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

K. Strijckmans, R. Schelfhout, F. Moens, D. Depla

Dedicated Research on Advanced Films, and Targets
Outline

1. Introduction
2. IV-characteristic
3. Reactive IV-model
4. Results
5. Conclusion
Flow controlled DCMS hysteresis

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 $\gamma_\text{see}$
c) decreasing sputter yield ($Y_c << Y_m$)

Easy process control
No access to transition mode!
Bad film/deposition control
Pressure controlled DCMS hysteresis

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

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

😊 Access to transition
😊 Better film/deposition control
😊 Easy process control

Wisely choose your operation parameter!


Succes is material dependent

Stability of voltage control

- Increase in $V$
- $I$ increases along $IV$-line
- Target gets less poisoned
- Decrease in current $I$
- Target gets more poisoned
- Stops decrease in current $I$
- Stabilize to new $IV$ point

Oxidation fraction
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 strongly influenced by target state

→ (ion-induced) secondary electron emission yield $\gamma_{\text{see}}$

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

Outline

1. Introduction
2. IV-characteristic
3. Reactive IV-model
4. Results
5. Conclusion
IV-dependencies

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

  target thickness ↑ magnetic field $B$ ↓

Low $B$: pressure ↑ impedance ↓

High $B$: pressure ↑ impedance ↑


IV-dependencies

- Target condition
  - sputter yield $\rightarrow$ gas rarefaction (HiPIMS $\leftrightarrow$ DCMS)
  - elemental composition $\rightarrow$ secondary electron emission yield $\gamma_{\text{se}}$
    - metal $\rightarrow$ electron reflection probability $r(p)$
    - compound
    - chemisorbed

---


Thornton relation extended

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

- \( W_0 \): 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

Values?
- \( \gamma_{\text{see}} \)
  - \( \heartsuit \) metals: empirical formulas + experimental data
  - \( \heartsuit \) compounds: limited data
- \( r \)
  - \( \heartsuit \) what makes it fit? \( \frac{1}{2} \)?
  - \( \heartsuit \) impact low for 0 to 0.6

Is sheath energization alone?
- \( \heartsuit \) modelling results: **Ohmic heating** has a role (HiPIMS \( \leftrightarrow \) DCMS)
  - but
  - **no** (direct) target dependency

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

Target dependency of \( V_D \) in \( \gamma_{\text{see}} \) and \( r \)

Secondary electron emission yields $\gamma_{\text{see}}$

Dominant target dependency of $V_D$ is on $\gamma_{\text{see}}$

Use this correlation for the $\gamma_{\text{see}}$ of compounds

but slope is pressure/current dependent for given sputter system

IV-relations

For diode sputtering:

\[ I = \beta (V - V_0)^{3/2} \quad \Rightarrow \quad \beta \sim \gamma_{\text{see}} \]


For DC magnetrons:

\[ I = \beta V^n \]

\[ \Rightarrow \text{overestimation at low voltage} \]


For DC and RF magnetrons:

\[ I = \beta (V - V_0)^2 \]

\[ \Rightarrow \text{valid for broad condition range} \]


Thornton idea of a \( V_{\text{min}} \)

\[ \beta \text{ (or } n\text{) define sharpness of IV or impedance of plasma} \]

2 magnetron designs, 11 metals, 7 pressures, 7 \( B \) strengths

IV-relations

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

How clean is your target? $\text{(}P_{\text{base}} < 4 \times 10^{-4} \text{ Pa)}$

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


Reactive IV-characteristic

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

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

\[ I = \beta (V - V_0)^2 \]
\[ \beta \sim \gamma_{\text{see}} \]

\[ \gamma_{\text{see}}^{\text{Al,chem}} < \gamma_{\text{see}}^{\text{Al}} < \gamma_{\text{see}}^{\text{Al,oxide}} \]

\[ \gamma_{\text{see}}^{\text{Ti,oxide}} < \gamma_{\text{see}}^{\text{Ti,chem}} < \gamma_{\text{see}}^{\text{Ti}} \]
Outline

