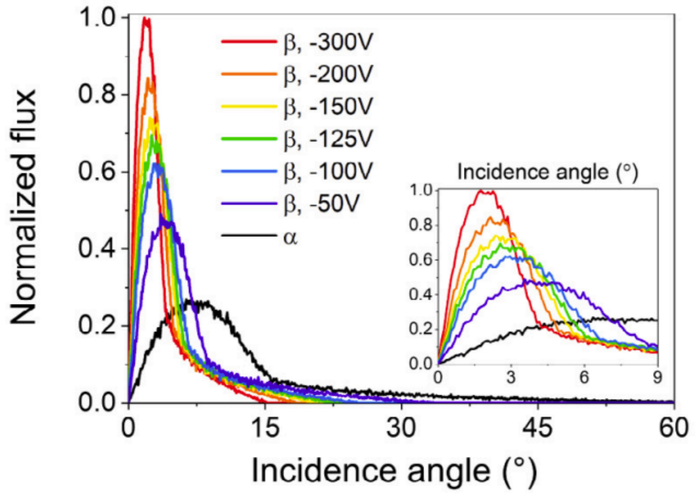
- **HiPIMS is useful ONLY IF the substrate is BIASED**
  - Densification
    - Ions' trajectory normal to the surface
    - Impinging energy control





Ions impinging angle at the plasma sheath surface



Ions impinging angle at the substrate's surface for various bias voltages

https://doi.org/10.1016/j.vacuum.2024.113354

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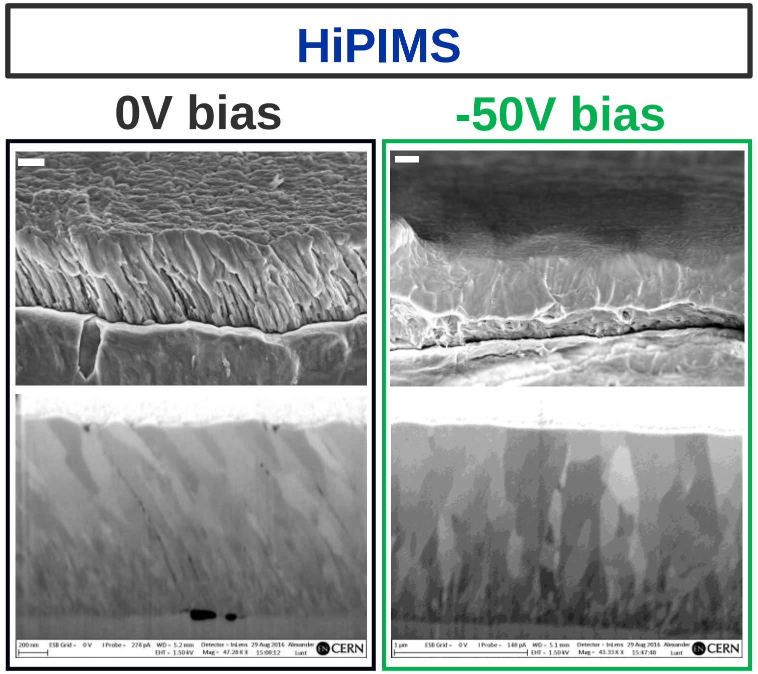
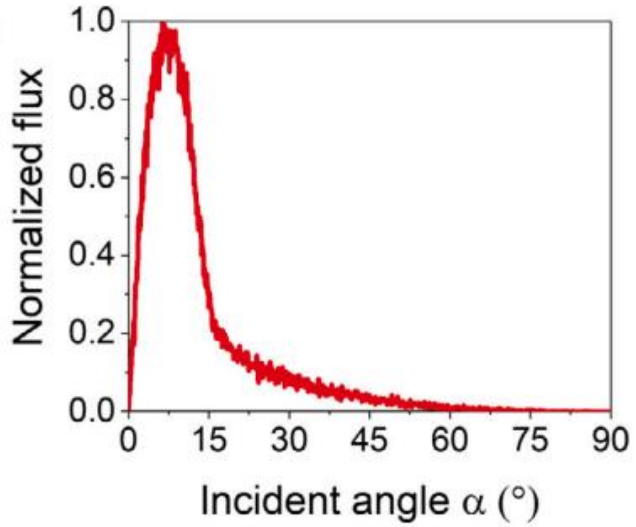


HOW IS THAT BIAS AFFECTING THE FILM’S PROPERTIES?



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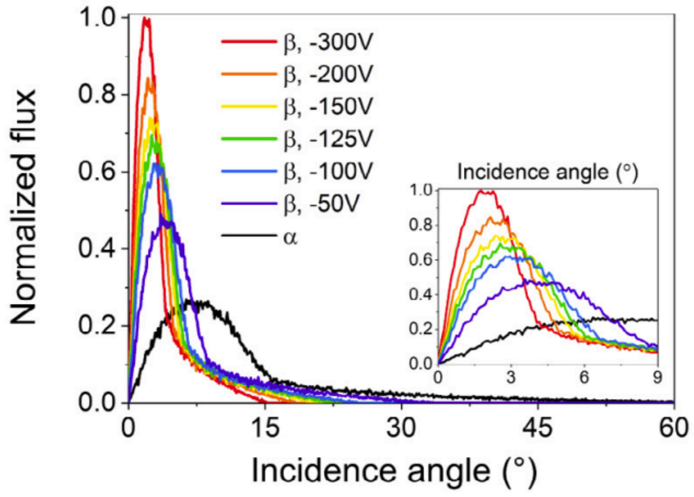

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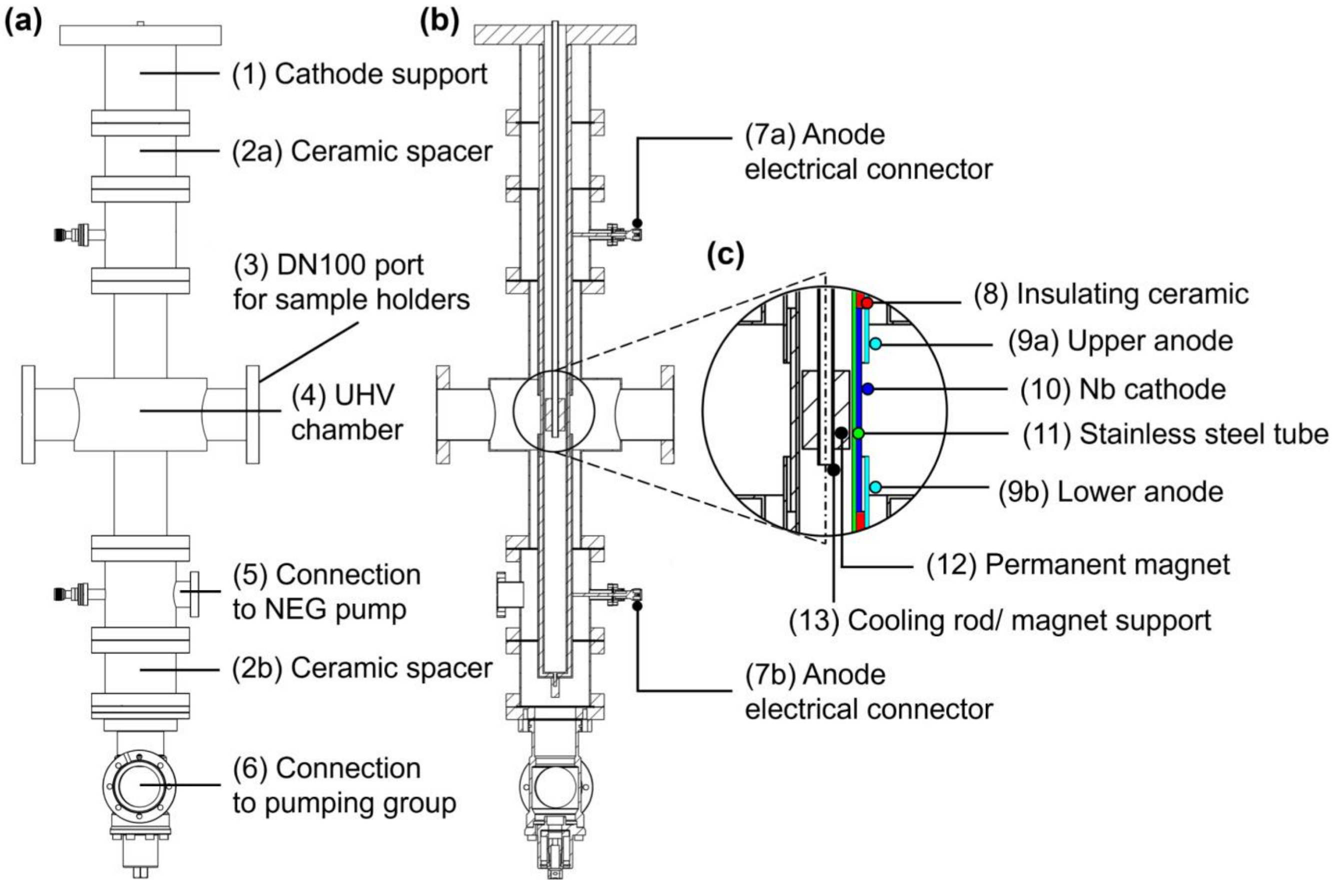
Coating



