Elektronenmikroskopie:

Prof. Dr. Thomas Hellweg

Dr. Yvonne Hannappel

Dr. Inga Ennen

AH

UNIVERSITÄT BIELEFELD

JEOL FS 2200

FEI Helios FIB

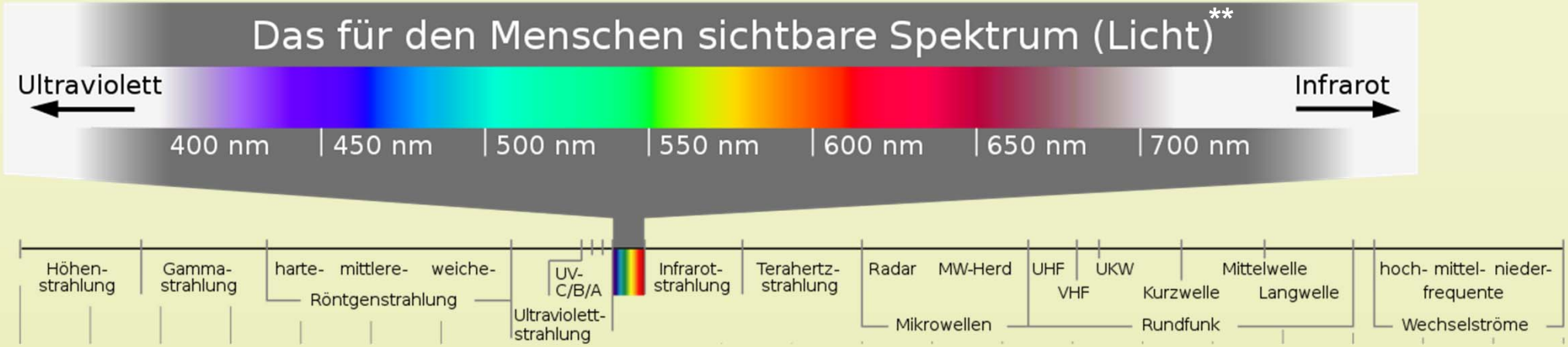
JEOL ARM

Prof. Dr. Jörg Lindner

Dr. Thomas Riedl

UNIVERSITÄT PADERBORN
Die Universität der Informationsgesellschaft

Eigenschaften von Elektronen:



** https://de.wikipedia.org/wiki/Elektromagnetisches_Spektrum

1923 hat Louis de Broglie (1892-1987) vorgeschlagen, dass die Wellenlänge eines „Materie-Teilchen“ in gleicher Weise wie die eines Photons mit dem Impuls zusammenhängt:

$$\lambda_{\substack{\text{de Broglie}\\ \text{eines Teilchens}}} = \frac{h_{\text{Plancksches Wirkungsquantum}}}{p_{\text{Impuls}}}$$

Eigenschaften von Elektronen:

$$e_{\substack{\text{Ladung}\\ \text{des Elektron}}} \cdot U_{\text{beschleunigende Spannung}} \overset{\substack{\text{Energie}\\ =\\ \text{Erhaltung}}}{=} \frac{1}{2} m_{\substack{\text{Masse}\\ \text{Elektron}}} \cdot v^2_{\substack{\text{Geschwindigkeit}\\ \text{Elektron}}}$$

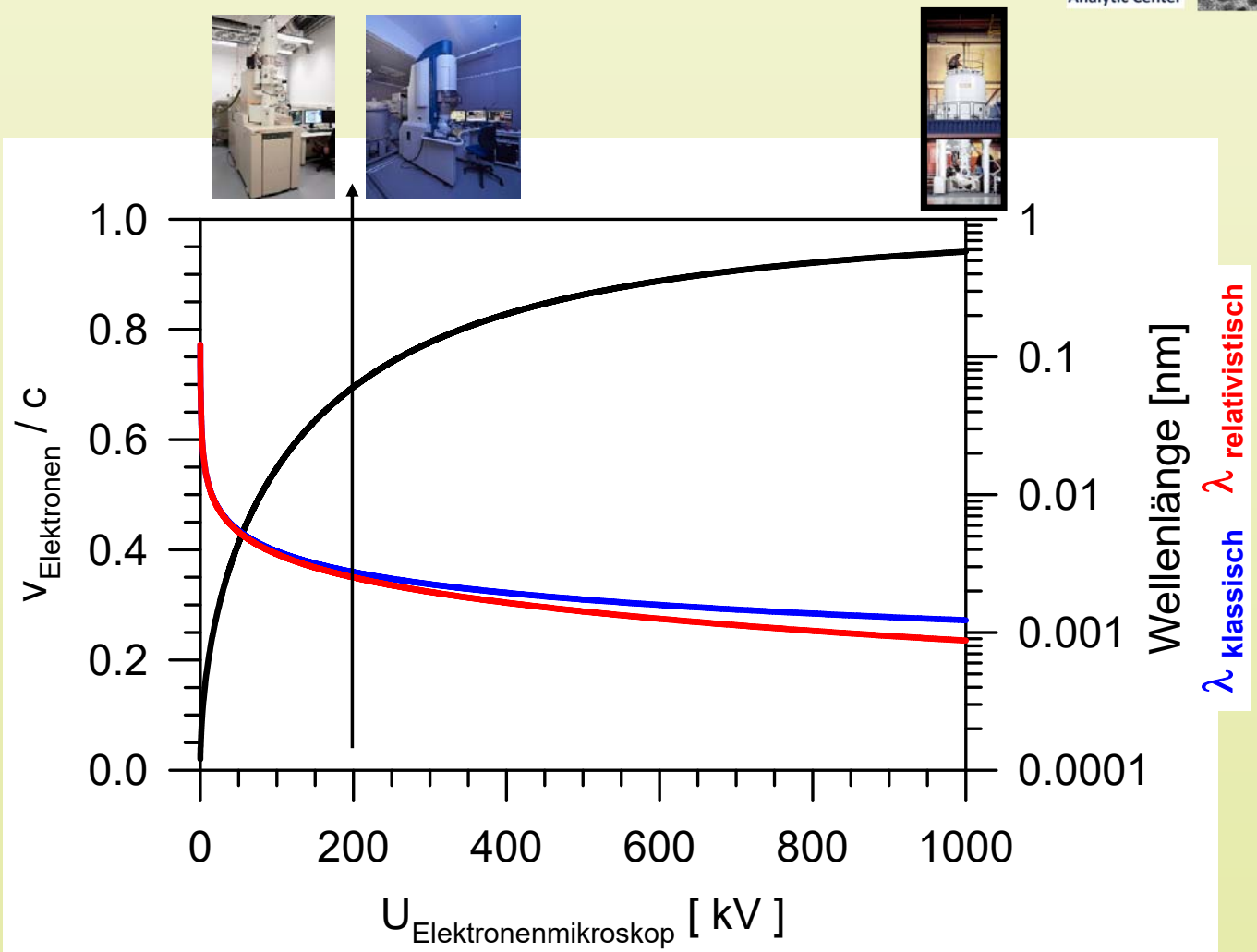
$$v_{\substack{\text{Geschwindigkeit}\\ \text{Elektron}}} = \sqrt{\frac{2 \cdot e_{\substack{\text{Ladung}\\ \text{des Elektrons}}} \cdot U_{\text{beschleunigende Spannung}}}{m_{\substack{\text{Masse}\\ \text{Elektron}}}}}$$

$$p_{\substack{\text{Impuls}\\ \text{Elektron}}} = m_{\substack{\text{Masse}\\ \text{Elektron}}} \cdot v_{\substack{\text{Geschwindigkeit}\\ \text{Elektron}}} = m_{\substack{\text{Masse}\\ \text{Elektron}}} \cdot \sqrt{\frac{2 \cdot e_{\substack{\text{Ladung}\\ \text{des Elektrons}}} \cdot U_{\text{beschleunigende Spannung}}}{m_{\substack{\text{Masse}\\ \text{Elektron}}}}}$$

$$\lambda_{\substack{\text{de Broglie}\\ \text{eines Teilchens}}} = \frac{h_{\text{Plancksches Wirkungsquantum}}}{p_{\substack{\text{Impuls}\\ \text{Elektron}}}} = \frac{h_{\text{Plancksches Wirkungsquantum}}}{\sqrt{2 \cdot e_{\substack{\text{Ladung}\\ \text{des Elektrons}}} \cdot m_{\substack{\text{Masse}\\ \text{Elektron}}} \cdot U_{\text{beschleunigende Spannung}}}}$$

$$\lambda_{\text{relativistisch}} = \frac{h}{p_{\text{relativistisch}}} = \frac{h}{\sqrt{2 \cdot e \cdot m \cdot U \cdot \left(1 + \frac{e \cdot U}{2 \cdot m \cdot c^2}\right)}}$$

Eigenschaften von Elektronen:



Entwicklung der Transmissionselektronenmikroskope:

September 1983:
30t - JEOL ARM mit U = 1,5 MeV
Mit 0,16 nm Auflösung

ncem

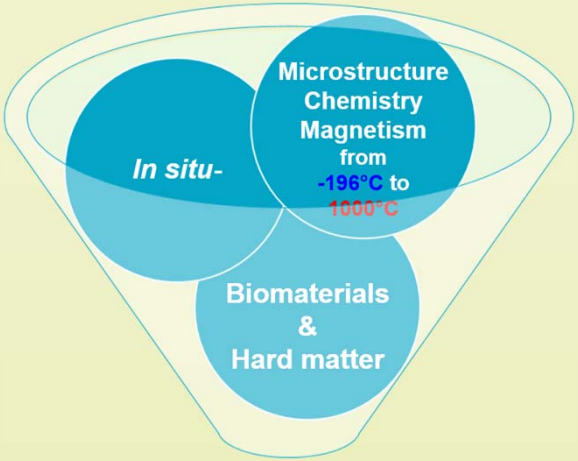
Welcome to the
National Center for Electron Microscopy

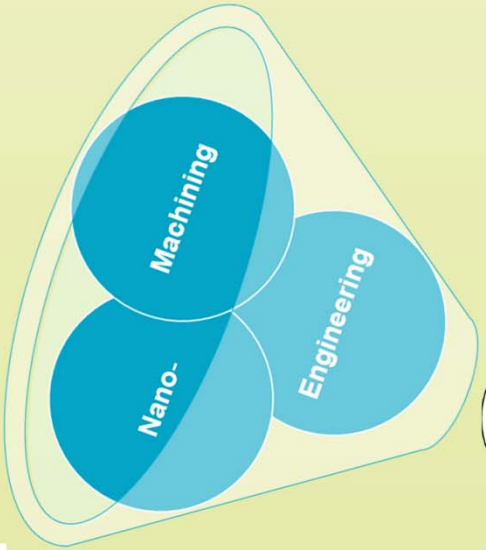
U.S. Department of Energy facility located at the University of California, Berkeley

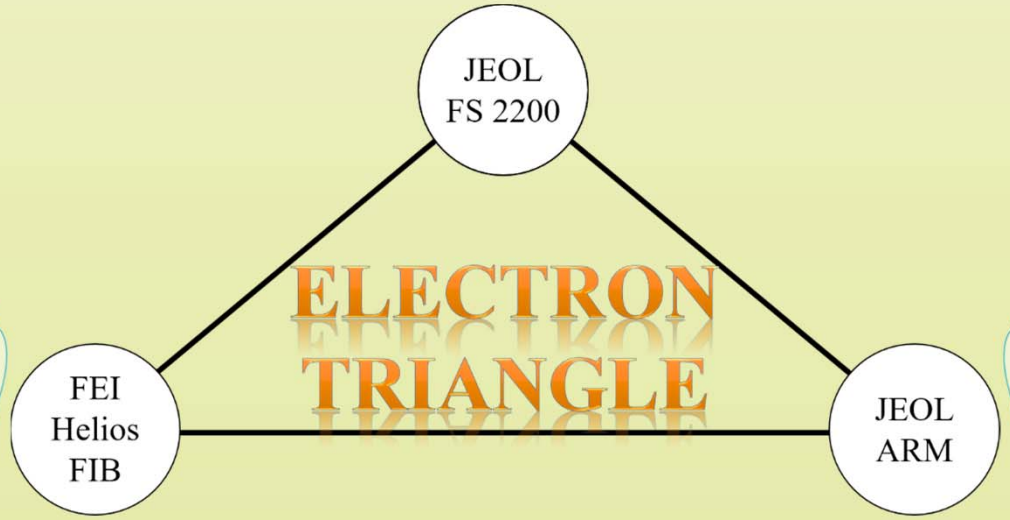
Oktober 2008 TEAM:
Transmissions-Elektronen-Mikroskop
mit Aberrations-Korrektur, FEI
U = 80 - 200 keV
Mit 0,1 nm – 0,05 nm Auflösung

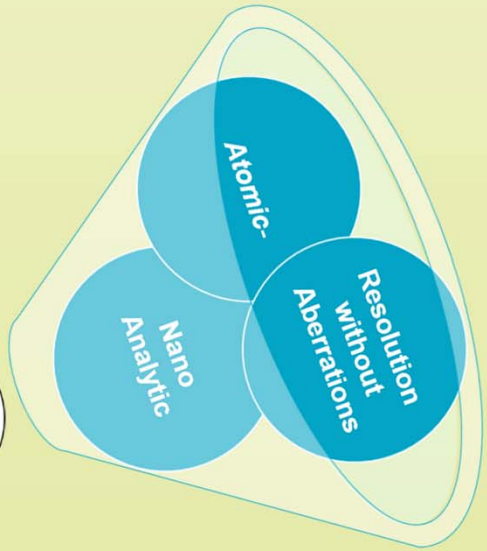
Probe muss dünn sein!

Die Möglichkeiten des OWL-Analytic Centers

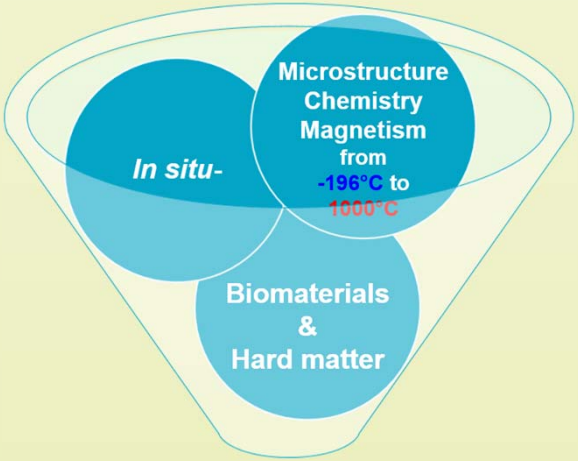


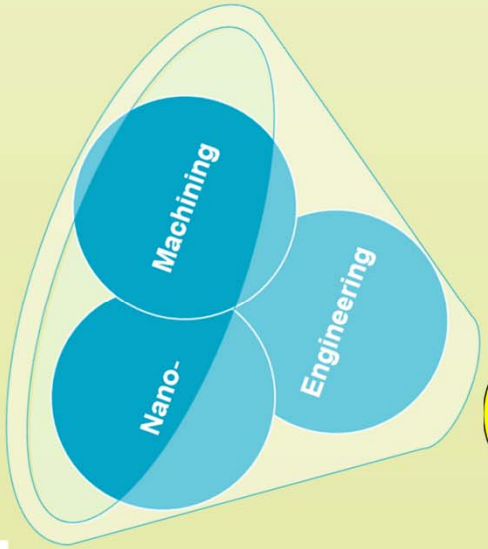


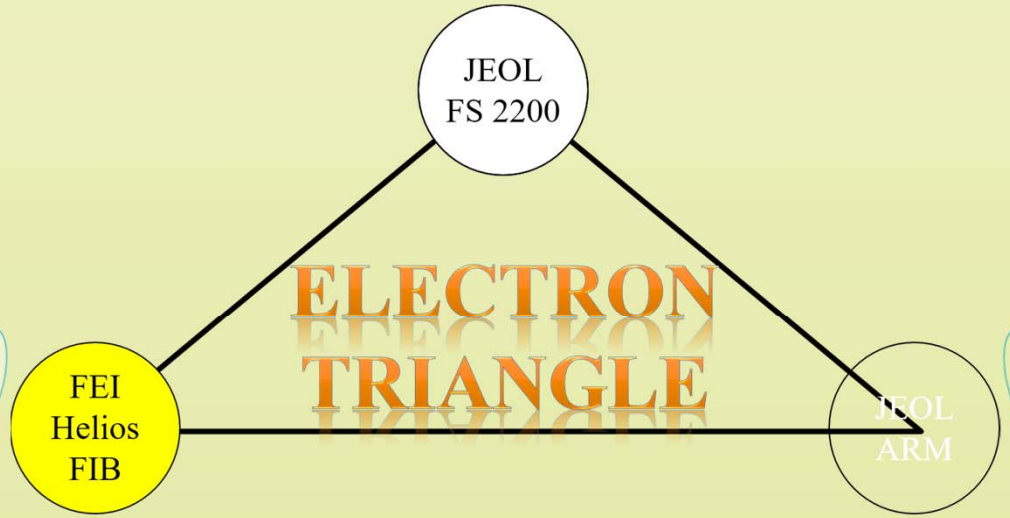


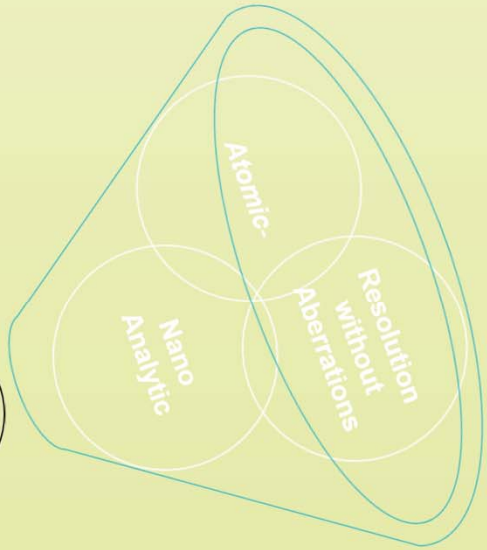


FEI Helios - Focused Ion Dual Beam

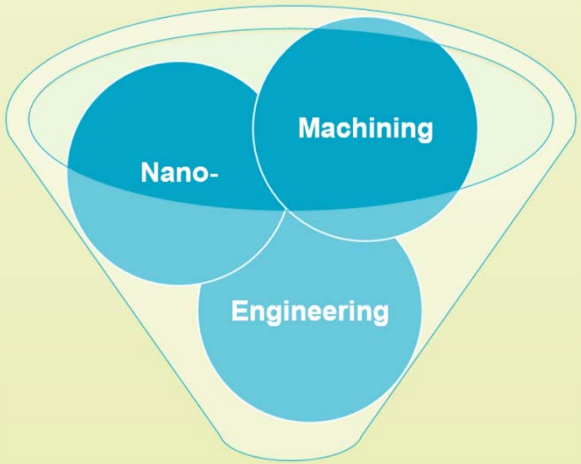








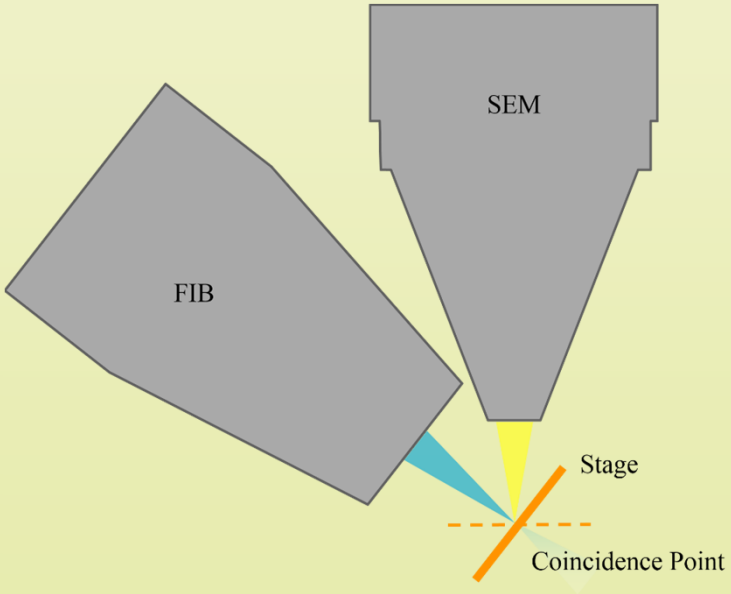
FEI Helios - Focused Ion Dual Beam

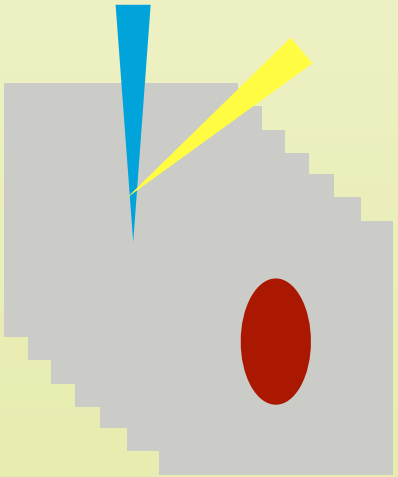


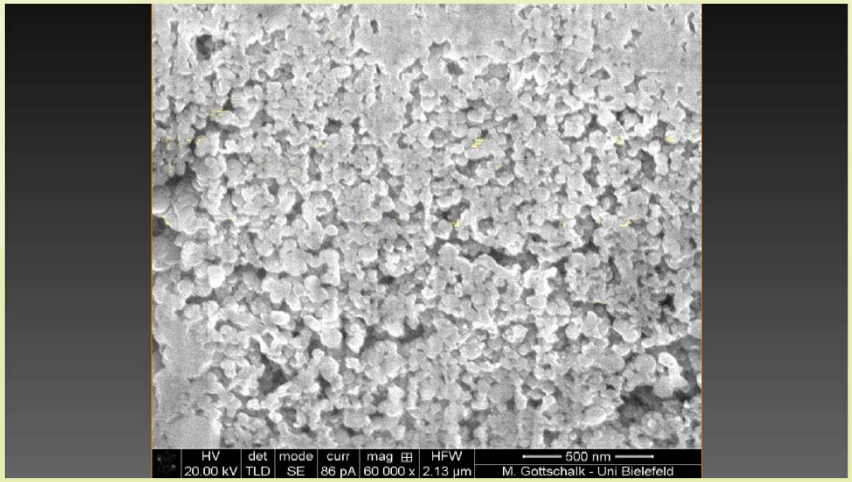
FEI Helios - Focused Ion Dual Beam

Physikalische Bedeutung von 3D-Anordnung magnetischer Nanopartikel in einer Matrix

→ 3D Rekonstruktion der Mikrostruktur



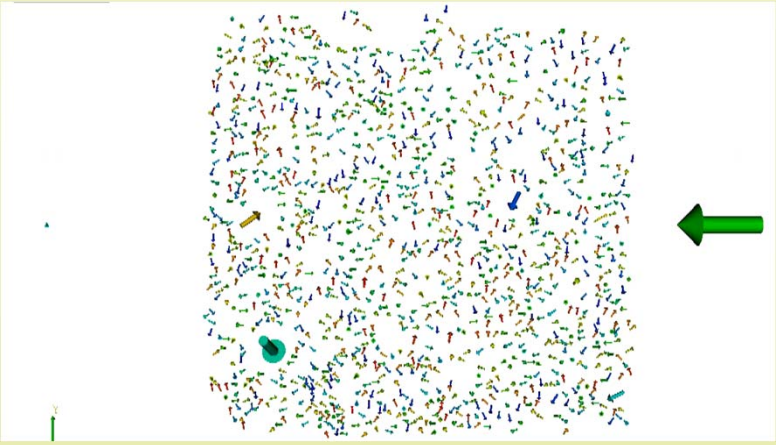


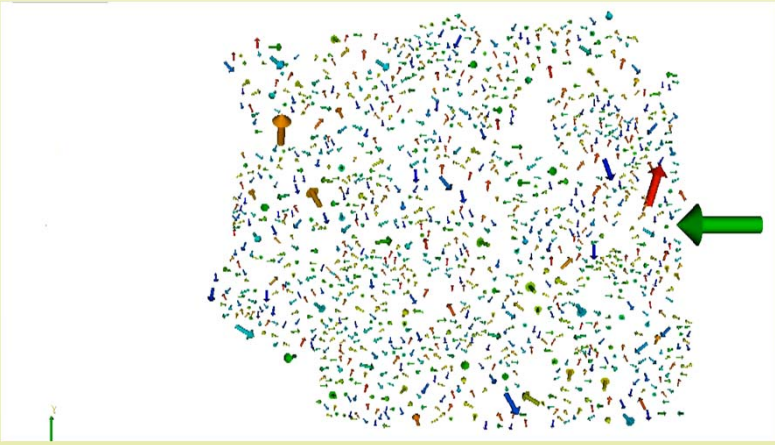


FEI Helios - Focused Ion Dual Beam

Vergleich von Co-Kern/C-Schale Nanopartikeln in Agarose Matrizes

Berechnung der magnetischen Wechselwirkungen über
die genaue Information der Mikrostruktur





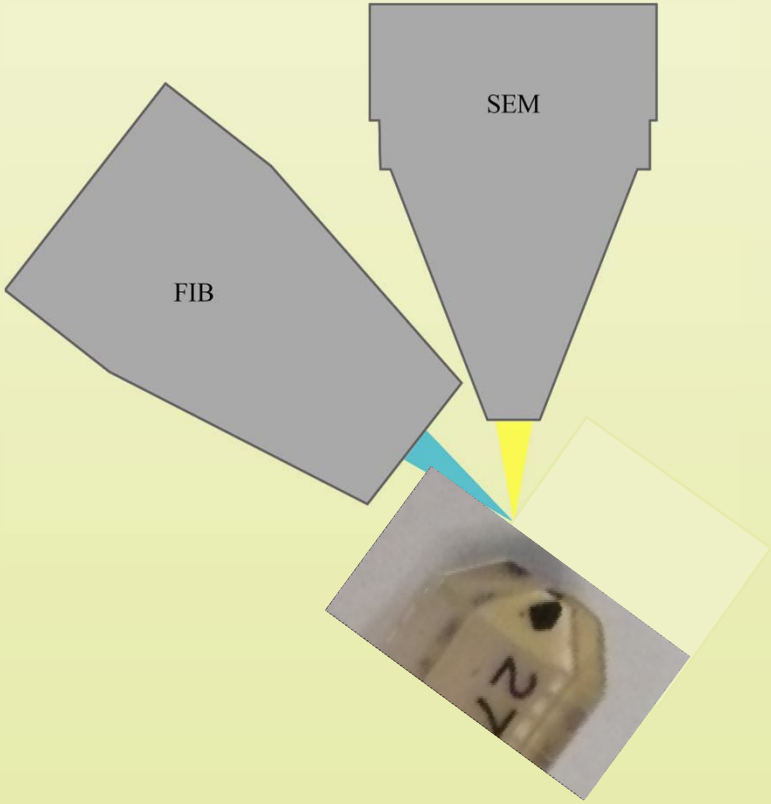
→ starke ferromagnetische Kopplung der Nanopartikel

