FELMI-ZFE
Work Group S³

Institute of Electron Microscopy and Nanoanalysis
Graz Centre for Electron Microscopy

Christian Doppler Forschungsgesellschaft

3D-Nano-Printing via Focused Electron Beams: From a Concept Towards Stable Nano-Fabrication.

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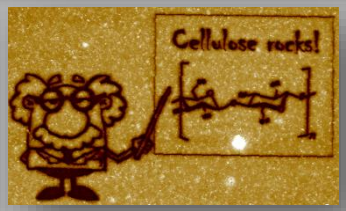
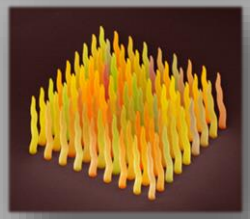
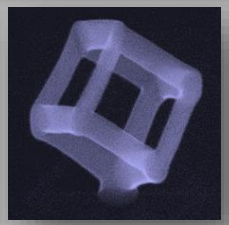
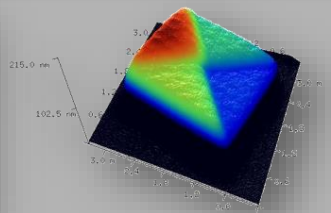
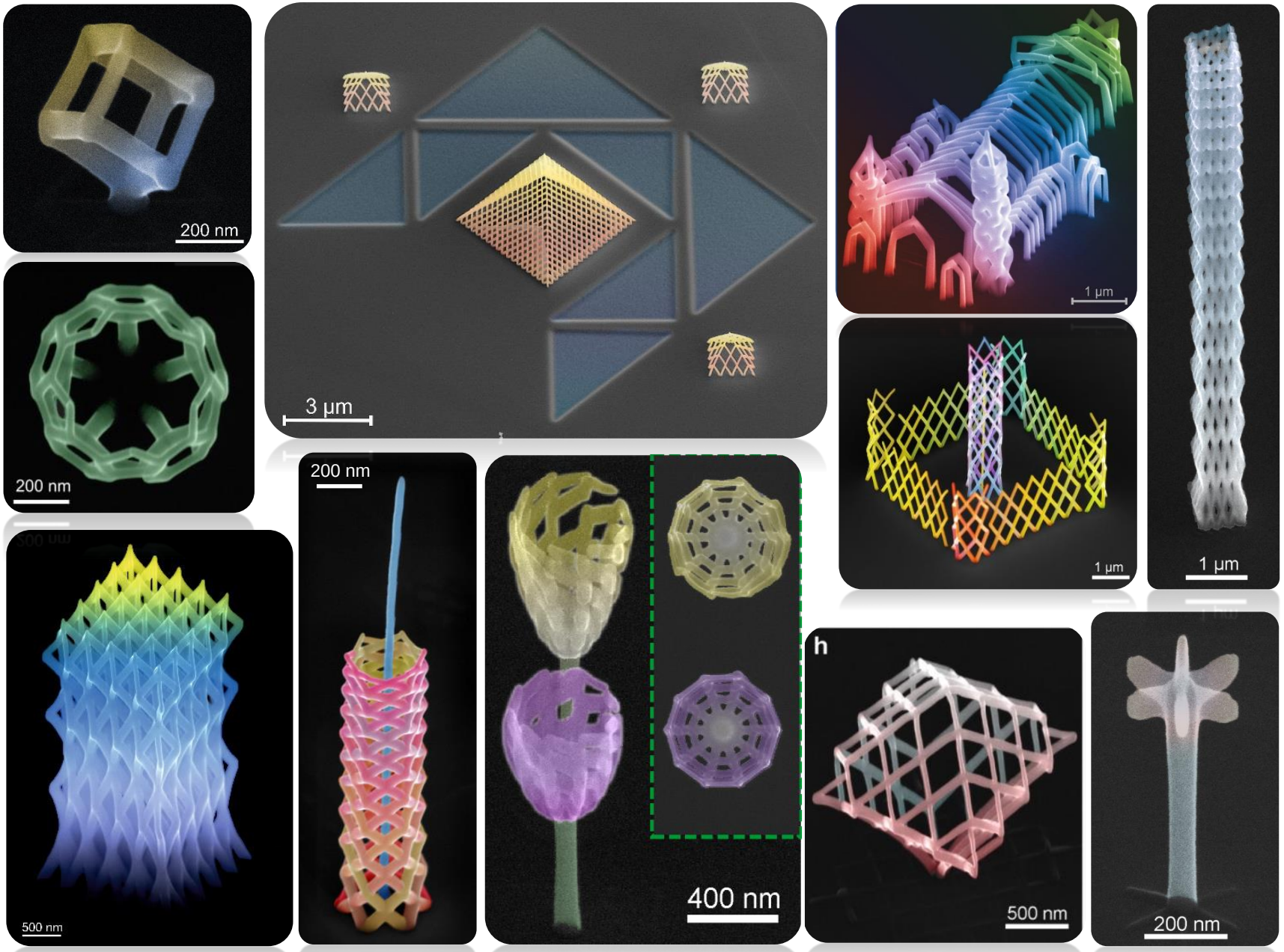




Did you know, your FIB can do this?



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

Hardware Requirements

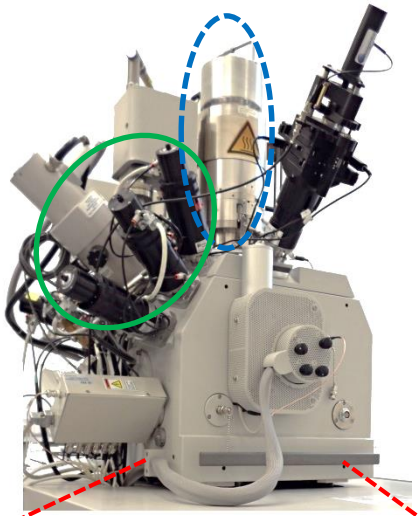
1) Focused Electron Beam

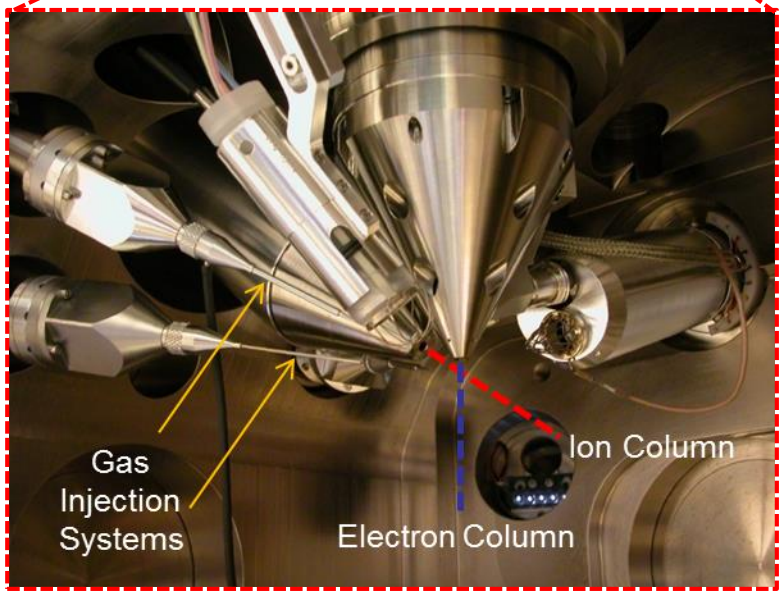
2) Gas Injection System (GIS)

3) Beam Control

(scanning coils, patterning)