Samples:

Surface treatment: SUBU 5
Size 10x35x1 mm^3

Sputtering

Process parameter	Value	Unit
Average Power	1.2	kW
Frequency	100	Hz
Pulse length	200	µs
Temperature	150	°C
Thickness	1-6	µm
Base Pressure (after BO)	3.10^{-10}	mbar
Process gas	Kr	-
Process pressure	$2.3.10^{-3}$	mbar
Bias Voltage	[-50 … -125]	V

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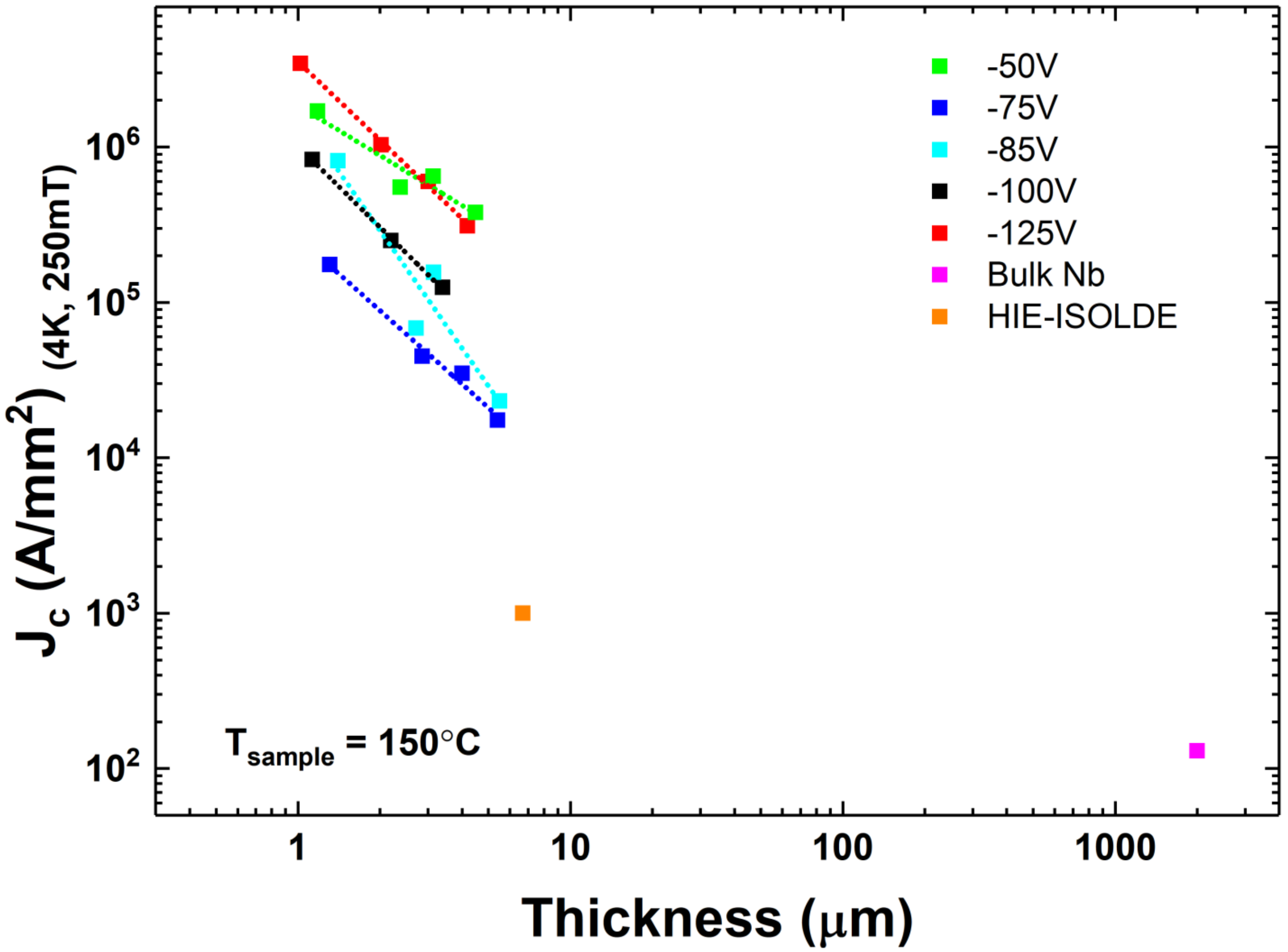
Jc vs Film Thickness and Bias