→ Kettenbildung von Nanopartikeln: $f_{vol} = 0.56$

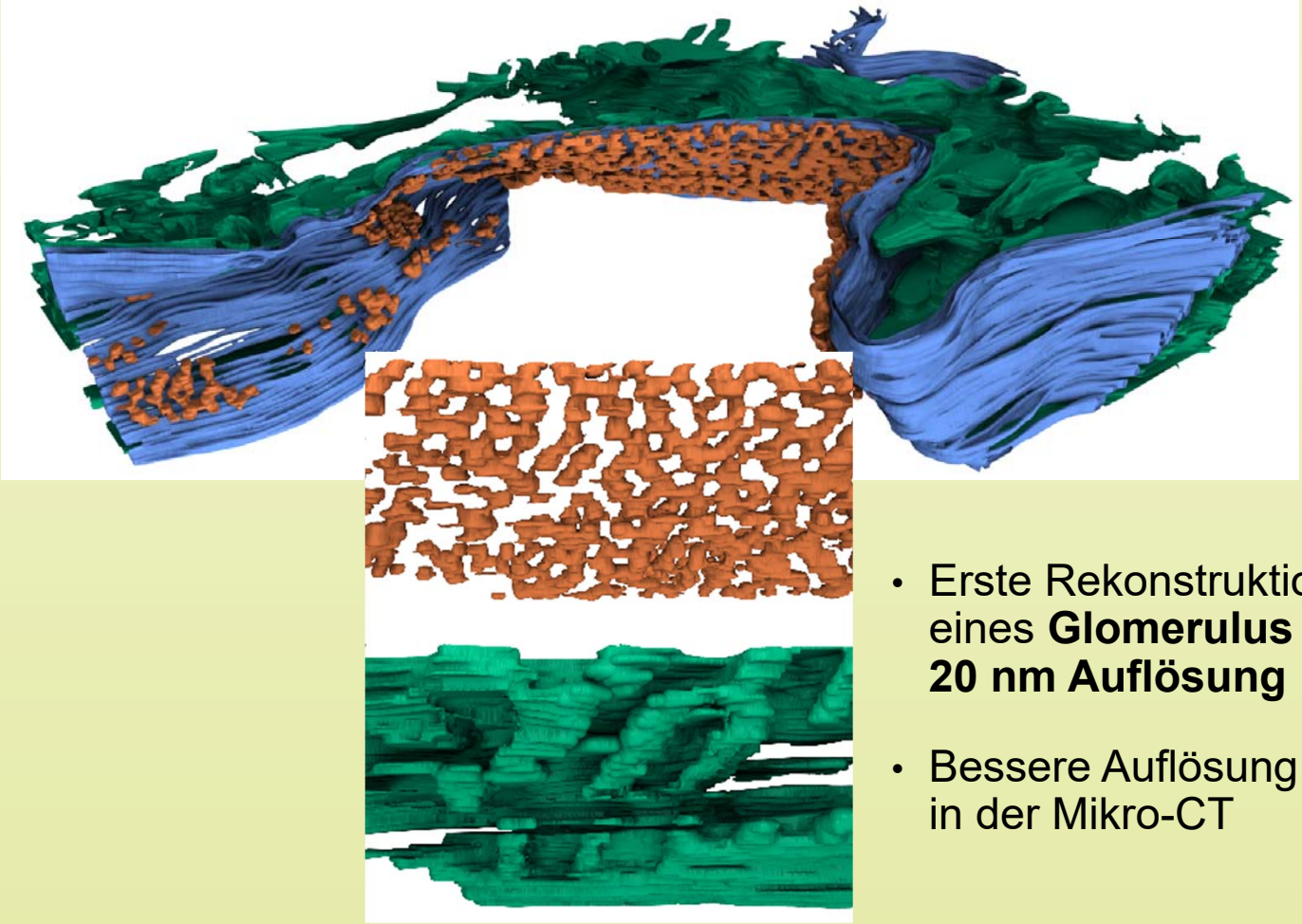
→ Nanopartikel sind dicht gepackt: $f_{vol} = 0.7$

FEI Helios - Focused Ion Dual Beam

Bio-Imaging: Wie sieht die Membran zwischen Blut- und Urin-Bereich der Niere bei Mäusen aus?



FEI Helios - Focused Ion Dual Beam



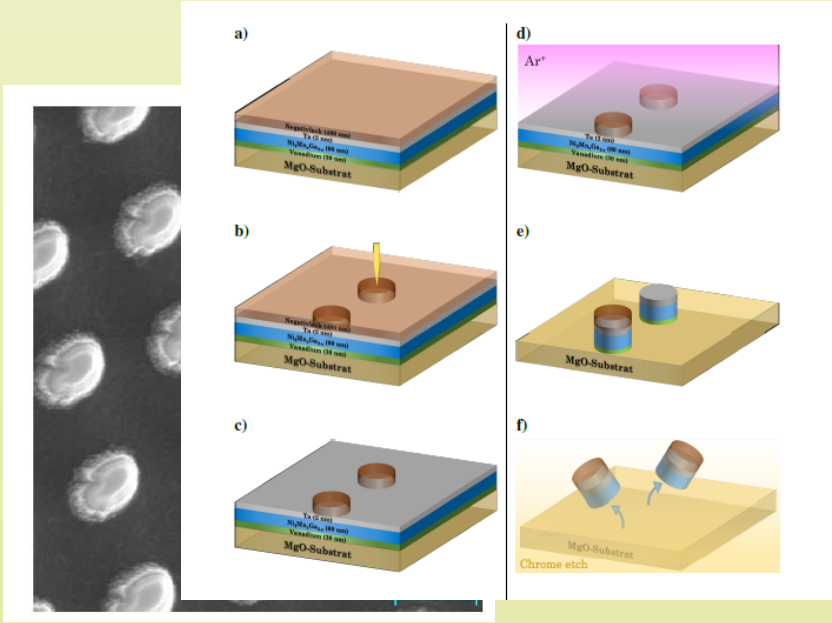
- Erste Rekonstruktion eines **Glomerulus** mit **20 nm Auflösung**
- Bessere Auflösung als in der Mikro-CT

FEI Helios - Focused Ion Dual Beam

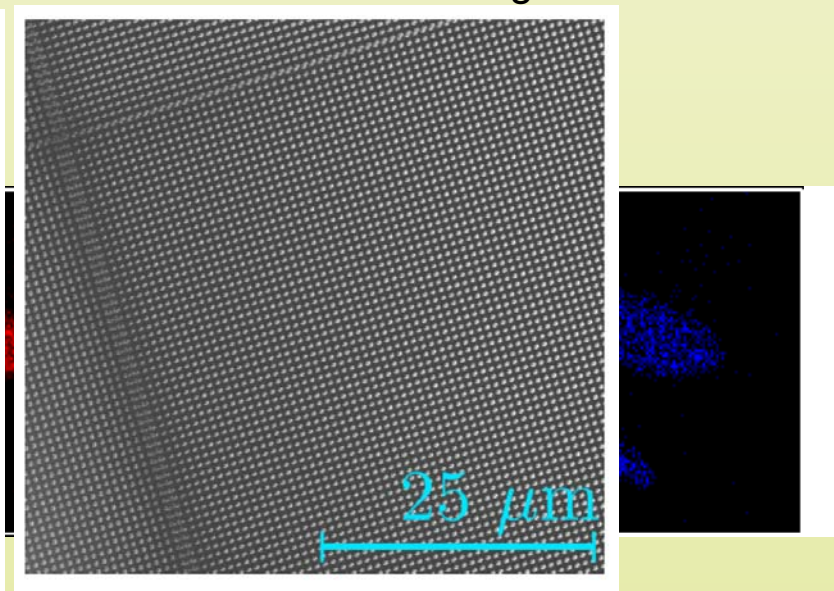
Nanopartikelherstellung aus ultradünnen Multilagen-Schichtsystemen:

Nanopartikuläre Magnetokalorik-Systeme: Ni_2MnGa

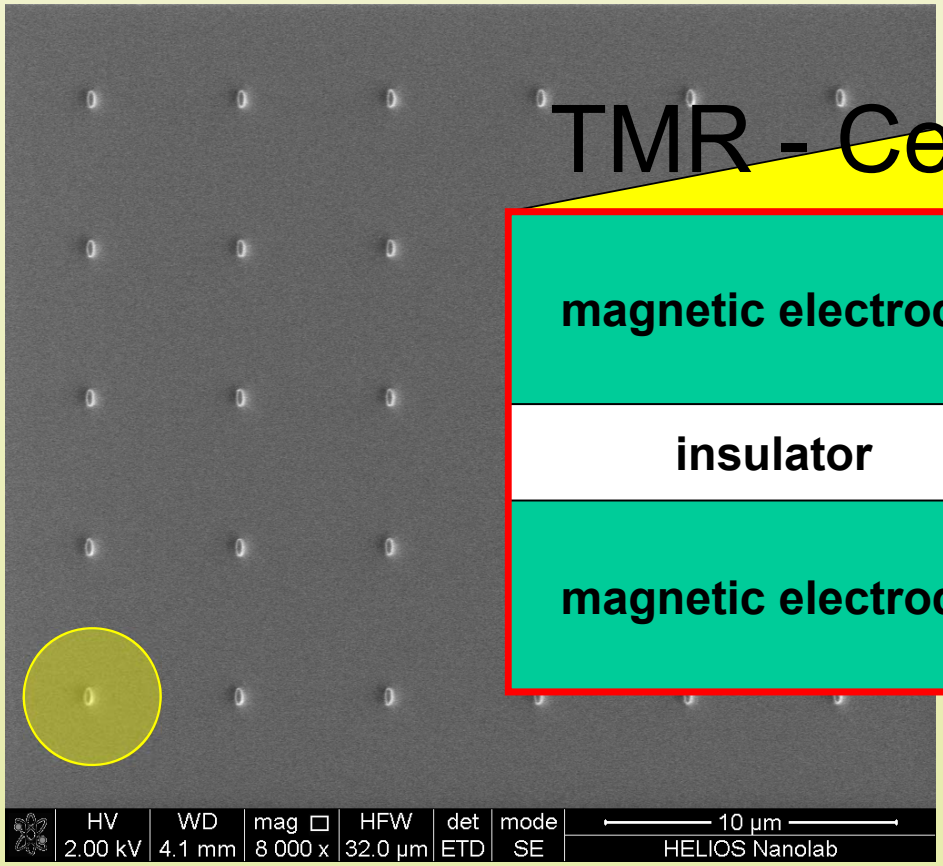
Schichtdesign:



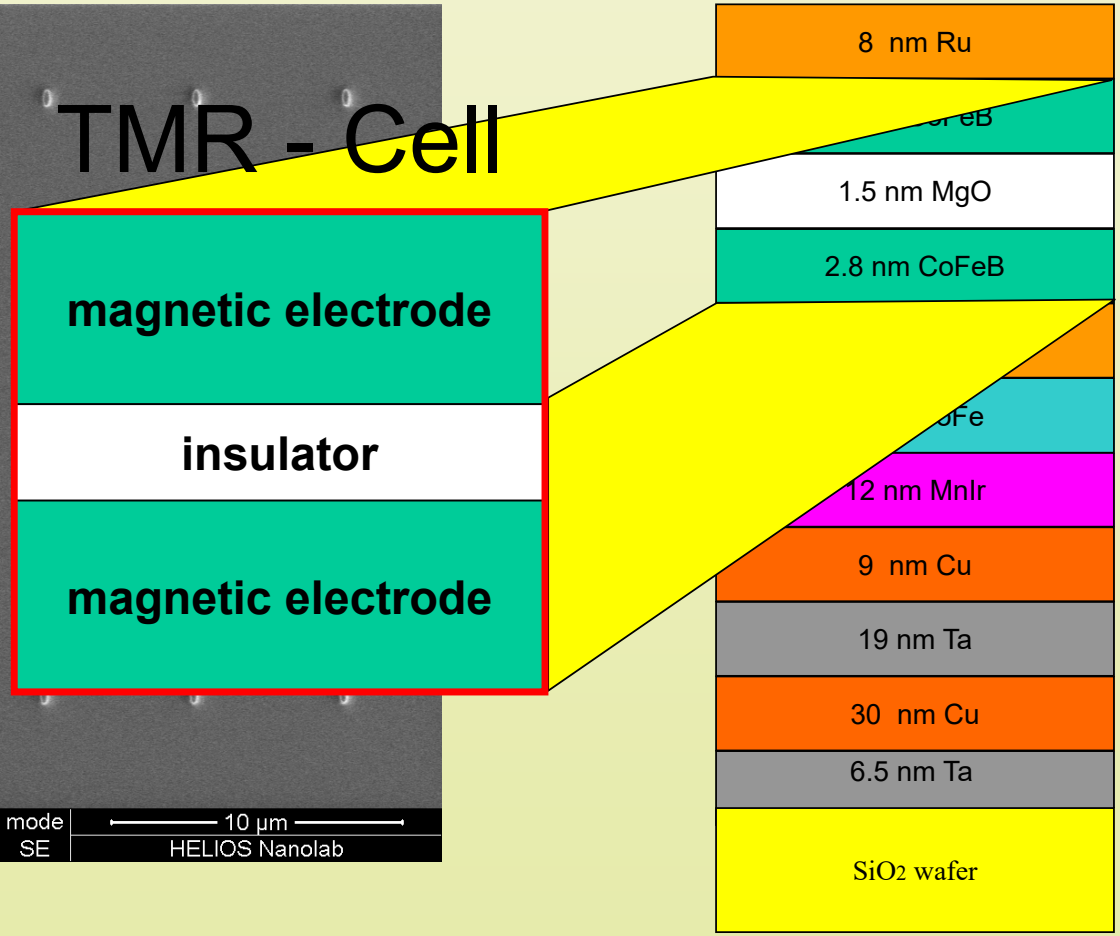
Nach der Strukturierung:

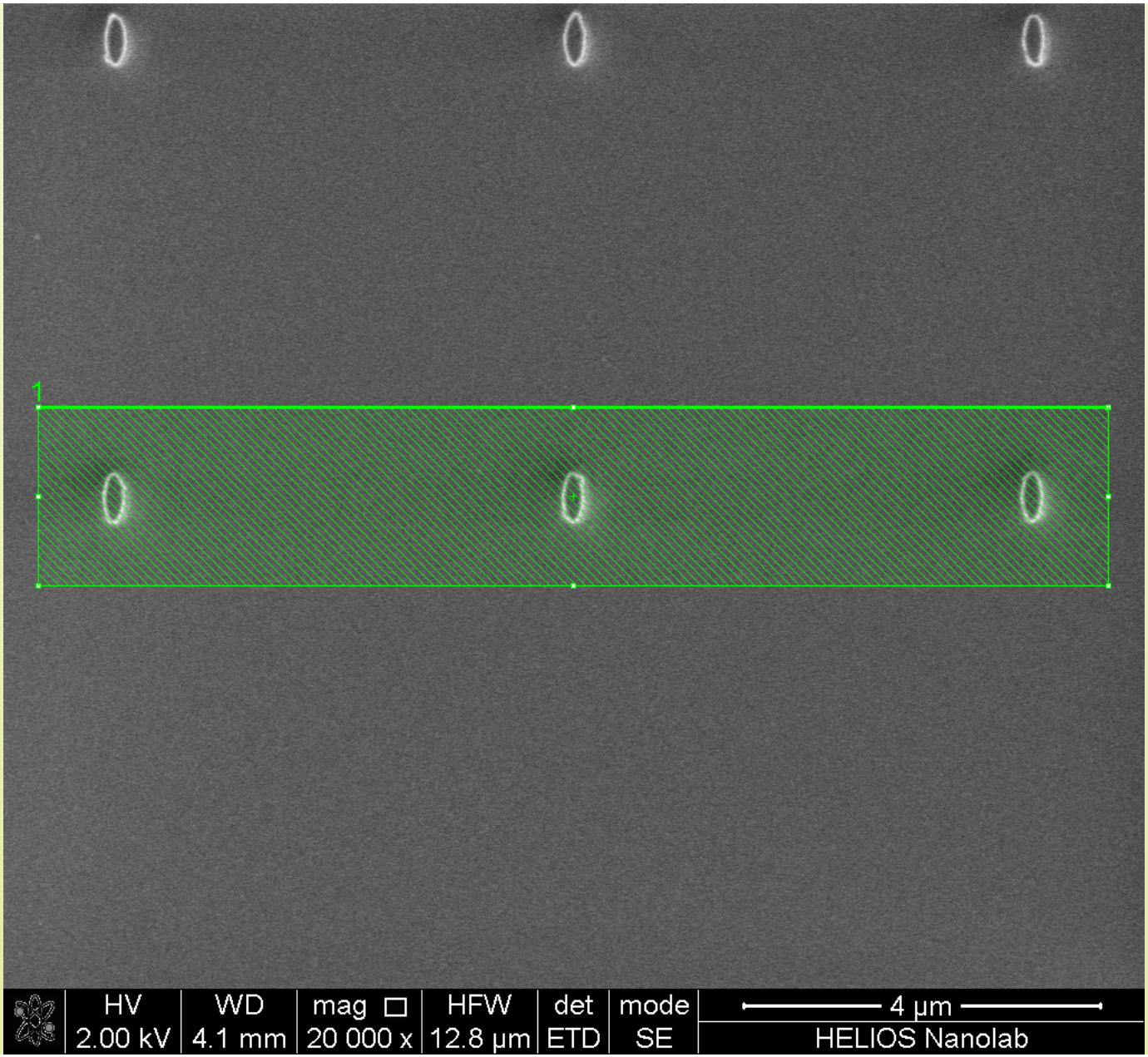


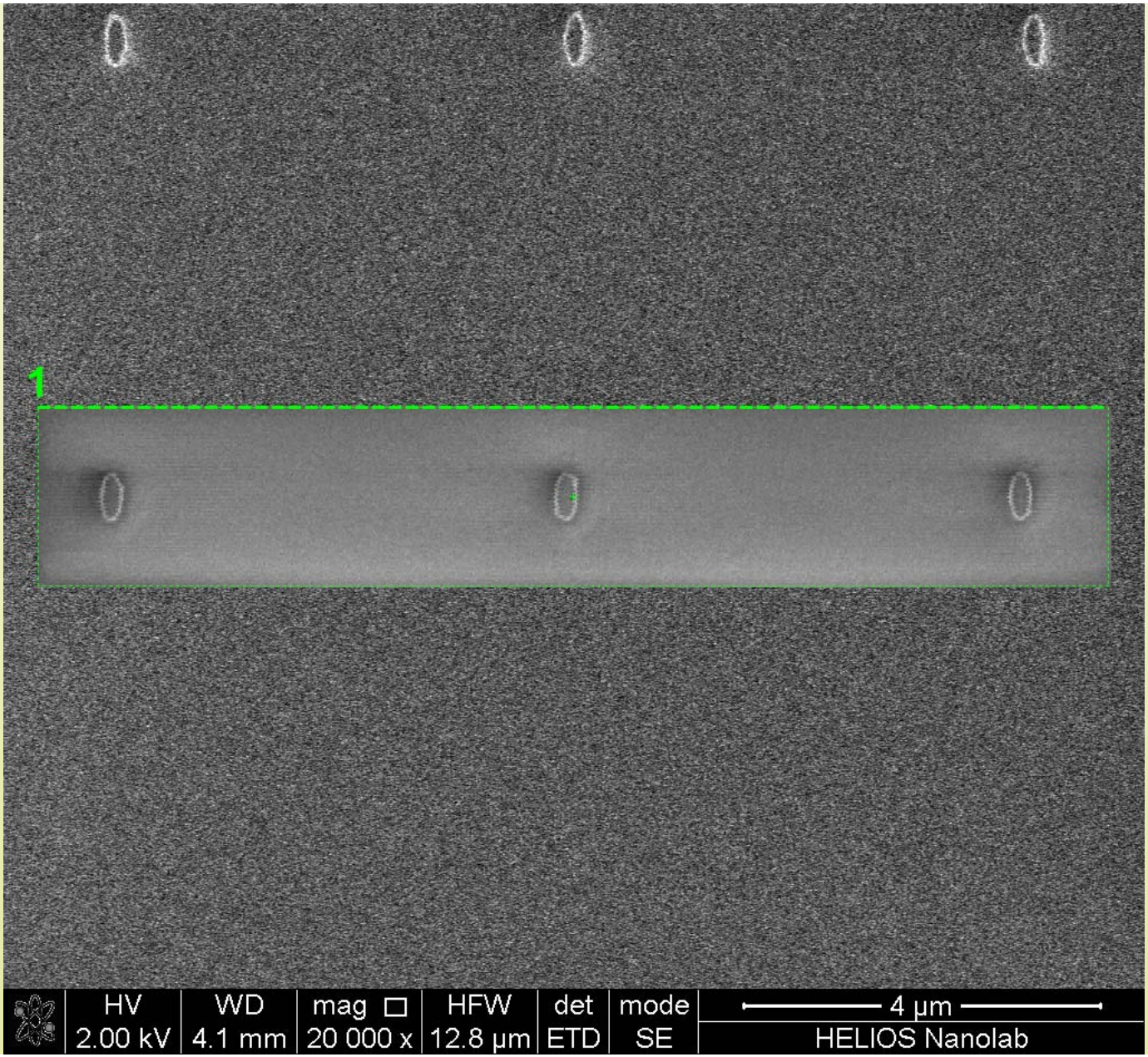
FEI Helios - Focused Ion Dual Beam: Proben für die hochauflösende Elektronenmikroskopie