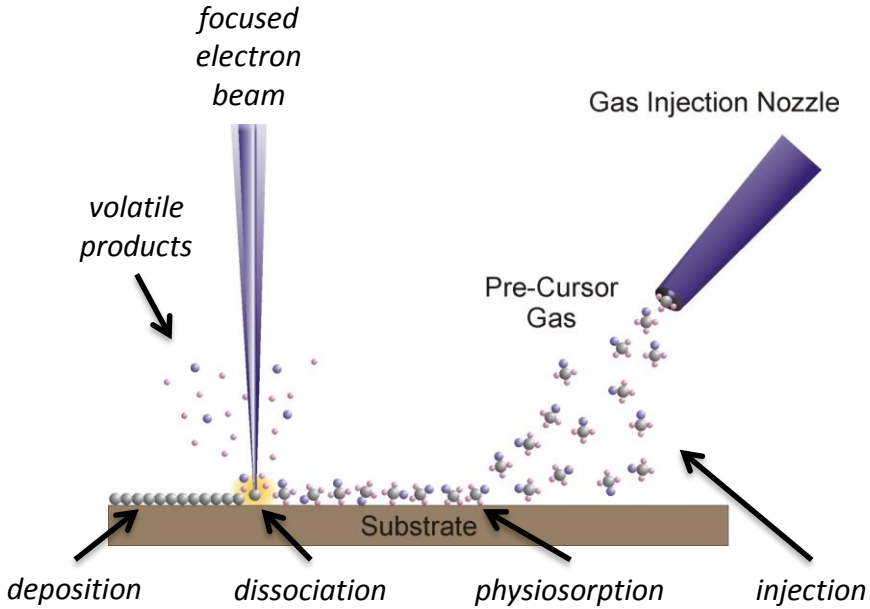
Electron / Ion Beam Microscope



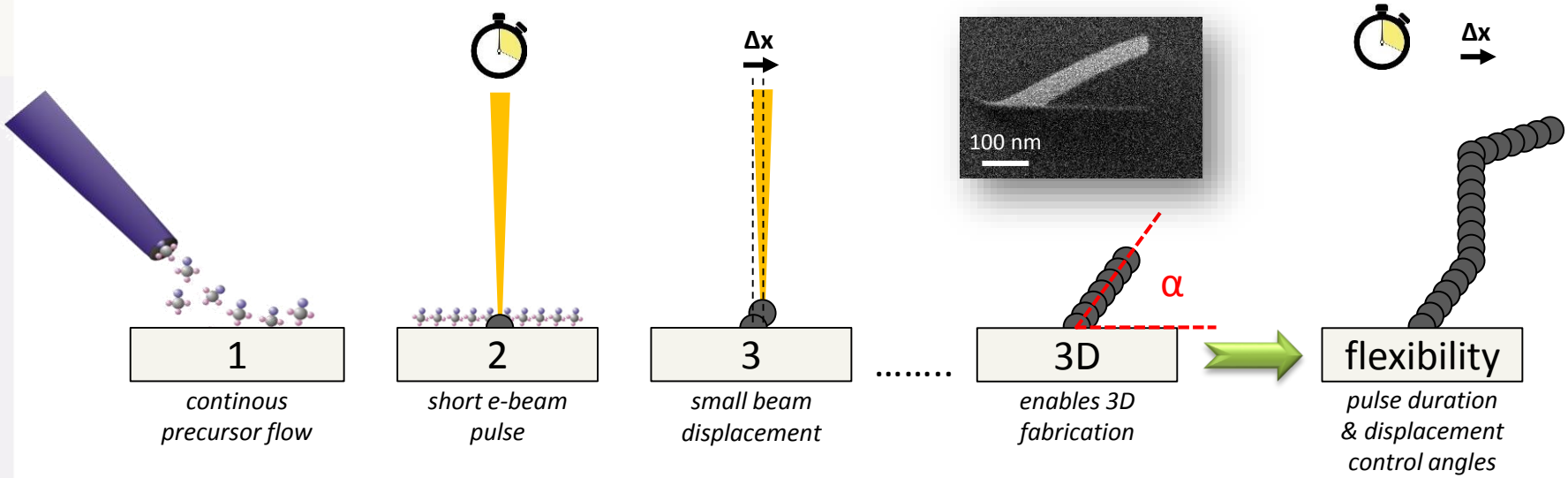


Focused Electron Beam Induced Deposition (FEBID)

Basic principle



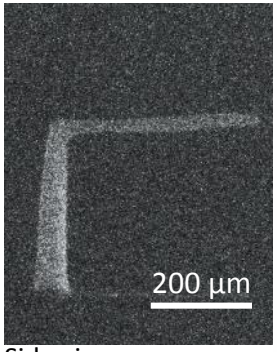
3D-Nanoprinting



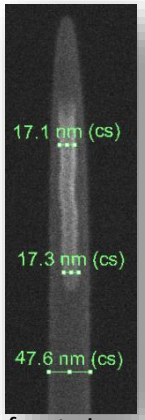
Unique selling points

1) Small feature size

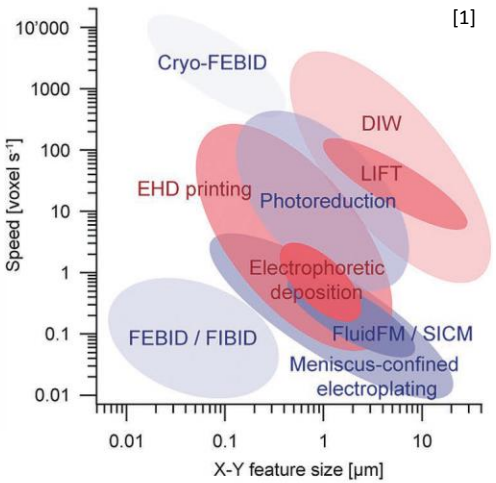
Typical around 60 nm
Down to Sub-20 nm



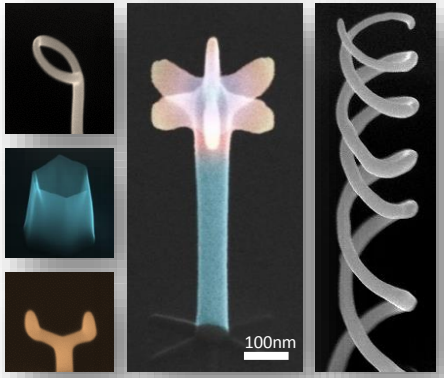
Side view

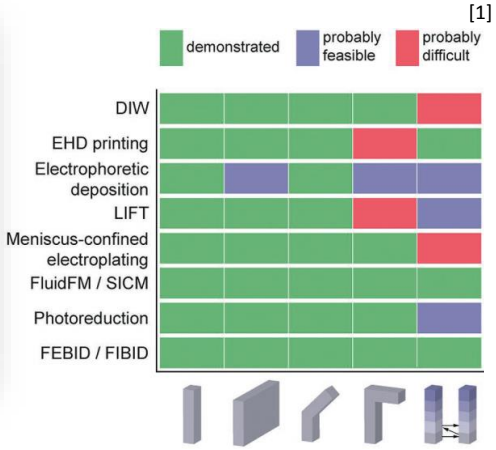


front view



2) Shape flexibility

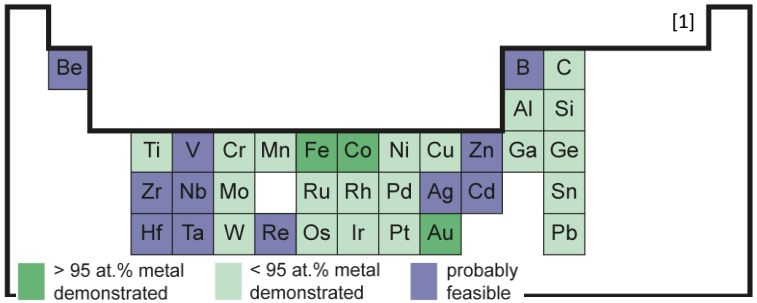




3) Many materials

→ Different functionalities of deposit are possible!