Thicker layers

- → lower J_c
- → lower defect density

HiPIMS films

- Still orders of magnitude higher than bulk Nb



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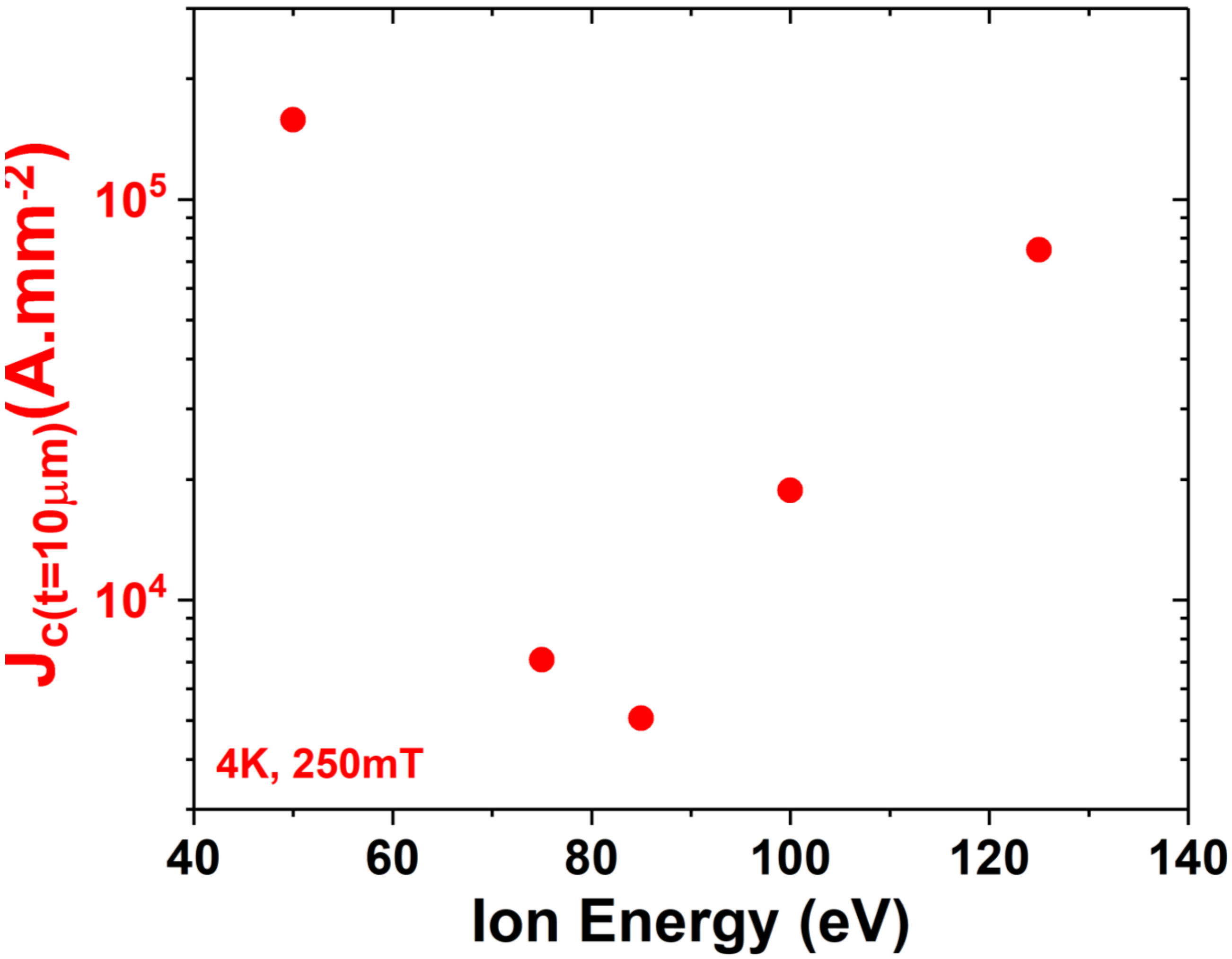
Extrapolated Jc for 10μm thick films

Non linear bias voltage dependency

Can we
1. identify the defects responsible for the trend?
2. Explain the bias optimum?



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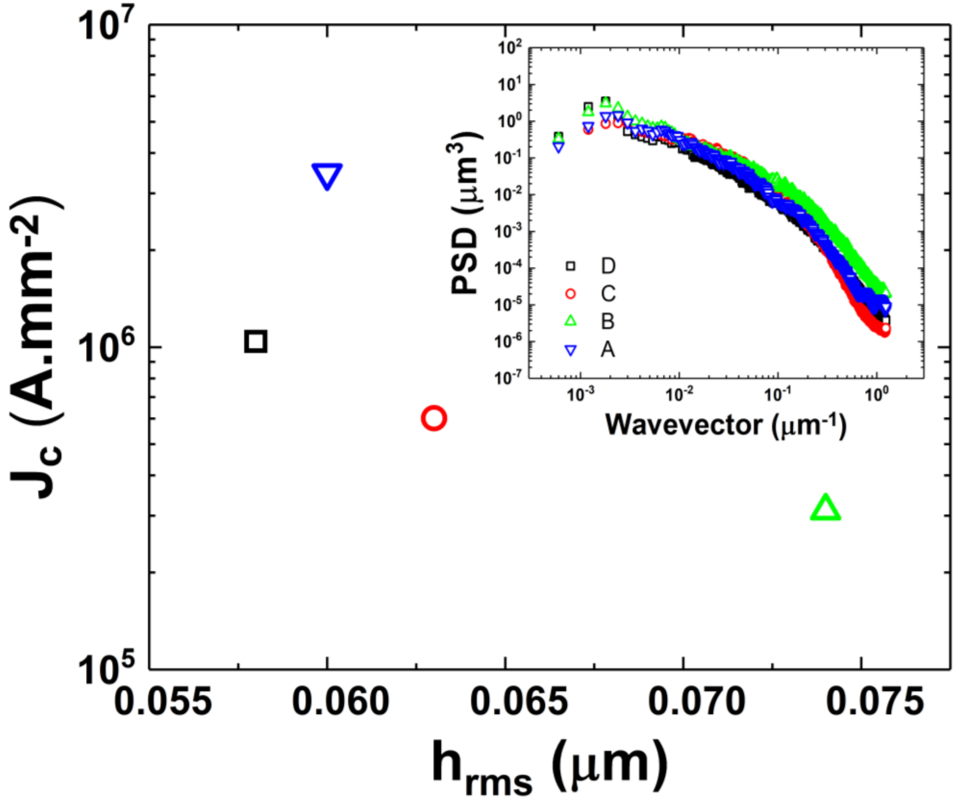
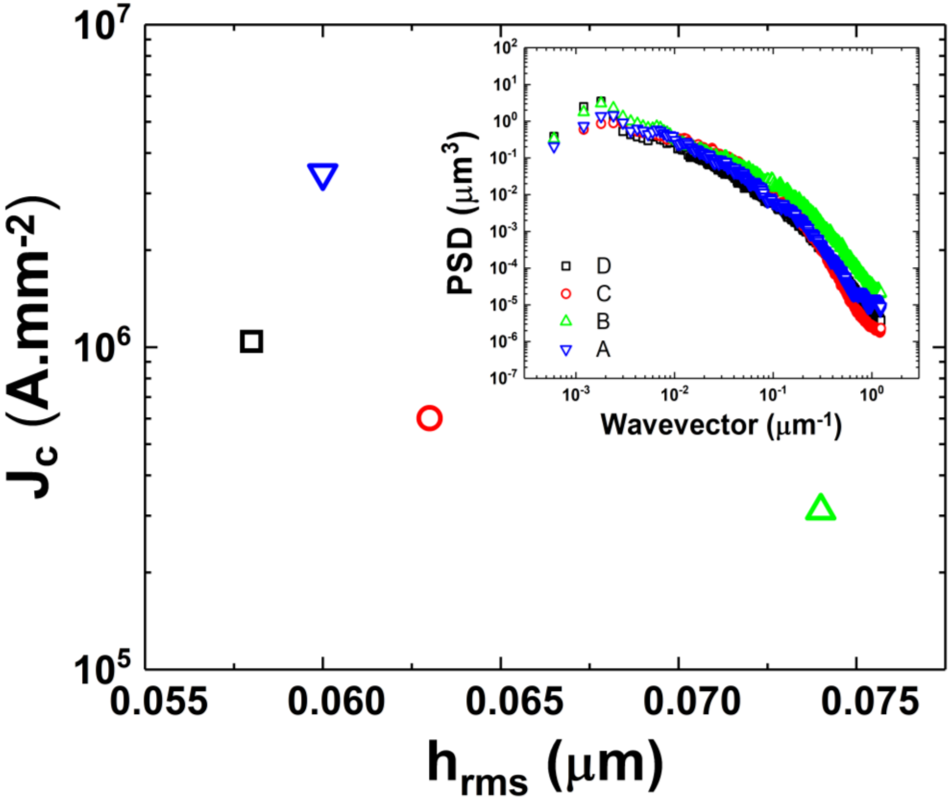
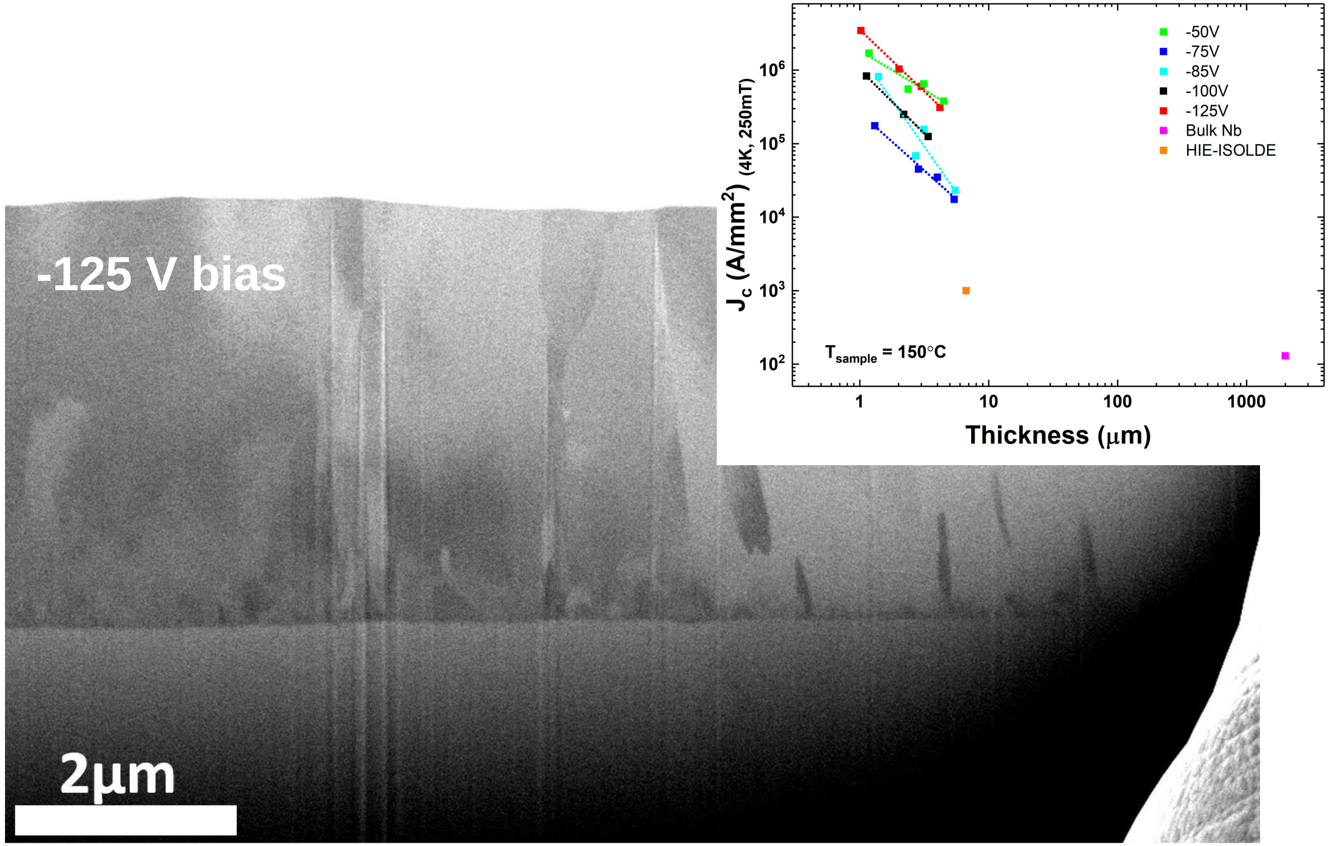
J_c vs film's thickness