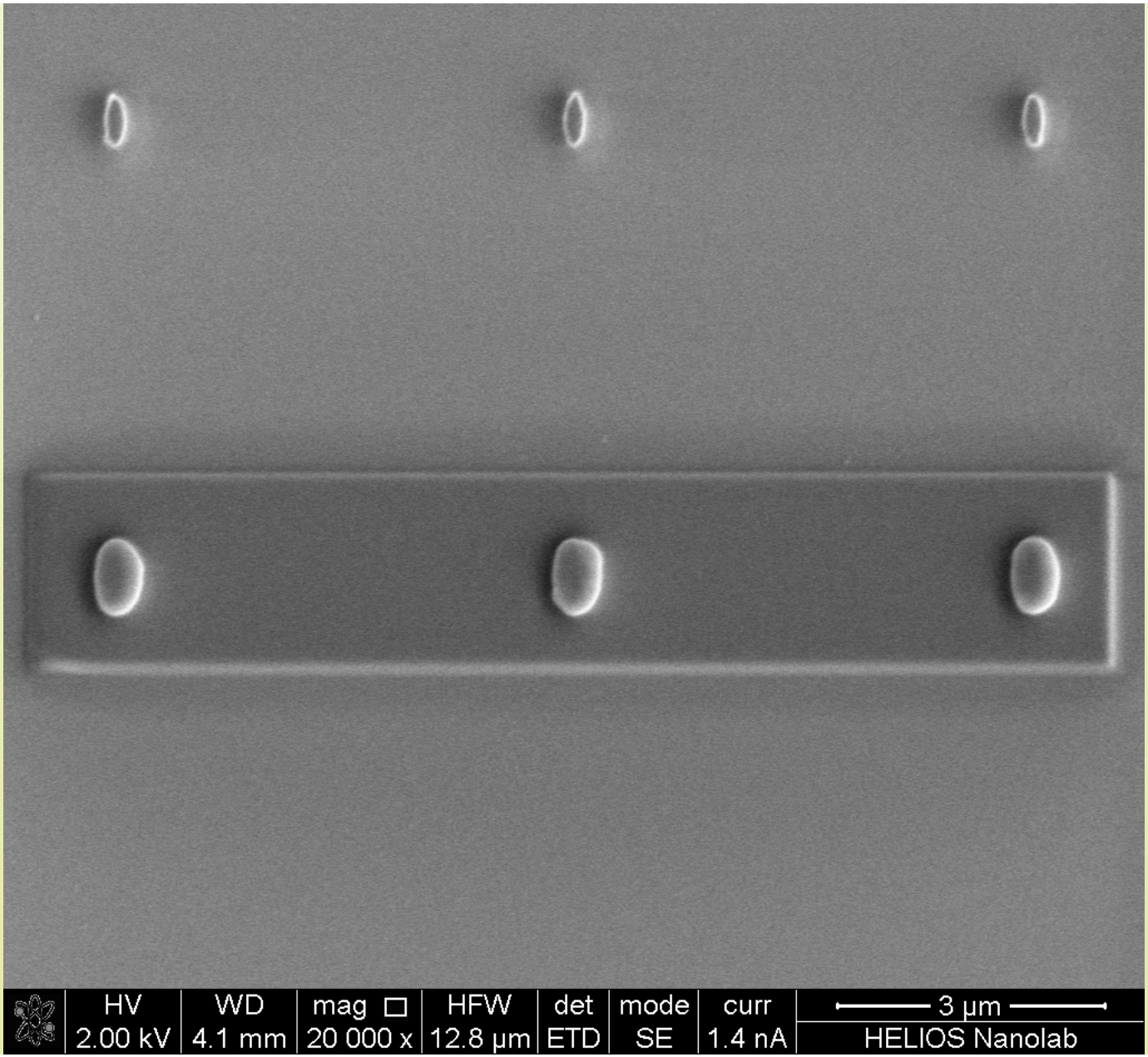


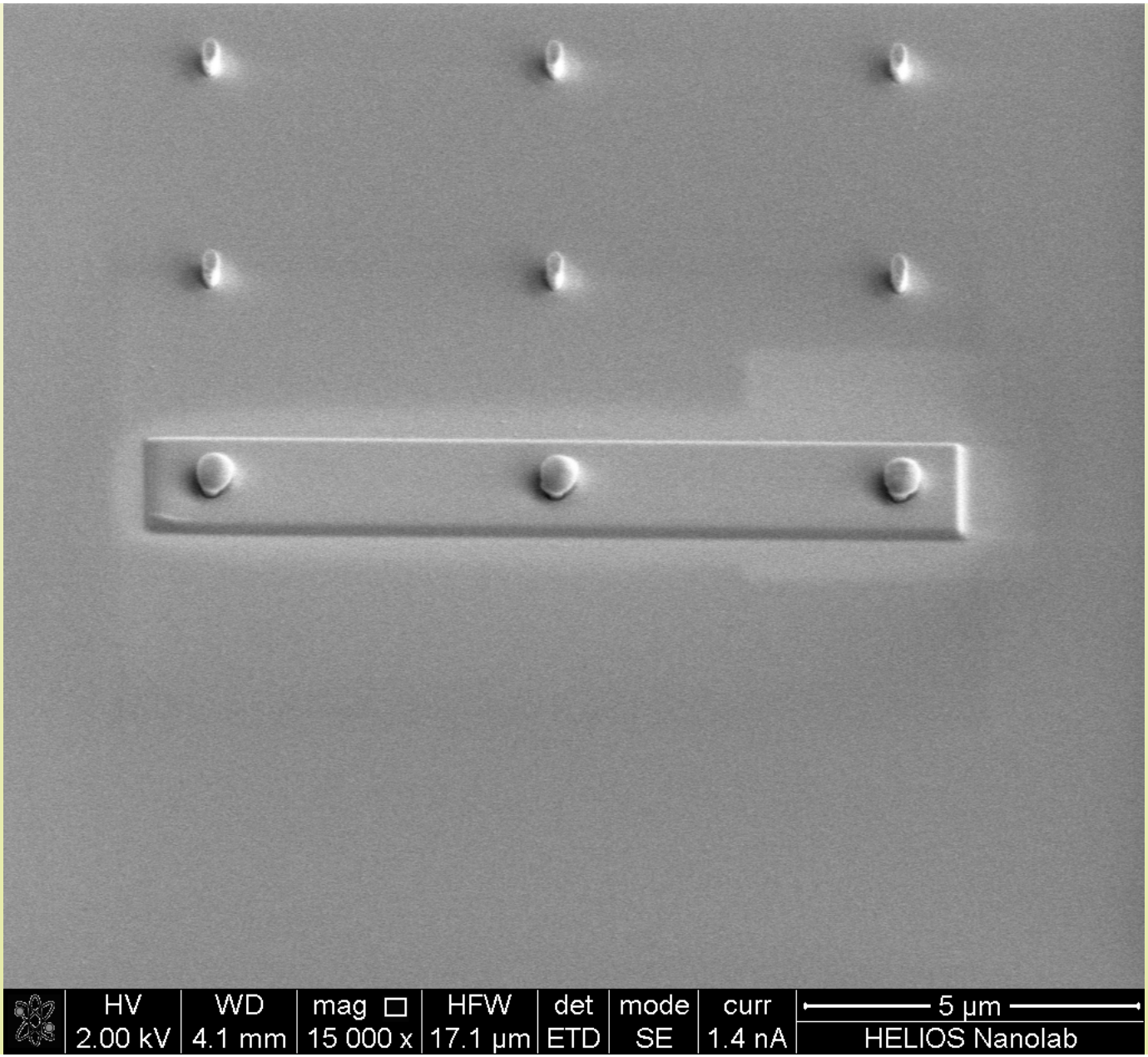
TMR - Cell

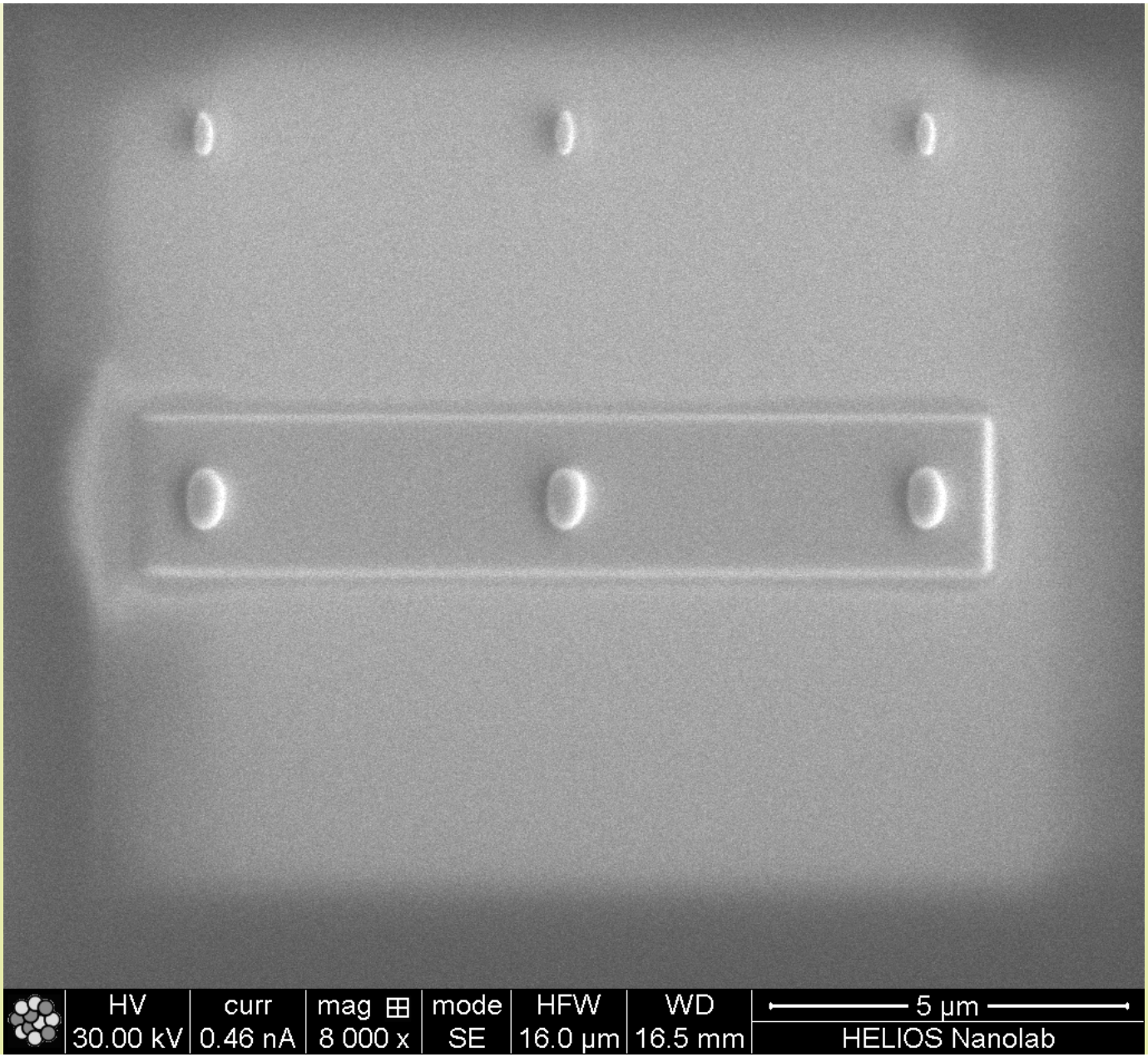


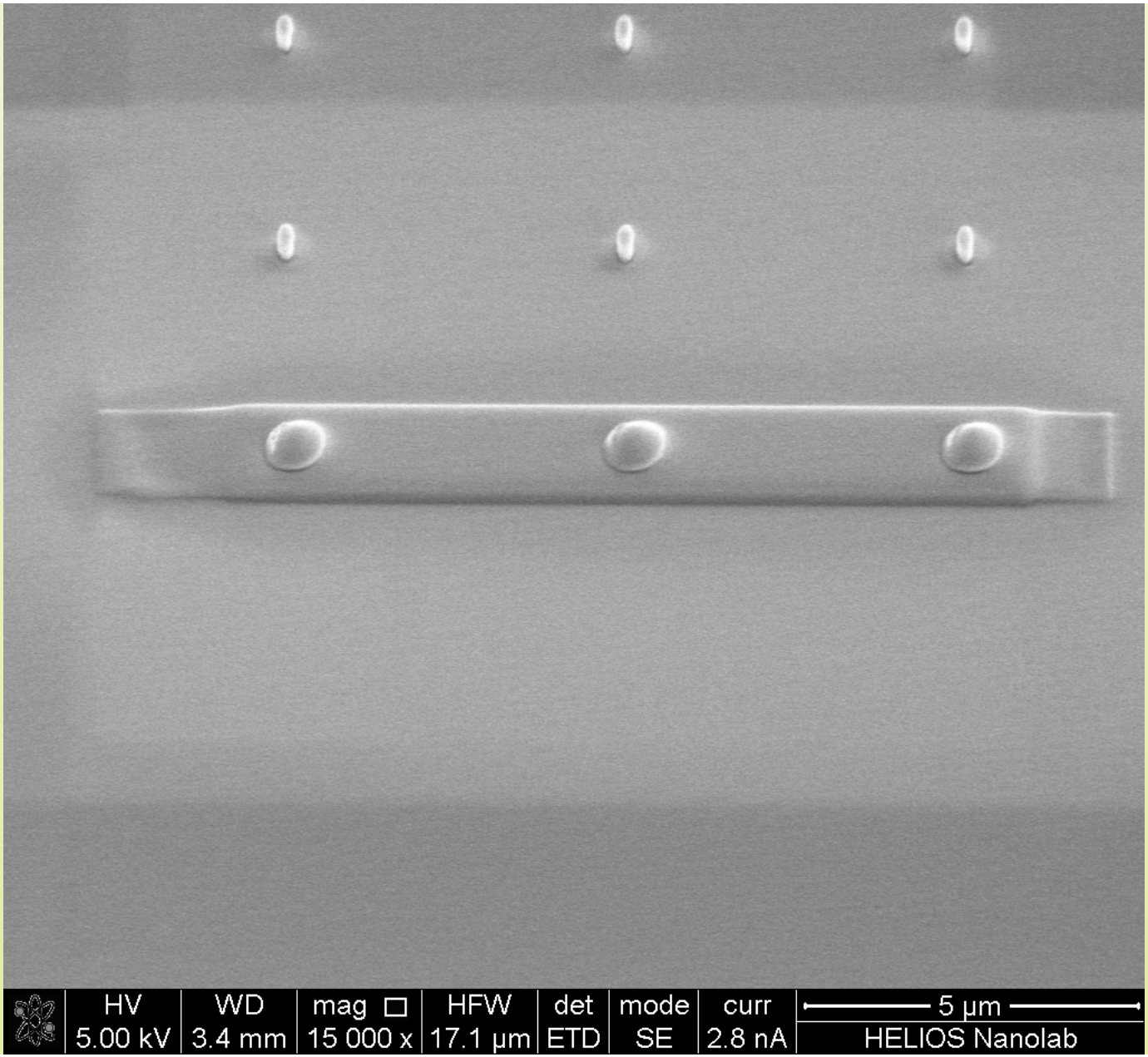

1
HV
2.00 kV
WD
4.1 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
4 µm
HELIOS Nanolab

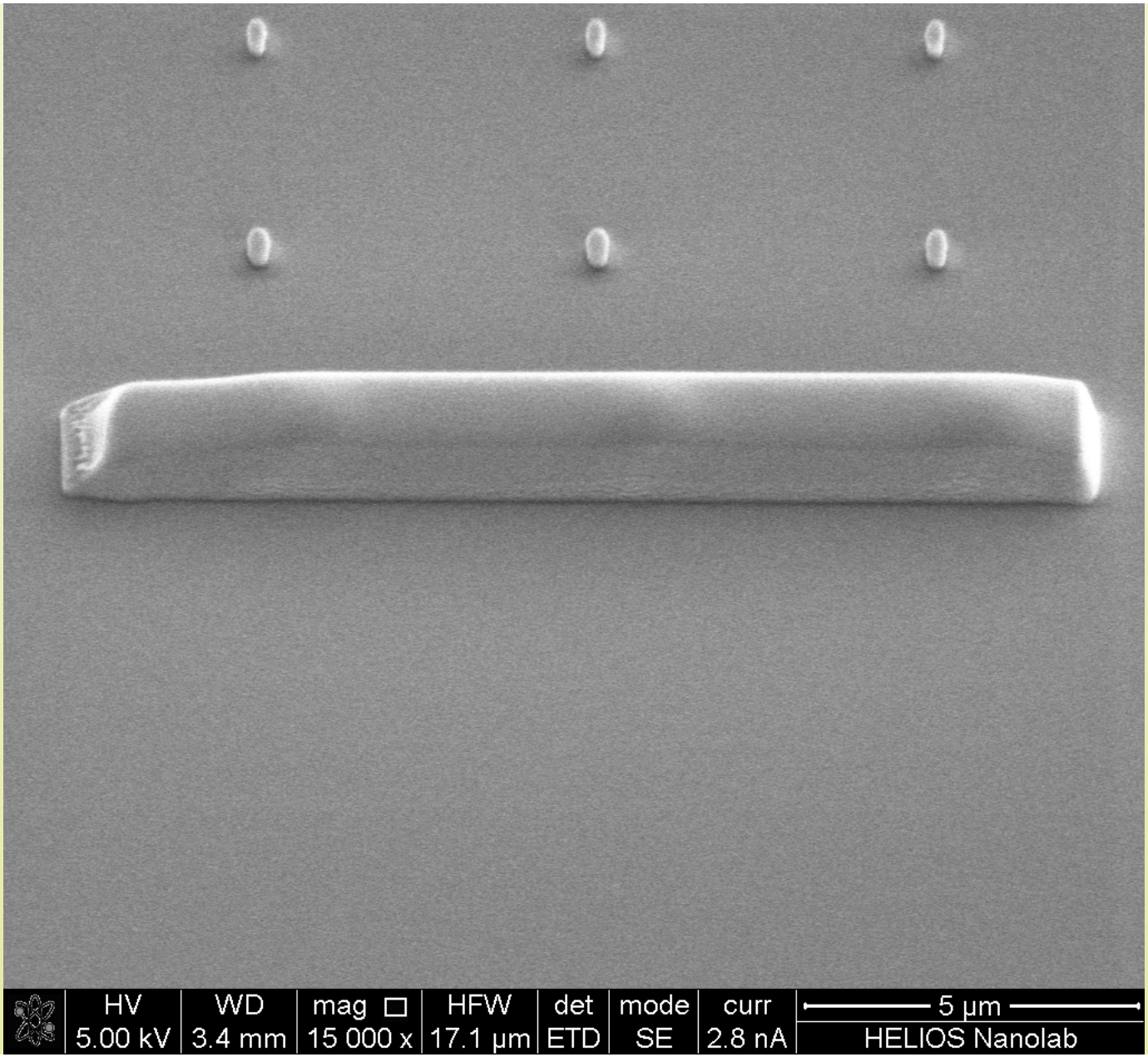

1
HV
2.00 kV
WD
4.1 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
4 µm
HELIOS Nanolab

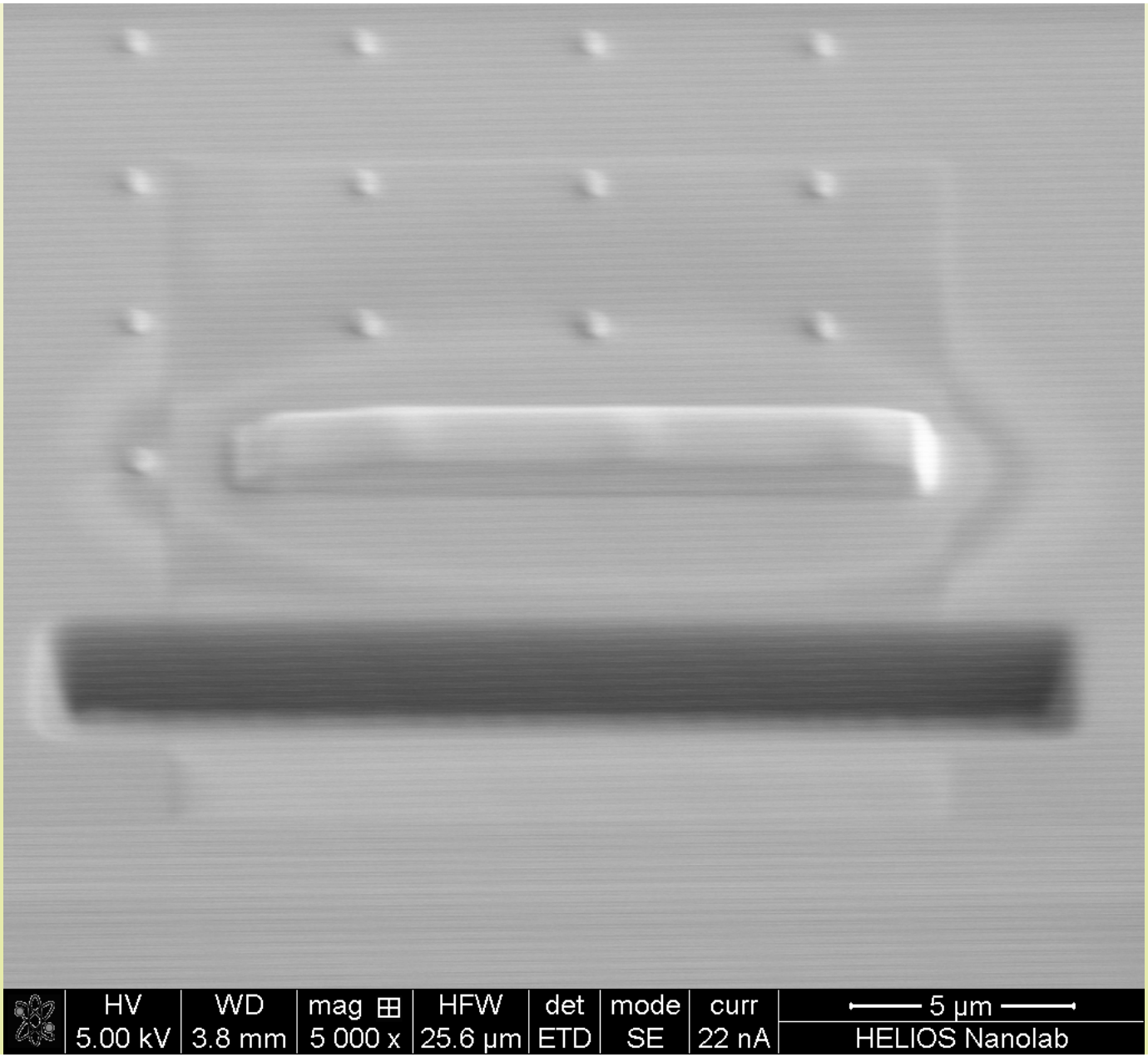

HV
2.00 kV
WD
4.1 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
curr
1.4 nA
3 µm
HELIOS Nanolab

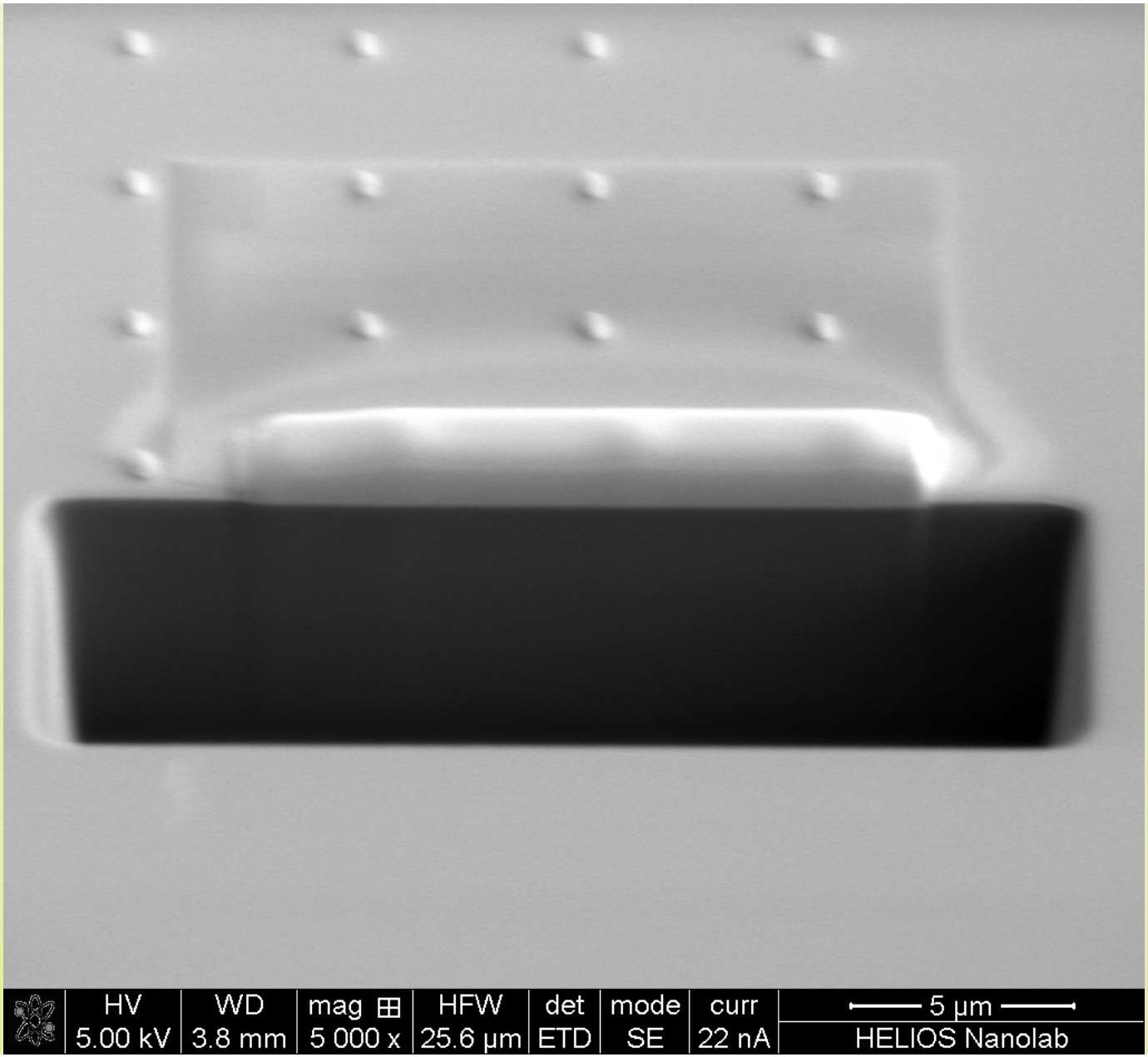

HV
2.00 kV
WD
4.1 mm
mag
15 000 x
HFW
17.1 µm
det
ETD
mode
SE
curr
1.4 nA
5 µm
HELIOS Nanolab

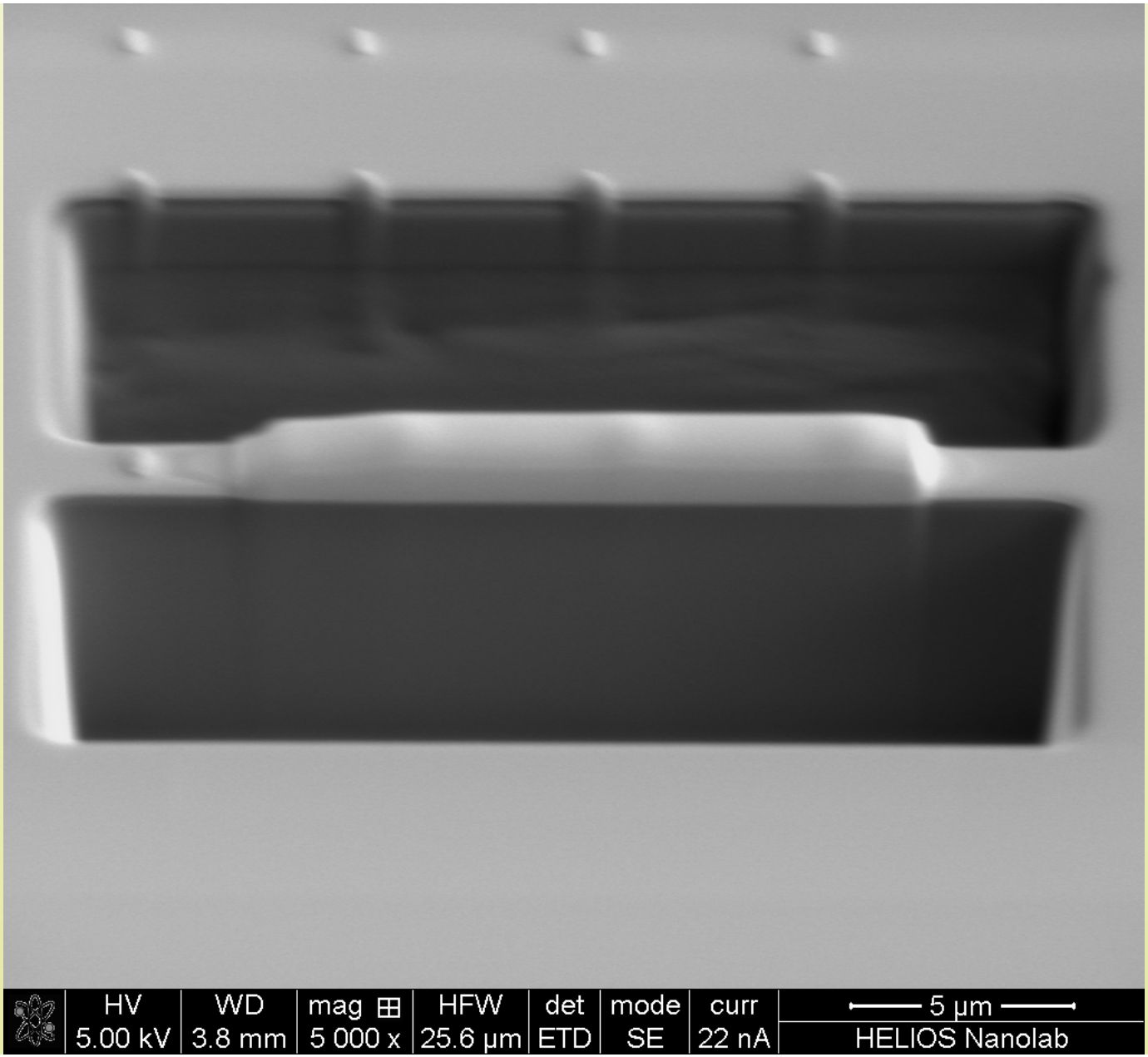

HV
30.00 kV
curr
0.46 nA
mag
8 000 x
mode
SE
HFW
16.0 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

