Reactive (HiPIMS) through the eyes of a ‘simple’ model

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Outline

❶ Introduction

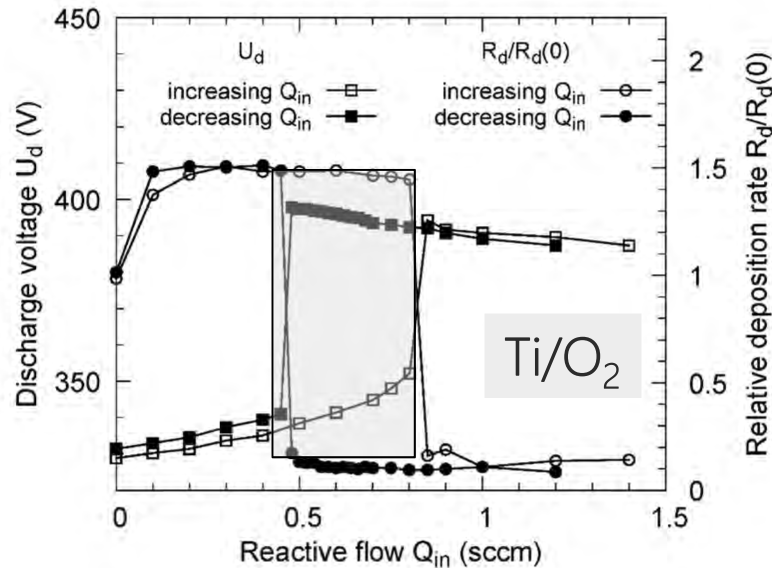
❷ IV-characteristic

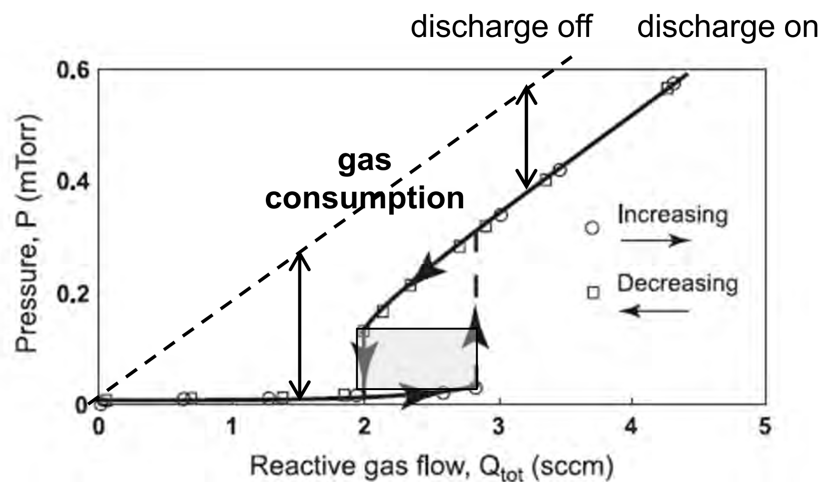
❸ Reactive IV-model

❹ Results

❺ Conclusion

Flow controlled DCMS hysteresis





S. Berg, et al., *Thin Solid Films 476*, 215 (2005)

Direct hysteresis experiment = stepwise in/decrease of single **operation** parameter

Hysteresis @ constant current

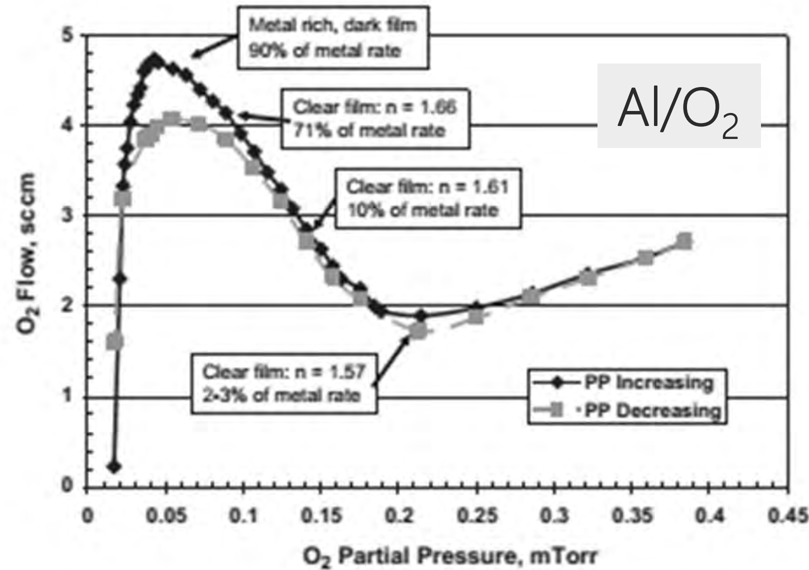
a) reactive gas pressure
b) discharge voltage
c) deposition rate

by **poisoning**

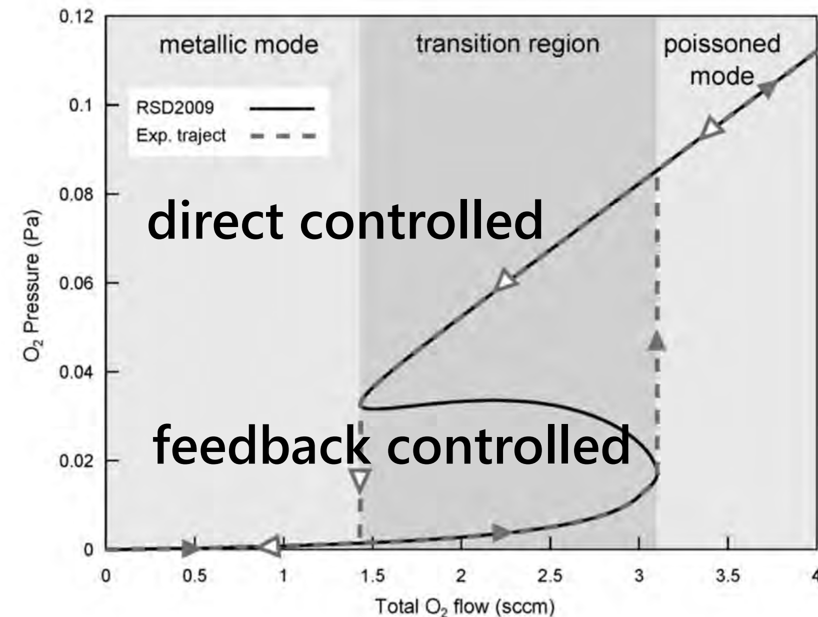
a) vanishing getter pump
b) changing γ_{see}
c) decreasing sputter yield ($Y_c << Y_m$)

☺ Easy process control
☹ No access to transition mode!
☹ Bad film/deposition control

Pressure controlled DCMS hysteresis



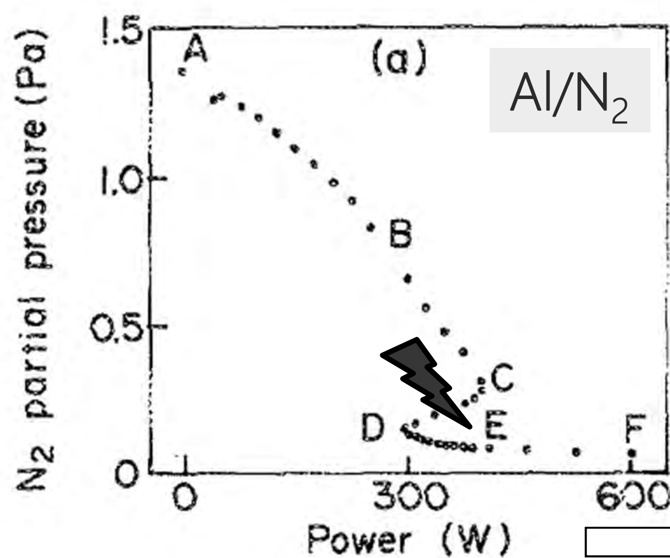
W.D. Sproul, et al., *Thin Solid Films 491*, 1 (2005)



Feedback hysteresis experiment =
stepwise in/decrease of **variable** by feedback controlled operation parameter

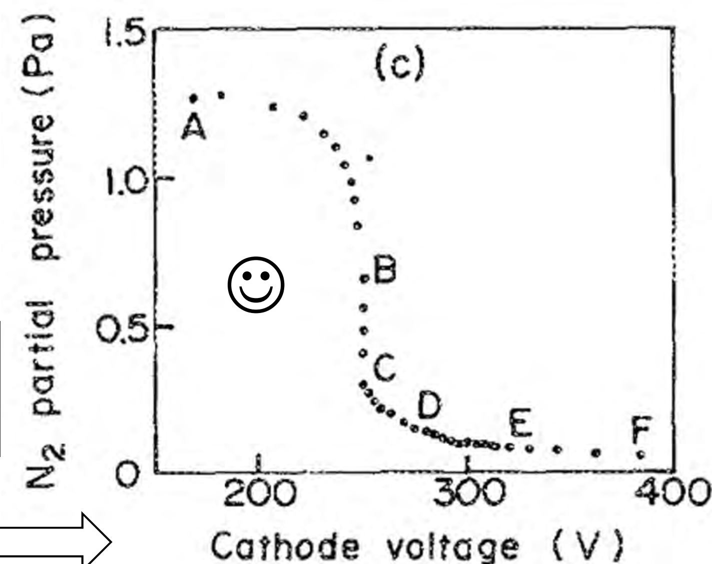
☺ Access to transition
☺ Better film/deposition control
☹ Harder process control

Voltage controlled DCMS hysteresis

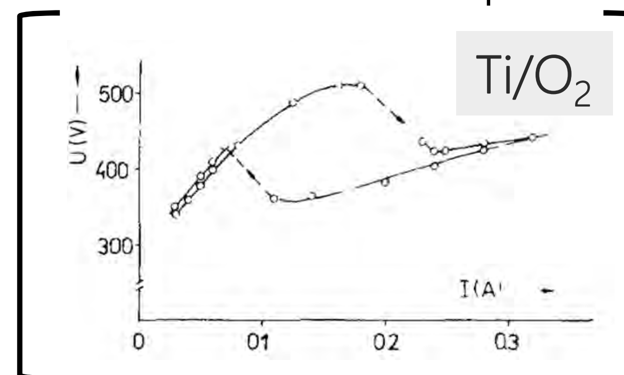


Wisely choose your operation parameter!