J_c does not correlate with roughness

J_c vs film's thickness

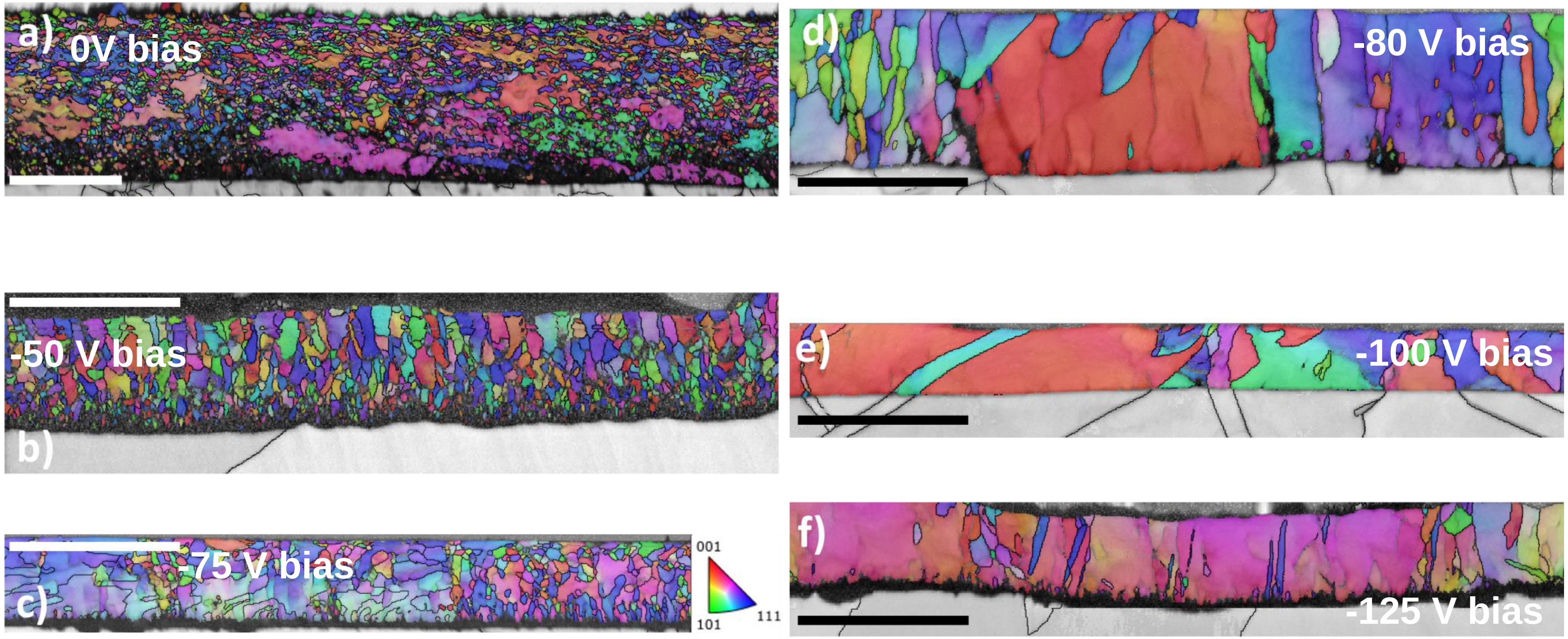




J_c does not correlate with roughness

J_c thickness dependency cannot be correlated with grain growth kinetics

J_c vs film's thickness



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J_c thickness dependency cannot be correlated with grain growth kinetics