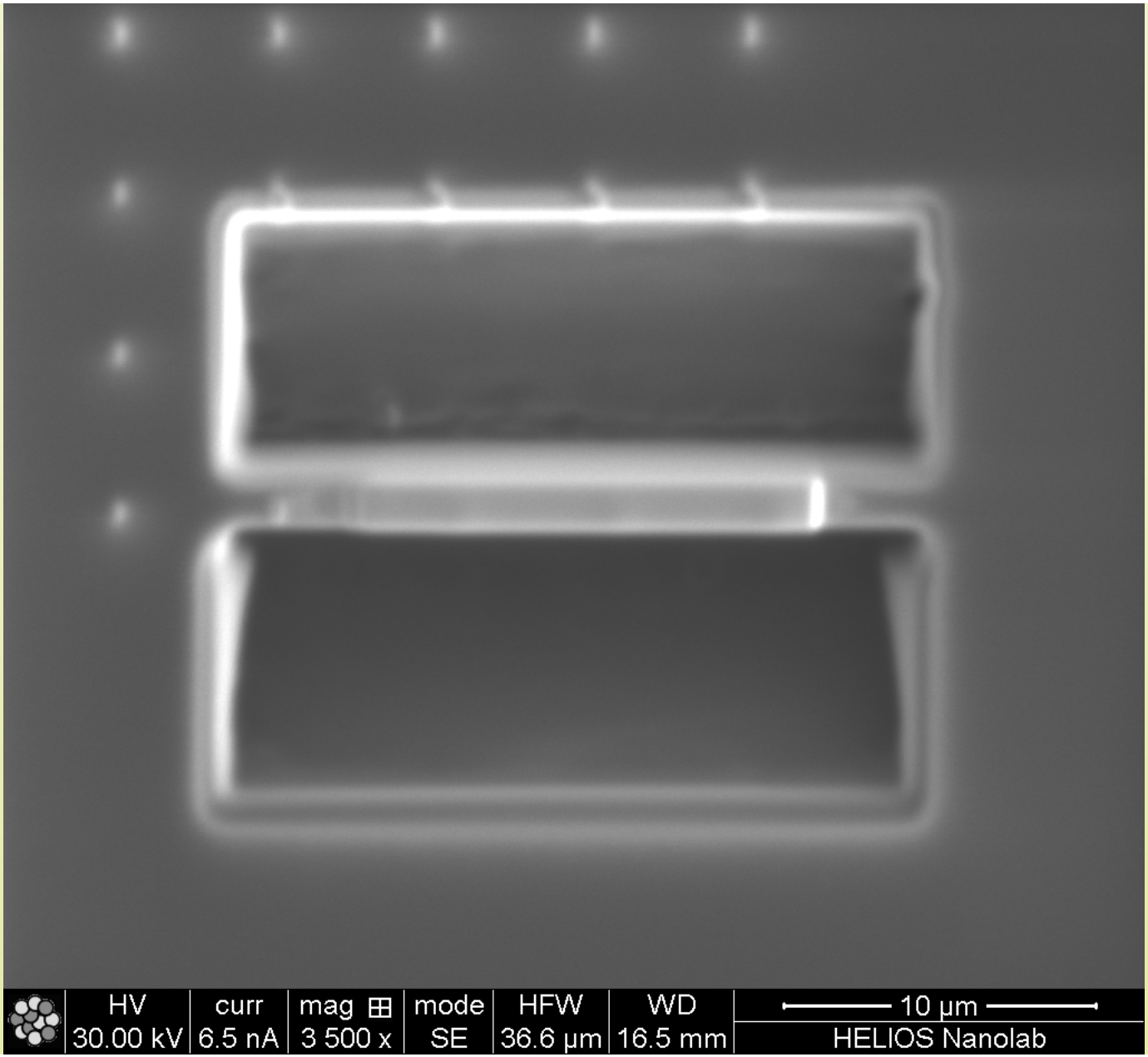

HV
5.00 kV
WD
3.4 mm
mag
15 000 x
HFW
17.1 µm
det
ETD
mode
SE
curr
2.8 nA
5 µm
HELIOS Nanolab

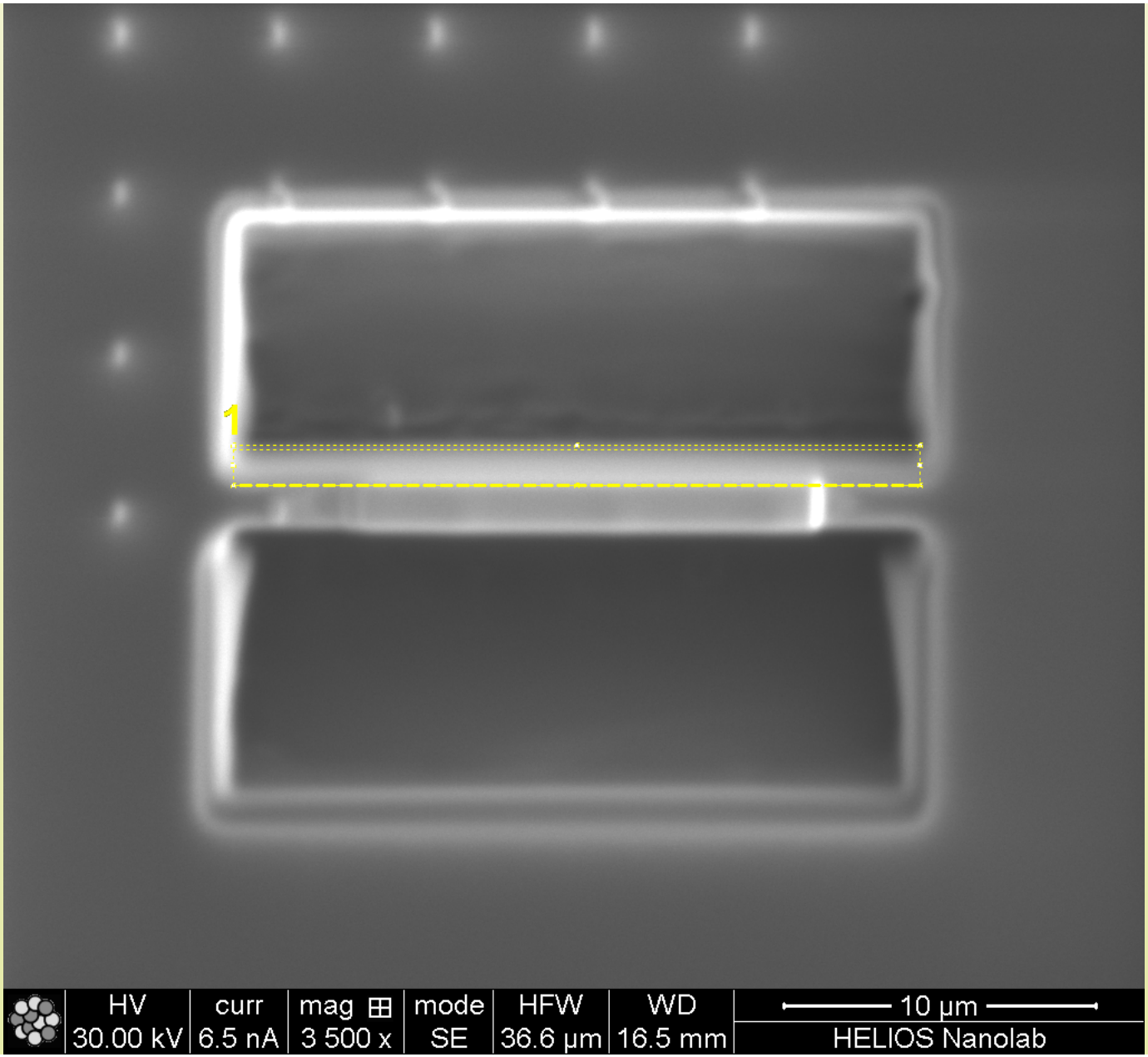

HV
5.00 kV
WD
3.4 mm
mag
15 000 x
HFW
17.1 µm
det
ETD
mode
SE
curr
2.8 nA
5 µm
HELIOS Nanolab

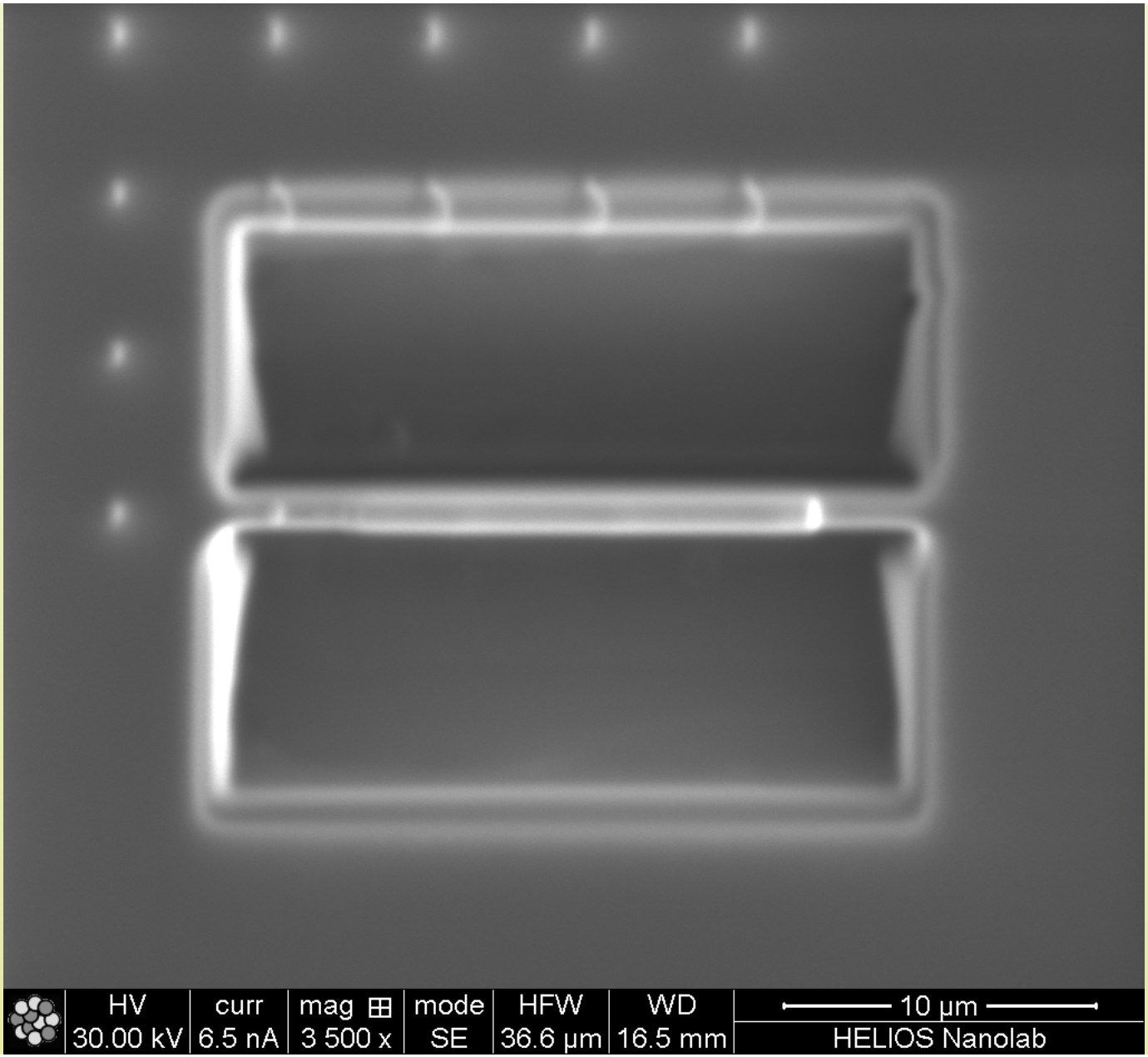

HV
5.00 kV
WD
3.8 mm
mag
5 000 x
HFW
25.6 µm
det
ETD
mode
SE
curr
22 nA
5 µm
HELIOS Nanolab

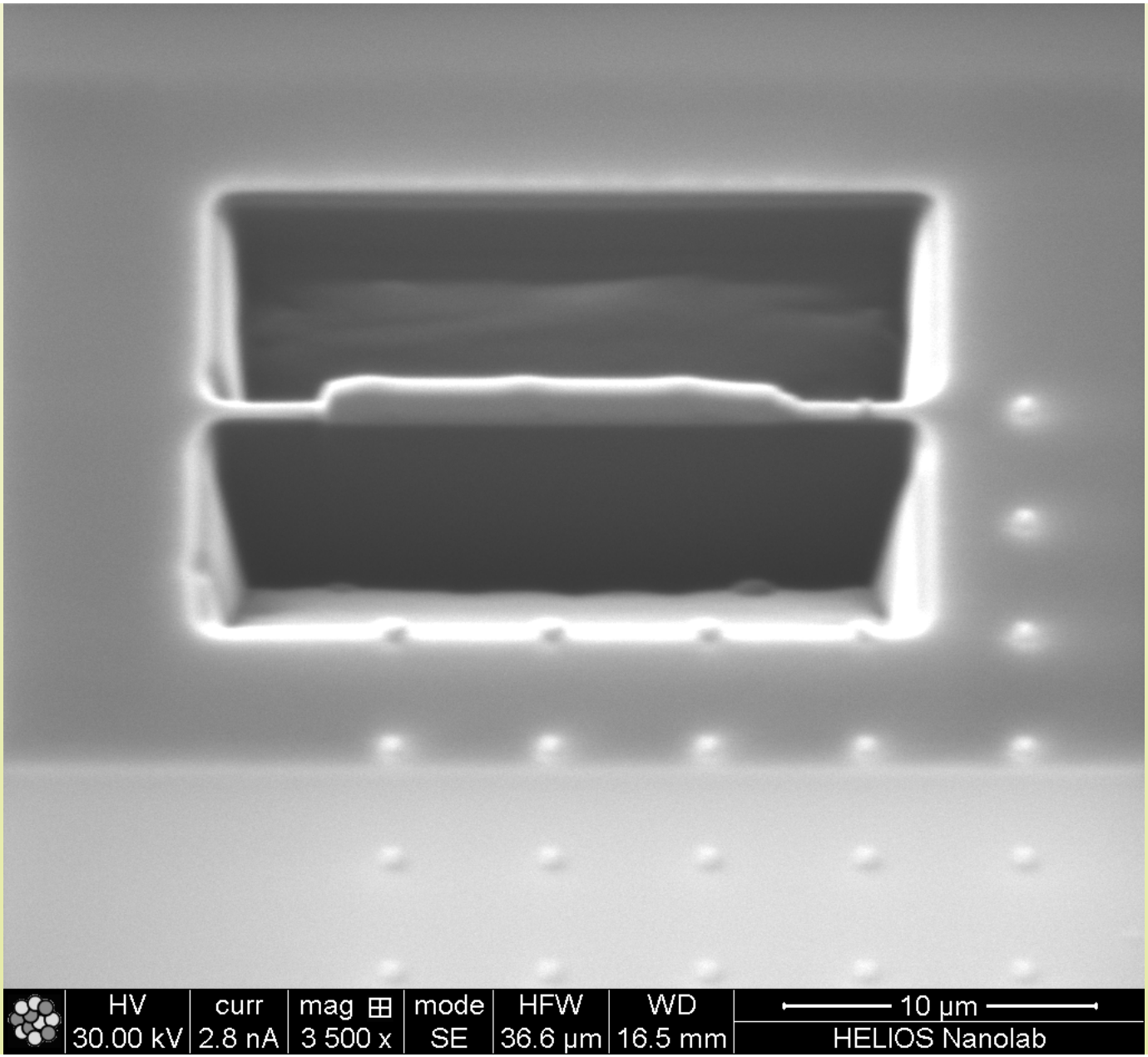

HV
5.00 kV
WD
3.8 mm
mag
5 000 x
HFW
25.6 µm
det
ETD
mode
SE
curr
22 nA
5 µm
HELIOS Nanolab

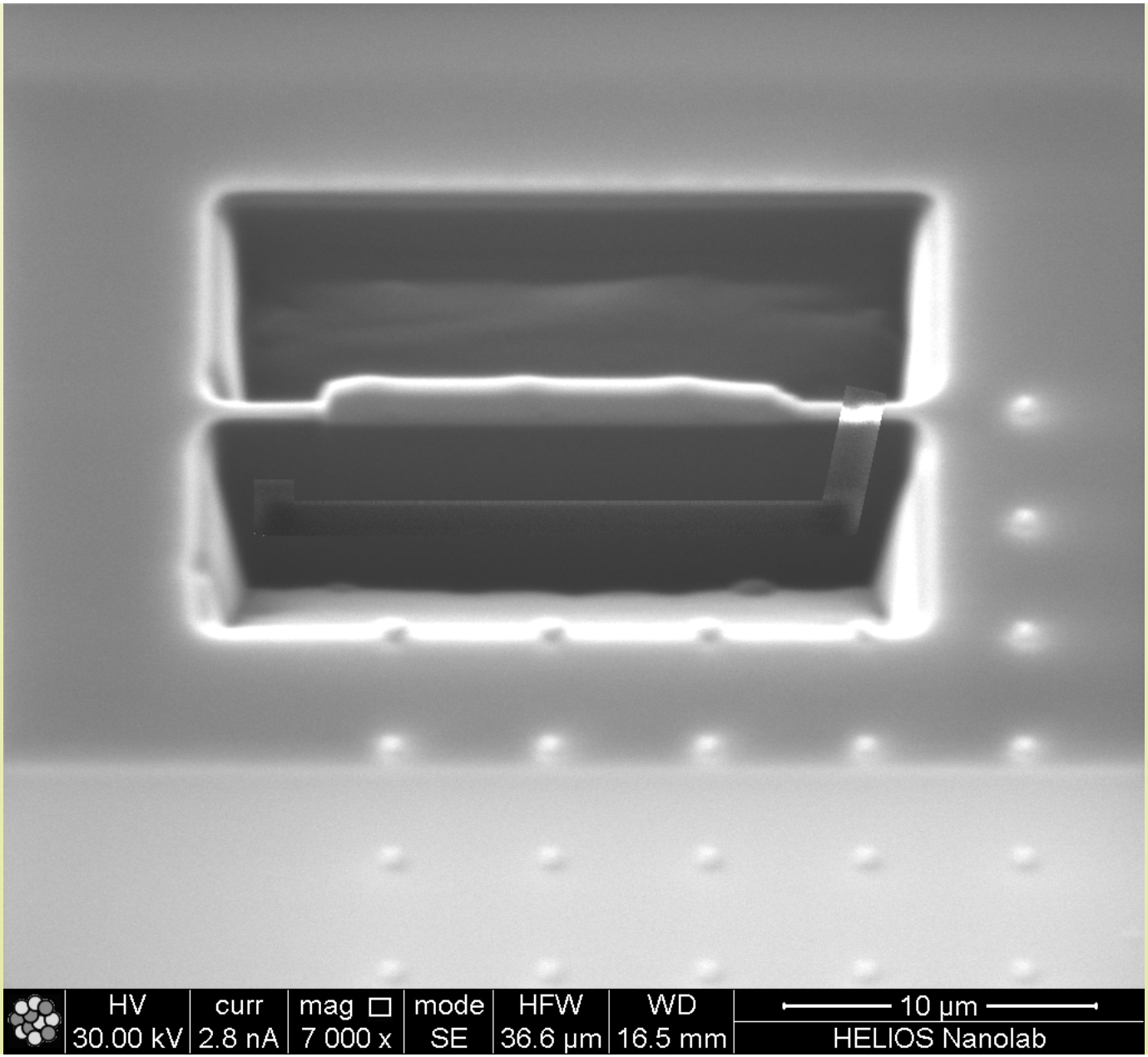

HV
5.00 kV
WD
3.8 mm
mag
5 000 x
HFW
25.6 µm
det
ETD
mode
SE
curr
22 nA
5 µm
HELIOS Nanolab

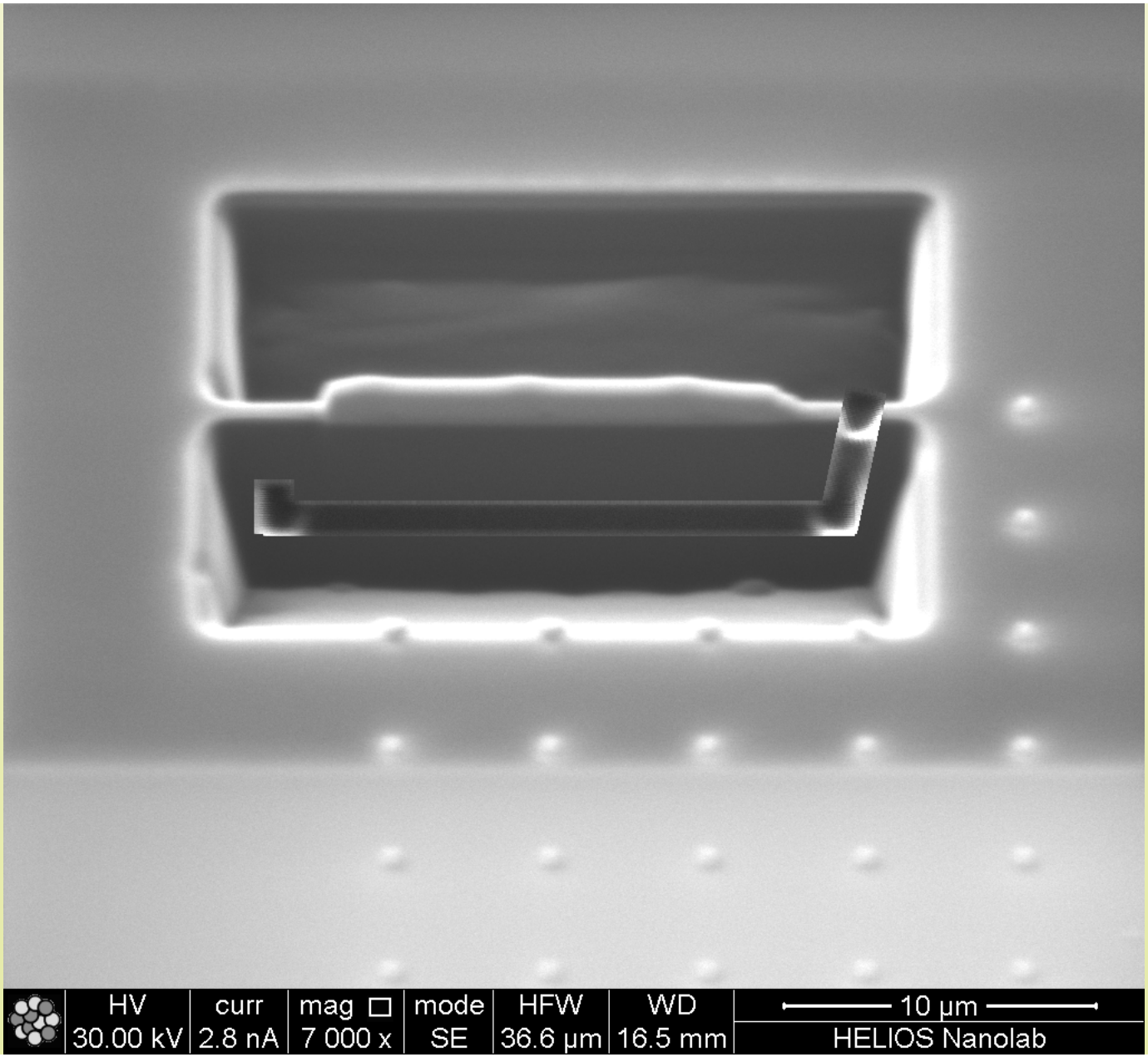

HV
30.00 kV
curr
6.5 nA
mag
3 500 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