McMahon, et al., *J. Vac. Sci. Technol. 20*, 376 (1982)



Succes is material dependent

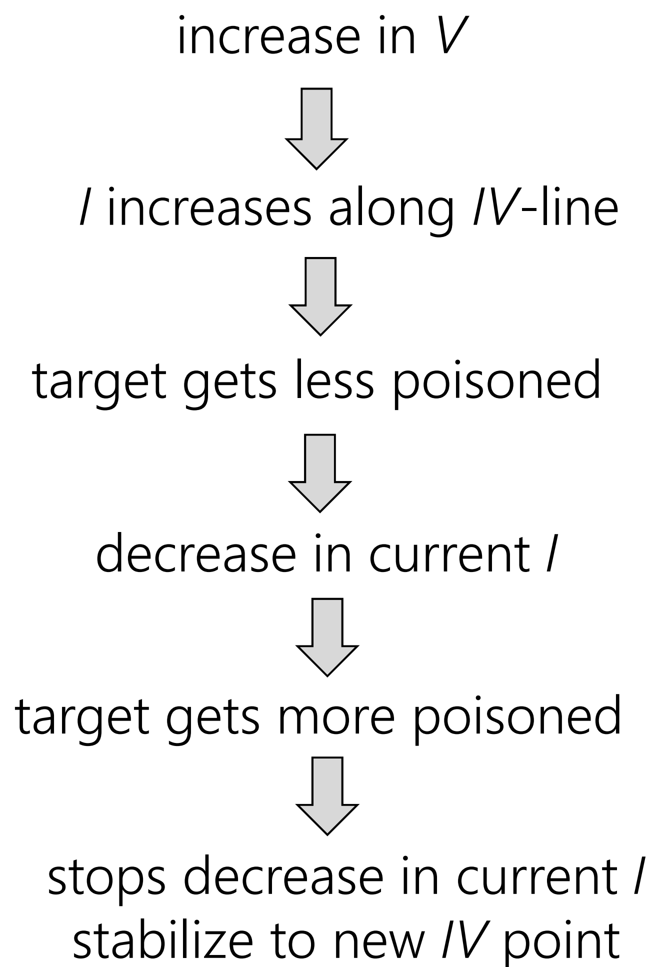


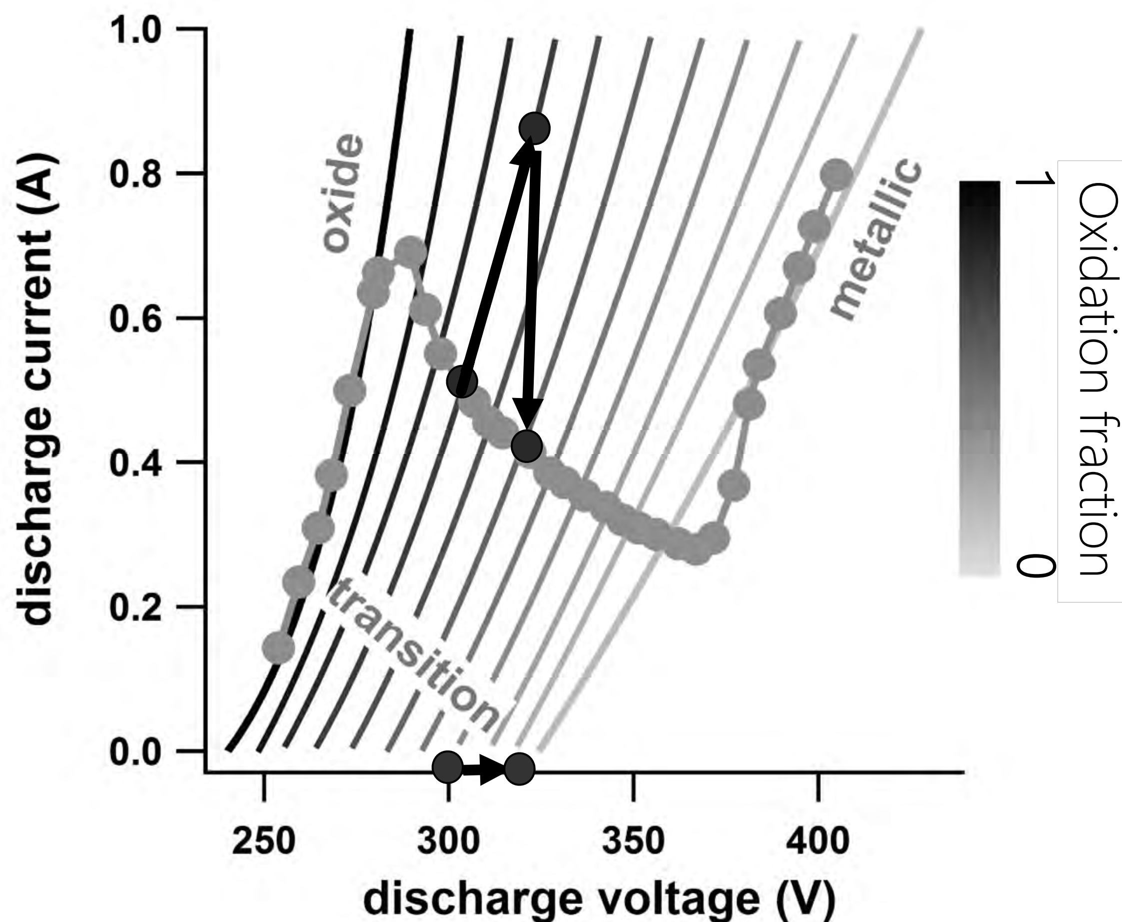
Steenbeck, et al., *Thin Solid Films 92*, 371 (1982)

Voltage control of reactive (O_2/N_2) Al deposition is the key to

- ☺ Access to transition
- ☺ Better film/deposition control
- ☺ Easy process control

Stability of voltage control





Voltage control for R-HiPIMS

Hysteresis study:

direct control on the reactive flow is common while fixing the current/power

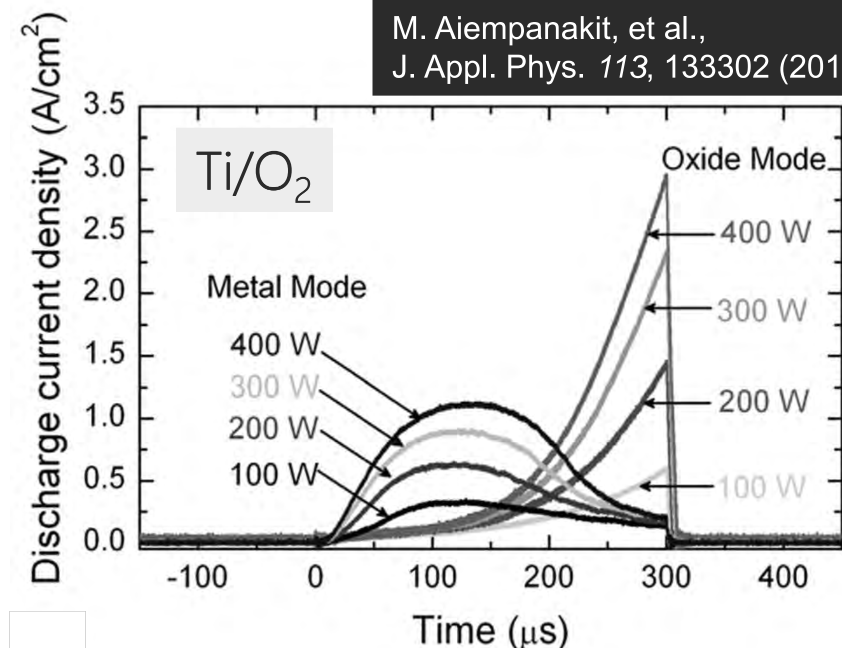
… but much can be learned from the **IV-characteristic** as it is stronly influenced by target state

➔ (ion-induced) secondary electron emission yield γ_{see}

As HiPIMS is typically voltage controlled, this way of looking to R-HiPIMS may be an interesting tool.