J_c vs Film Thickness and Bias

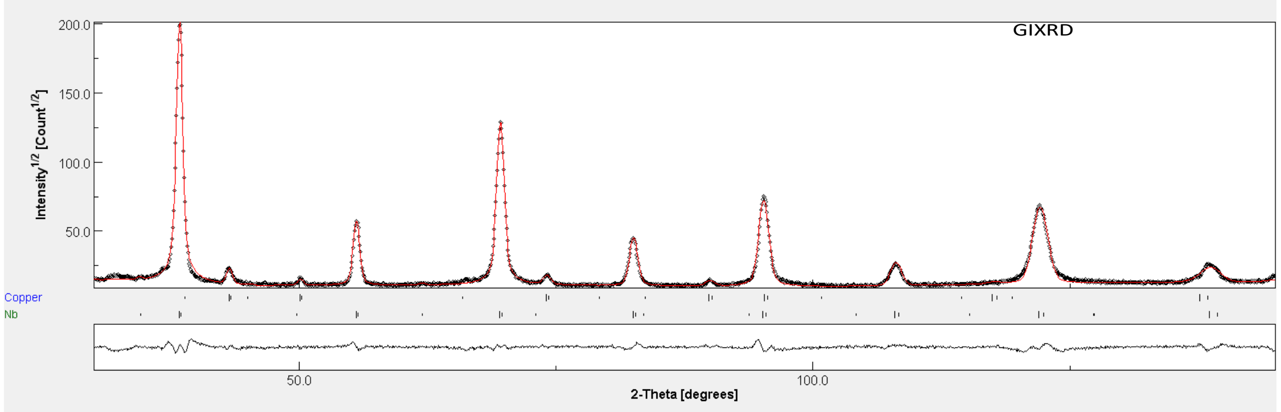
Grazing incidence XRD analysis

Surface defects density evaluation

Diffractogram Rietveld refinement

- crystallites size
- microstrain

$$\rho_D = 2\sqrt{3}\frac{\langle \varepsilon^2 \rangle^{1/2}}{D \times b}$$



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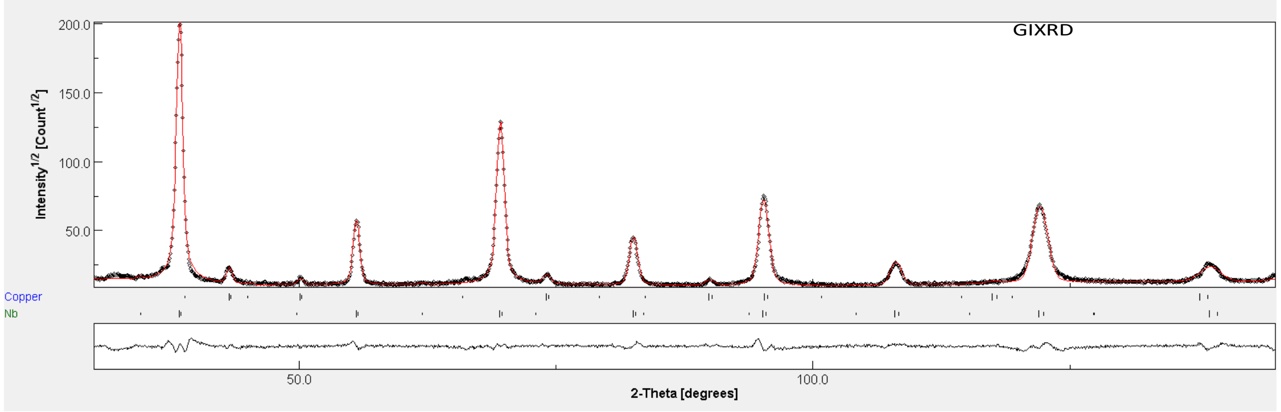

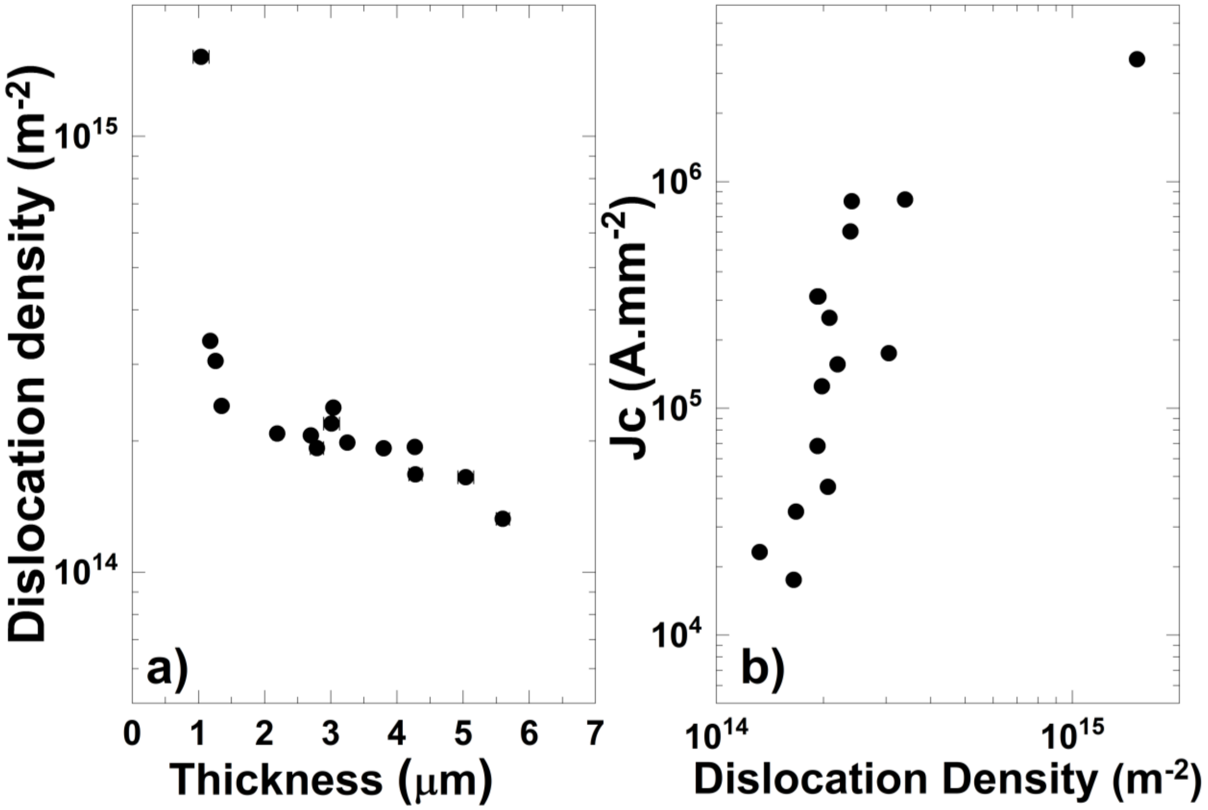

Dislocations appear to be a good candidate for explaining the Jc modultaion

GIXRD DO NOT «SEE» ALL THE DISLOCATIONS

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Energy deposition during growth

D.K. Brice, J.Y. Tsao, S.T. Picraux, Nucl. Instr. Meth. B 44 Ž1989. 68.