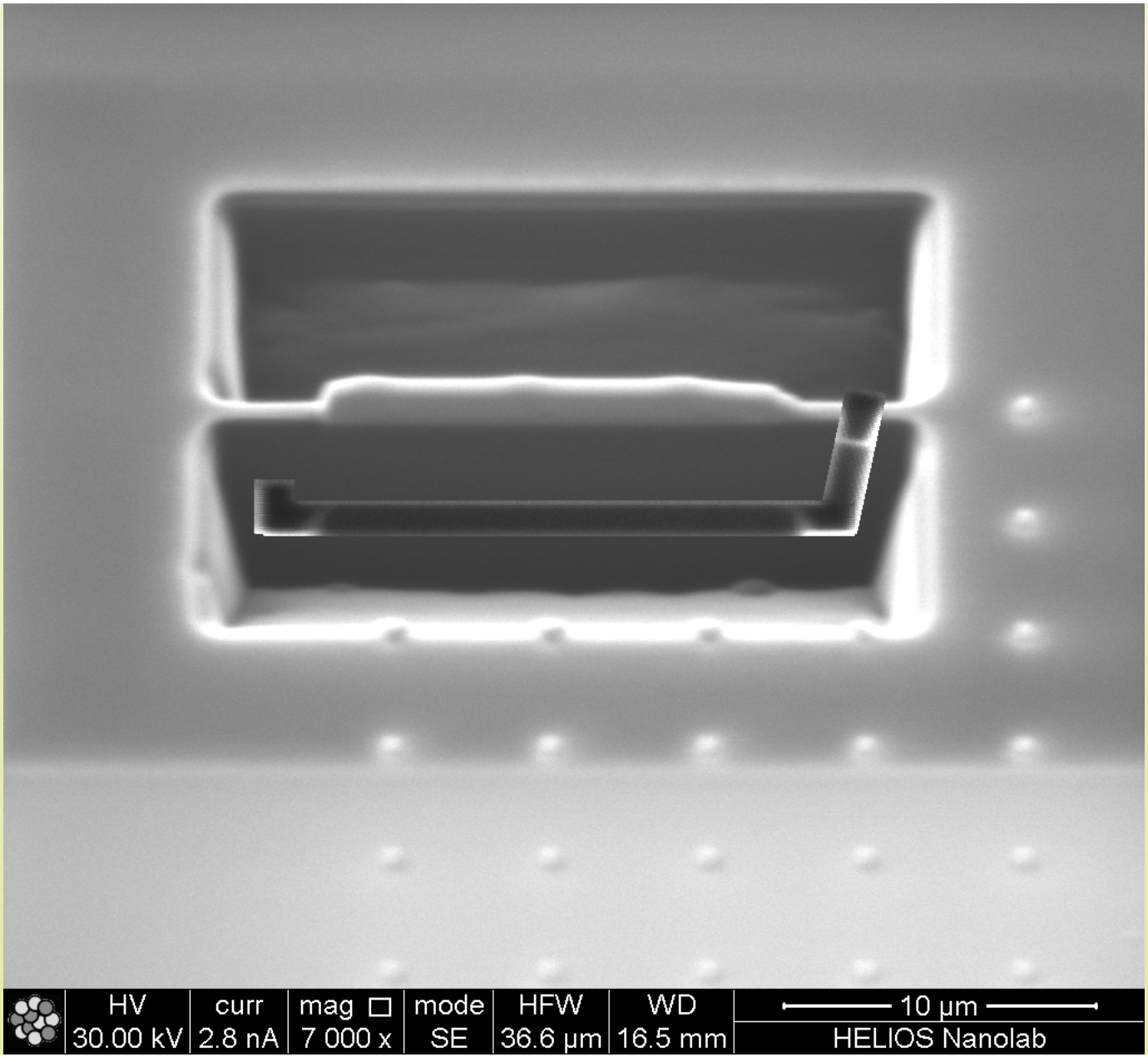

1
HV
30.00 kV
curr
6.5 nA
mag
3 500 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

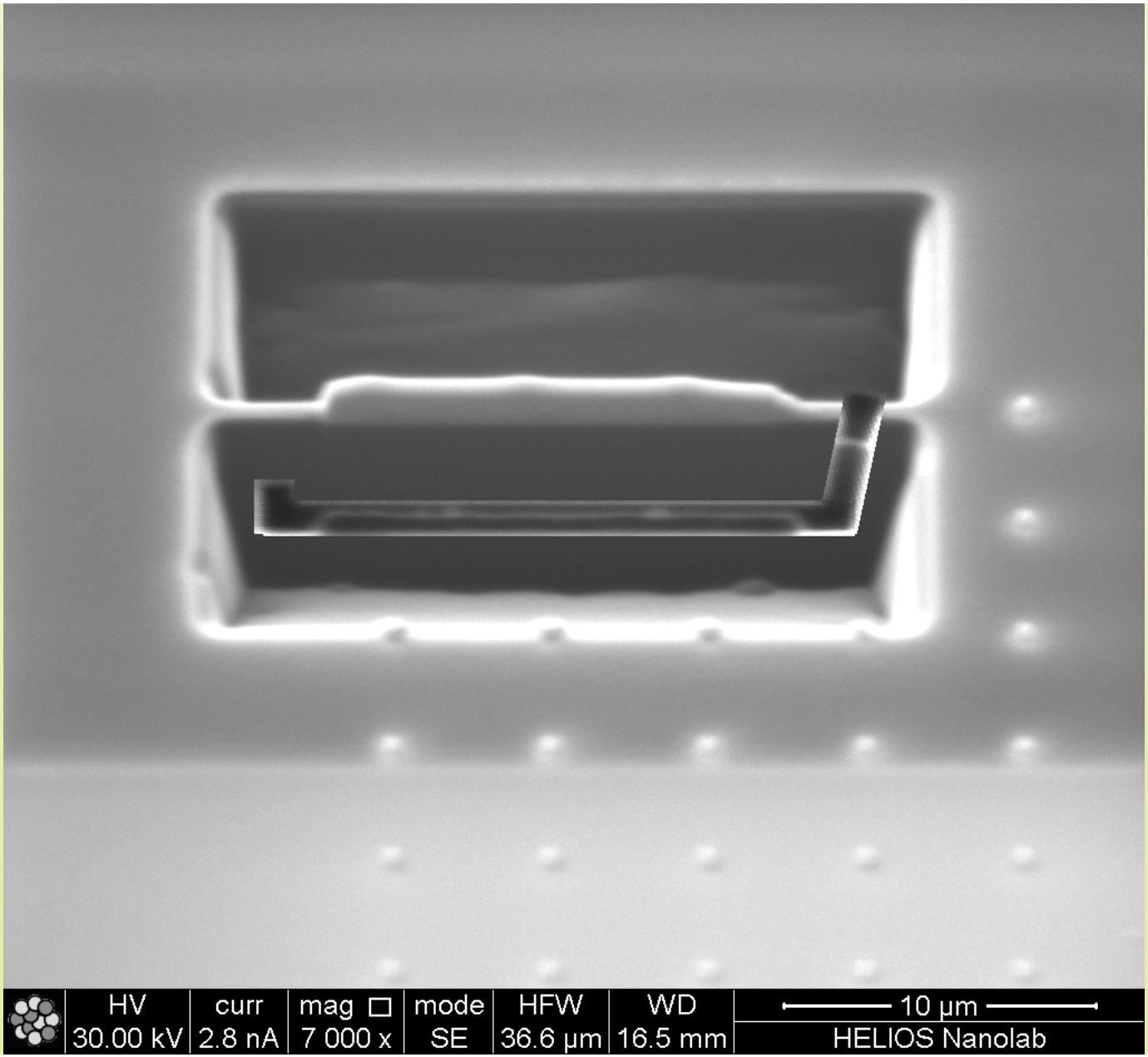

HV
30.00 kV
curr
6.5 nA
mag
3 500 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

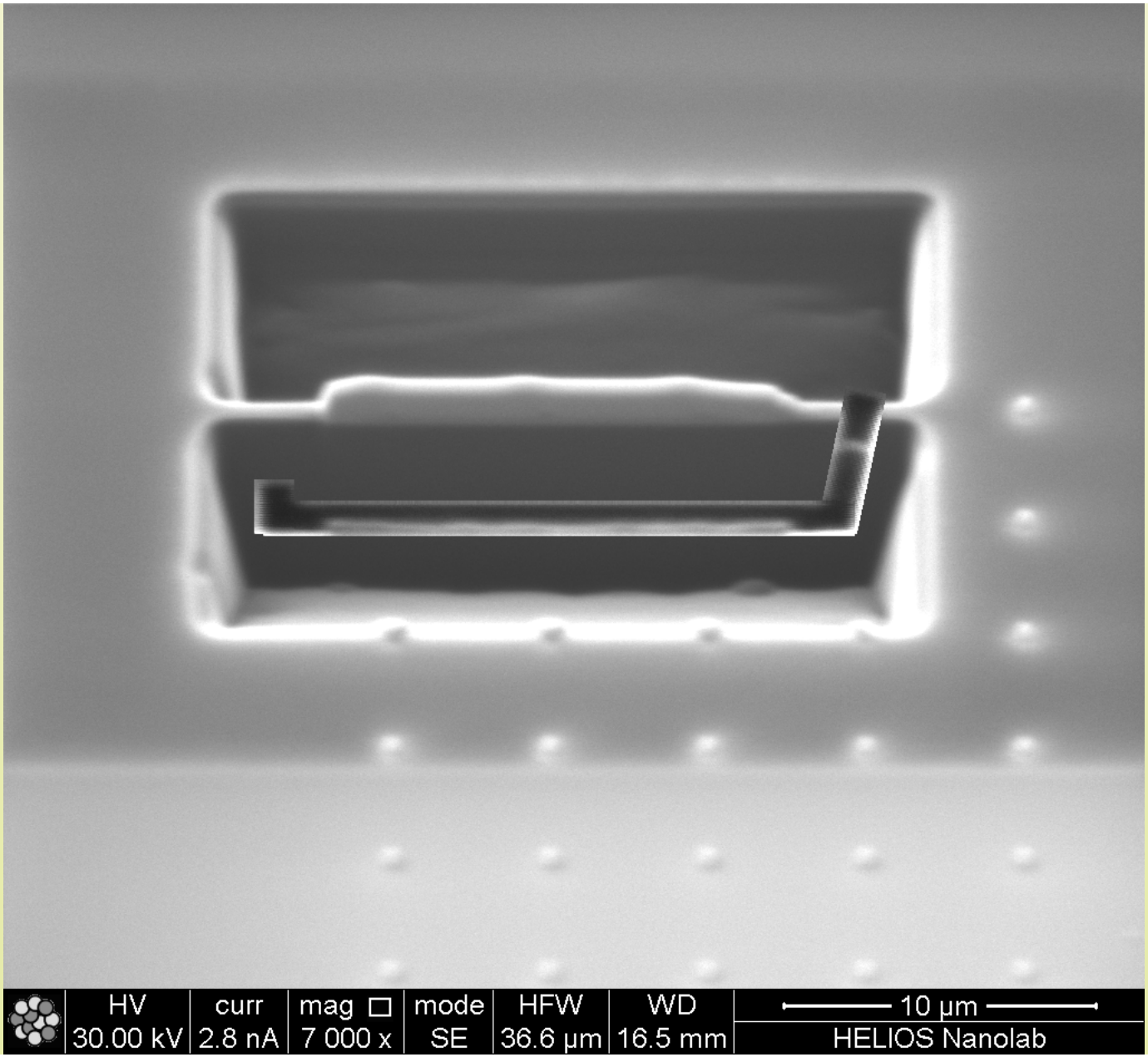

HV
30.00 kV
curr
2.8 nA
mag
3 500 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

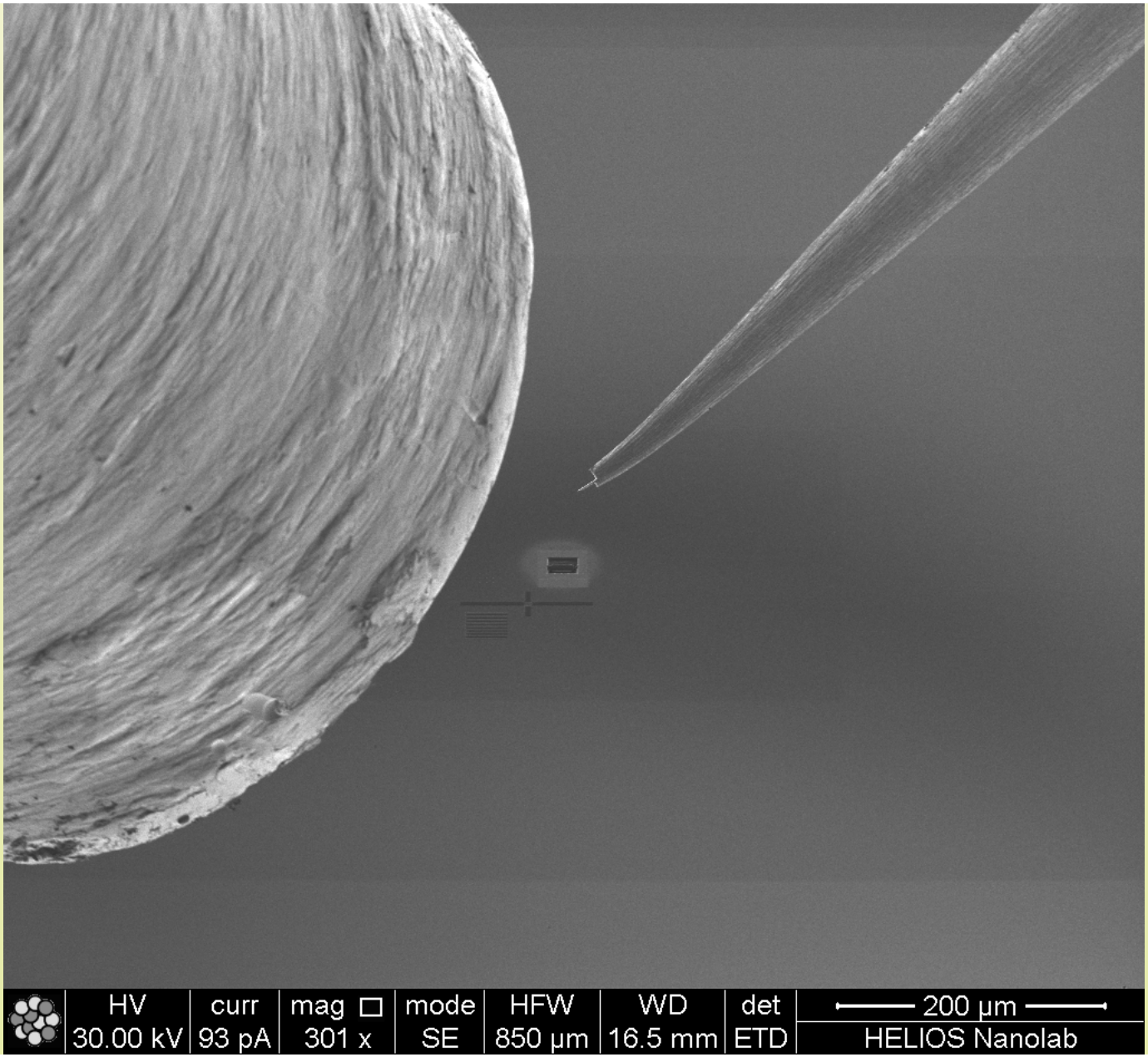

HV
30.00 kV
curr
2.8 nA
mag
7 000 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

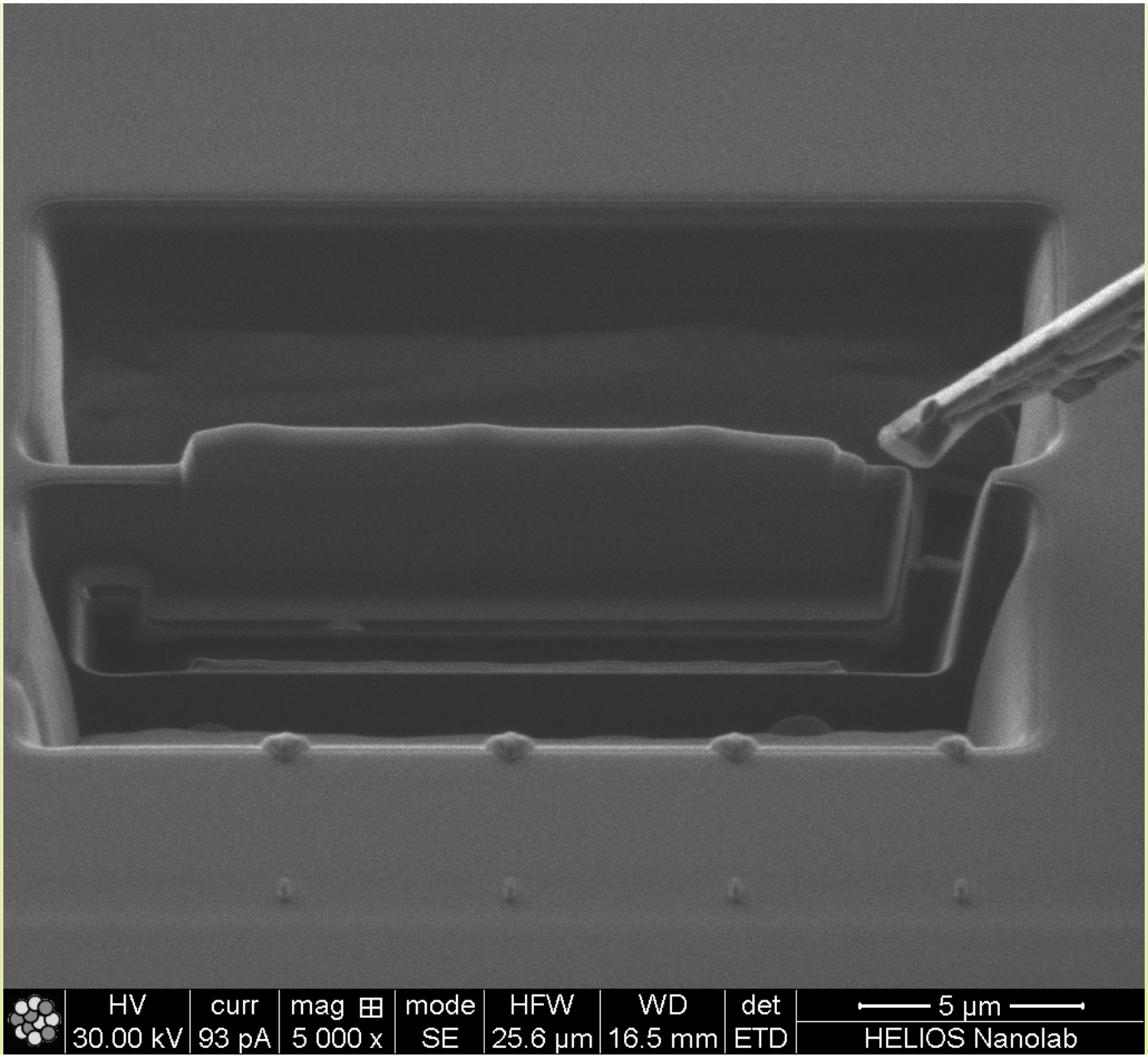

HV
30.00 kV
curr
2.8 nA
mag
7 000 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

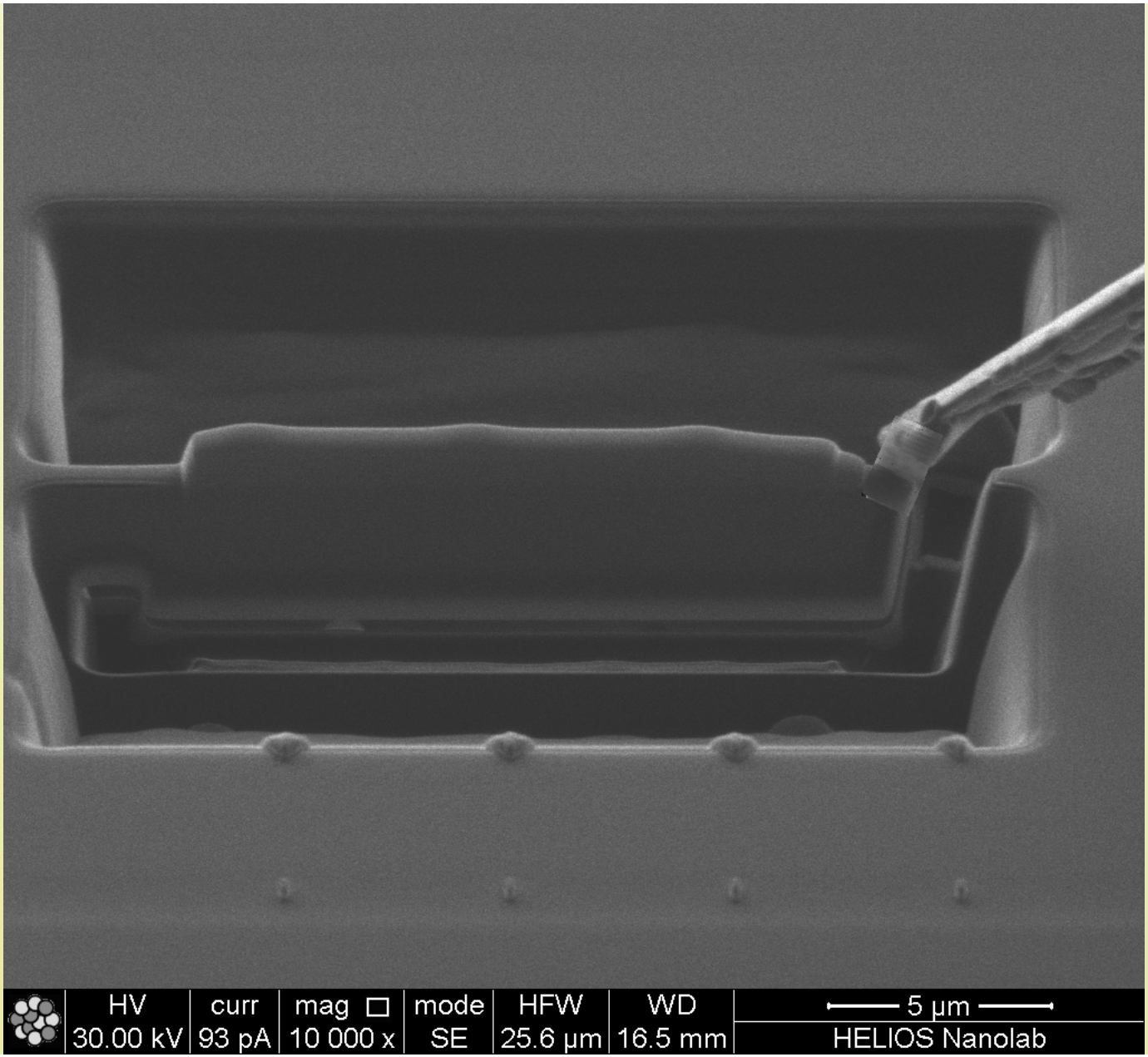

HV
30.00 kV
curr
2.8 nA
mag
7 000 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

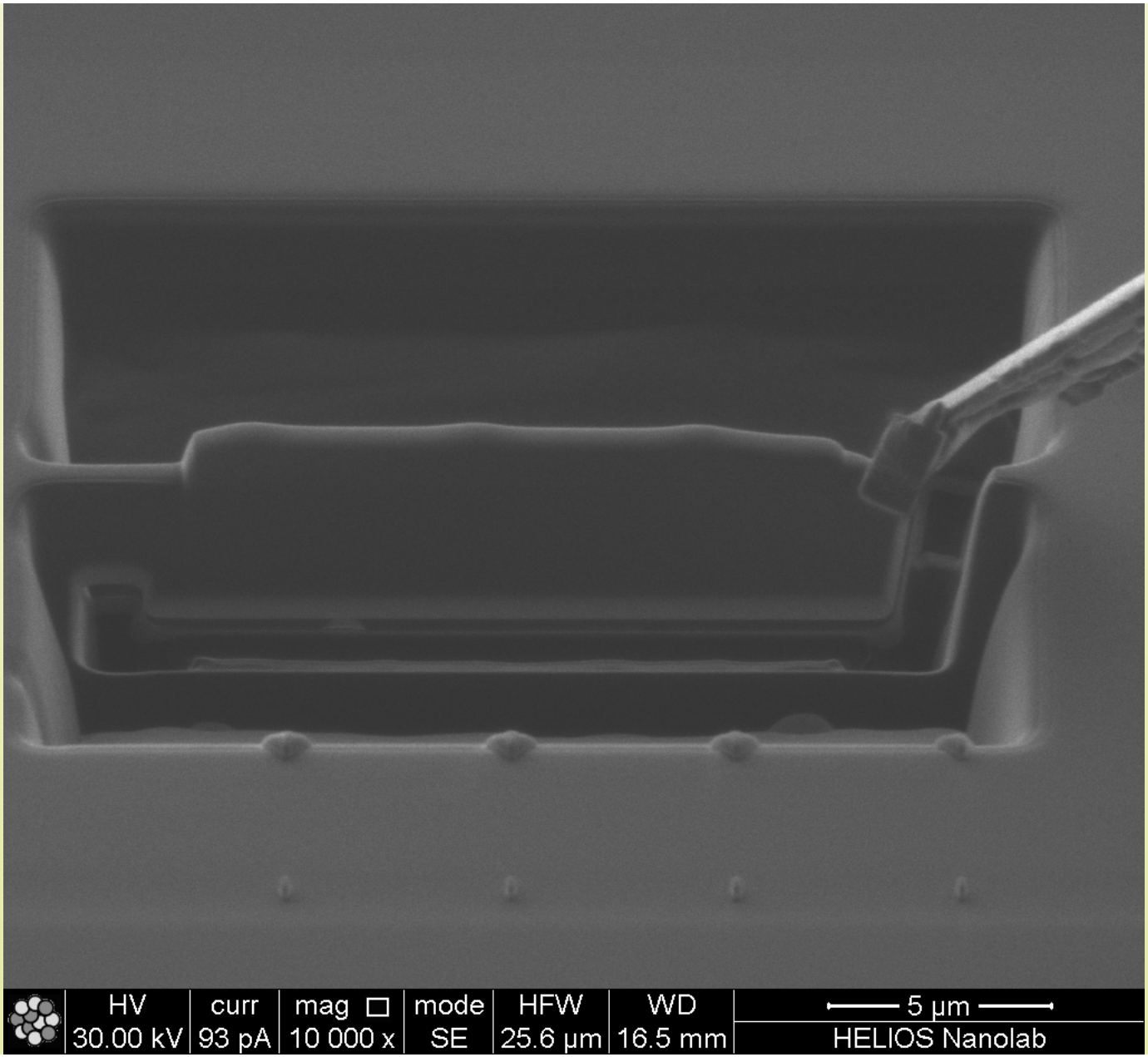

HV
30.00 kV
curr
2.8 nA
mag
7 000 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

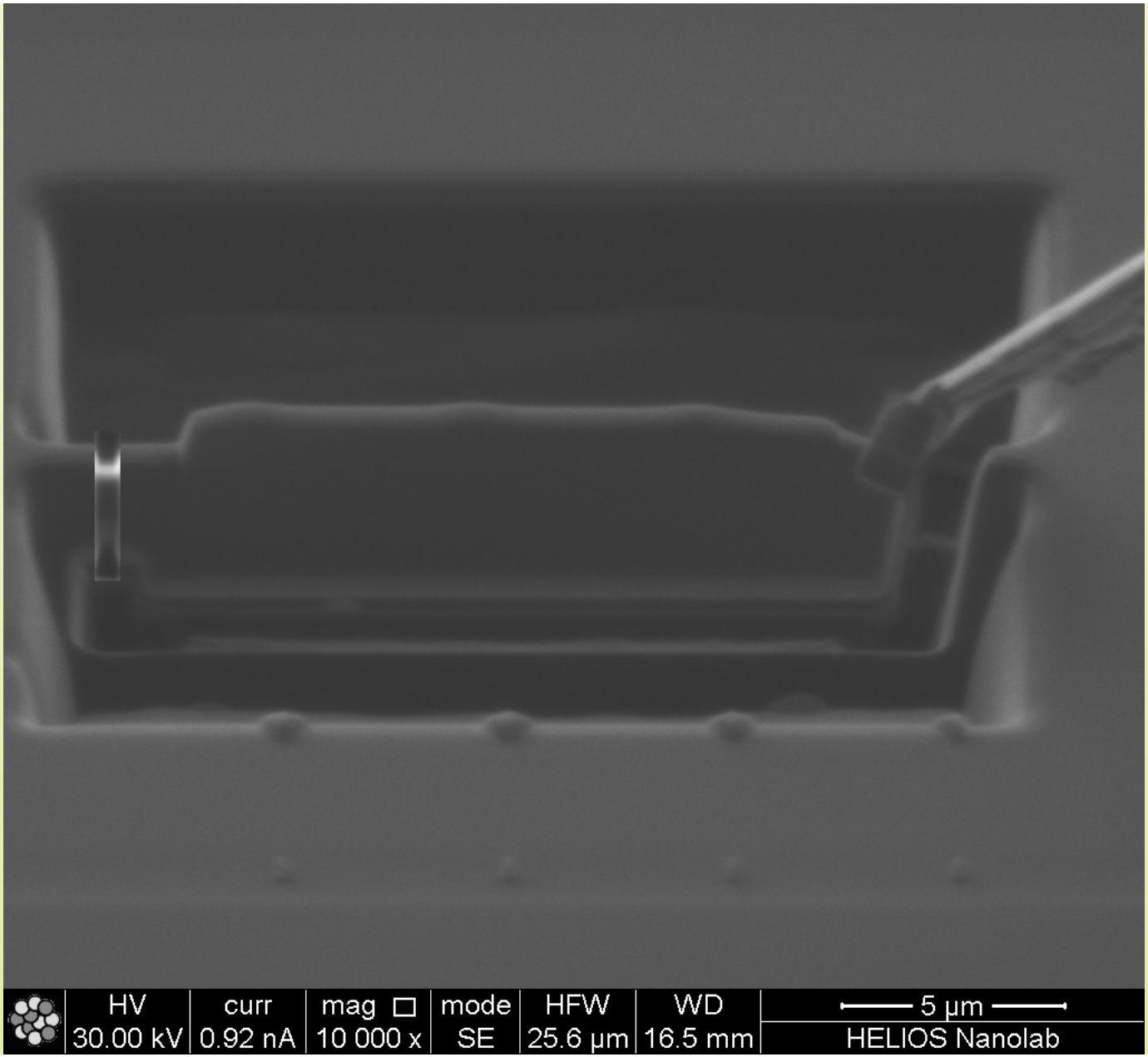

HV
30.00 kV
curr
2.8 nA
mag
7 000 x
mode
SE
HFW
36.6 µm
WD
16.5 mm
10 µm
HELIOS Nanolab