➔ shift of a 'pure' **plasma** viewpoint to a more **target** oriented viewpoint

M. Aiempanakit, et al.,
J. Appl. Phys. *113*, 133302 (2013)



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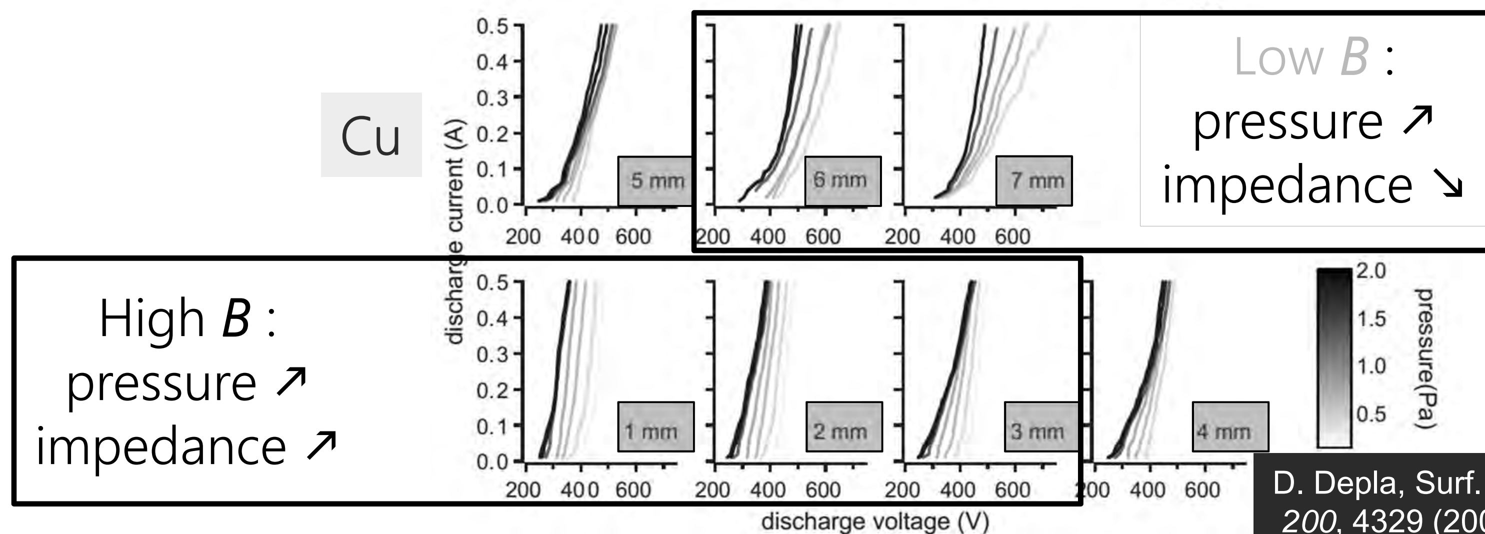
IV-dependencies

- Process parameters
  - ✓ **gas pressure**/composition ➔ reactive gas fraction < 20 %
  - ✓ magnetron design
    - ➢ **magnetic field *B*** ➔ target erosion
    - ➢ anode position ➔ shielding or disappearing

I. Petrov, et al., J. Vac. Sci. Technol. A 11, 2733 (1993)

target thickness ↗ magnetic field *B* ↘

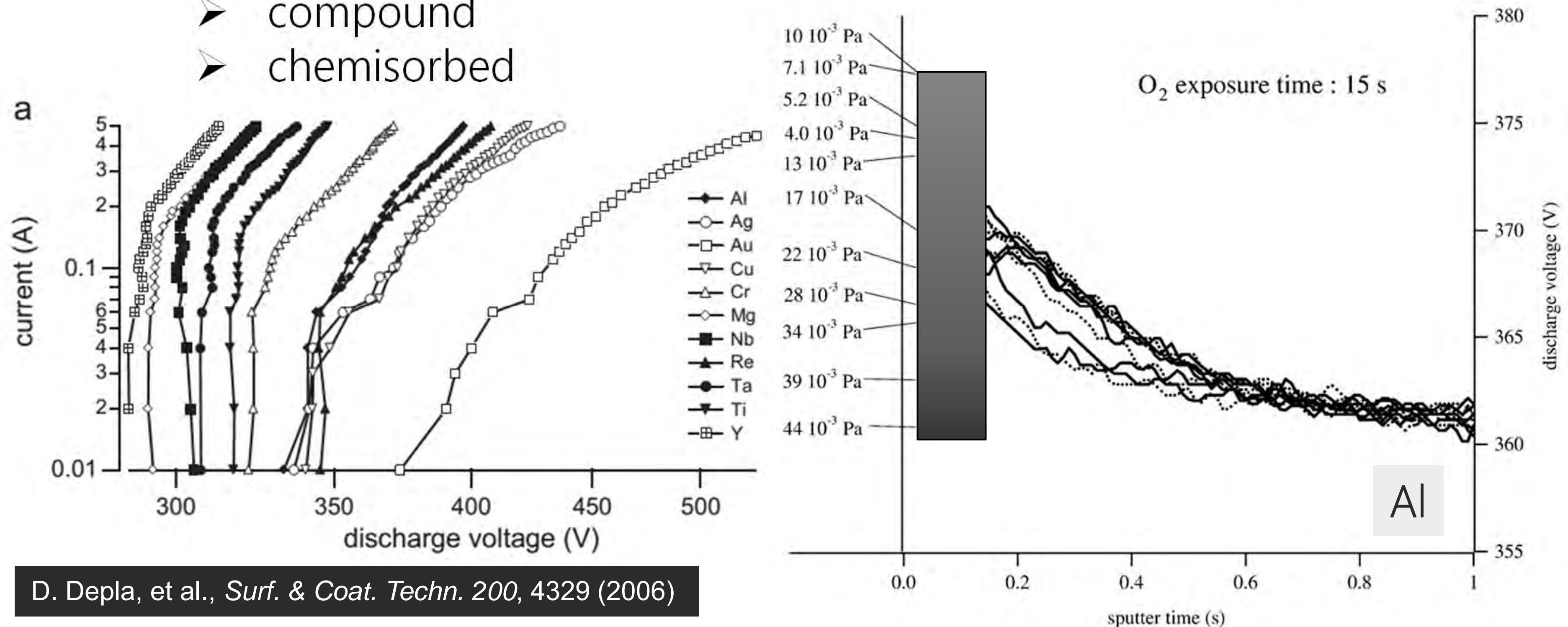


D. Depla, Surf. & Coat. Techn. *200*, 4329 (2006)

IV-dependencies

- Target condition
  - ✓ sputter yield ➔ gas rarefaction (HiPIMS ↔ DCMS)
  - ✓ elemental composition ➔ secondary electron emission yield γ_{see}
    - ➢ metal ➔ electron reflection probability r(p)
    - ➢ compound
    - ➢ chemisorbed

F. Moens, et al., *Frontiers in Physics 5*, 51 (2017)



D. Depla, et al., *Surf. & Coat. Techn. 200*, 4329 (2006)

Thornton relation extended

D. Depla, et al., *Thin Solid Films 517*, 2825 (2009)

$$V_D = \frac{W_0}{E(p,B,I)\gamma_{see}[\varepsilon_i\varepsilon_e](p,B,I)}$$

$\gamma_{eff,see}$

W_0: effective ionization energy
$\varepsilon_i(p,B,I)$: ion collection efficiency
$\varepsilon_e(p,B,I)$: e⁻ ionization efficiency
$E(p,B,I) = \langle mr \rangle$:
effective gas ionization probability
m: e^- multiplication factor
r: e^- reflection probability

Target dependency of V_D in γ_{see} and r

Values? γ_{see}
☺ metals: emperical formulas
+ experimental data
☹ compounds:
limited data

r
☹ what makes it fit? ½?
☺ impact low for 0 to 0.6

G. Buyle, Phd Thesis, Ghent (2005)

Is sheath energization alone?

A. Anders, *Appl. Phys. Lett. 105*, 244104 (2014)

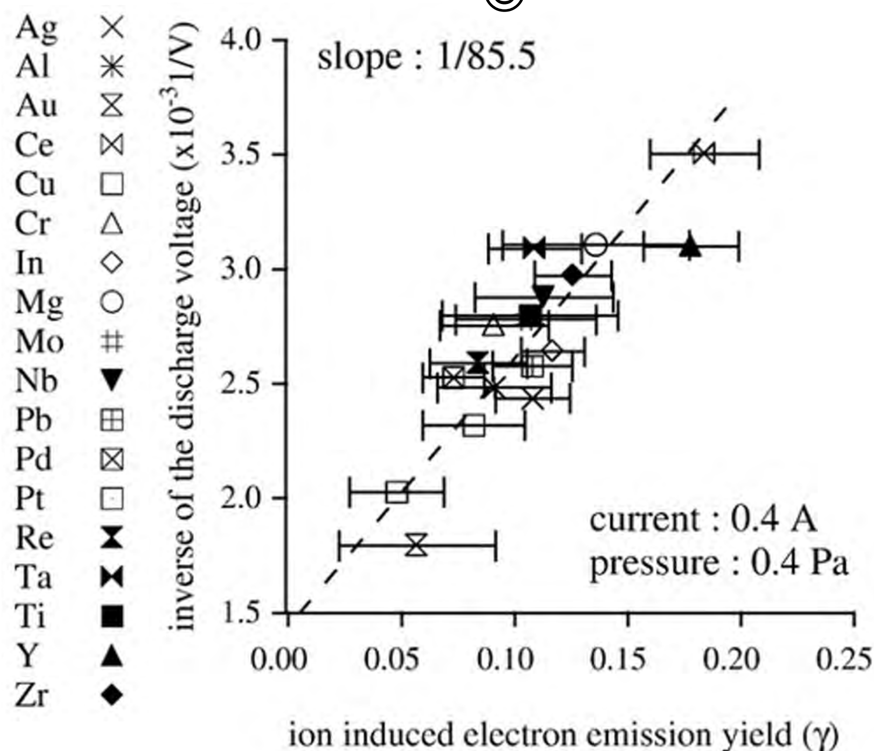
☞ modelling results : **Ohmic heating** has a role (HiPIMS ↔ DCMS)
but
no (direct) target dependency