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



[1] Hirt, L. *et al.* Adv. Mater. (2017), 1604211

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**

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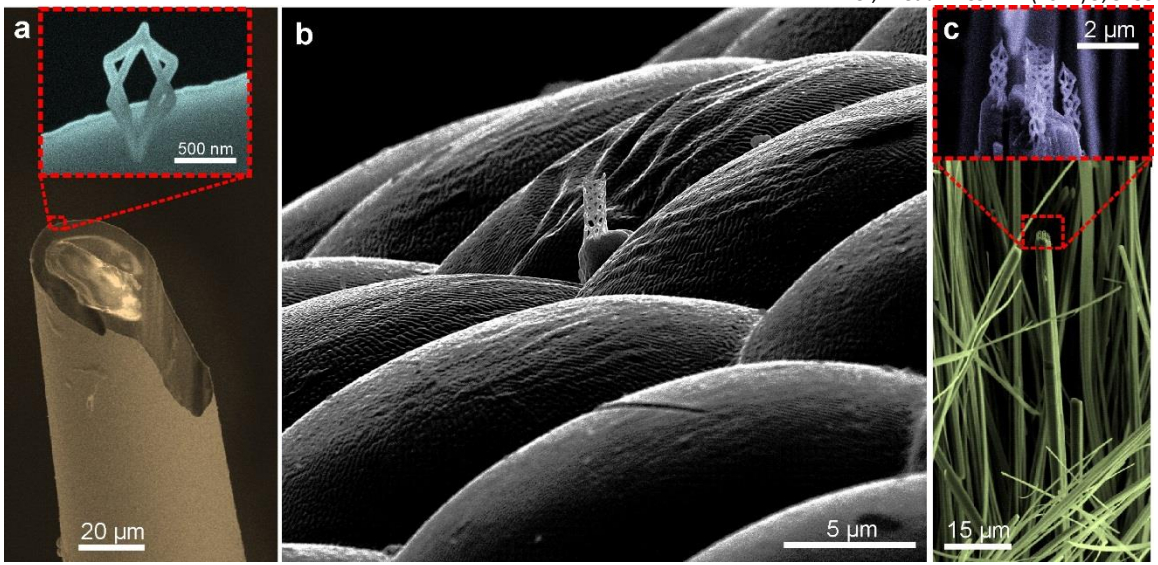


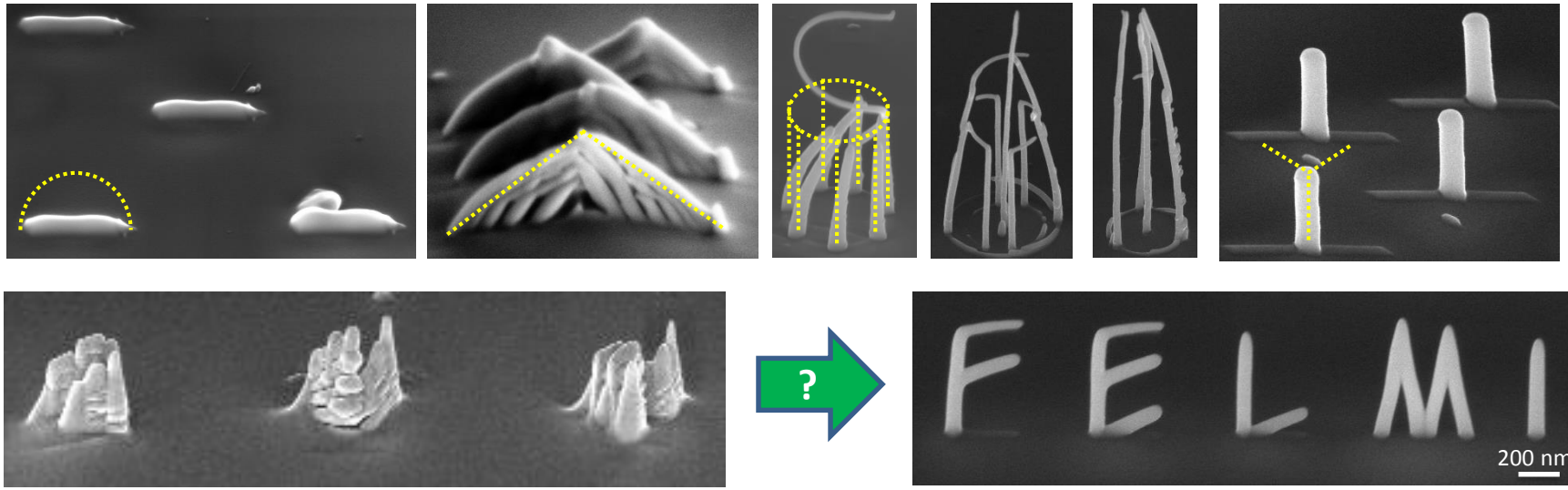
6) Very low temperature rise

7) No unwanted sputtering

8) No unwanted material implantation

Important Parameters

outtakes

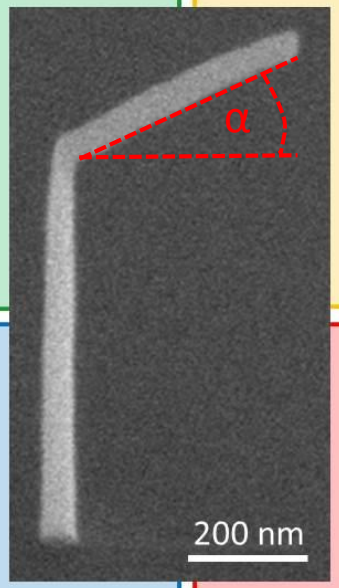


Beam parameter

Primary energy (U)
Beam current (I)
Beam focus and astigmatism
Immersion Mode
Beam shift

Patterning parameter

Point Pitch (PoP) } patterning velocity (v)
Beam current (DT) } patterning velocity (v)
Pattern direction
Patterning sequence
Engine accuracy



Gas parameter

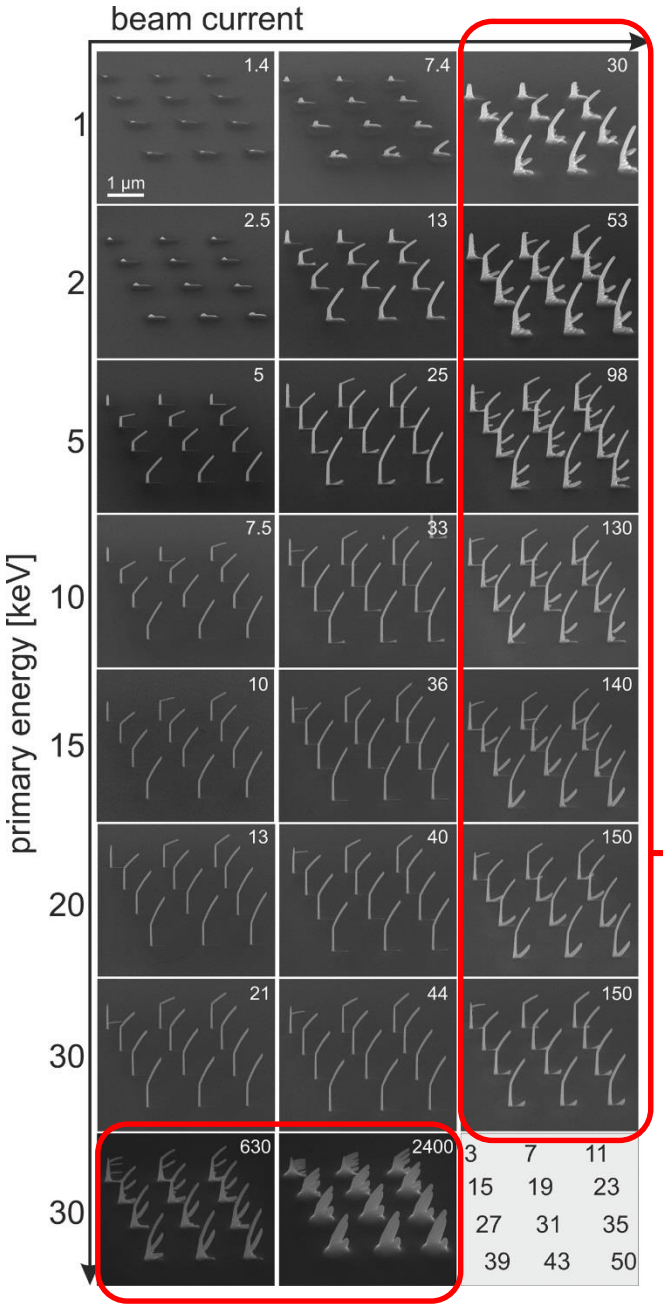
Precursor type
GIS alignment
Precursor temperature
Refresh time (RT)