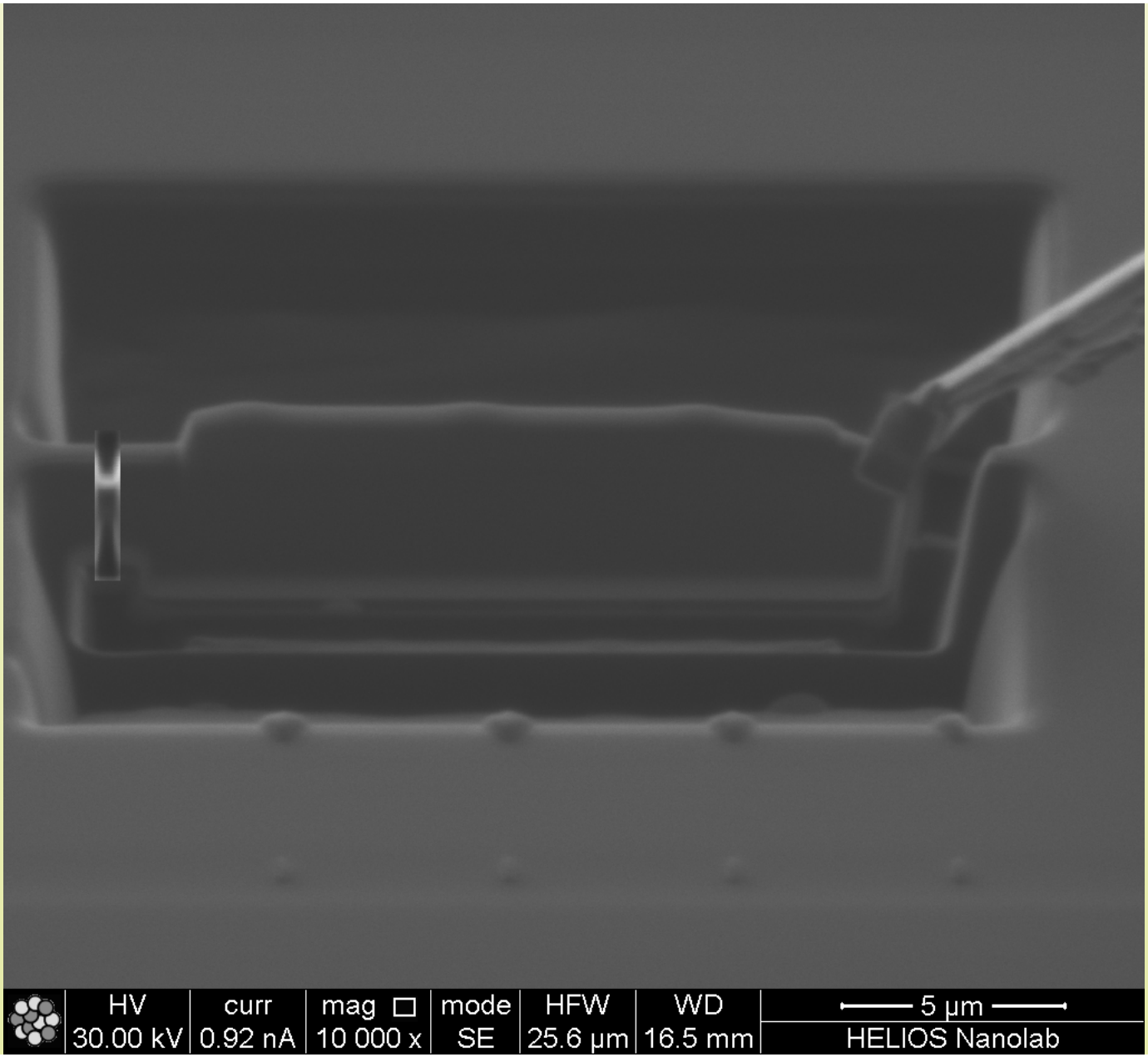

HV
30.00 kV
curr
93 pA
mag
301 x
mode
SE
HFW
850 µm
WD
16.5 mm
det
ETD
200 µm
HELIOS Nanolab

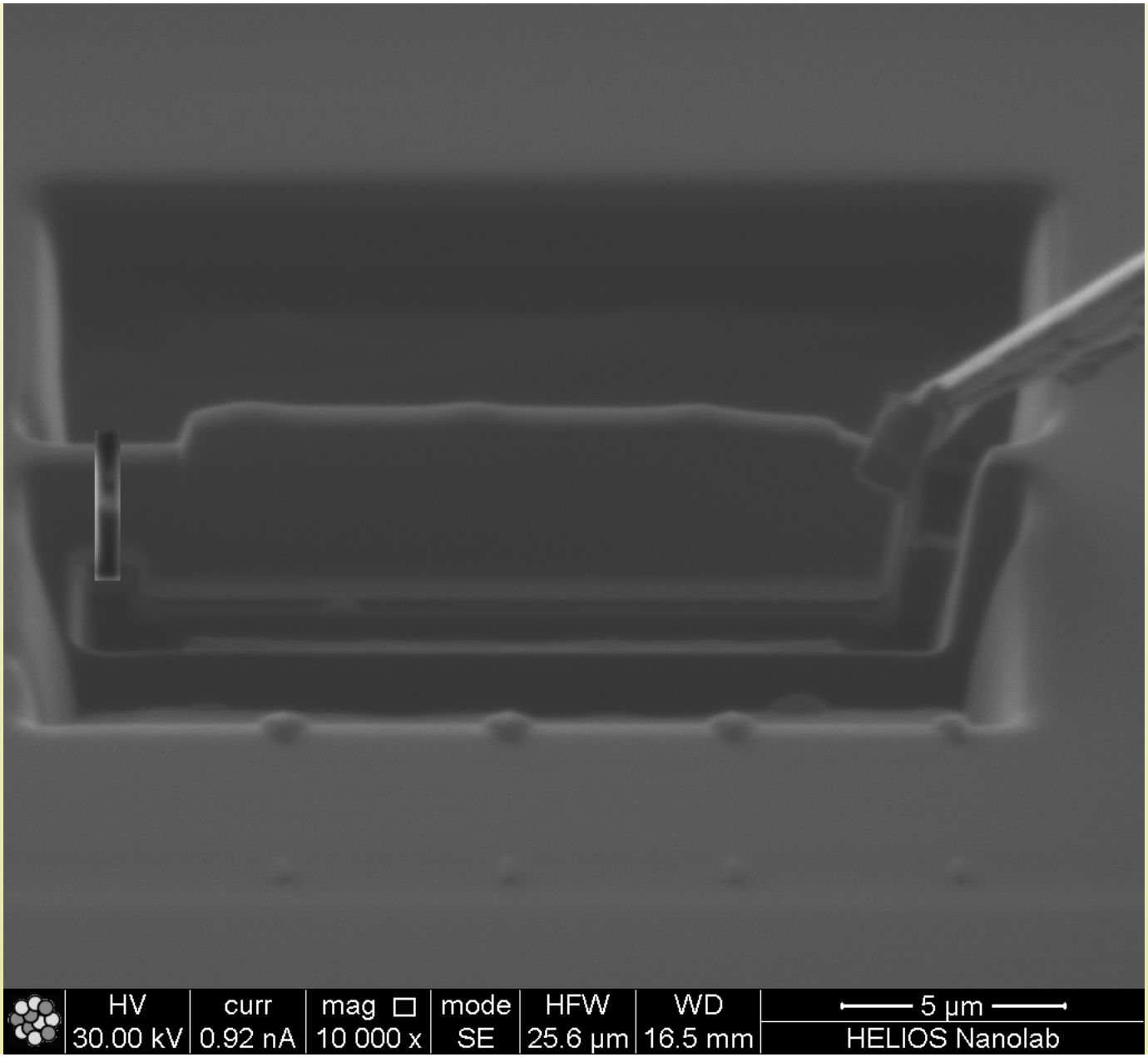

HV
30.00 kV
curr
93 pA
mag
5 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
det
ETD
5 µm
HELIOS Nanolab