$$\frac{1}{V_D} = (A(r)\gamma_{see})_{sheath} + B_{Ohmic}$$

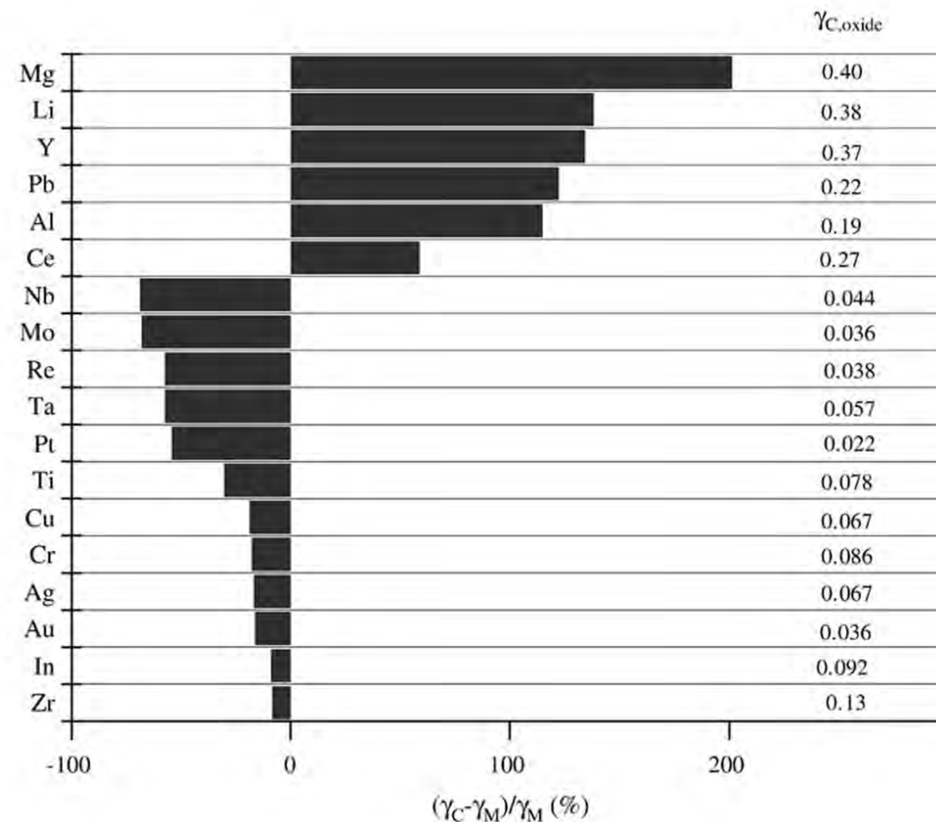
N. Brenning, et al., *Plasma Sources Sci. Technol. 25* (2016) 065024

Secondary electron emission yields γ_{see}

Dominant target dependency of V_D is on γ_{see}



Use this correlation for the γ_{see} of compounds



but slope is pressure/current dependent for given sputter system

D. Depla, et al., *Thin Solid Films 517*, 2825 (2009)

IV-relations

For diode sputtering:

$$I = \beta(V - V_0)^{3/2} \quad \rightarrow \quad \beta \sim \gamma_{see}$$

E.J. Soxman, *Proc. of 7th Int. Vacuum Congress*, p. 309 (1977)

For DC magnetrons:

$$I = \beta V^n$$

➔ overestimation at low voltage

J. A. Thornton, *J. Vac. Sci. Technol. 15*, 171 (1978)

For DC and RF magnetrons:

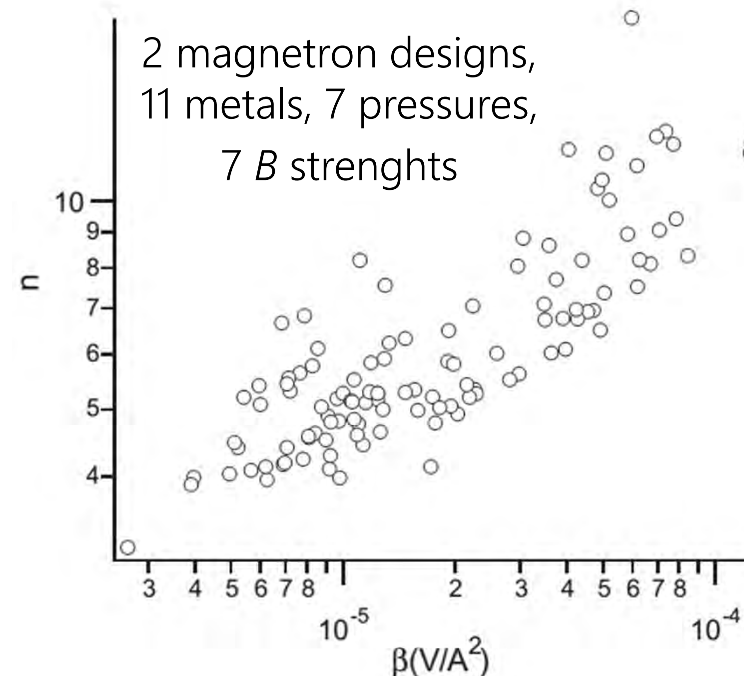
$$I = \beta(V - V_0)^2$$

➔ valid for broad condition range

W.D. Westwood et al., *J. Appl. Phys. 54*, 6841 (1983)

Thornton idea of a V_{min}

β (or n) define sharpness of IV or impedance of plasma

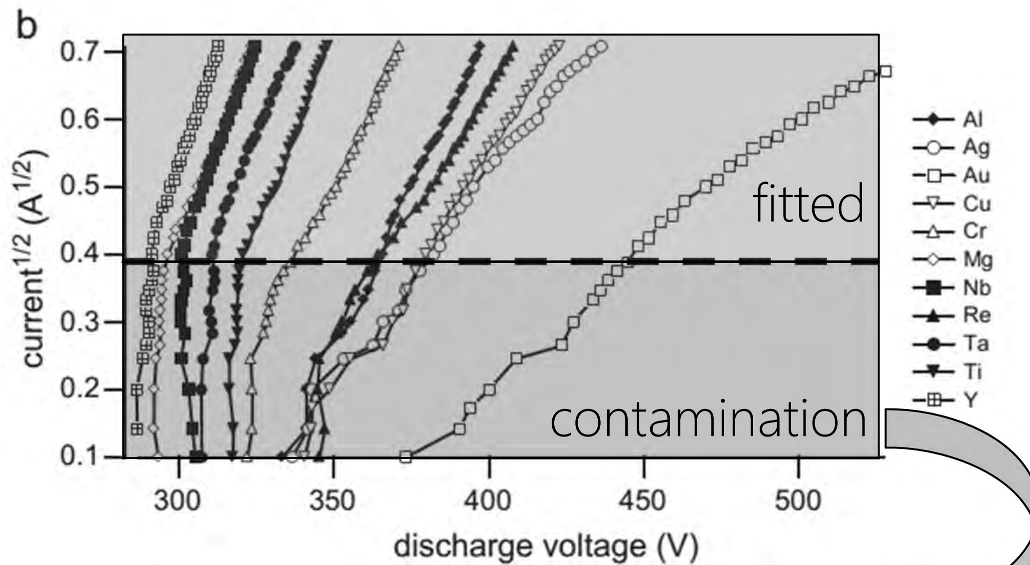


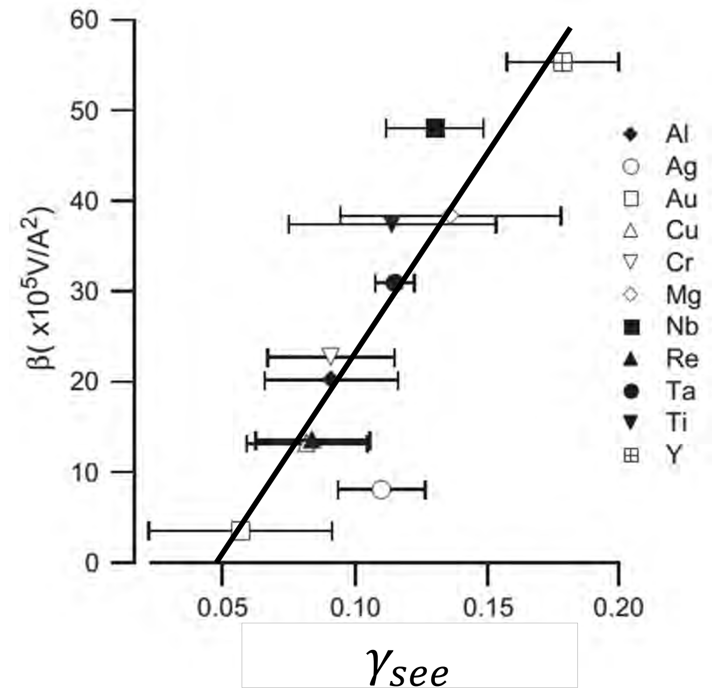
D. Depla, et al., Surf. & Coat. Techn. *200*, 4329 (2006)

IV-relations

Best fitted by $I = \beta(V - V_0)^2$

$\beta \sim \gamma_{see}$





How clean is your target?

(P_{base} < 4×10^{-4} Pa)

R. Schelfhout, et al., *Appl. Surf. Science 355*, 743 (2015)

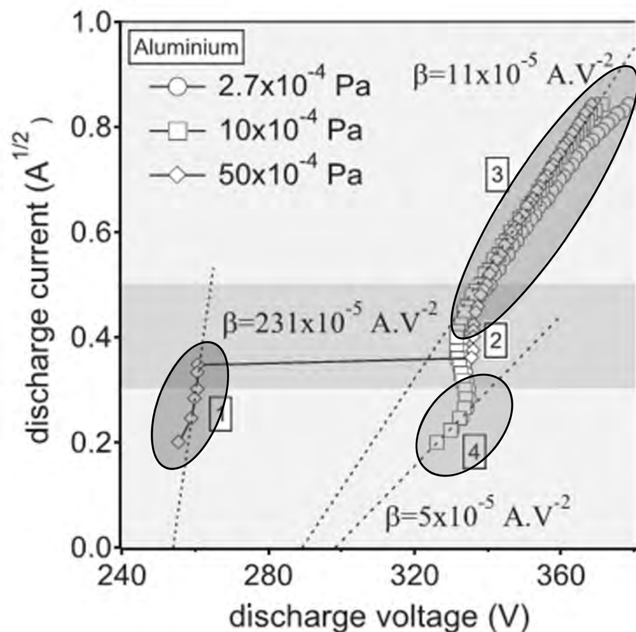
D. Depla, et al., Surf. & Coat. Techn. *200*, 4329 (2006)

Reactive IV-characteristic

A rotating cylindrical magnetron with varied oxygen pressure modifying the target composition between