Ma *et al*. Sketching of preferred energy regime for ion beam assisted epitaxy Applied Surface Science 137 (1999) 184–190

Brice *et al* model further optimized/simplified by Ma *et al*

Surface = 1st monolayer
Underneath = bulk

Crystalline plane family {hkl}	Coordination number	$E_d^{(s)}$ (eV)	d^{hkl} (Å)
{110}	6	27	$a/\sqrt{2}$
{100}	4	18	$a/2$
{211}	5	22.5	$a/\sqrt{6}$
{310}	4	18	$a/\sqrt{10}$
{111}	4	18	$a/\sqrt{12}$
{321}	5	22.5	$a/\sqrt{14}$
{210}	4	18	$a/\sqrt{20}$

$$E(x_s) = \left(\sqrt{E} - \frac{1.7 + K_l}{2R_c} x_s \sqrt{E_c}\right)^2$$

Ion's energy at depth x_s (here taken as plane inerspacing)

$$\Psi_d^{(s)}(E, E_d^{(s)}) = \frac{1.7}{1.7 + K_l}\left[E - E(x_s) - 4\left(\frac{E_d^{(s)}}{\gamma}\right)^{3/4}(E^{1/4} - E(x_s)^{1/4})\right]$$

Energy deposited within the surface per incident ion

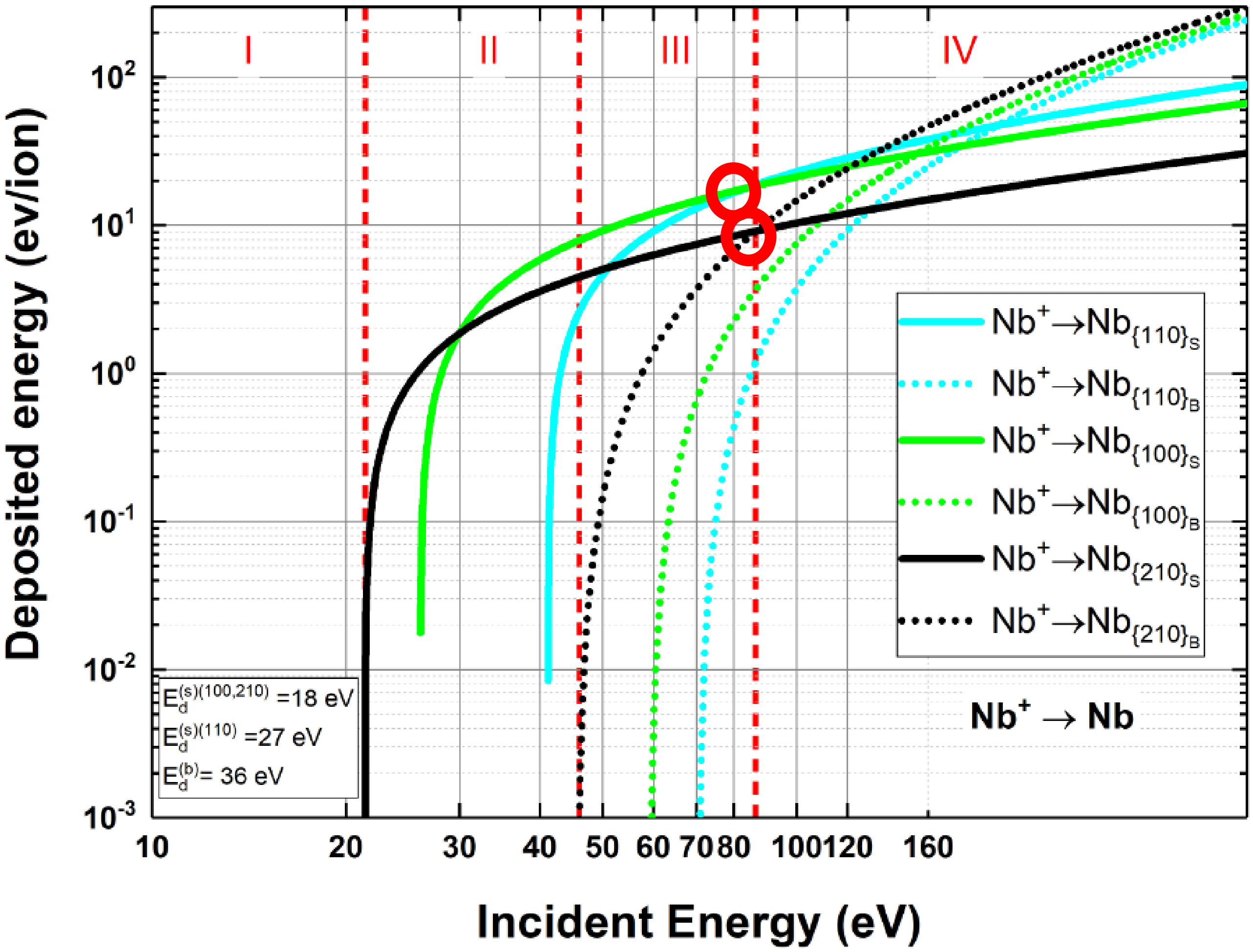
$$\Psi_d^{(b)}(E, E_d^{(b)}) = \frac{1.7}{1.7 + K_l}\left[E(x_s) - \frac{E_d^{(b)}}{\gamma} - 4\left(\frac{E_d^{(b)}}{\gamma}\right)^{3/4}\left(E(x_s)^{1/4} - \left(\frac{E_d^{(b)}}{\gamma}\right)^{1/4}\right)\right]$$