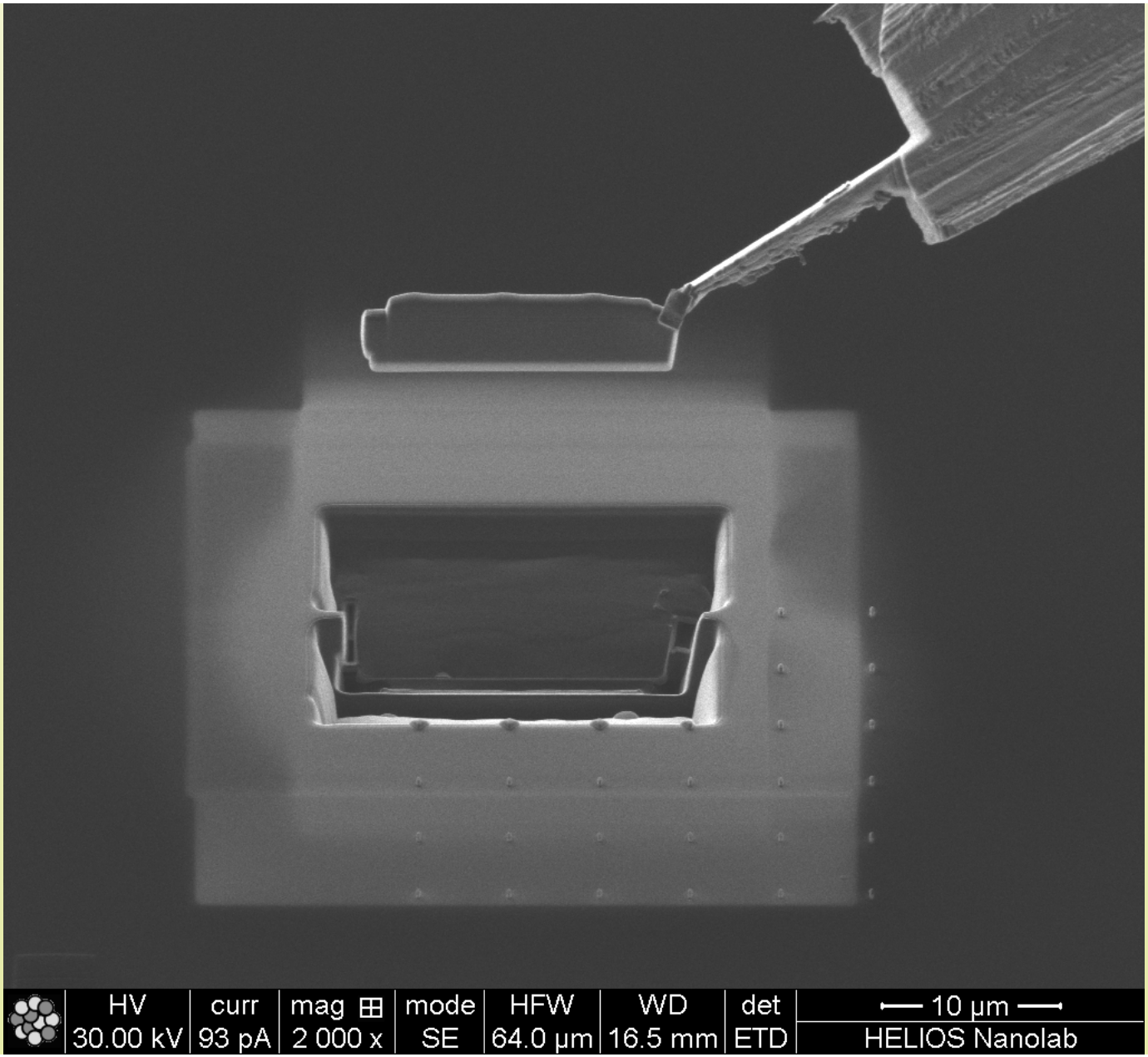

HV
30.00 kV
curr
93 pA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

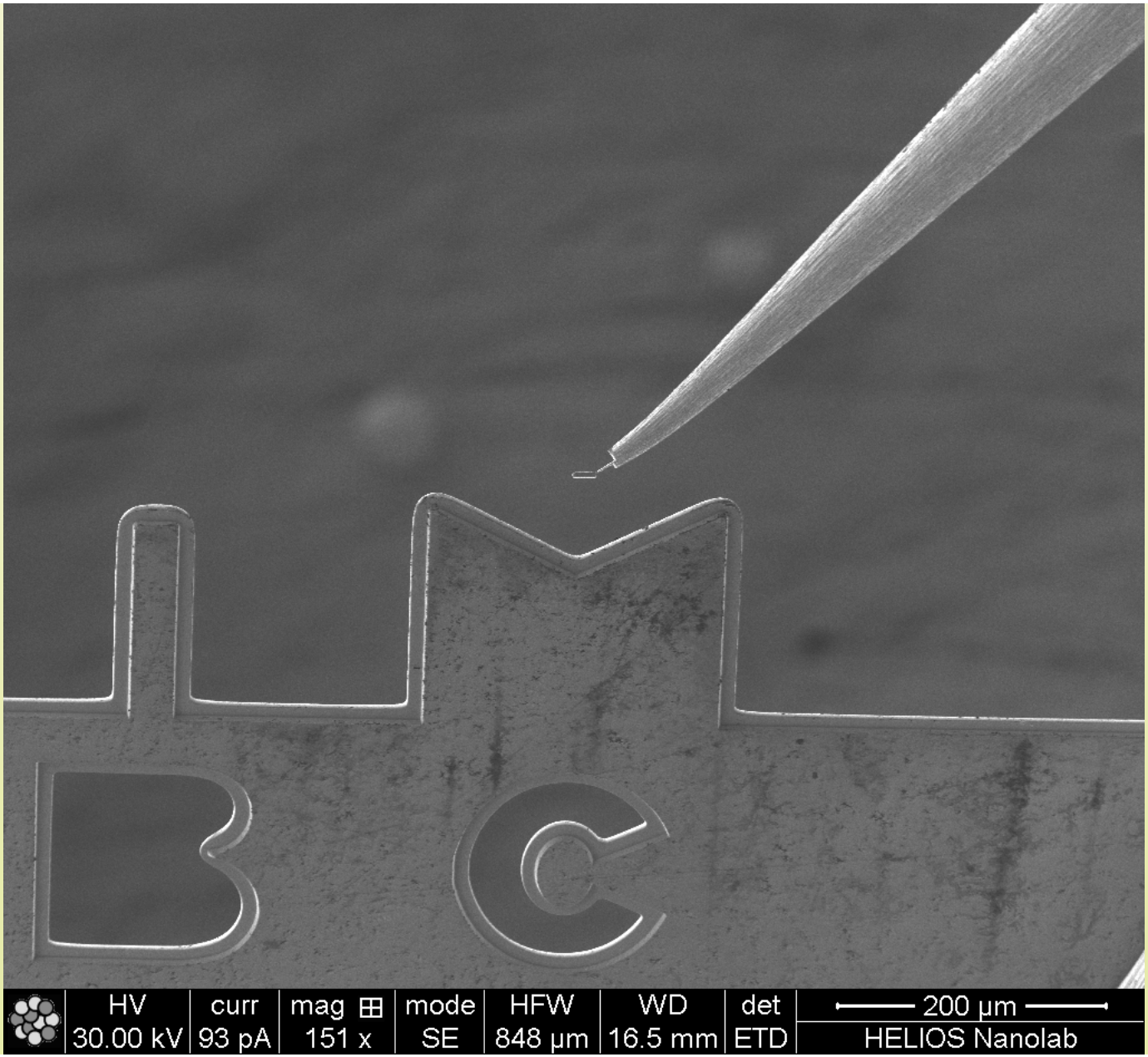

HV
30.00 kV
curr
93 pA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

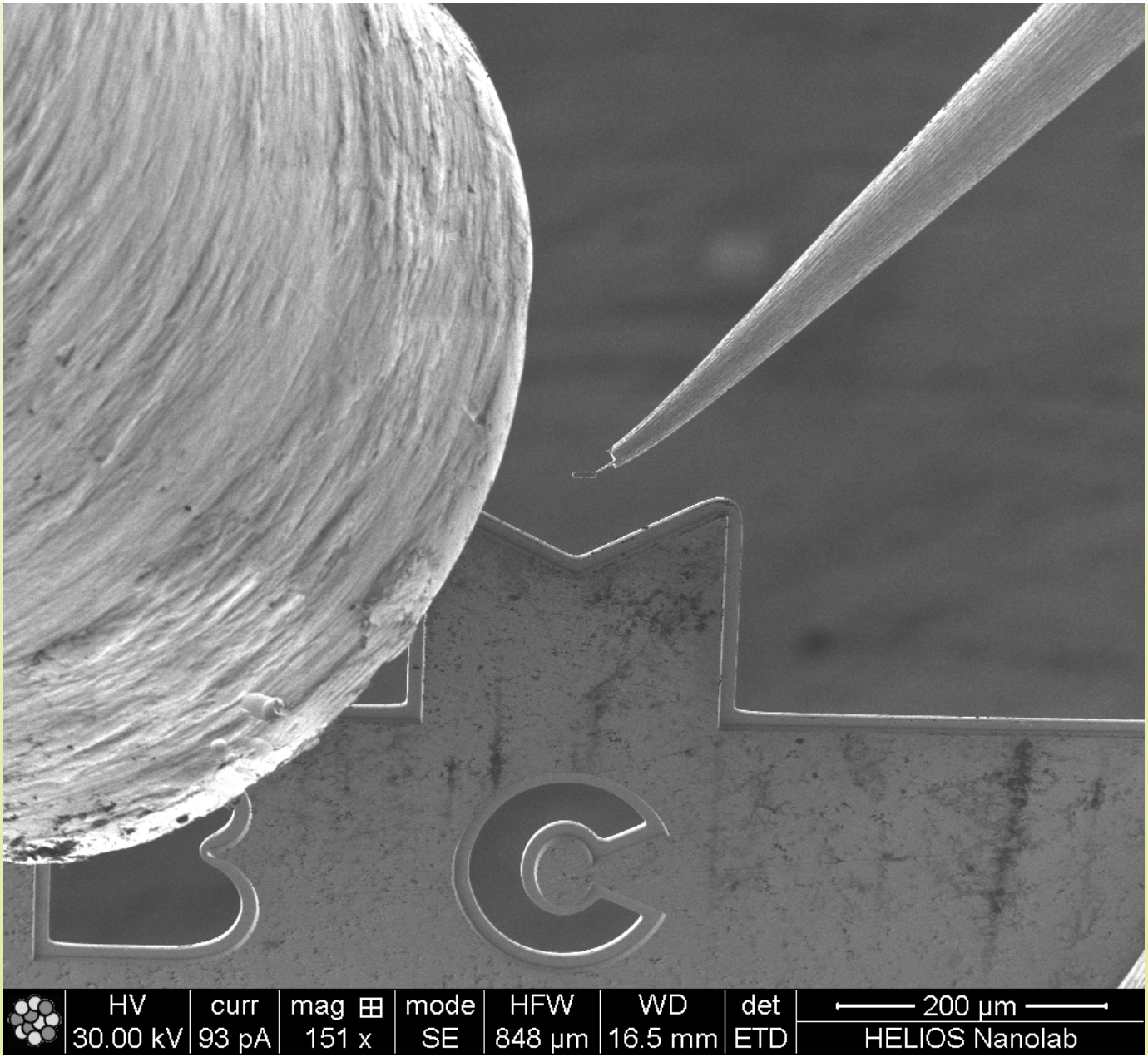

HV
30.00 kV
curr
0.92 nA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

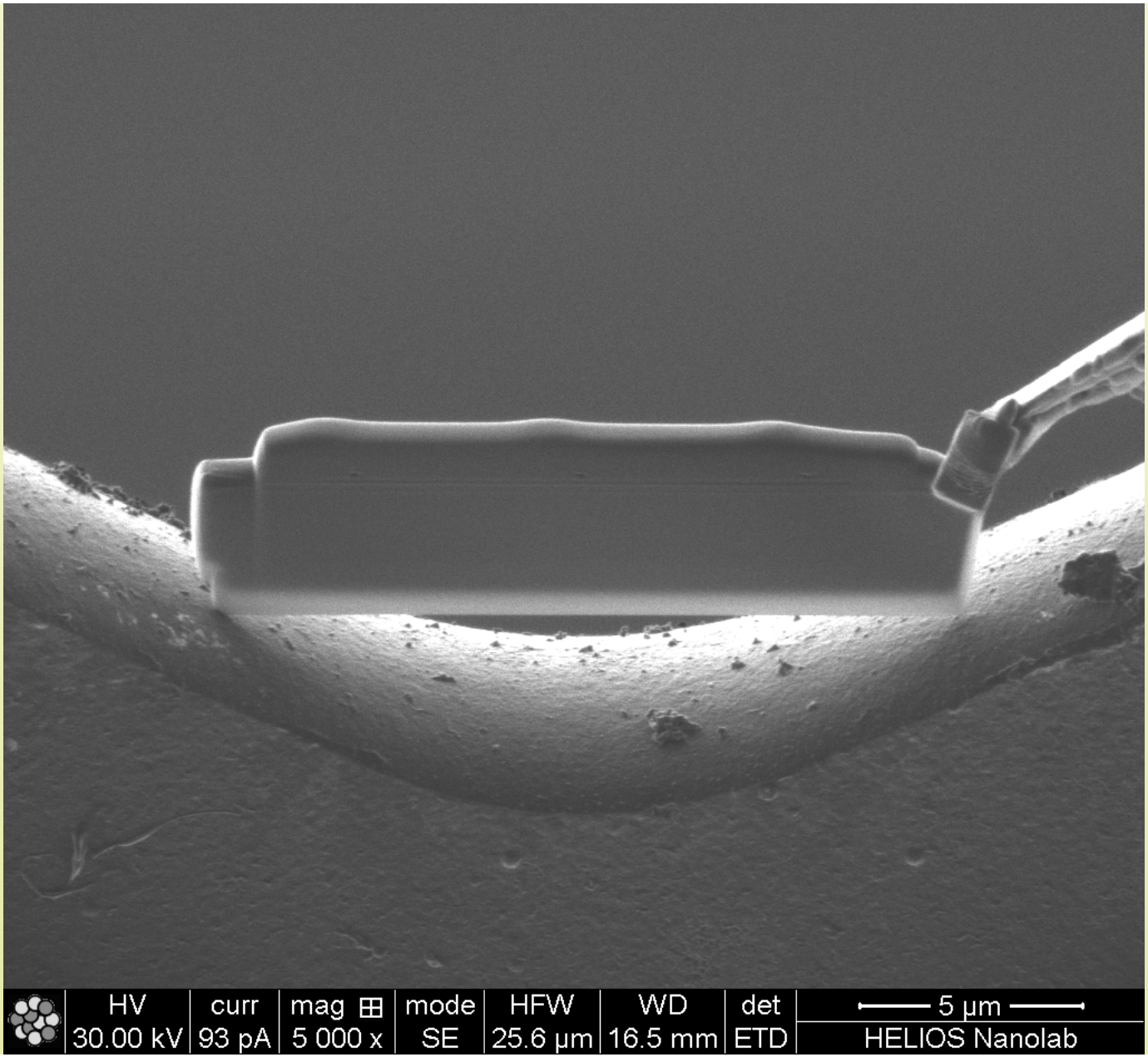

HV
30.00 kV
curr
0.92 nA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

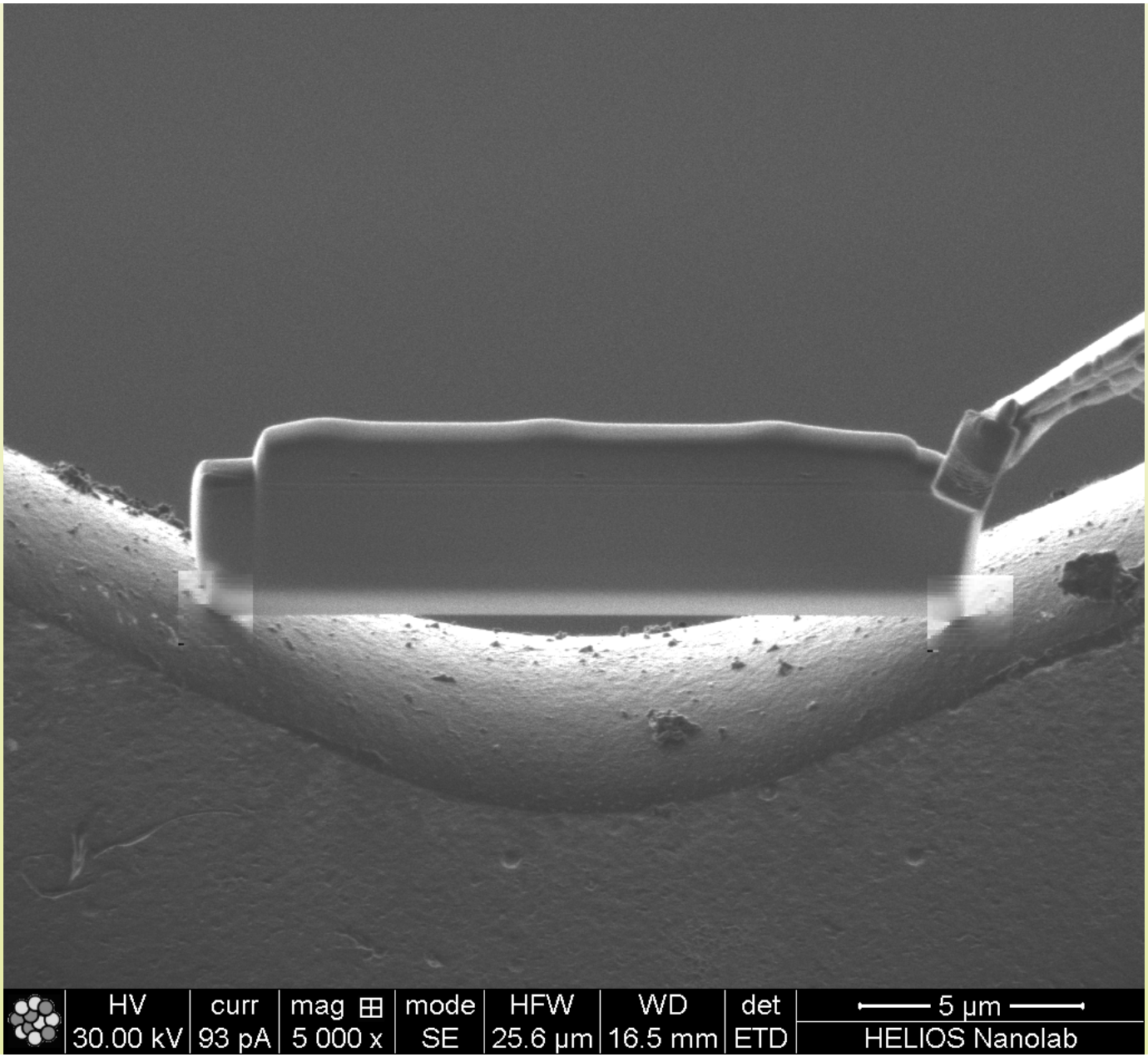

HV
30.00 kV
curr
0.92 nA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

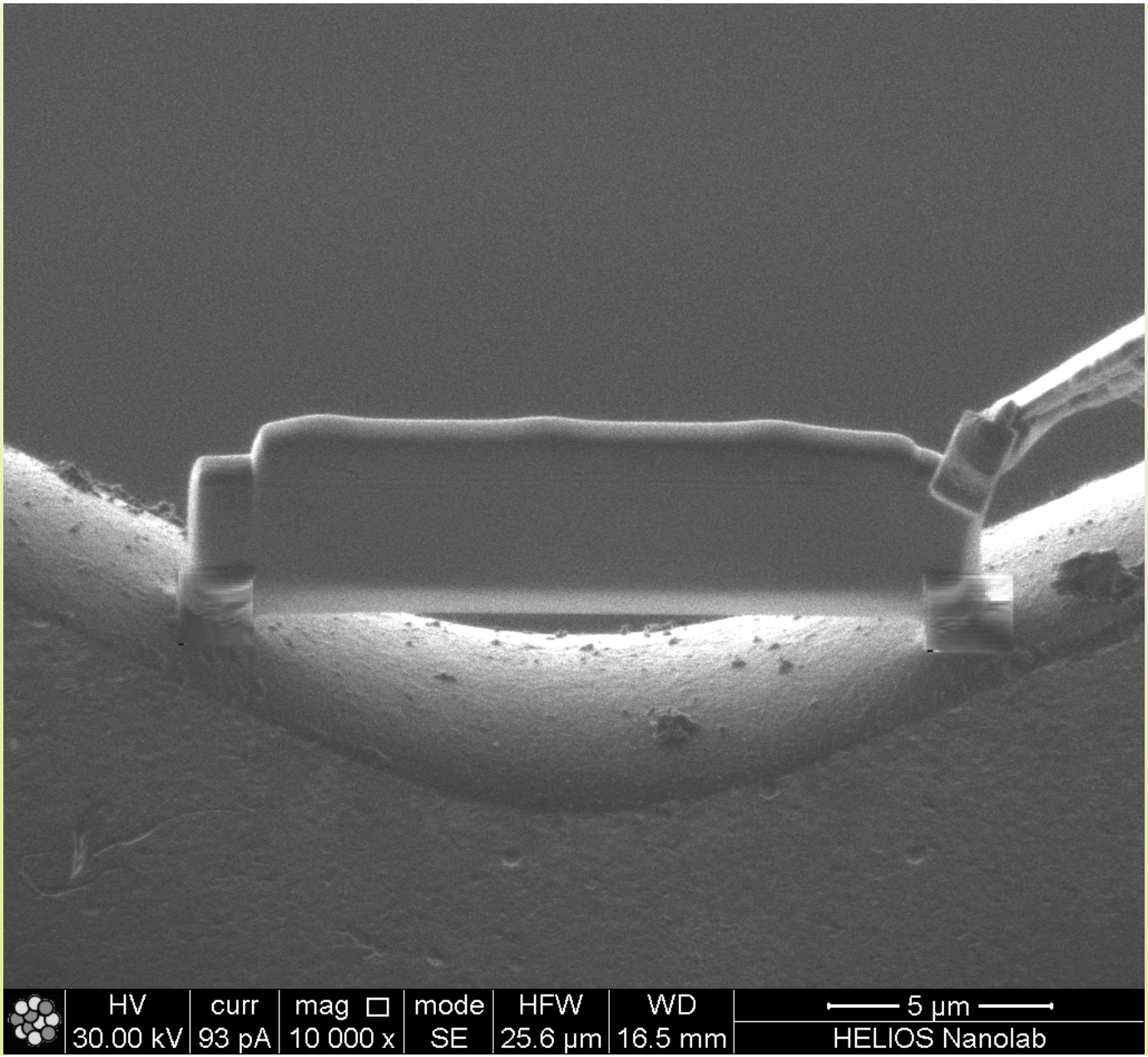

HV
30.00 kV
curr
93 pA
mag
2 000 x
mode
SE
HFW
64.0 µm
WD
16.5 mm
det
ETD
10 µm
HELIOS Nanolab

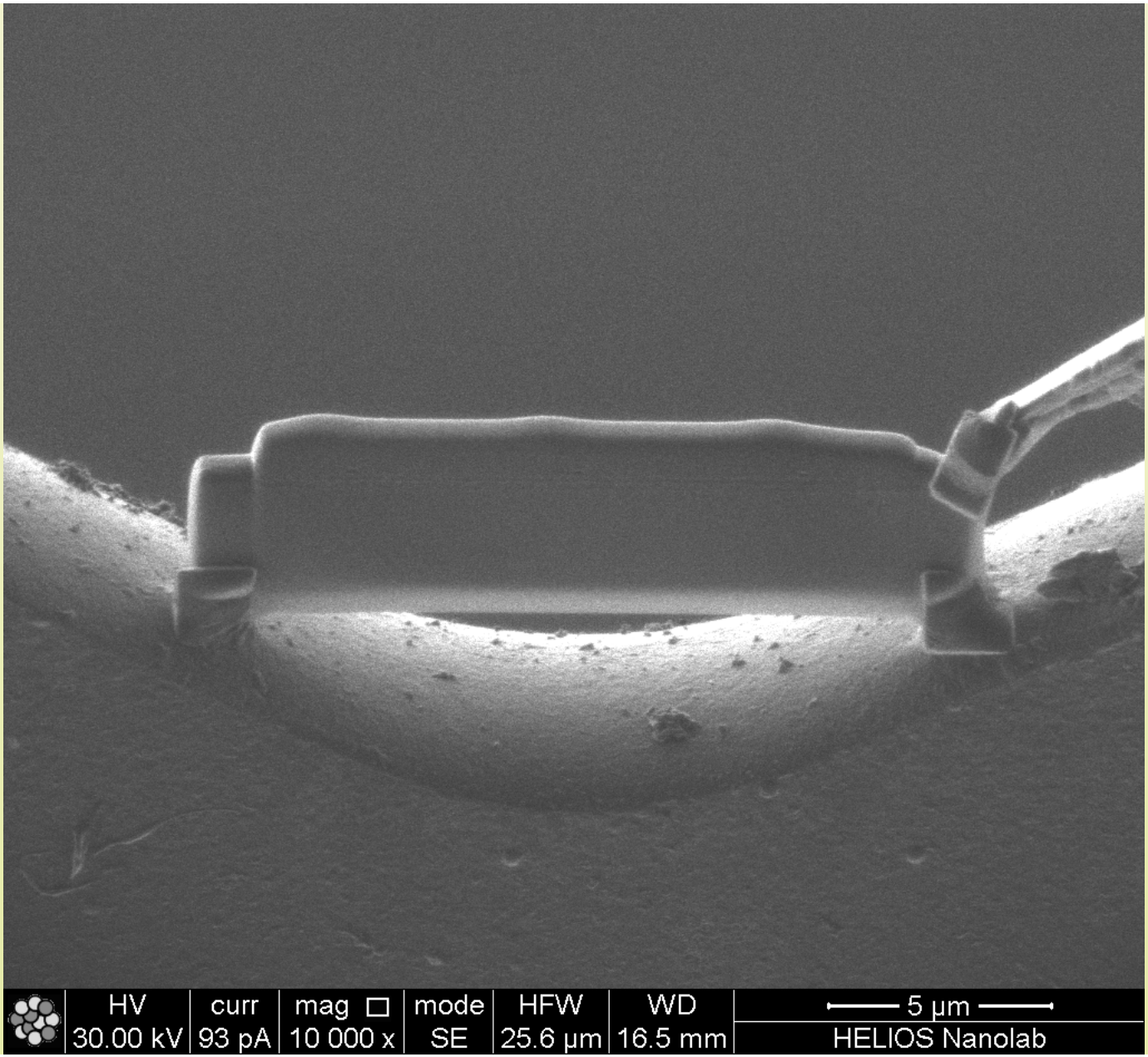

HV
30.00 kV
curr
93 pA
mag
151 x
mode
SE
HFW
848 µm
WD
16.5 mm
det
ETD
200 µm
HELIOS Nanolab

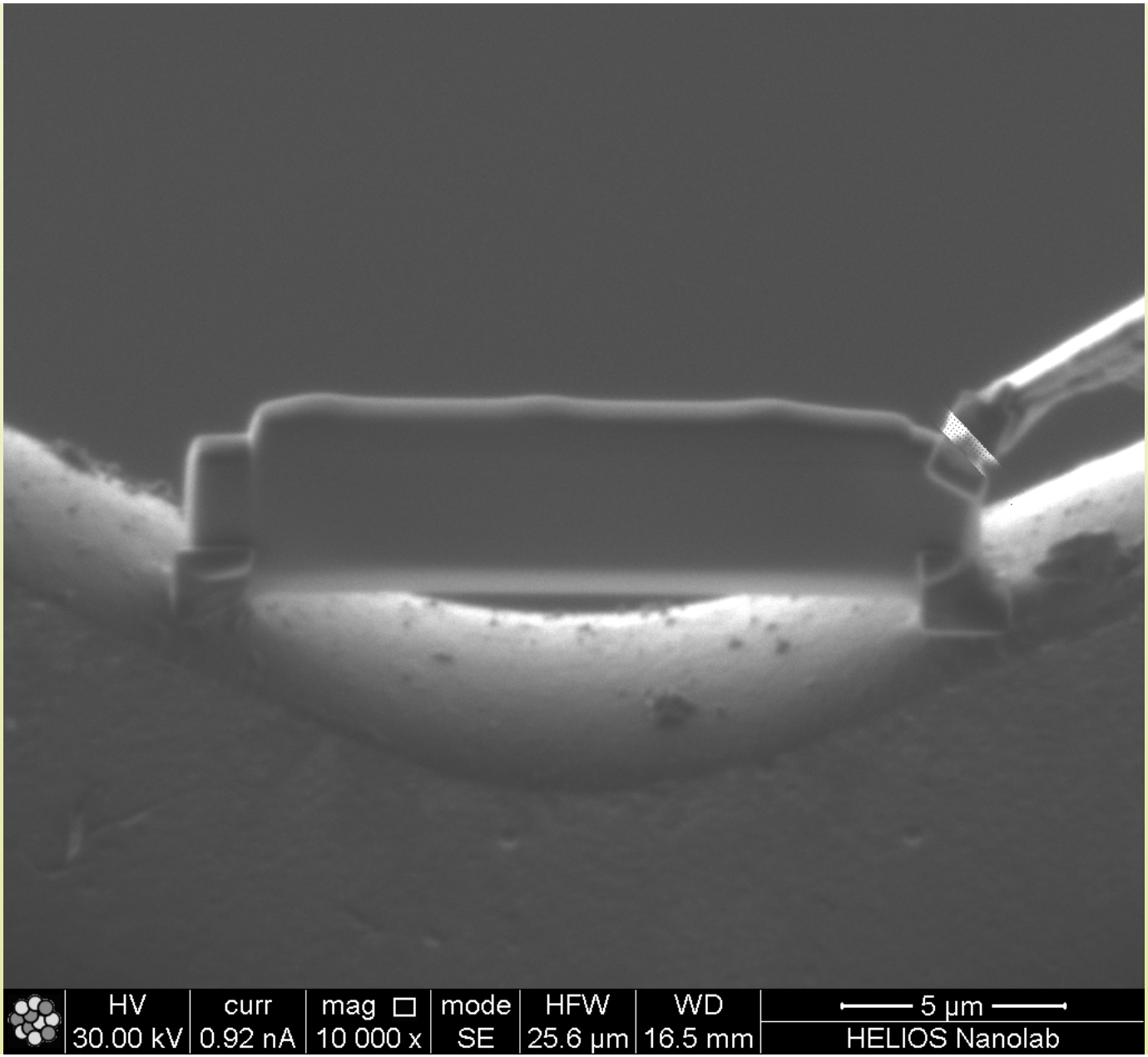

HV
30.00 kV
curr
93 pA
mag
151 x
mode
SE
HFW
848 µm
WD
16.5 mm
det
ETD
200 µm
HELIOS Nanolab

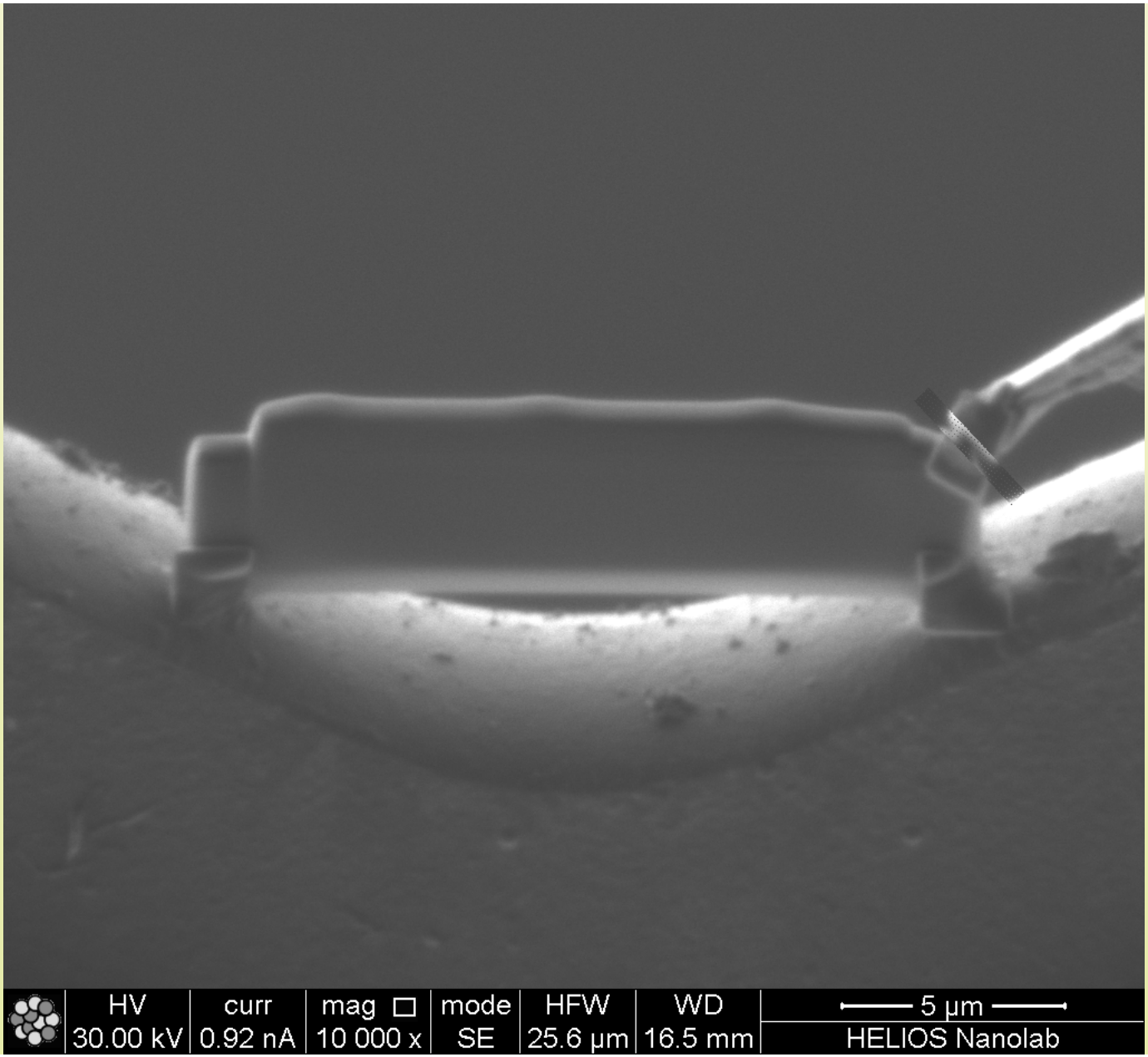

HV
30.00 kV
curr
93 pA
mag
5 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
det
ETD
5 µm
HELIOS Nanolab

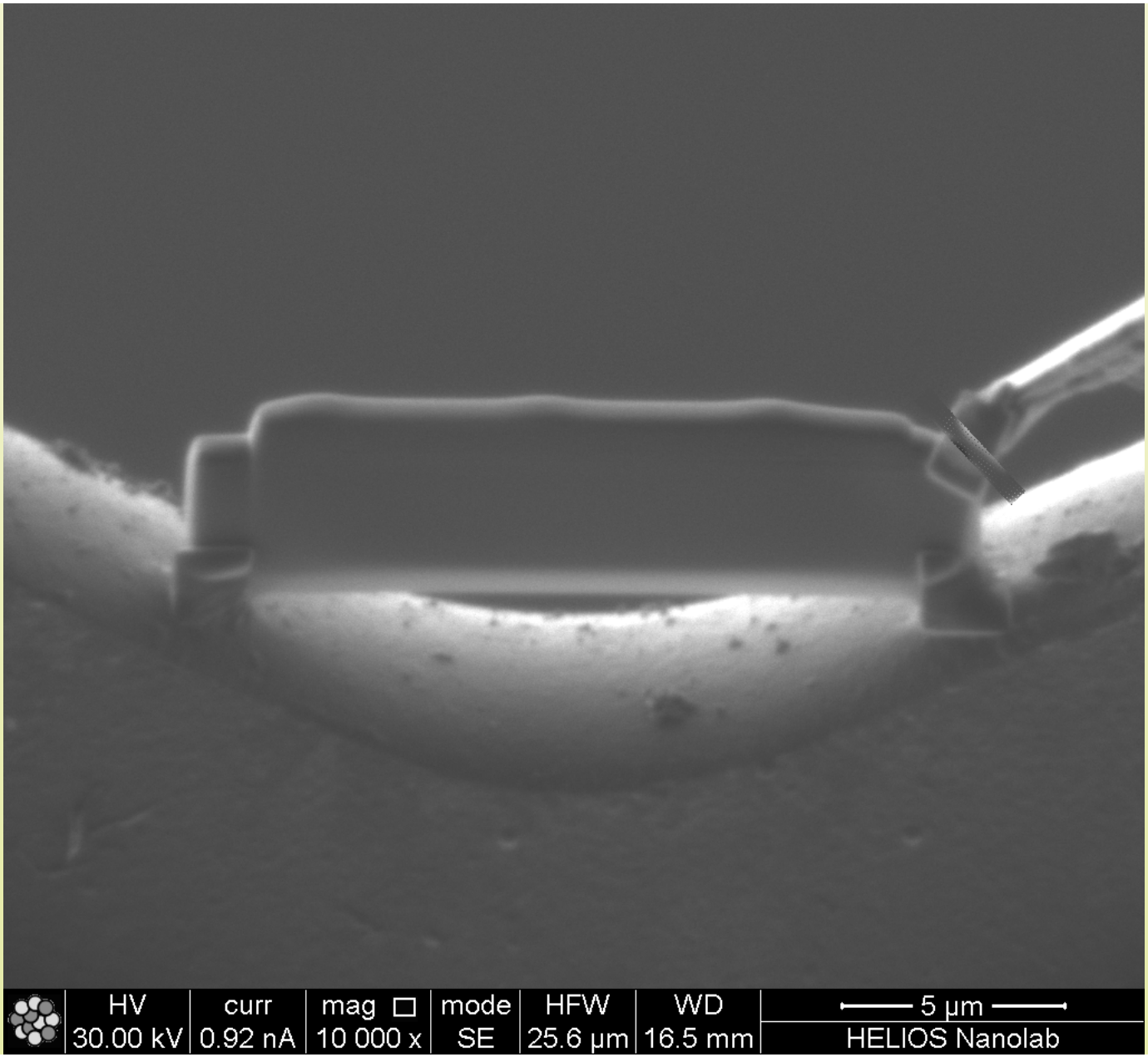

HV
30.00 kV
curr
93 pA
mag
5 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
det
ETD
5 µm
HELIOS Nanolab