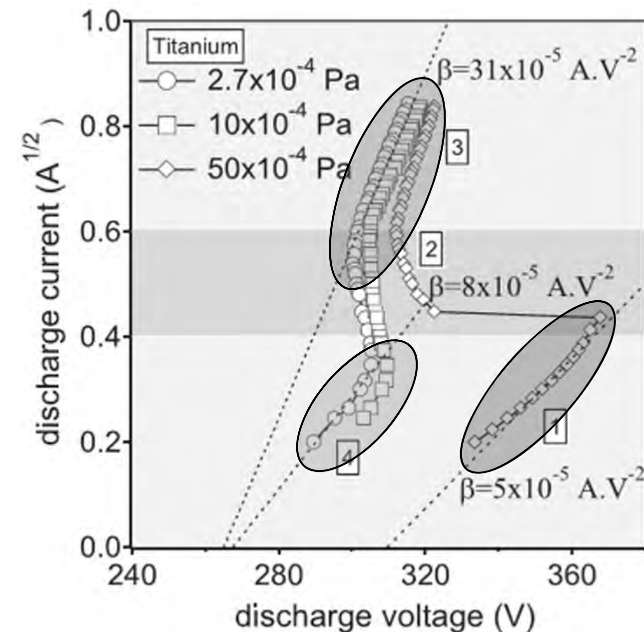
- ✓ metal mode
- ✓ oxide mode
- ✓ (partial?) chemisorbed mode

De Gryse, et al., *Thin Solid Films 520*, 5833 (2012)



$$I = \beta(V - V_0)^2$$

$$\beta \sim \gamma_{see}$$



$$\gamma_{see}^{Al,chem} < \gamma_{see}^{Al} < \gamma_{see}^{Al,oxide}$$

$$\gamma_{see}^{Ti,oxide} < \gamma_{see}^{Ti,chem} < \gamma_{see}^{Ti}$$

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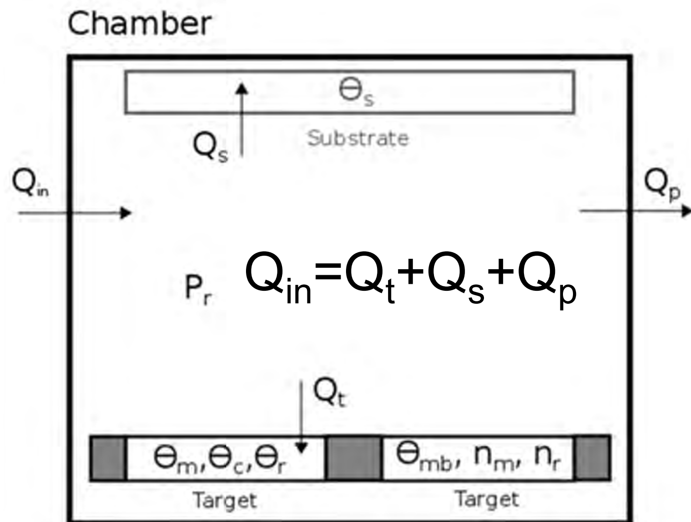
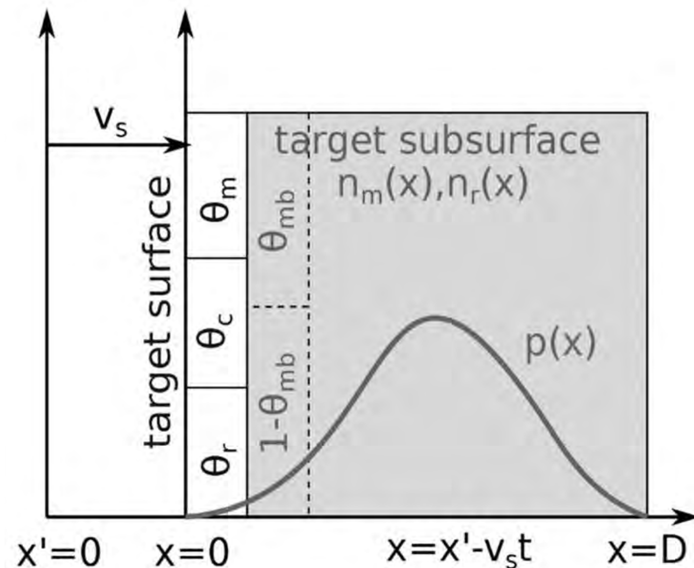
RSD model

= Berg model + advanced target model

model 'engine' = set of balance equations





System part	Resolved variable		Model approach
Chamber	P_r Q_p	reactive partial pressure gas flow to pump	one-cell
Target • Surface	θ_m θ_c θ_r Q_t	metallic fraction chemisorbed fraction compound fraction gas flow consumption	one-cell uniform current multi-cell non-uniform current redeposition profile
• Subsurface	θ_{mb} $n_m(x)$ $n_r(x)$	subsurface metal fraction metal depth concentration reactive depth concentration	continuum in depth
Substrate	θ_s Q_s	chemisorbed concentration gas flow consumption	one-cell multi-cell deposition profile

Strijckmans, et al., *J. Phys. D: Appl. Phys. 47*, 235302 (2014)

Towards RSD model for IV

General IV-relation : $I = \beta(V - V_0)^n$

- assumption: only target surface condition influences IV by changing γ_{see}

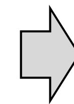
metal (θ_m, γ_m)
compound (θ_r , γ_r)
chemisorbed (θ_c, γ_c)

Generalized Thornton relation

$$\gamma_{see} = \frac{a}{V} + b$$

$$\gamma_{see} = \sum_{m,r,c} \theta_i \gamma_i \qquad \gamma_i = \frac{a}{V_i} + b$$

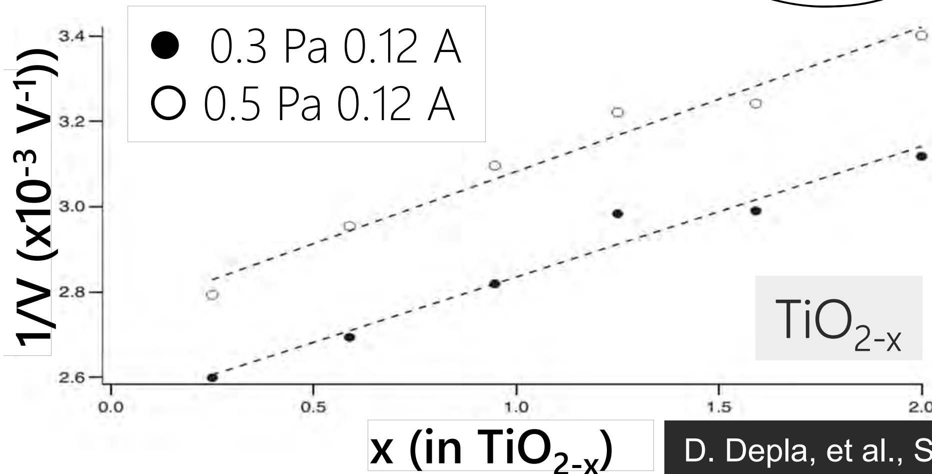
$$\gamma_{see} = a \sum_{m,r,c} \frac{1}{V_i} \theta_i + b \sum_{m,r,c} \theta_i \quad (=1)$$



$$\frac{1}{V} = \sum_{m,r,c} \frac{1}{V_i} \theta_i$$

$$I = \beta_i \left(V_i - V_{0,i}\right)^{n_i}$$

$$\frac{1}{V} = \sum_{m,r,c} \frac{\theta_i}{\left(I/\beta_i\right)^{1/n_i} + V_{0,i}}$$



D. Depla, et al., Surf. & Coat. Techn. *201*, 848 (2006)

Target: single versus multi-cell

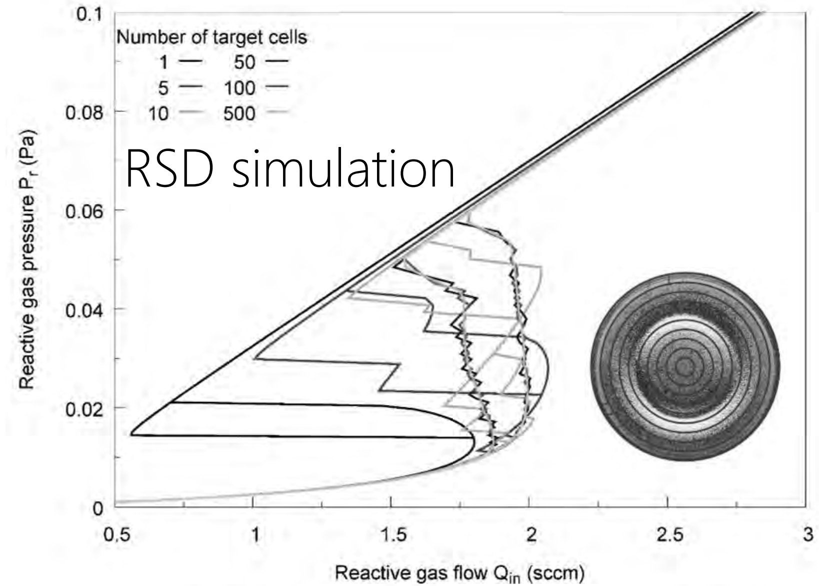
Current on target is non-uniform!