Other influences

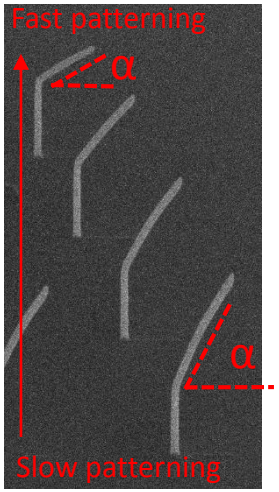
Drift
Substrate

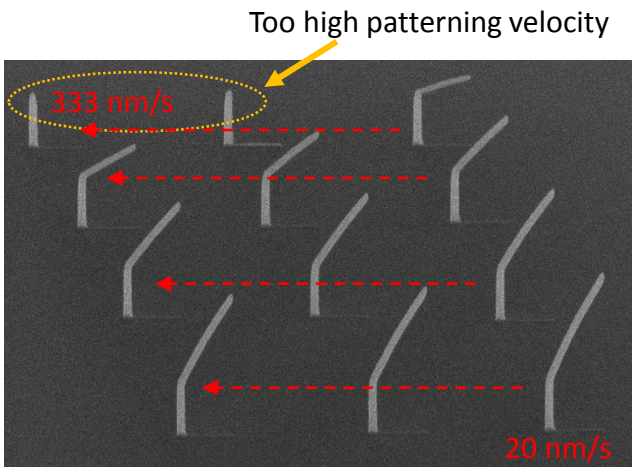
Winkler, R. *et al.* in submission

Beam Parameter: Beam Current

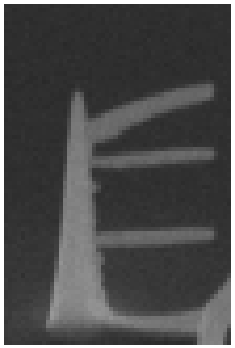


Winkler, R. *et al.* in submission





Co-Branching



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

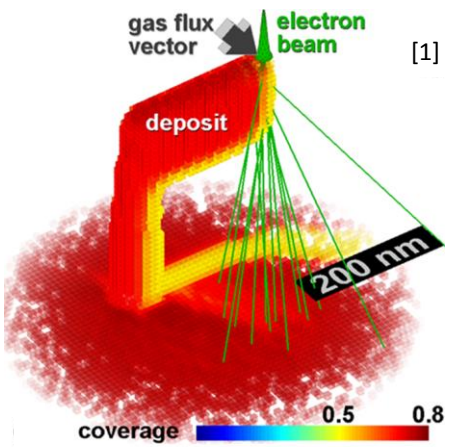
Gas/Beam Parameter: Working Regime

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

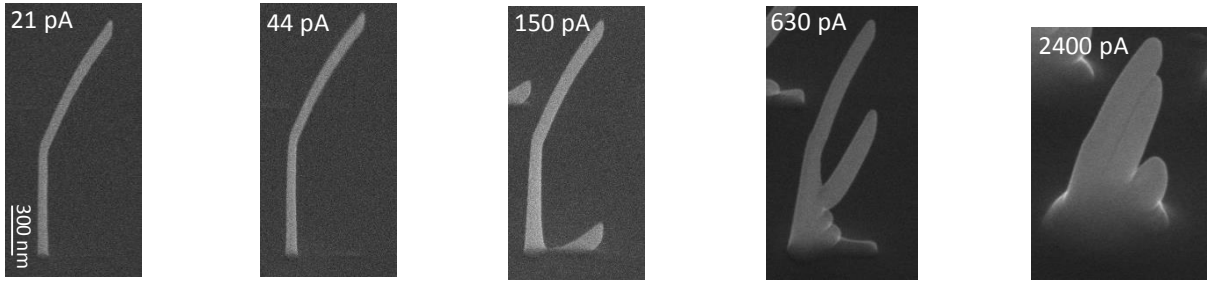


[1]

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

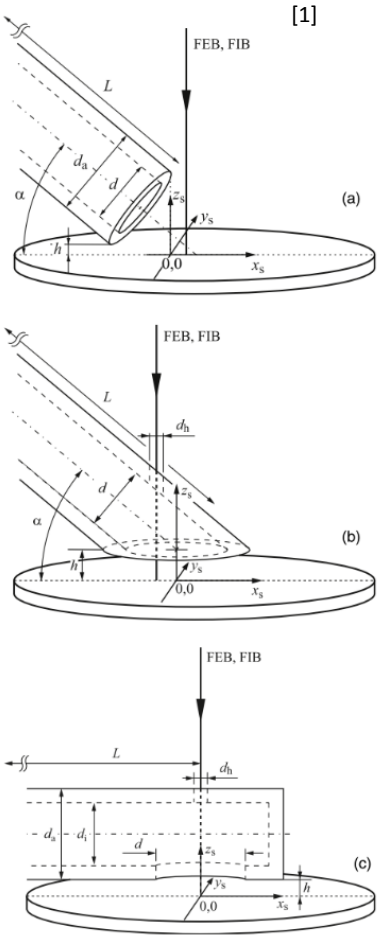
Fowlkes *et al.*, ACS Nano (2016) Winkler, R. *et al.* in submission

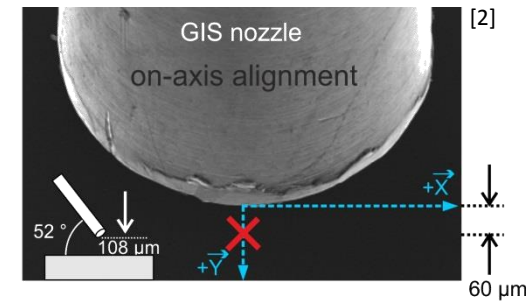
Gas Parameter: Enhance Precursor Supply

How to increase precursor coverage

Nozzle geometry

[1]





