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Did you know, your FIB can do this?

Winkler, R. et al. ASC Appl. Mater. Interfaces (2017) 9, 8233
Hardware Requirements

1) **Focused Electron Beam**

2) **Gas Injection System (GIS)**

3) **Beam Control**  
   (scanning coils, patterning)
Focused Electron Beam Induced Deposition (FEBID)

**Basic principle**

1. **Continuous precursor flow**
2. **Short e-beam pulse**
3. **Small beam displacement**

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**3D-Nanoprinting**

- **Allows for 3D printing**
- **Enables 3D fabrication**
- **Flexibility in pulse duration and displacement control angles**

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**Focused electron beam injection**

**Pre-Cursor Gas**

**Gas Injection Nozzle**

**Substrate**

**Deposition**

**Dissociation**

**Physisorption**

**Volatile products**

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**Δx**

100 nm
Unique selling points

1) **Small feature size**
   
   Typical around 60 nm  
   Down to Sub-20 nm

2) **Shape flexibility**

3) **Many materials**

   → Different functionalities of deposit are possible!

   e.g. insulating, semi-conducting, conducting, magnetic, resonant optics, ...

Unique Selling Points

4) **Direct-write** in one process step
   - No mask needed
   - No resist needed

5) **Substrate independent**
   - On almost any substrate **material**
   - On almost any **morphology**

6) Very low temperature rise
7) No unwanted sputtering
8) No unwanted material implantation

Winkler, R. et al. ACS AMI (2017) 9, 8233
**Beam Parameter: Beam Current**

1. **Patterning velocity** defines the segment angle
2. Use **LOWEST** beam currents

Winkler, R. et al. in submission
Gas/Beam Parameter: Working Regime

2 extreme conditions

- **electron limited**
  - excess of precursor molecules (electrons are limiting the growth)
  - Reduced proximal co-deposition
  - Better growth control
  - No influence of gas flux direction

- **precursor limited**
  - too less precursor molecules (excess of electrons)
  - Proximal deposition due to excess electrons
  - Strong influence of patterning direction in relation to the gas flux

**Go towards electron limited conditions**

LOW beam currents, high primary energies
HIGH precursor supply, refresh times, 3D-interlacing

HIGH beam currents, low primary energies
long stationary beams

Fowlkes et al., ACS Nano (2016)  Winkler, R. et al. in submission
Gas Parameter: Enhance Precursor Supply

How to increase precursor coverage

Nozzle geometry

GIS alignment

Optimize:

1) **Height**
   Bring nozzle closer to the substrate (< 100 µm)

2) **X position**
   Align nozzle main axis along Y axis (X = 0 µm)

3) **Y position**
   Bring E-beam center close to nozzle edge (Y ~60 µm)

4) **Nozzle angle**
   If possible, use high angle port

1) **Patterning velocity** defines the segment angle
2) Use **LOWEST** beam **currents**
3) Use **HIGHEST** primary **energy**
Patterning Parameter: Pattern Velocity $v$

\[ \text{Pattern velocity} (v) = \frac{\text{Point Pitch (PoP)}}{\text{Dwell time (DT)}} \]

1) Record calibration curve
2) Good start values: PoP = 1 nm, DT = 12 ms; equal to 83 nm/s
Beam Parameter: Focus and Astigmatism

An empirical approach to set focus and correct astigmatism for 3D-nanoprinting:

**Step 1:** Deposit a *Test-pillar*

**Step 2:** Improve *Focus* and *Astigmatism*

... Repeat Step 1 and 2 until you are satisfied with shape & diameter...

**Step 3:** Deposit a *Reference-array* of critical segments

**Step 4:** Adjust, until *desired segment* is just growing

Optimize beam quality until critical angle of your reference array is stable
Patterning Sequence for Complex 3D Structures

Problems:

- **Structure bending:**
  First fabricated elements are affected by forward scattered electrons arising from later deposited elements, which leads to an unwanted bending and thickening.

- **Drift issues:**
  Sample/charging drift often prevent that branches finally grow together.

Solution:

Alternating patterning sequence (**3D-interlacing**)

→ No inhomogeneous structure bending
→ Higher growth rates due to intrinsic, increased refresh times
→ Minimized effects of drift

Use alternating point sequence (**3D-interlacing**)
Simple 3D Structures – File Preparation

Defining patterning sequence via file (FEI - stream file)

Concrete Example: Gothic arcs

<table>
<thead>
<tr>
<th>Header</th>
<th>Concrete Example: Gothic arcs</th>
</tr>
</thead>
<tbody>
<tr>
<td>s16</td>
<td>1</td>
</tr>
<tr>
<td>#patterning points</td>
<td></td>
</tr>
<tr>
<td>Dwell-Time</td>
<td>X-Coordinate</td>
</tr>
<tr>
<td>Dwell-Time</td>
<td>X-Coordinate</td>
</tr>
<tr>
<td>Dwell-Time</td>
<td>X-Coordinate</td>
</tr>
</tbody>
</table>

Synchronization point (optional)