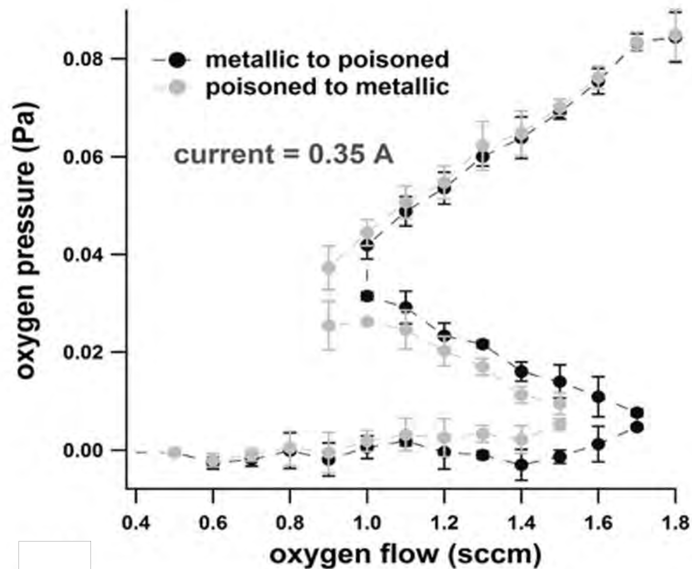
➔ crucial impact on the pQ-hysteresis simulation

➔ emergence of a **double hysteresis** which has been experimentally proved

R. Schelfhout, et al., *Appl. Phys. Lett. 109*, 111605 (2016)

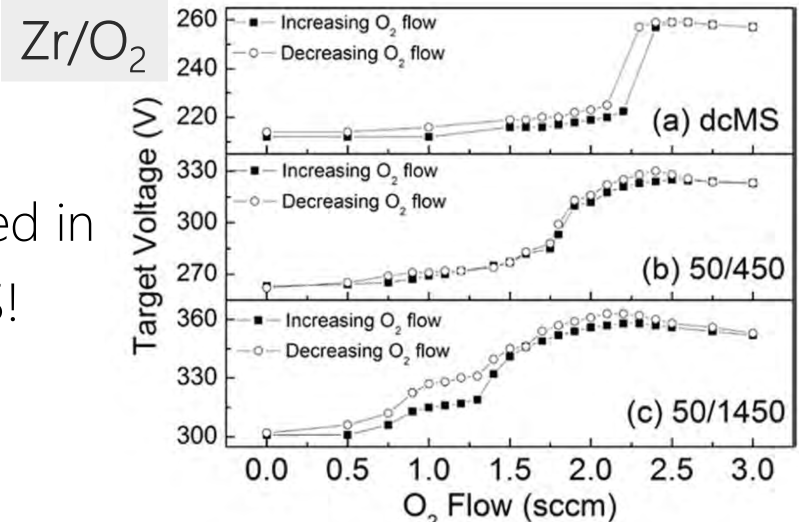
… where the procedure is based on voltage controlled measurement of the IV!





Also observed in R-HiPIMS!

Zr/O_2



Sarakinos, *Surf. Coat. Technol. 202*, 5033 (2008)

Extra's to RSD2013 model

- Linear voltage dependency of the sputter yield : $Y_i(V) = c_i V + d_i \quad i = m, r, c$
- Transport flux J_R of unbounded implanted reactive gas in target bulk
  - pressure-driven transport: $J_R = -T(x) n_R(x)$
    where $T(x)$ scales with implantation profile
  - damage-driven diffusion: $J_R = -D_{dam}(x) \frac{\partial n_R(x)}{\partial x}$
    where $D_{dam}(x)$ scales with damage profile
  - thermic diffusion: $J_R = -D_{therm} \frac{\partial n_R(x)}{\partial x}$
- Specification of IV-relations for metal, compound & chemisorbed (?) target

$$I = \beta_i \left(V - V_{0,i}\right)^{n_i}$$

[
- Gas dilution of sputter yield: $Y_i(n_R, V) = Y_i(V) \frac{n_0}{n_0 + n_R}$ n_0: metal density
  n_R: gas concentration
]

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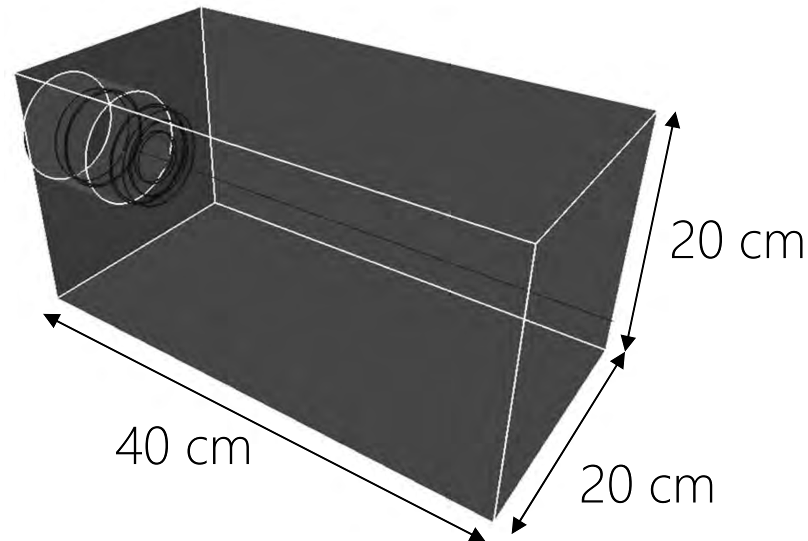
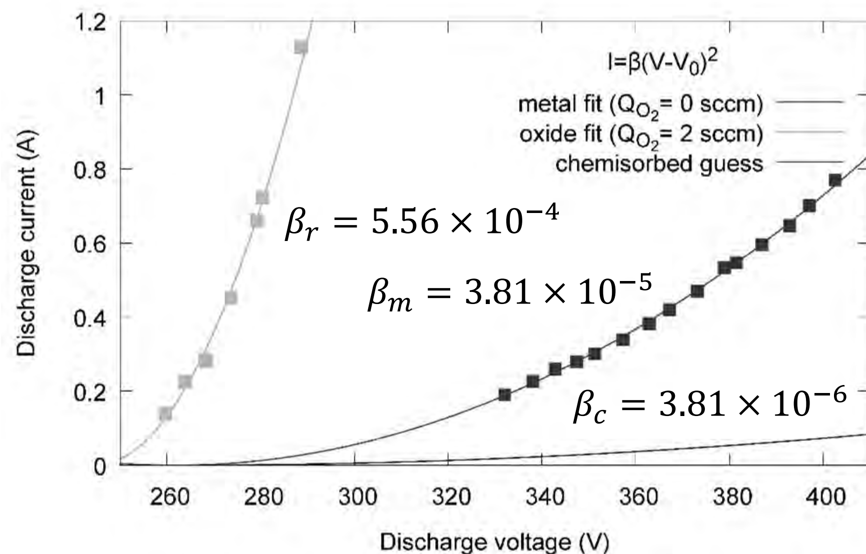
Reference system



Sputter conditions:

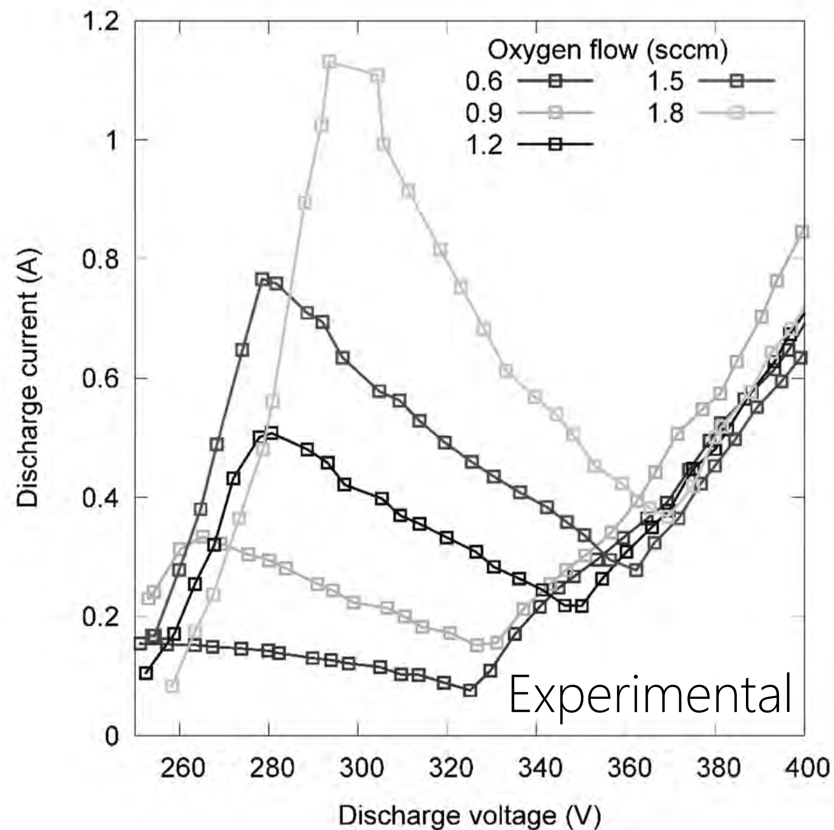
- Target Al
- Process gas Ar
- Reactive gas O_2
- Target diameter = 2 inch (20 cm^2)
- Pumping speed S = 30 L/s
- Argon pressure P_{Ar} = 0.4 Pa
- Oxygen flow Q_{O_2} = 1.2 sccm

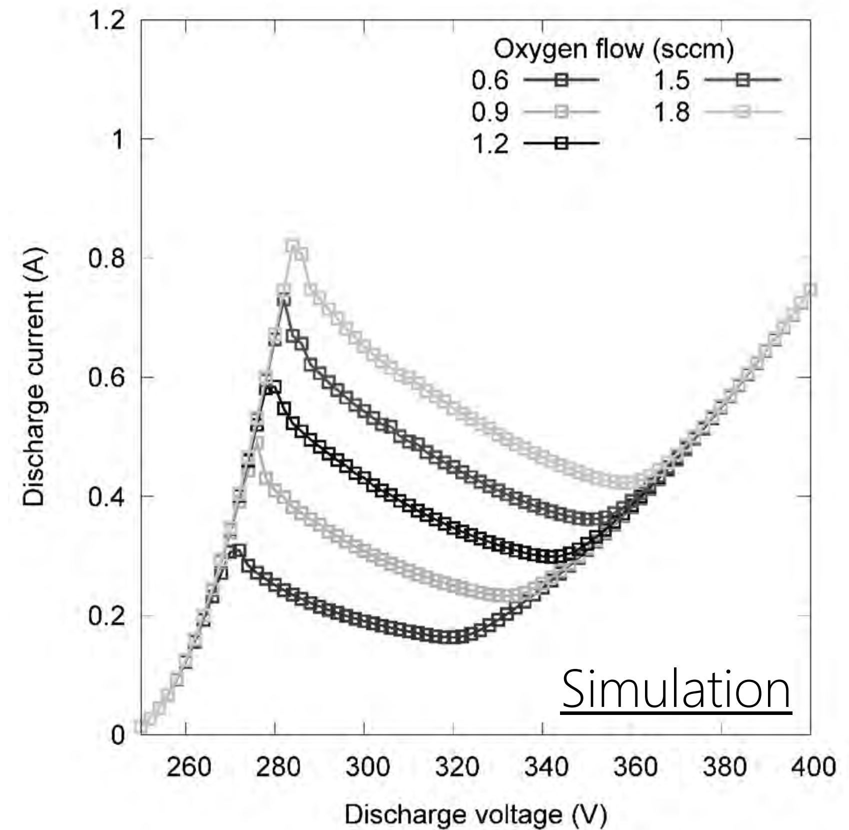


➔ Experimental and simulated IV-relation under variation of these operation parameters

➔ Fitted IV-relation for metal and oxide state
Guess for chemisorbed state!