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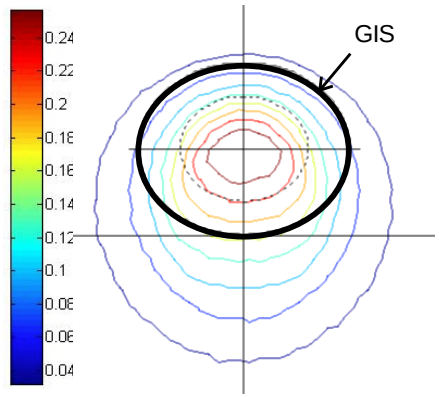
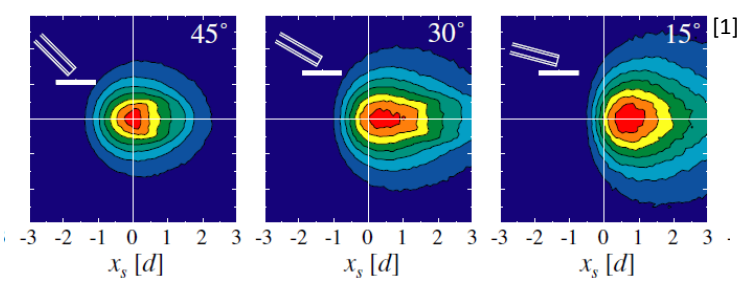




[1]

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)

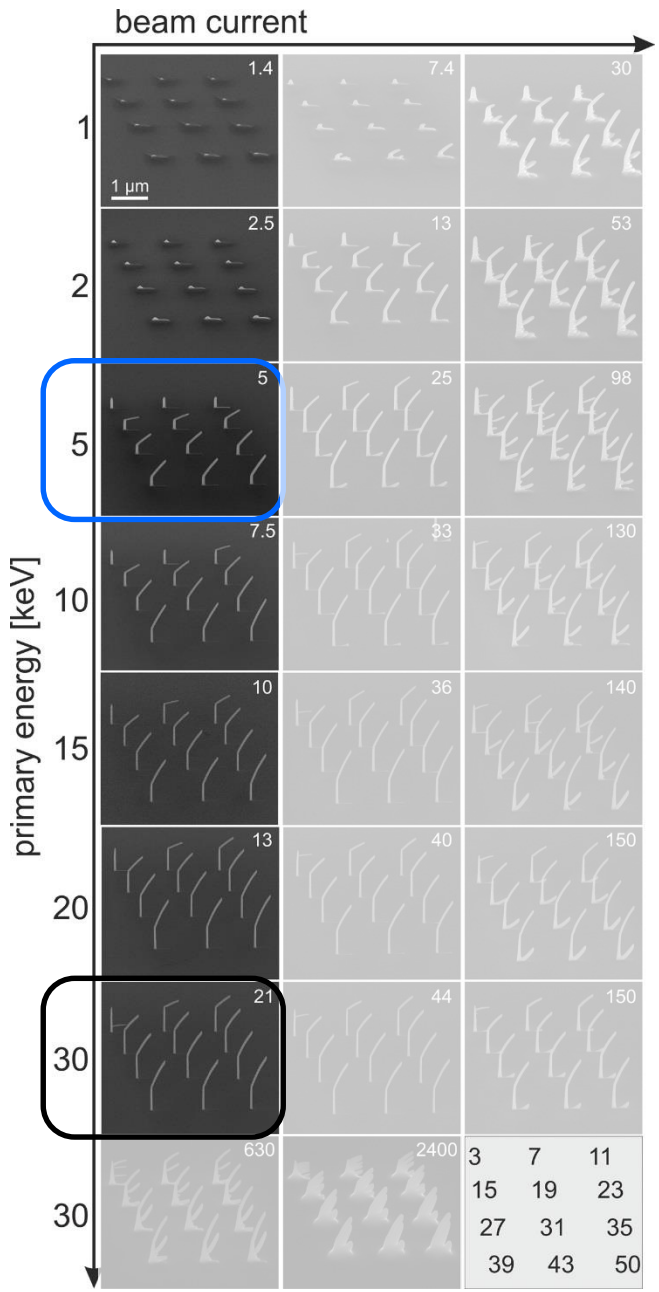
3) **Y position**
Bring E-beam center close to nozzle edge (Y ~60 µm)

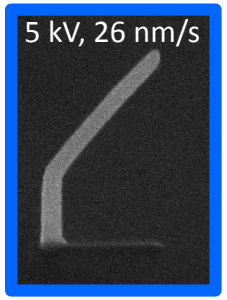
4) **Nozzle angle**
If possible, use high angle port

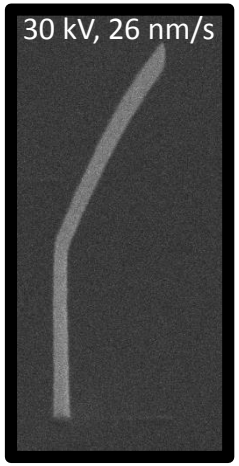
Optimize your Gas Injection System

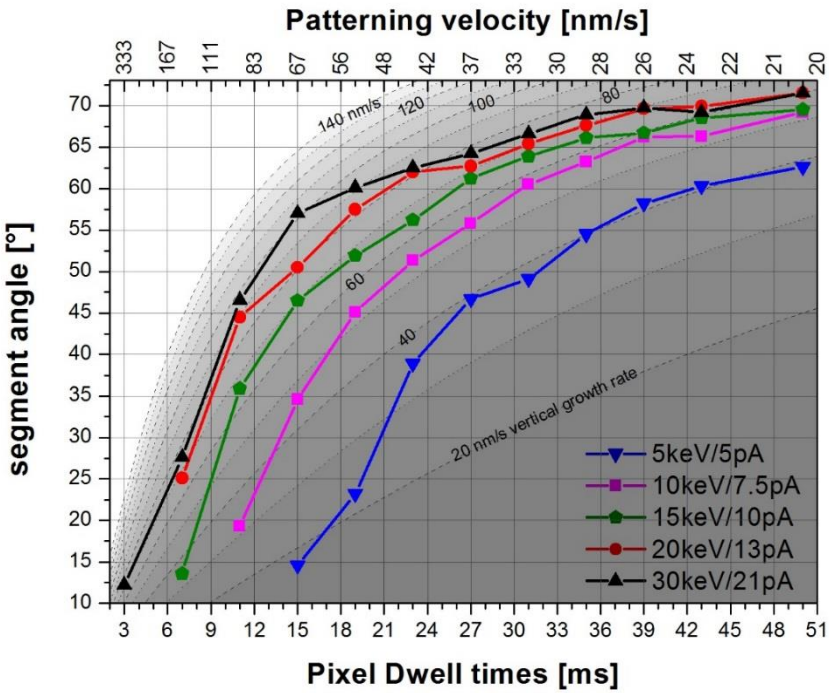
[1] V. Friedli and I. Utke, J. Phys. D: Appl. Phys. 42, 125305 (2009) [2] Winkler et al., ASC Appl. Mater. Interfaces, 6, 2987-2995 (2014)

Beam Parameter: Beam Current and Primary Energy







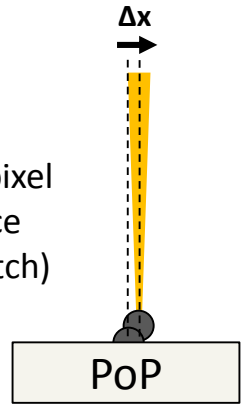


1) **Patterning velocity** defines the segment **angle**
2) Use **LOWEST** beam **currents**
3) Use **HIGHEST primary energy**

Winkler, R. *et al.* in submission

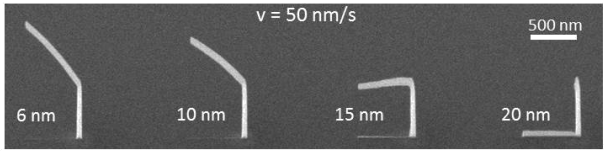
Patterning Parameter: Pattern Velocity v