Concrete Example:

<table>
<thead>
<tr>
<th>s16</th>
<th>1</th>
</tr>
</thead>
<tbody>
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<td>250000  1000  0</td>
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<tr>
<td>140000  999  0</td>
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</tr>
<tr>
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<tr>
<td>...</td>
<td></td>
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<tr>
<td>140000  499  0</td>
<td></td>
</tr>
<tr>
<td>140000  501  0</td>
<td></td>
</tr>
<tr>
<td>140000  500  0</td>
<td></td>
</tr>
</tbody>
</table>

(1 0 1000)
Complex 3D Structures – File Preparation

3D-FEBID generator software (available soon)

Winkler, R. et al. ASC Appl. Mater. Interfaces (2017) 9, 8233
Fowlkes et al., in preparation
Preparation Guide

1) GIS Optimization [1]

- a) steeper angle
- b) closer
- c) on-axis

Hints, tips and tricks

Good values to start with:
- Angle: 52°
- Distance GIS nozzle to substrate < 100 µm
- E-beam center at X = 0 µm, Y ≈ 60 µm

2) Stream File Preparation

- a) Harmonize structure dimensions with Horizontal Field Width (→ Magnification)
- b) Translate real dimensions to pixel
- c) Calculate patterning points according to your point pitch
- d) Adjust dwell time for each point according to the desired angle (calibration upfront!)
- e) Arrange patterning points in an alternating point sequence (3D interlacing)
- f) Alternatively to a) - e), use 3D-generator software (coming soon) [2], or contact us:
  Robert.winkler@felmi-zfe.at
  Harald.Plank@felmi-zfe.at

Hints, tips and tricks

- To enable desired PoP size and to avoid potential rounding errors use high magnifications
- What is the resolution of your patterning engine?
  12bit: 4096 points
  16bit: 65536 points
  \( \rightarrow \) Horizontal Field Width/Resolution = 1 Pixel
- 3D-interlacing is highly recommended for stable architectures

3) Setup microscope

- a) Heat precursor at least 30 minutes before deposition
- b) Bring sample surface into ideal position relative to the optimized GIS
- c) Set highest primary energy \(^{[1,2]}\) and
- d) Set lowest beam current \(^{[1,2]}\)

4) Beam focus

- a) Rough beam alignment with inserted GIS
- b) Switch to high magnification (~ 500 nm HFW)
- c) Deposit testspot
- d) Adjust focus and correct astigmatism
- e) Repeat step c) and d) until diameter is satisfying
- f) Deposit test-array and monitor sample current concerning stability and repeat c-f if needed

5) Deposition

- a) Select deposition area in close proximity to focused area
- b) Select necessary magnification
- c) Load and arrange file(s)
- d) Think about fabrication order (shadowing/co-deposition)
- e) Wait for mechanical drift stabilization
- f) Open GIS valve and wait until pressure is in equilibrium
- g) Start deposition
- h) Wait with imaging until vacuum level is low again to avoid contamination

Hints, tipps and tricks

- @b) Eucentric height or defined GIS-surface distance
- @c) at FELMI: 30 keV
- @d) at FELMI: 21 pA
- @a) otherwise no visible pillar might grow
- @b) so that you can measure the deposited testspot afterwards with high accuracy
- @e) at FELMI: diameter about 52 nm for MeCpPtMe\(_3\) precursor and 30 keV/21 pA
- @f) at FELMI: A horizontal segment should grow at 4 ms and 1 nm PoP (= 250 nm/s)

- @a) this ensures almost identical focal quality
- @b and c) so that the designed file has the correct PoP
- @d) for multiple files always pattern towards GIS to avoid gas flux shadowing.
- @e) depends on stage stability. At FELMI ~ 15 minutes
- @f) at FELMI at least 3 minutes

- Be careful with beam shift option!
- For very high structures: precursor supply gets complicated \(\rightarrow\) different growth rates

[1] Winkler et al., in submission
Summary

**Focused Electron Beam induced deposition (FEBID)** is capable for **3D-nanoprinting** of freestanding, complex geometries.

The main **advantages** are
- Feature sizes below 20 nm
- Manifold materials and functionalities
- Almost substrate independent (morphology, material)
- Direct-write of complex structures

Reliable fabrication is challenging due to the **high number of process parameters** involved. Evaluating the **most important factors** revealed high-fidelity 3D-printing at:

- Low beam current
- High primary energy
- High precursor coverage
- Excellent beam focus
- 3D-interlacing patterning strategy
- A lot of patience!

**Good values to start with**
- MeCpPtMe$_3$
- $U = 30$ keV
- $I = 21$ pA
- Testspot size @5s: 52 nm
- PoP = 1 nm or lower
- DT (@PoP 1 nm) = 12 ms for ~ 45° segment angle

For further question contact us: Robert.winkler@felmi-zfe.at Harald.Plank@felmi-zfe.at
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Thank you for your attention!

Science Meets Art:
3D-Nano-model of the glass pyramids of the Louvre (Paris) in a scale of 1:8,000,000