Energy deposited within the bulk per incident ion

Energy deposition during growth

Four regimes identified

I
No energy transfer to the lattice – full recoil
No growth assistance provided



Energy deposition during growth

Four regimes identified

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No energy transfer to the lattice – full recoil
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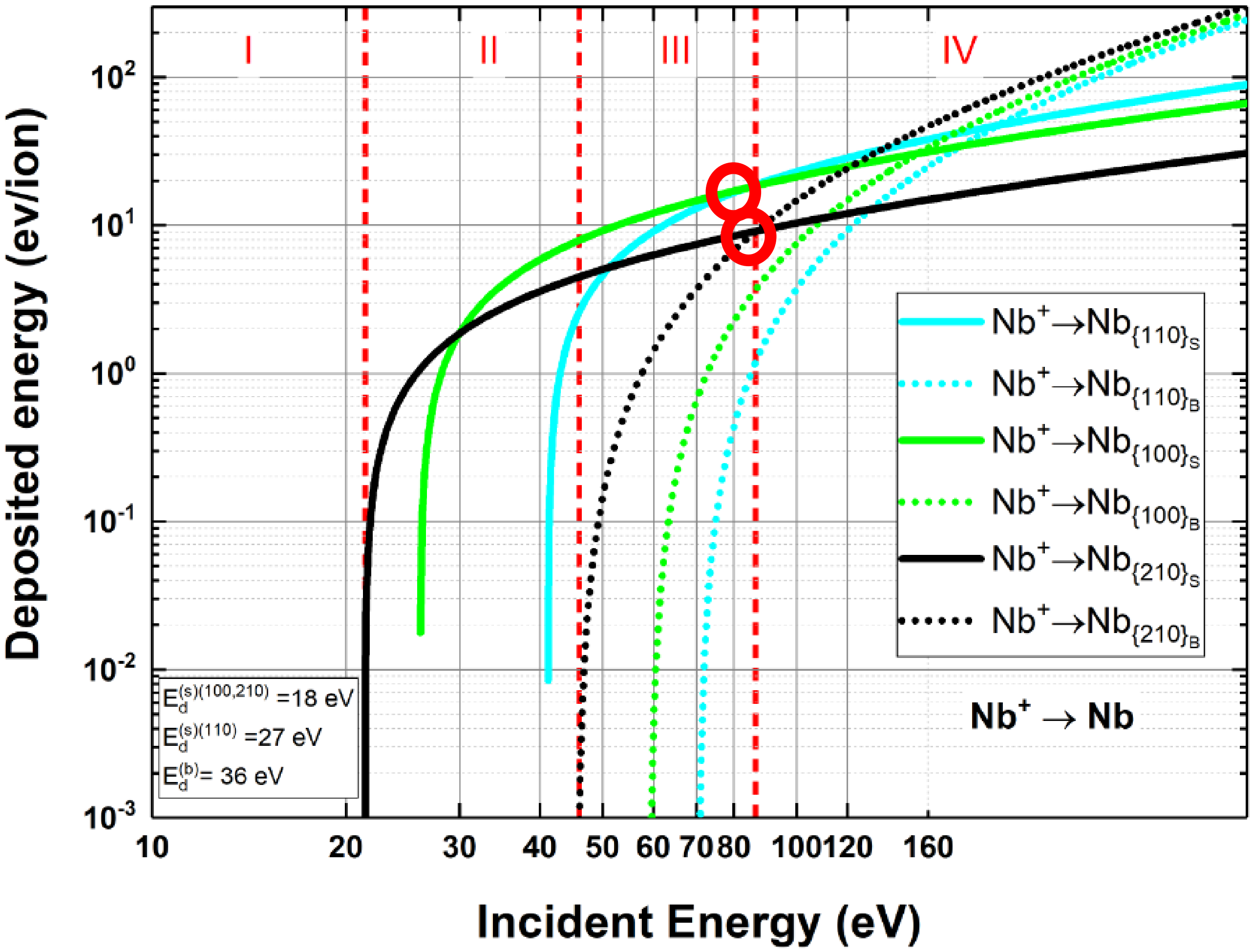
II
Only the surface layers are impacted.
Low densification
Possibility of buried defects not annealed by further bombardment (out of reach)



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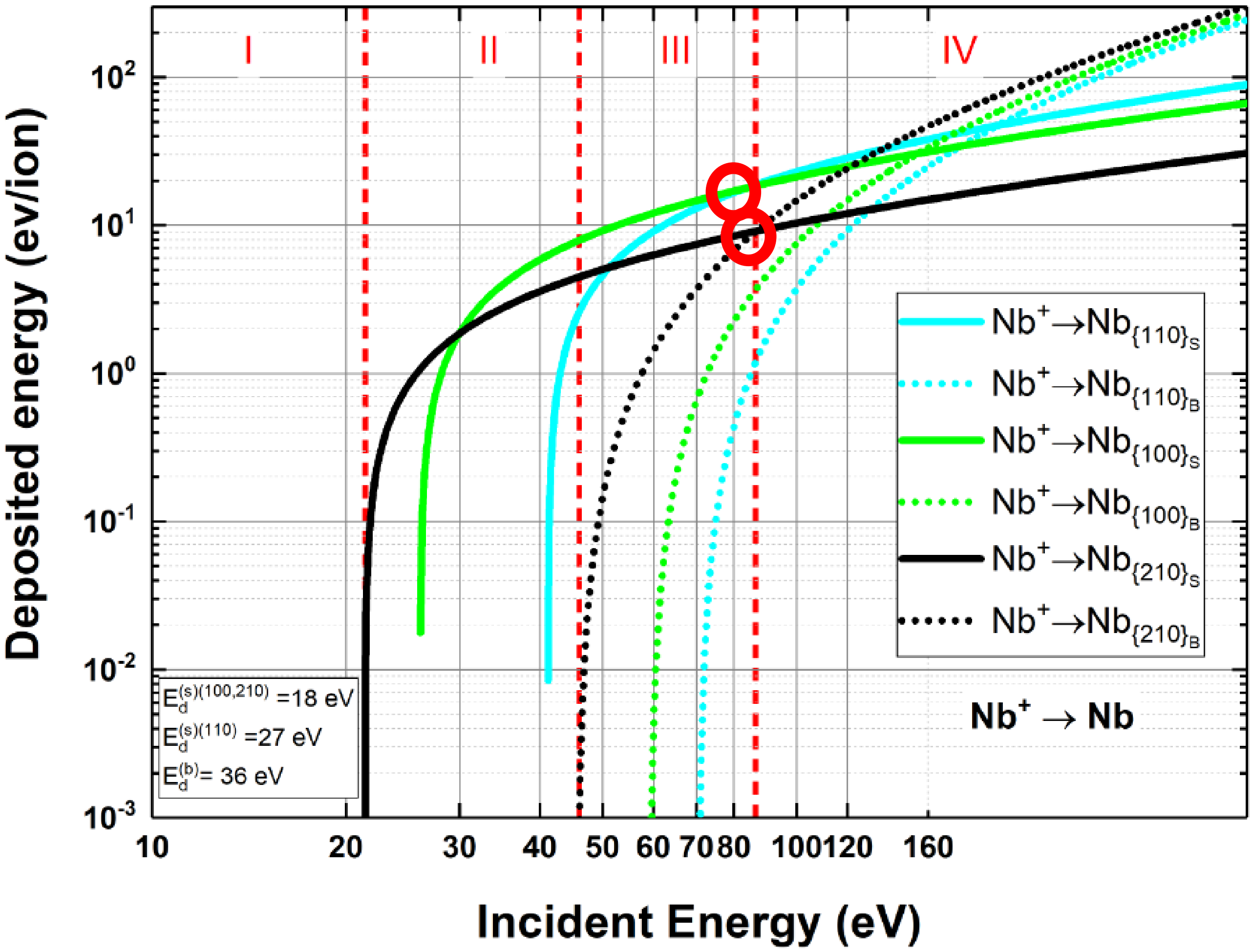
III
Both surface and bulk energy deposition