1. Introduction
2. IV-characteristic
3. Reactive IV-model
4. Results
5. Conclusion
### RSD model

= Berg model + advanced target model

model ‘engine’ = set of balance equations

<table>
<thead>
<tr>
<th>System part</th>
<th>Resolved variable</th>
<th>Model approach</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chamber</td>
<td>$P_r$, $Q_p$</td>
<td>one-cell</td>
</tr>
<tr>
<td>Target</td>
<td>$\theta_m$, $\theta_c$, $\theta_r$, $Q_t$</td>
<td>one-cell</td>
</tr>
<tr>
<td>• Surface</td>
<td></td>
<td>uniform current</td>
</tr>
<tr>
<td></td>
<td></td>
<td>multi-cell</td>
</tr>
<tr>
<td></td>
<td></td>
<td>non-uniform current</td>
</tr>
<tr>
<td></td>
<td></td>
<td>redeposition profile</td>
</tr>
<tr>
<td>• Subsurface</td>
<td>$\theta_{mb}$, $n_m(x)$, $n_r(x)$</td>
<td>continuum in depth</td>
</tr>
<tr>
<td>Substrate</td>
<td>$\theta_s$, $Q_s$</td>
<td>one-cell</td>
</tr>
<tr>
<td></td>
<td></td>
<td>multi-cell</td>
</tr>
<tr>
<td></td>
<td></td>
<td>deposition profile</td>
</tr>
</tbody>
</table>

Towards RSD model for IV

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

- assumption: only target surface condition influences IV by changing \( \gamma_{\text{see}} \)

\[ \gamma_{\text{see}} = \sum_{m,r,c} \theta_i \gamma_i \]

\[ \gamma_{\text{see}} = a \sum_{m,r,c} \frac{1}{V_i} \theta_i + b \sum_{m,r,c} \theta_i \]

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

\[ I = \beta_i (V_i - V_{0,i})^{n_i} \]

Generalized Thornton relation

\[ \gamma_{\text{see}} = \frac{a}{V} + b \]

```
Introduction IV-characteristic Reactive IV-model Results Conclusion
```

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


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

Also observed in R-HiPIMS!

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: \( J_R = -D_{\text{dam}}(x) \frac{\partial n_R(x)}{\partial x} \)
    where \( D_{\text{dam}}(x) \) scales with damage profile
  - thermic diffusion: \( J_R = -D_{\text{therm}} \frac{\partial n_R(x)}{\partial x} \)

- Specification of IV-relations for metal, compound & chemisorbed (?) target
  \( I = \beta_i (V - V_{0,i})^{n_i} \)

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

1. Introduction
2. IV-characteristic
3. Reactive IV-model
4. Results
5. Conclusion
Reference system

Sputter conditions:
- Target Al
- Process gas Ar
- Reactive gas O₂
- Target diameter = 2 inch (20 cm²)
- Pumping speed S = 30 L/s
- Argon pressure P_{Ar} = 0.4 Pa
- Oxygen flow Q_{O₂} = 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

\[ Q_{O_2} = 0.8 \text{ sccm} \]

\[ \rightarrow \text{increasing pumping speed removes hysteresis by decreasing current of 2}^{\text{nd}} \text{ critical point} \]
→ 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:

- **not** same as current density by current

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

---

Double IV-hysteresis

R-DCMS

\[ P_{Ar} = 1.6 \text{ Pa} \]
\[ Q_{O_2} = 1.2 \text{ sccm} \]
\[ S = 32 \text{ L/s} \]

R-HiPIMS

\[ P_{Ar} = 2 \text{ Pa} \]
\[ Q_{O_2} = 0.5 \text{ sccm} \]
\[ S = 59 \text{ L/s} \]
\[ f = 495 \text{ Hz} \]
\[ t_{on} = 20 \mu s \]

➔ Independent of technique (HiPIMS ↔ DCMS)
➔ Double hysteresis is in RSD model but not yet satisfactory
   ➔ the responsible mechanism in RSD is identified as a second criticality
➔ Hysteresis behavior in R-HiPIMS?

Outline

1 Introduction
2 IV-characteristic
3 Reactive IV-model
4 Results
5 Conclusion
Conclusion

What we have learned...

✓ Direct **voltage control** for the reactive sputtering of Al₂O₃ 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
Contributing colleagues:

Roeland Schelfhout
Diederik Depla
Filip Moens

Find this presentation on www.DRAFT.ugent.be soon...