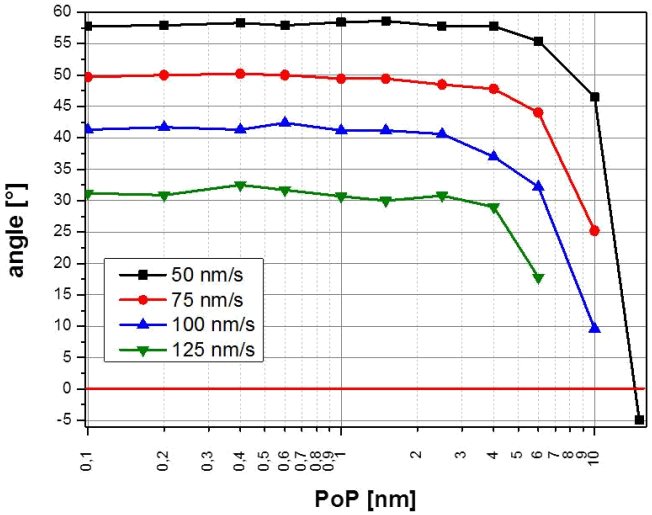
Pixel to pixel distance (Point Pitch)

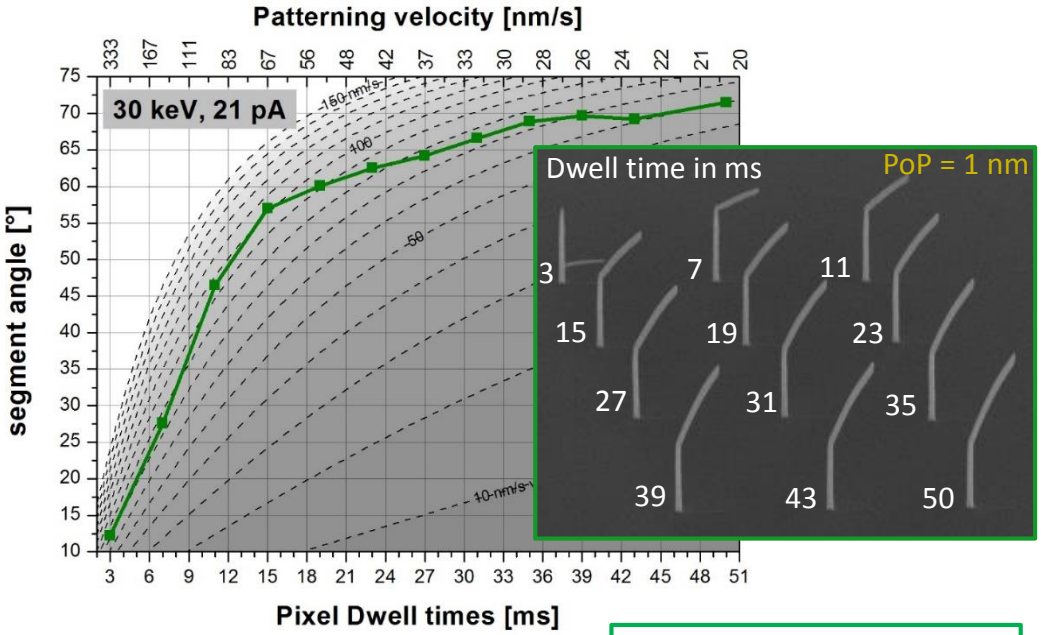


$$Pattern\ velocity\ (v) = \frac{Point\ Pitch\ (PoP)}{Dwell\ time\ (DT)}$$

Stationary beam exposure time (Dwell Time)







Record calibration curve
→ Angle α = f(PoP, DT)

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

Winkler, R. *et al.* in submission

Beam Parameter: Focus and Astigmatism

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

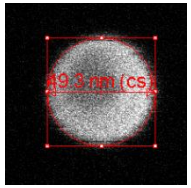
Step 1: Deposit a *Test-pillar*

Step 2: Improve *Focus* and *Astigmatism*

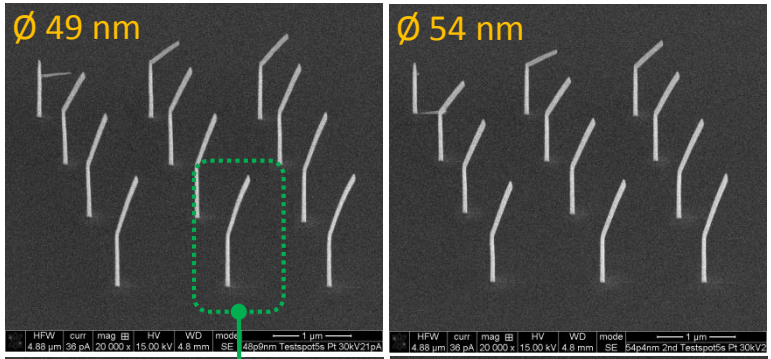
… Repeat Step 1 and 2 until you are satisfied with shape & diameter…

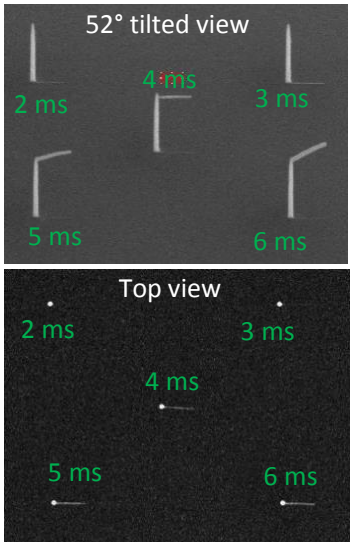
Step 3: Deposit a *Reference-array* of critical segments

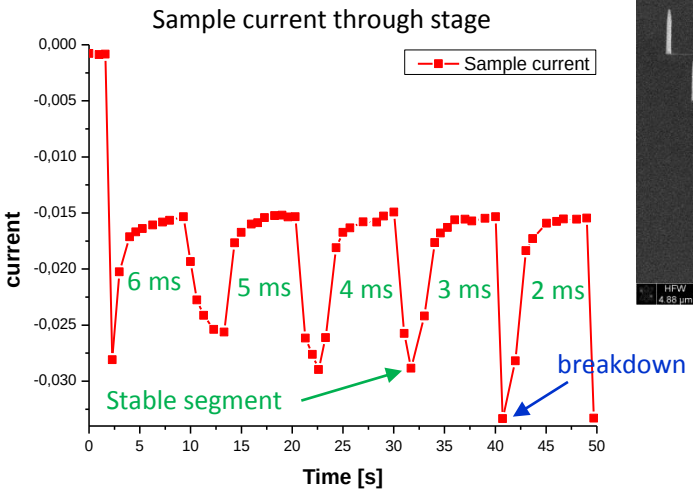
Test pillar top view

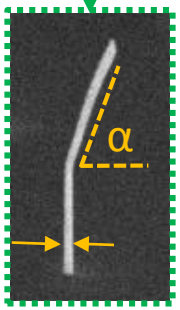


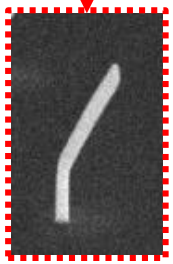
Variation of test-pillar diameter











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

parameter @FELMI:

U = 30 keV
I = 21 pA
5 seconds stationary beam
→test pillar diameter (Pt) ~ 52 nm

Optimize beam quality until critical angle of your reference array is stable

Winkler, R. *et al.* in submission

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:

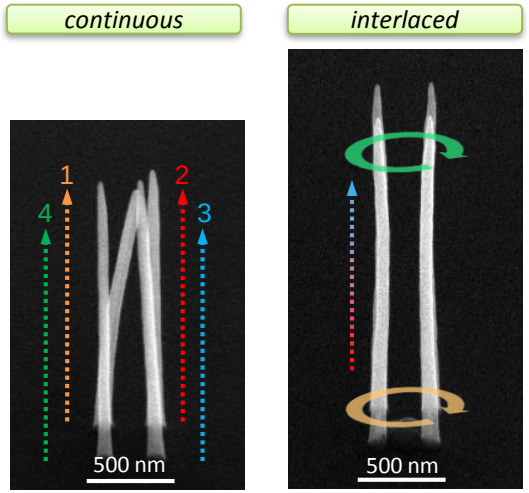
Alternating patterning sequence (**3D-interlacing**)

→ No inhomogeneous structure bending

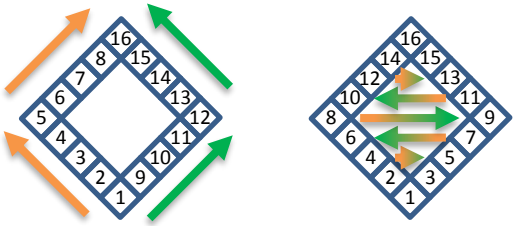
→ Higher growth rates due to intrinsic, increased refresh times

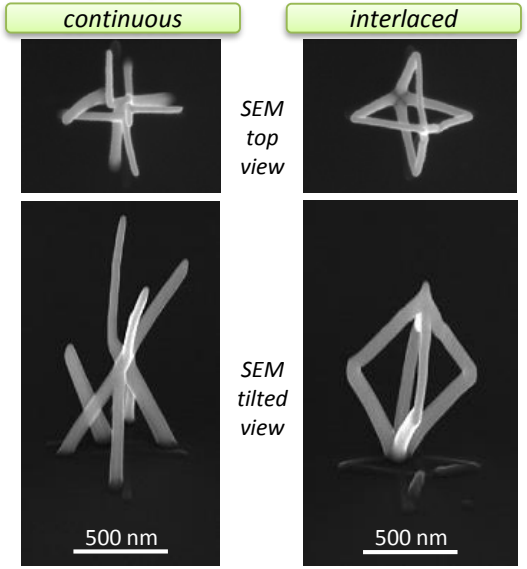
→ Minimized effects of drift

Use alternating point sequence (3D-interlacing)



patterning sequence of two branches in side view





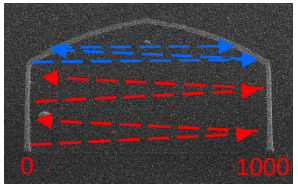
Winkler, R. *et al.* ASC Appl. Mater. Interfaces (2017) 9, 8233

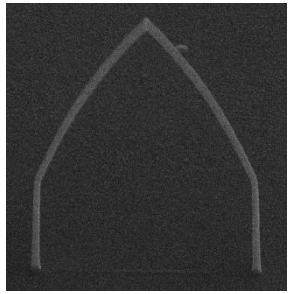
Simple 3D Structures – File Preparation

Defining patterning sequence via file (FEI - stream file)

```
s16
1                                  Header
#patterning points
Dwell-Time   X-Coordinate   Y-Coordinate
Dwell-Time   X-Coordinate   Y-Coordinate
Dwell-Time   X-Coordinate   Y-Coordinate
...          ...            ...
```

Concrete Example: Gothic arcs

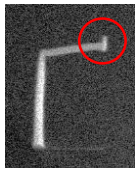


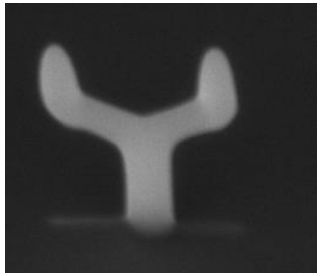


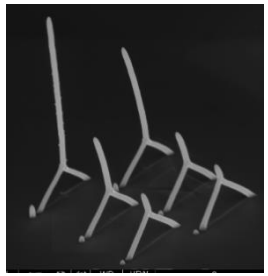
```
s16
1
8307
250000  0     0
250000  1000  0
250000  0     0
250000  1000  0
...
140000  1     0
140000  999   0
140000  2     0
140000  998   0
...
140000 499    0
140000 501    0
140000 500    0
(1 0 1000)
```

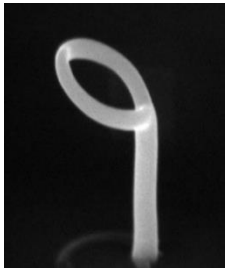
Synchronization point (optional)

```
s16
1
8307
250000  0     0
250000  1000  0
250000  0     0
250000  1000  0
...
240000  1     0
240000  999   0
240000  2     0
240000  998   0
...
240000 499    0
240000 501    0
240000 500    0
```



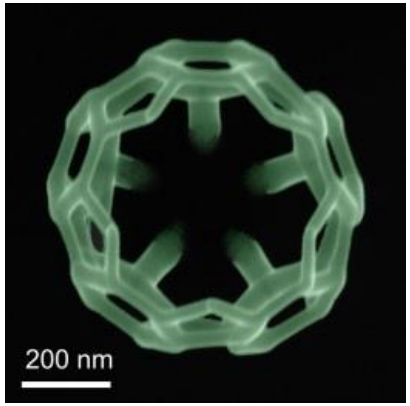






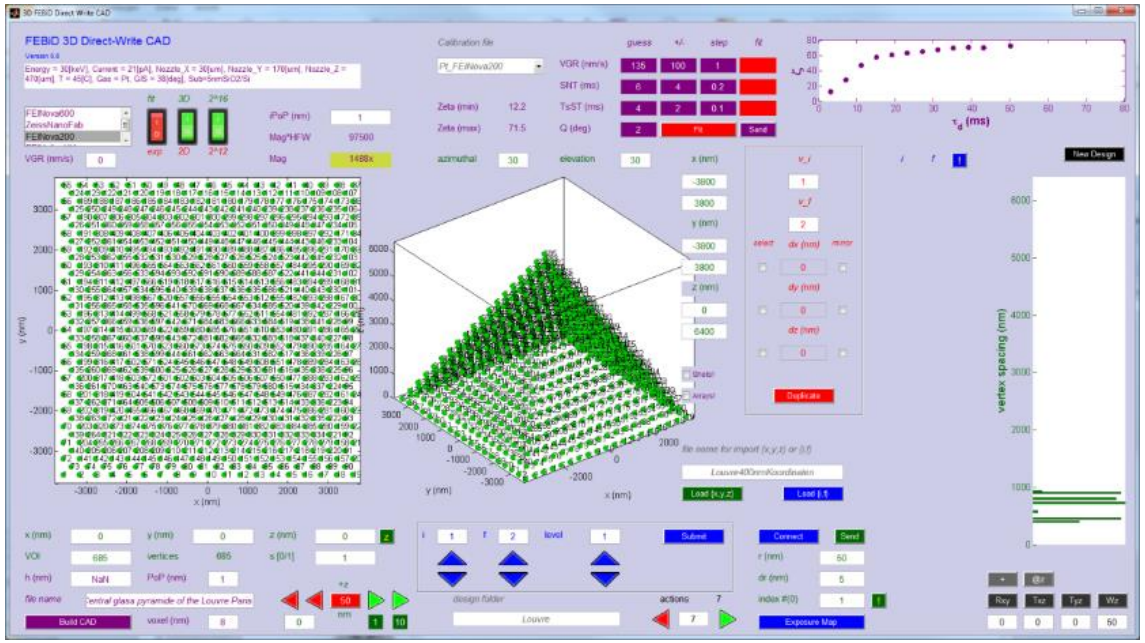
Complex 3D Structures – File Preparation

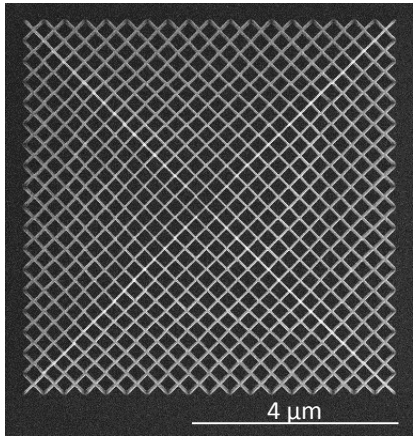
?