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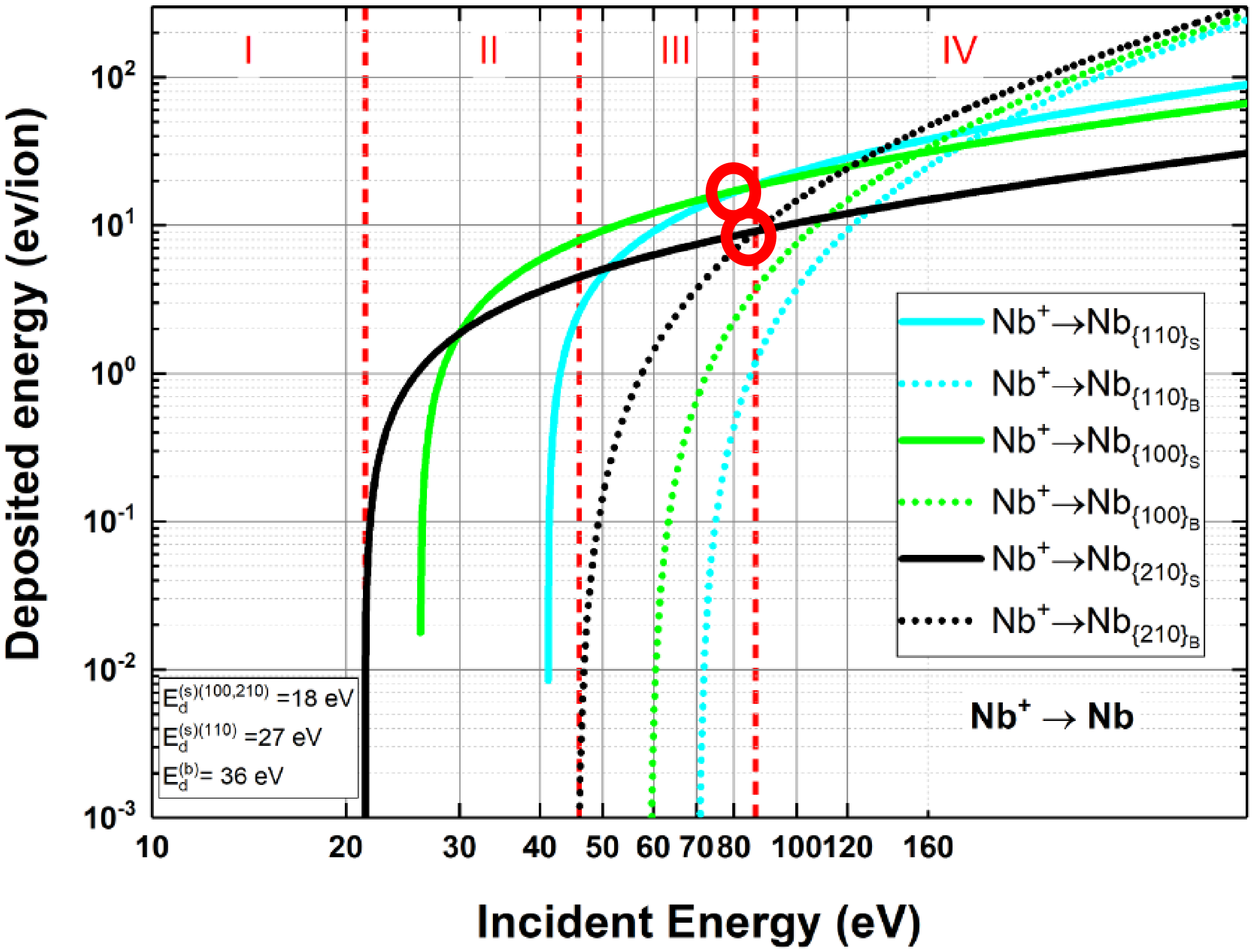
III
Both surface and bulk energy deposition

IV
More energy deposited within the bulk
Buried defects
Gas incorporation



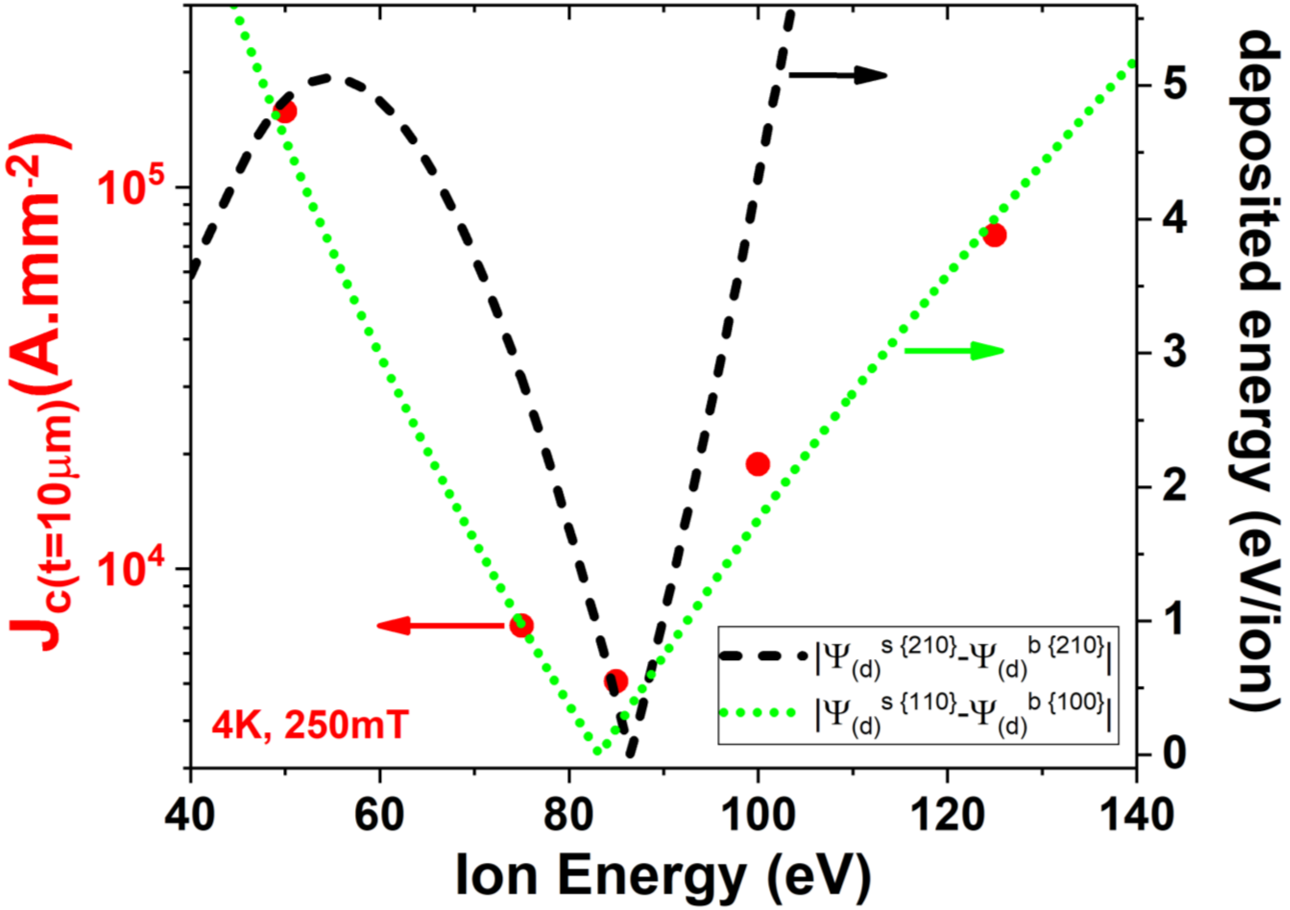
Comparison to experimental data

J_c vs energy seems correlated to the energy spread across {110} and {100} crystalline planes.

Optimum in Jc matches with the minimum energy spread across {110} and {100} crystalline planes.

Further investigations foreseen:
- EBSD
- Nano-indentation

And of course: RF testing of 1.3GHz cavities



Conclusion

- J_c is used to optimize the quality of Nb/Cu films in view of reducing field trapping and subsequent dissipation during RF operation
- Thickness dependency of Jc cannot be linked to neither roughness nor grain boundaries density (grain size)
- Dislocations appear as a good candidate to explain the Jc mitigation with layer thickening
- An optimum in J_c is found close to 80eV and appears to be attributed to the energy transferred to the various crystalline planes during the growth.
- Optimal point also confirmed by a separate study using 3rd harmonic RF test
  https://arxiv.org/abs/2305.07746
- More analysis are needed to support further the model

«God made the bulk; the surface was invented by the devil»

W. Pauli

Stay motivated ;)

Thank you

Setups

UNIVERSITÉ DE GENÈVE

VSM-SQUID J_c

FIB-SEM XB 540
Milling / Cross-sections

SEM Σigma (*Zeiss*)
EBSD