Influence of oxygen flow

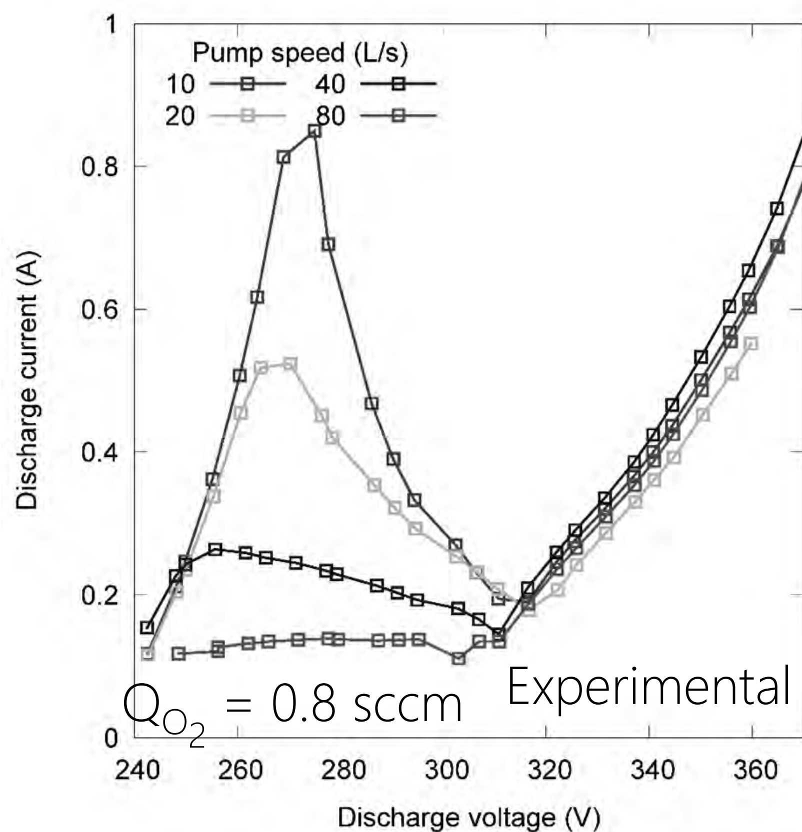


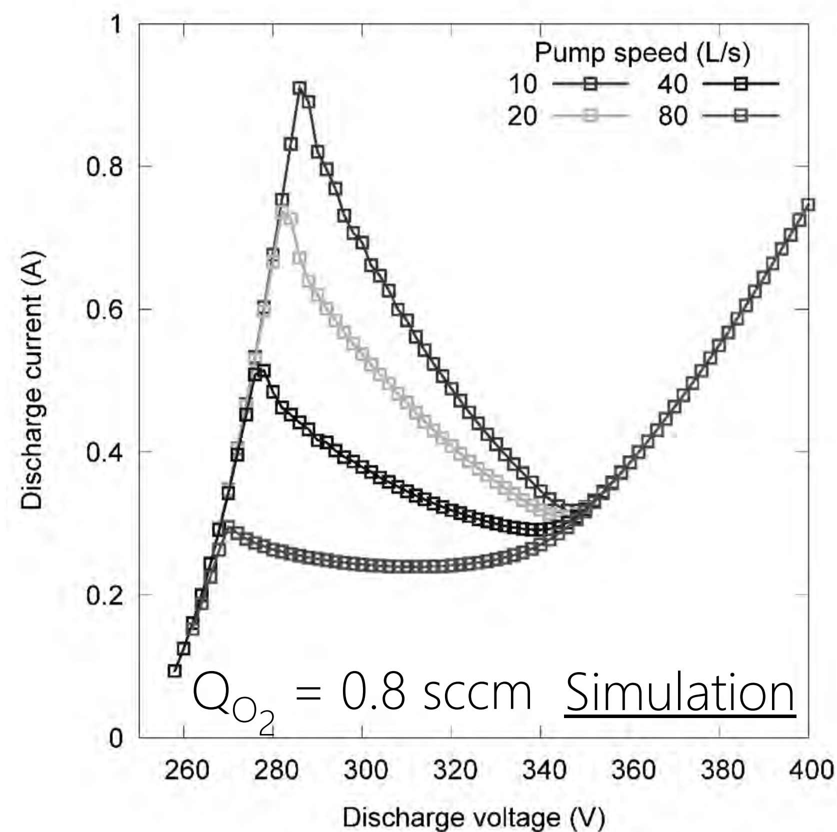


➔ higher oxygen flow makes hysteresis wider

➔ critical points shift to higher discharge currents

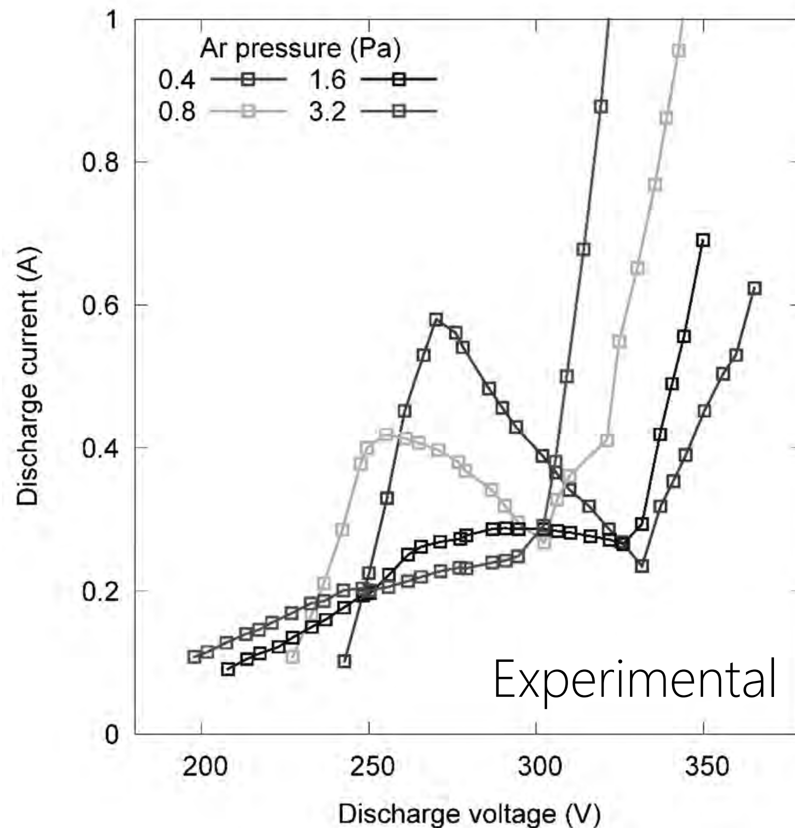
Influence of pumping speed

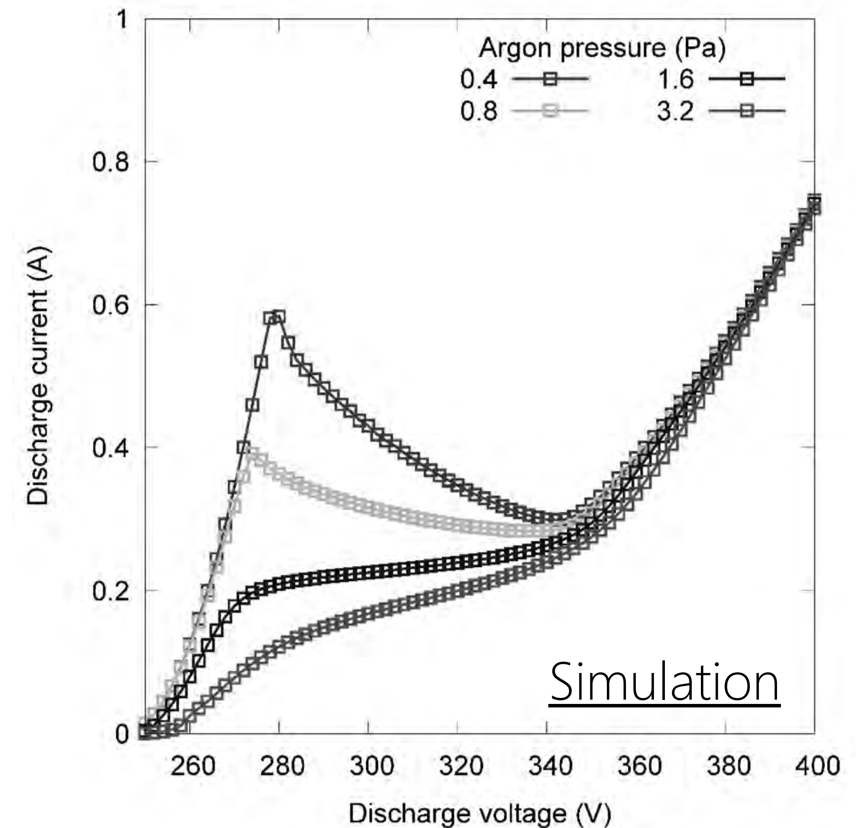




➔ increasing pumping speed removes hysteresis by decreasing current of 2nd critical point