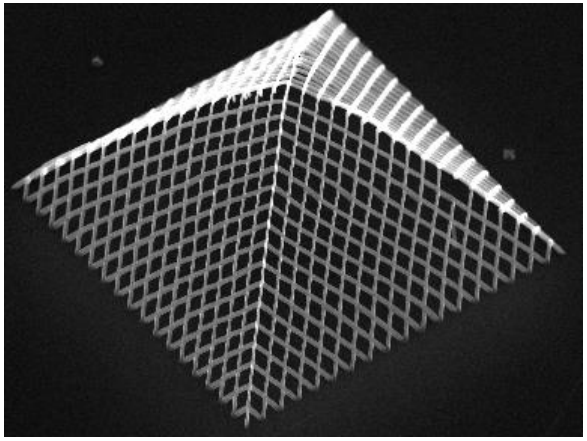


use

3D-FEBID generator software (availabe soon)





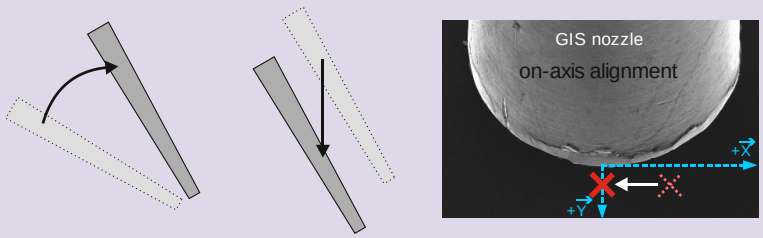


3D generator coming soon

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, tipps 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 (callibration 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

- To enable desired PoP size and to avoid potential rounding errors use high maginifications
- What is the resolution of your patterning engine?
  12bit: 4096 points
  16bit: 65536 points

→ Horizontal Field Width/Resolution = 1 Pixel

- 3D-interlacing is *highly recommended* for stable architectures

[1] Winkler et al., ASC Appl. Mater. Interfaces (2014), 6, 2987-2995
[2] Fowlkes et al., in preparation

FELMI-ZFE

Fabrication Guide

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]

Hints, tipps and tricks

@b) Eucentric height or defined GIS-surface distance

@c) at FELMI: 30 keV
@d) at FELMI: 21 pA

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

@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)

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

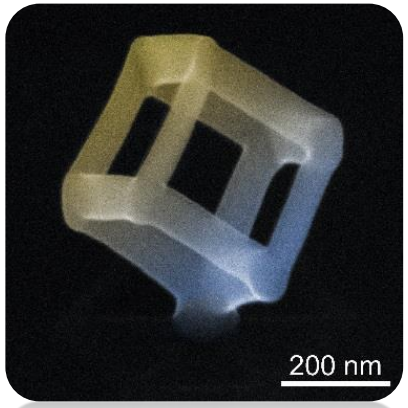
@a) this ensures almost identical focal quality
@b and c) so that the designed file has the correct PoP
@d) for multiple files *always pattern towards GIS* to avoid gas flux shadowing.
@e) depends on stage stability. At FELMI ~ 15 minutes
@f) at FELMI at least 3 minutes

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

[1] Winkler et al., in submission
[2] Winkler, R. *et al.* ASC Appl. Mater. Interfaces (2017) 9, 8233

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

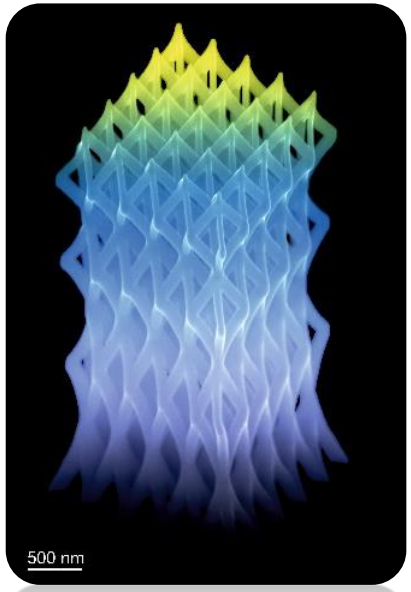
High primary energy

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 querstion contact us: Robert.winkler@felmi-zfe.at
Harald.Plank@felmi-zfe.at

FELMI-ZFE

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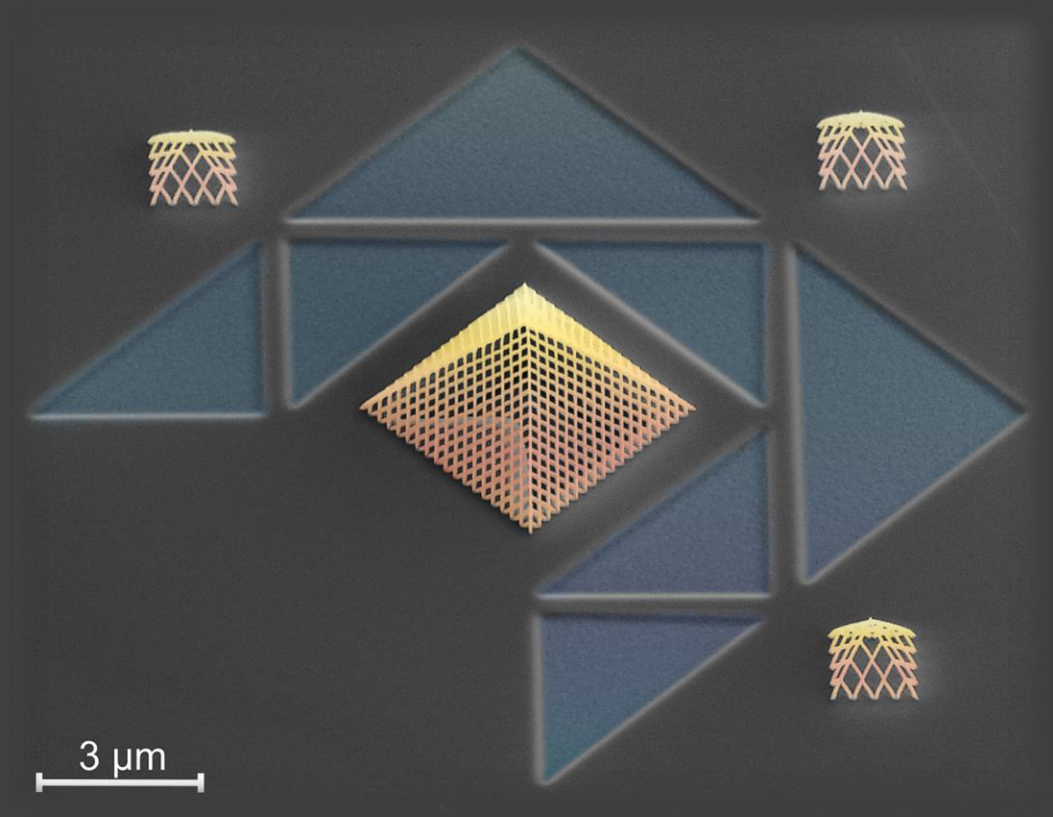
Thank you for your attention!



Science Meets Art:
3D-Nano-model of the glass pyramids of the Louvre (Paris) in a scale of 1:8.000.000