Influence of argon pressure





➔ Ar pressure influences metal IV-relation (not included in simulation)

➔ increasing Ar pressure removes hysteresis

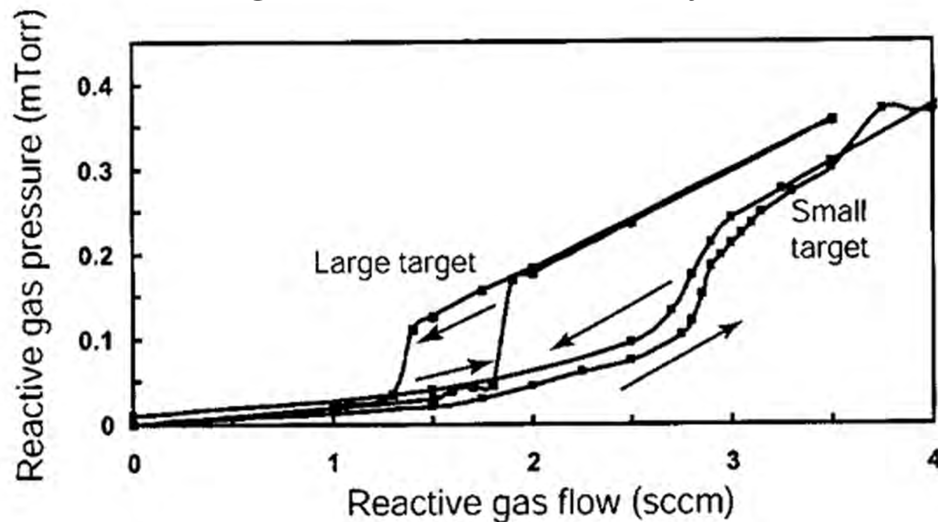
➔ simulation indicates that full poisoning is not reachable at high pressure

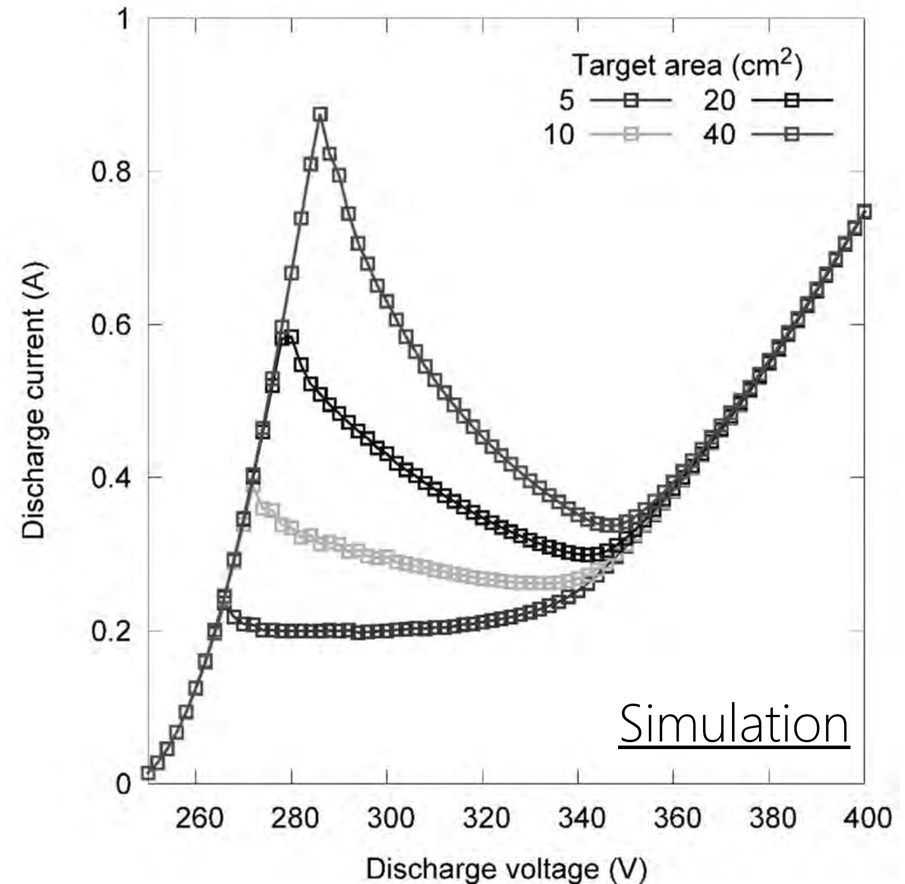
Influence of racetrack

Reduction of hysteresis possible by shrinking the erosion area:

✋ <u>not</u> same as current density ↗ by current ↗

- 1st critical point: amount of sputtered material ~ current
- 2nd critical point: compound removal on target ~ current density

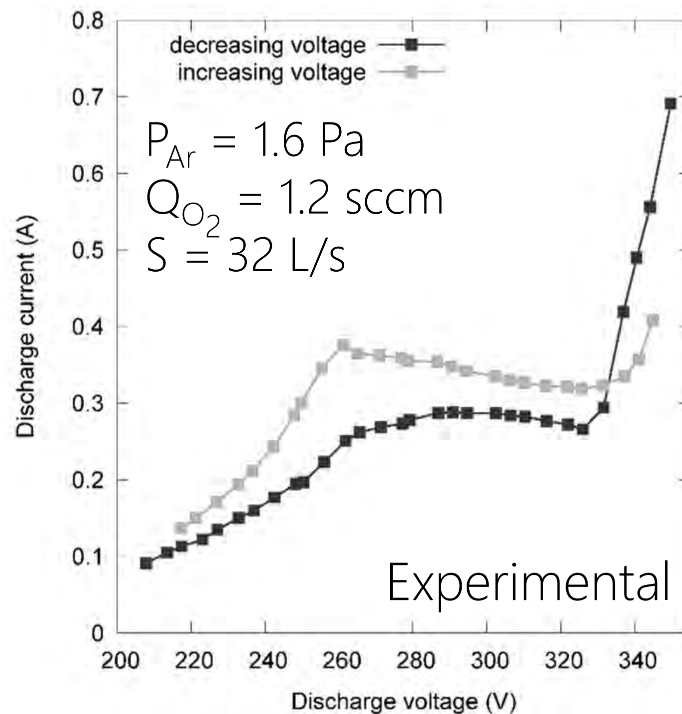




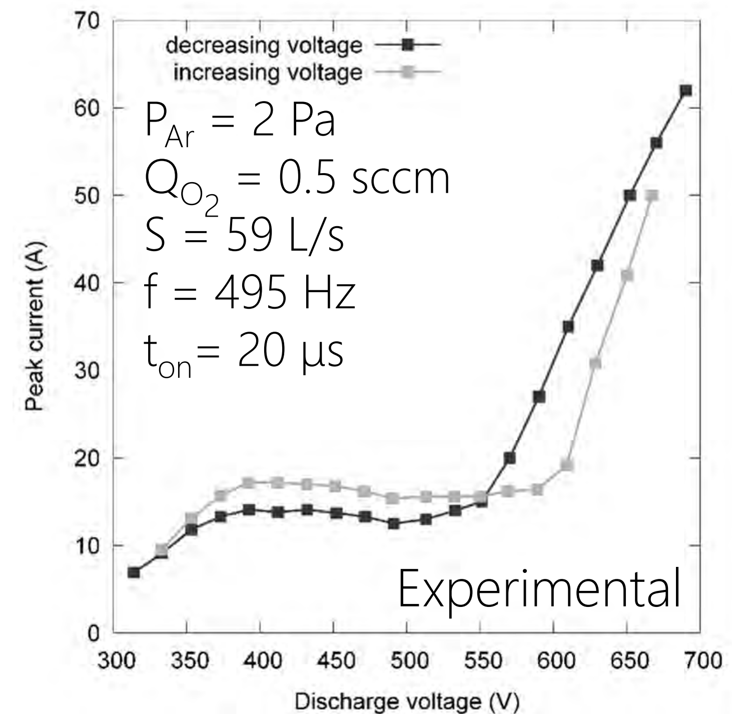
Nyberg, *Appl. Phys. Lett. 86*, 164106 (2005)

Double IV-hysteresis

R-DCMS



R-HiPIMS



➔ Independent of technique (HiPIMS ↔ DCMS)

➔ Double hysteresis is in RSD model but not yet satisfactory

☞ the responsable mechanism in RSD is identified as a second criticality

➔ Hysteresis behavior in R-HiPIMS?

K. Strijckmans, et al., *J. Appl. Phys. 121*, 080901 (2017)

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Conclusion

What we have learned…

- ✓ Direct **voltage control** for the reactive sputtering of Al_2O_3 enables **stable process control** in the transition mode of the hysteresis curve.
- ✓ Discharge **voltage** measurements are a monitor for the **target condition** during reactive sputtering via its relation with the **secondary electron emission yield**.

Ready for the future…

- ✓ The **RSD model** is extended to include simulation of the **IV-hysteresis** based on the Thornton relation.
- ✓ Simulated **IV-results** for **R-DCMS** are in line with experimental data.

The future…

- ✓ **More advanced model** to predict the experimental **double hysteresis** behaviour.
- ✓ **IV-modelling** of the **R-HiPIMS** hysteresis curve
- ✓ **IV-relation** for a target in **chemisorbed** state

Acknowledgements

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Roeland Schelfhout

Filip Moens

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