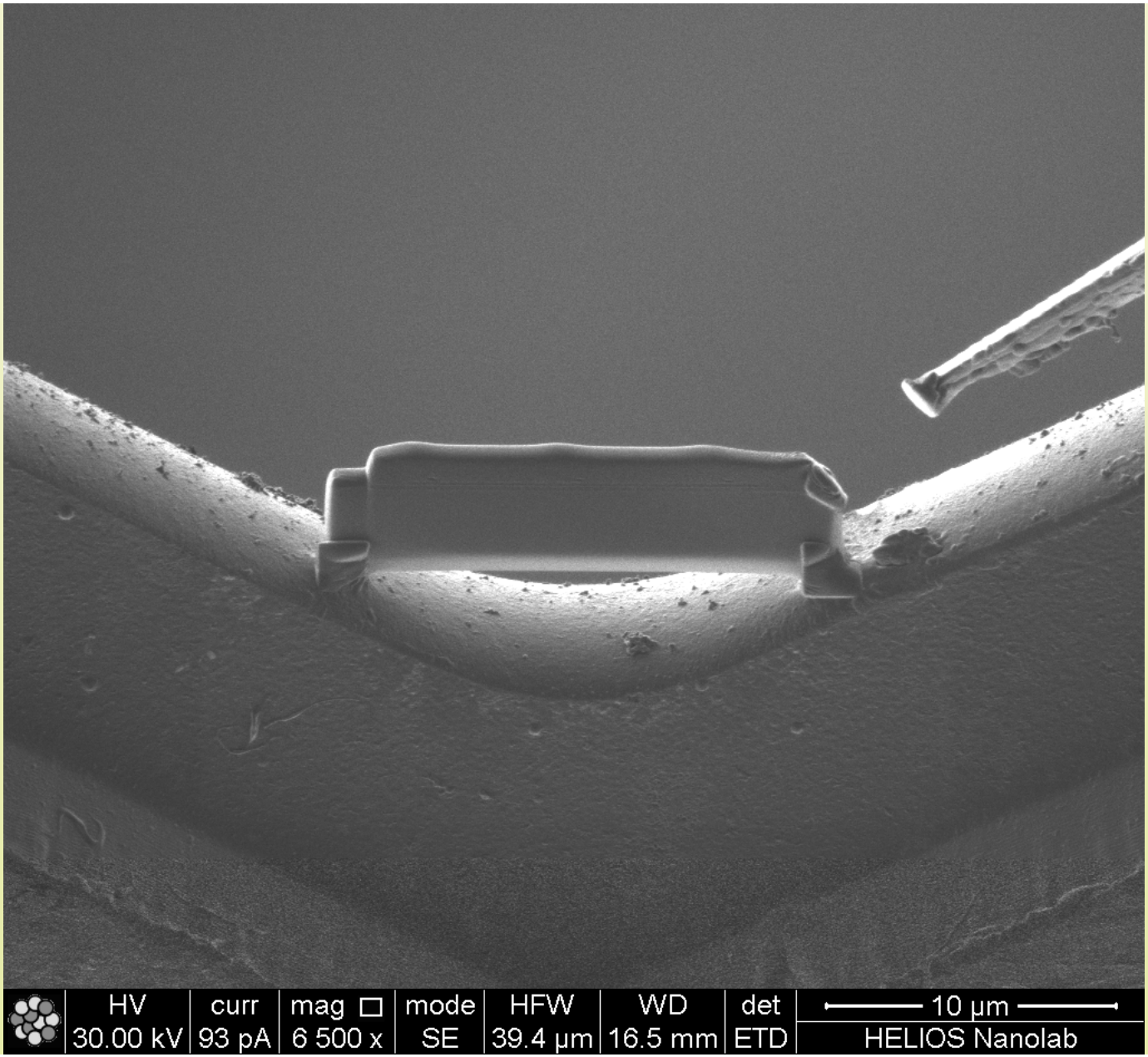

HV
30.00 kV
curr
93 pA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

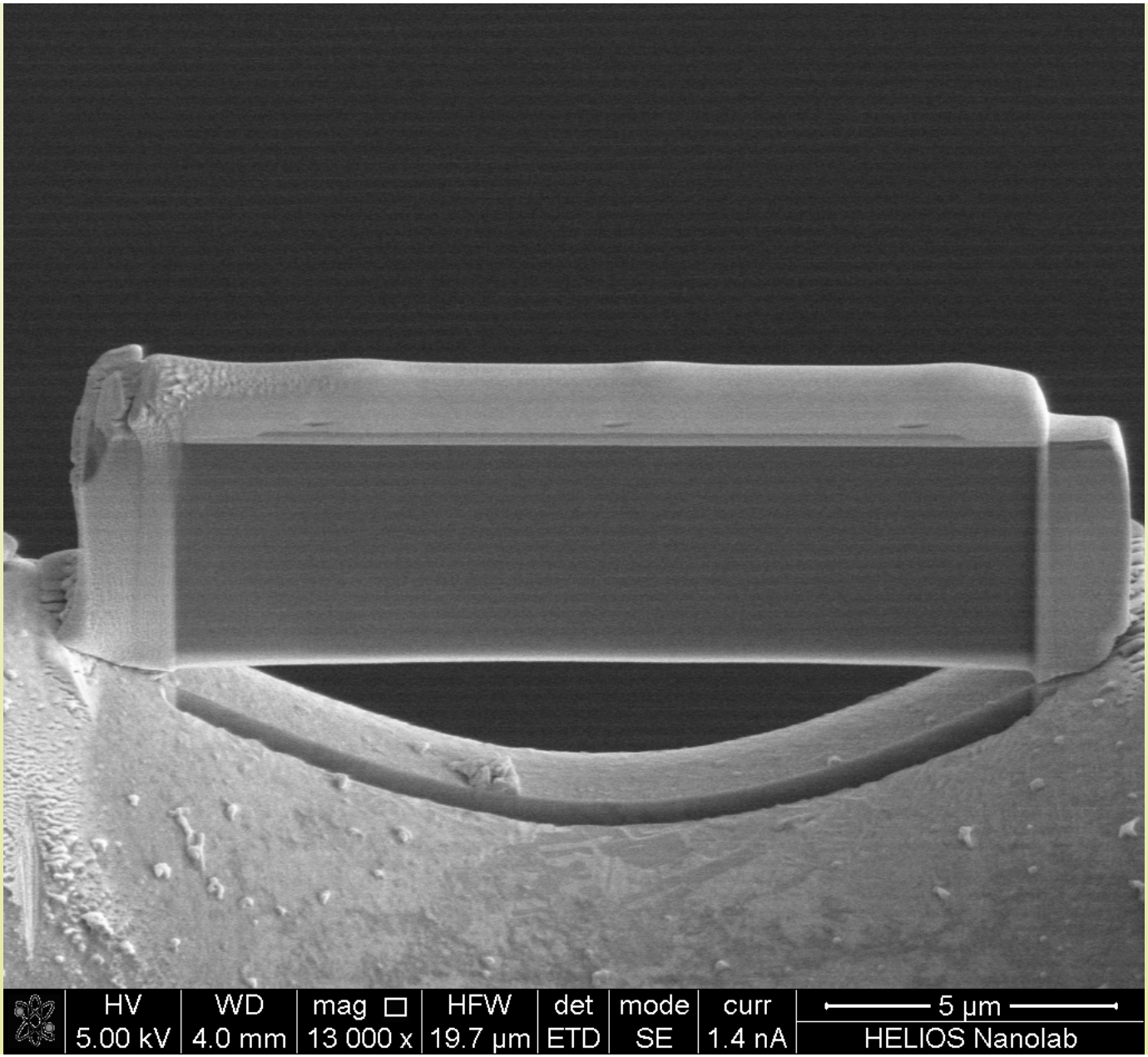

HV
30.00 kV
curr
93 pA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

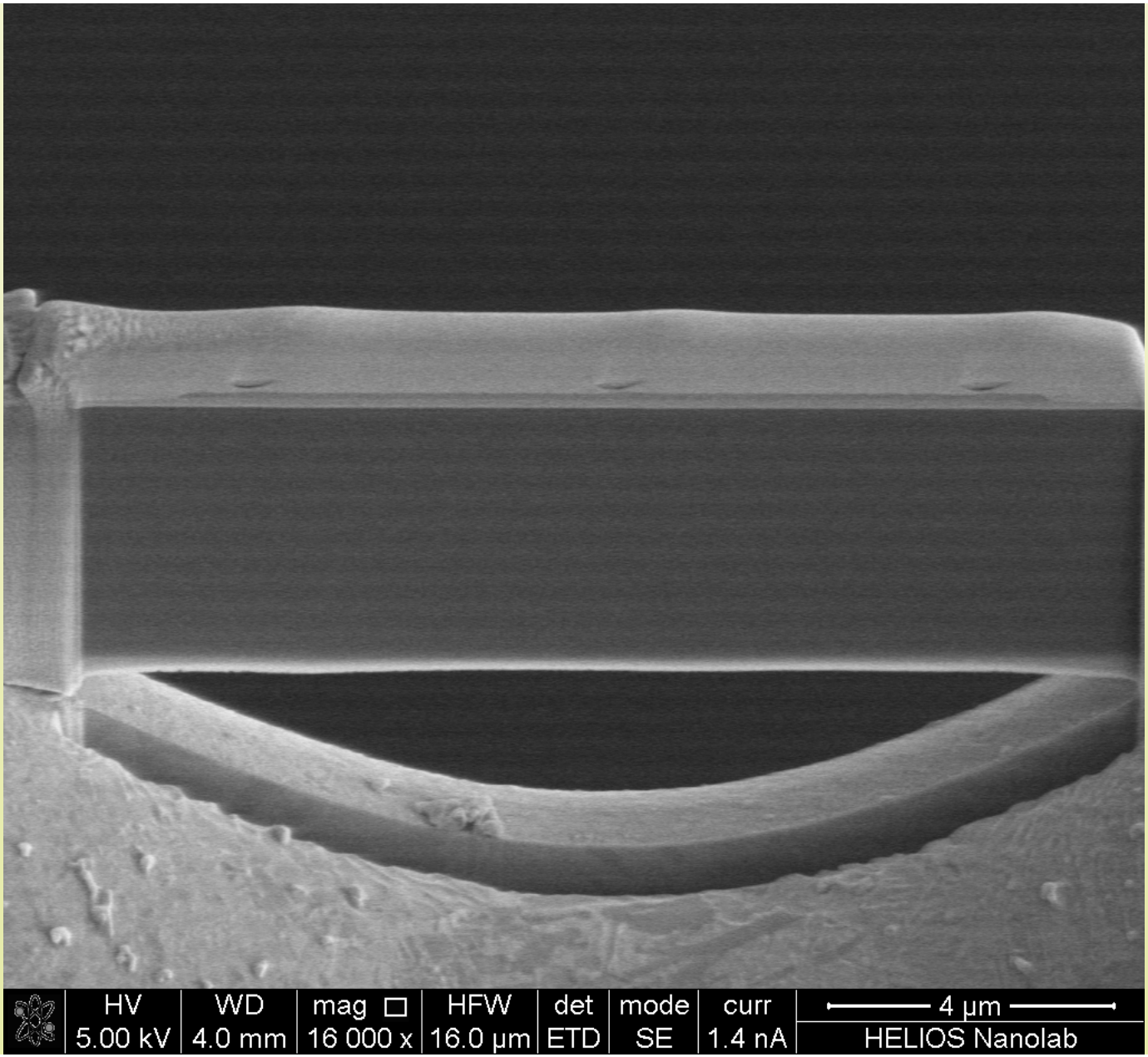

HV
30.00 kV
curr
0.92 nA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

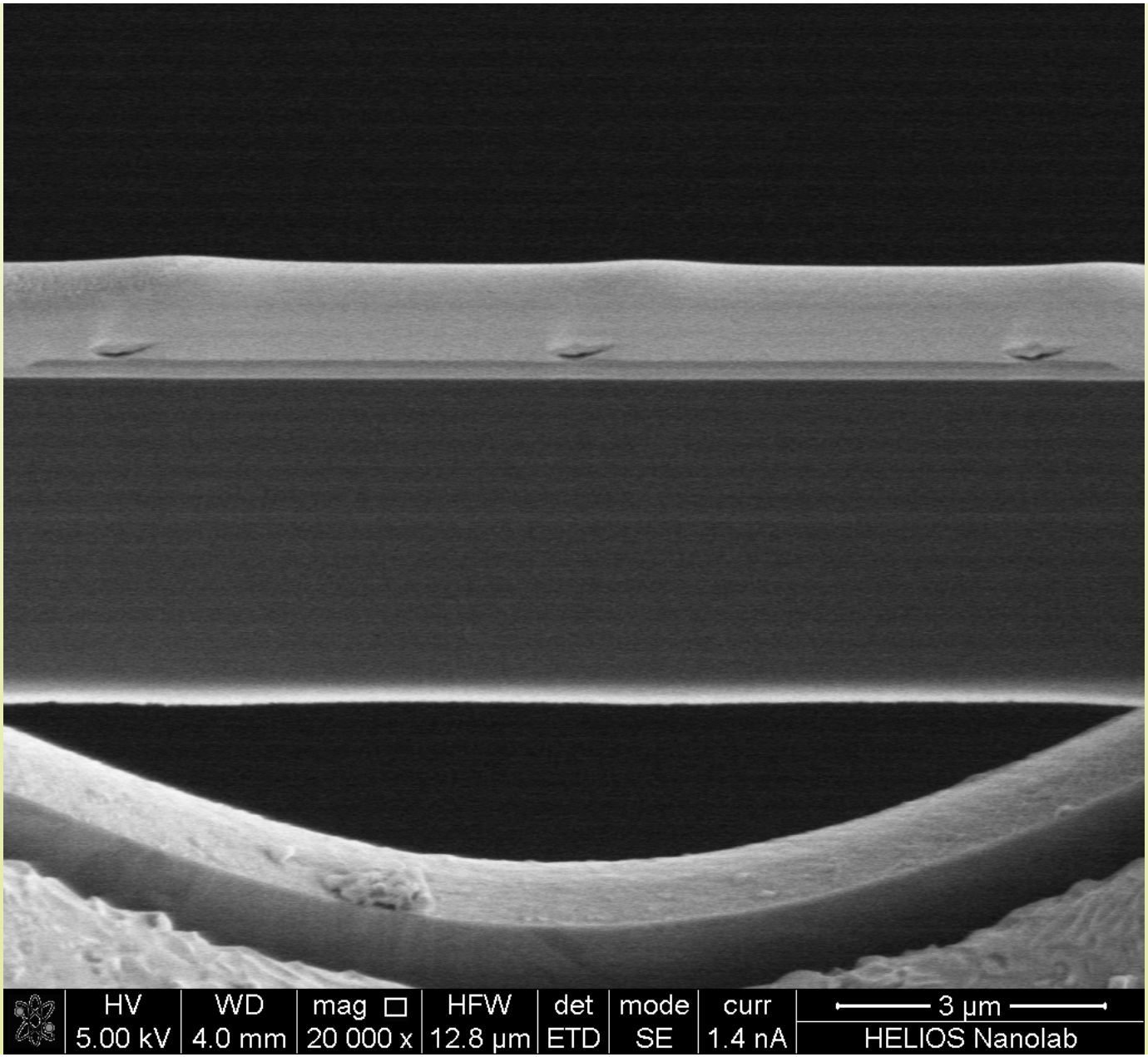

HV
30.00 kV
curr
0.92 nA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

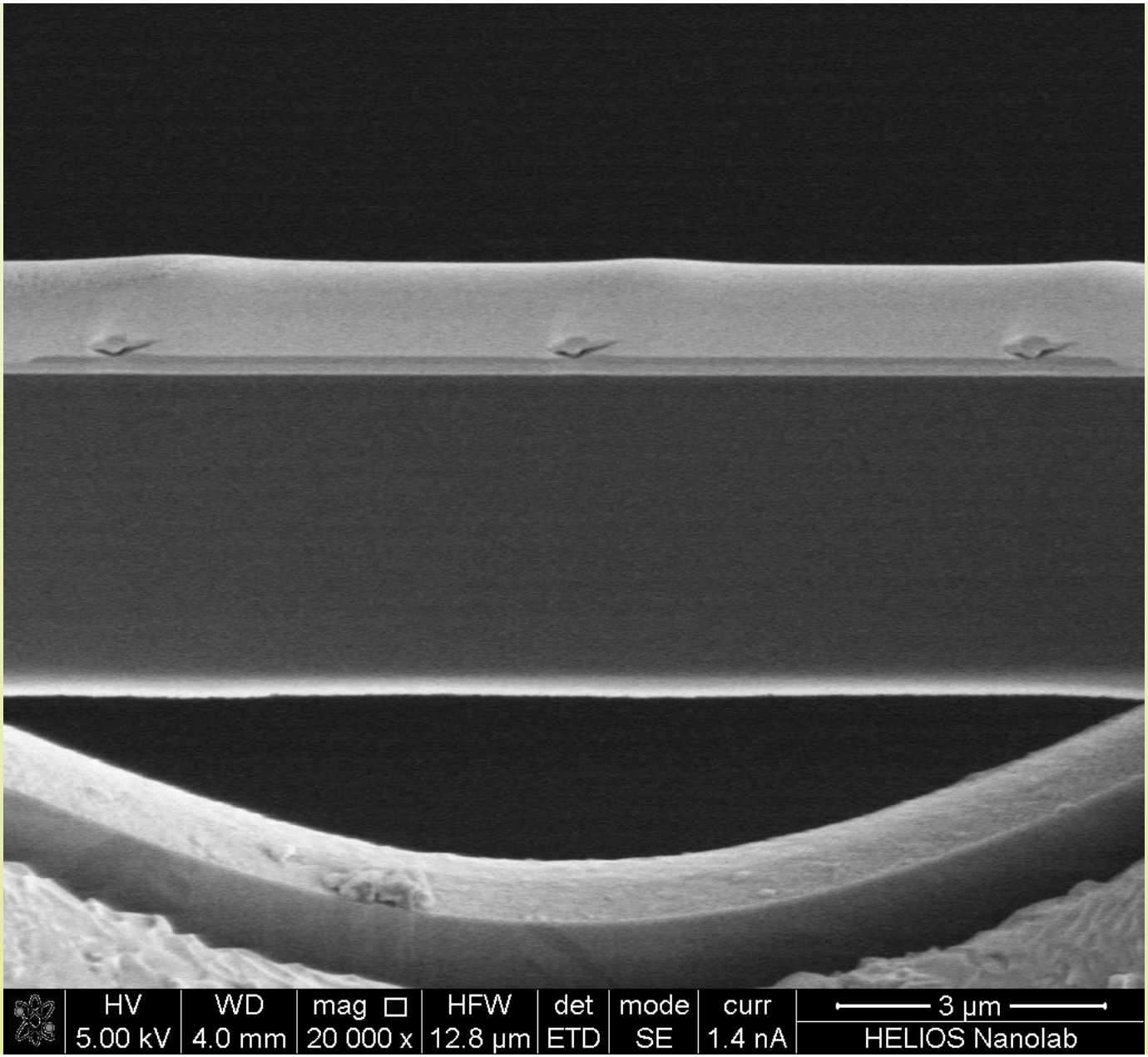

HV
30.00 kV
curr
0.92 nA
mag
10 000 x
mode
SE
HFW
25.6 µm
WD
16.5 mm
5 µm
HELIOS Nanolab

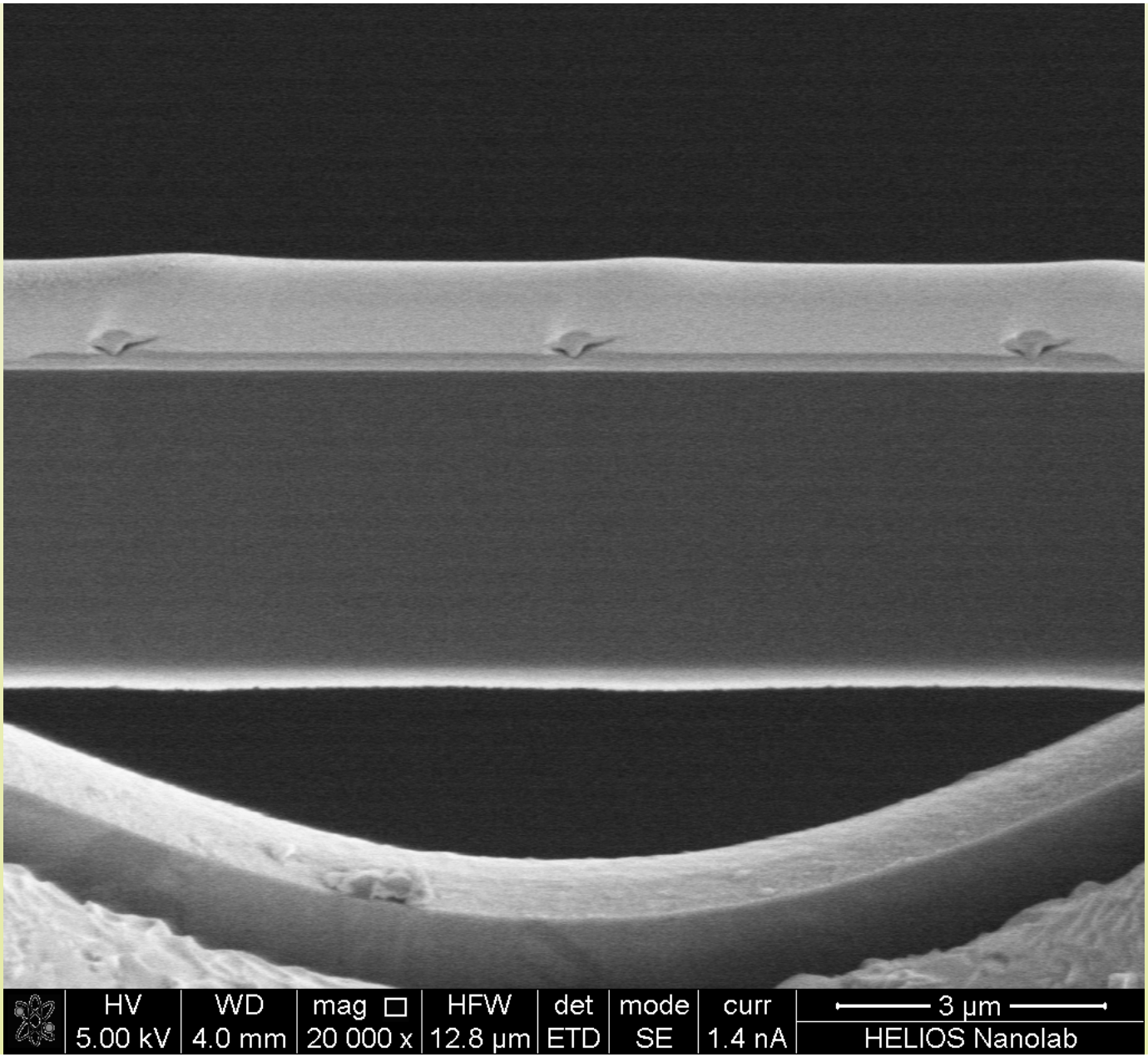

HV
30.00 kV
curr
93 pA
mag
6 500 x
mode
SE
HFW
39.4 µm
WD
16.5 mm
det
ETD
10 µm
HELIOS Nanolab

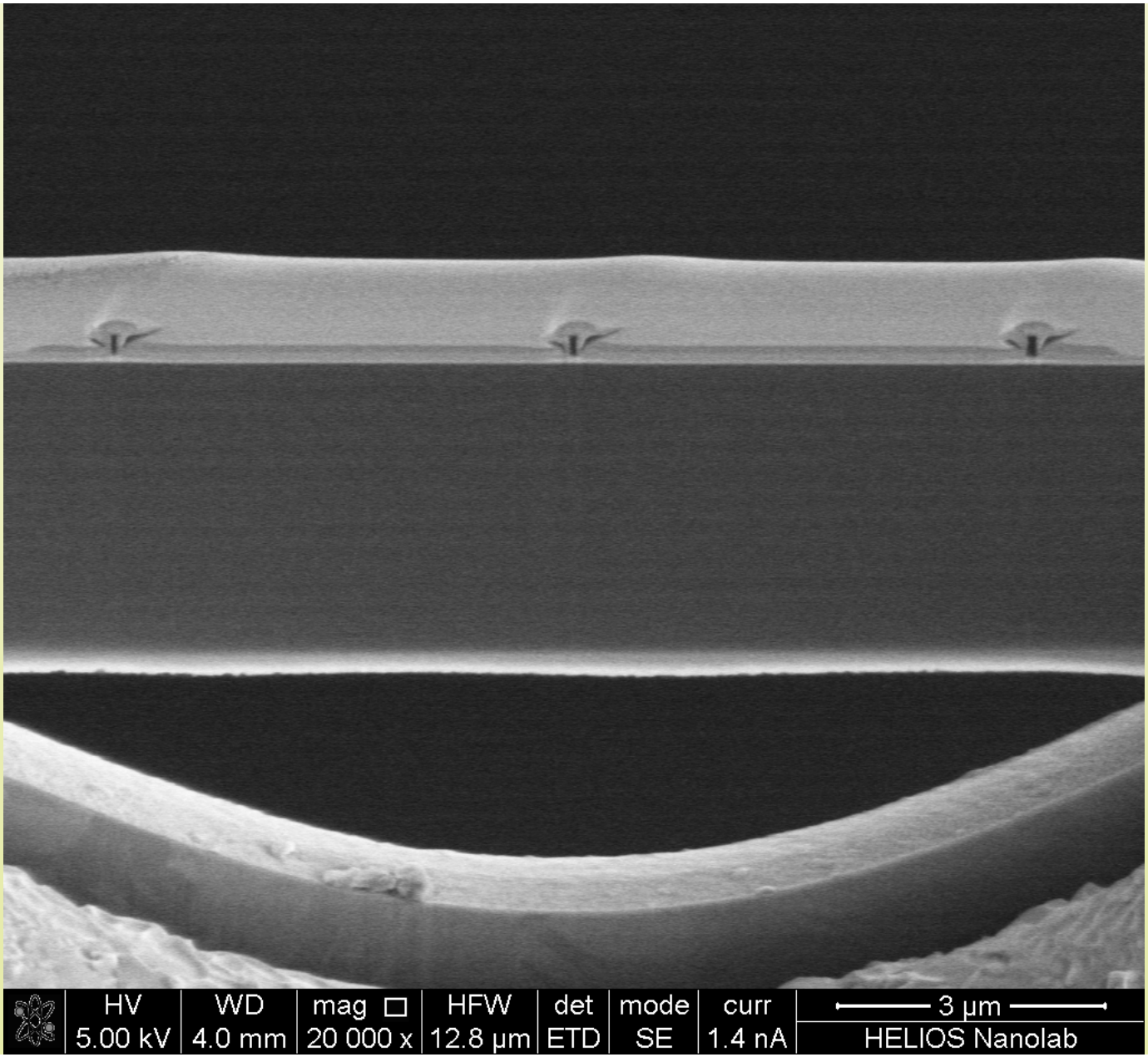

HV
5.00 kV
WD
4.0 mm
mag
13 000 x
HFW
19.7 µm
det
ETD
mode
SE
curr
1.4 nA
5 µm
HELIOS Nanolab

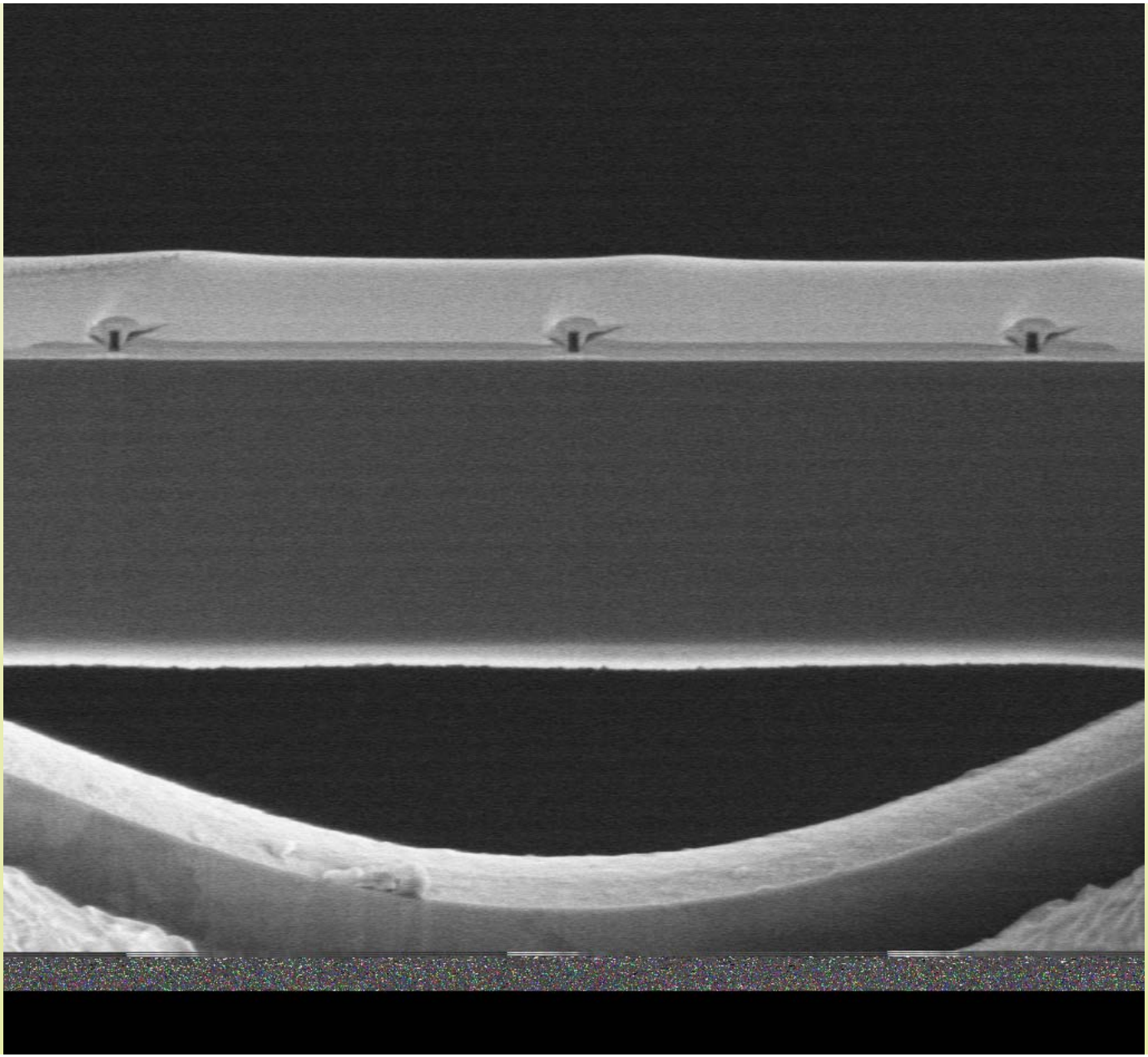

HV
5.00 kV
WD
4.0 mm
mag
16 000 x
HFW
16.0 µm
det
ETD
mode
SE
curr
1.4 nA
4 µm
HELIOS Nanolab

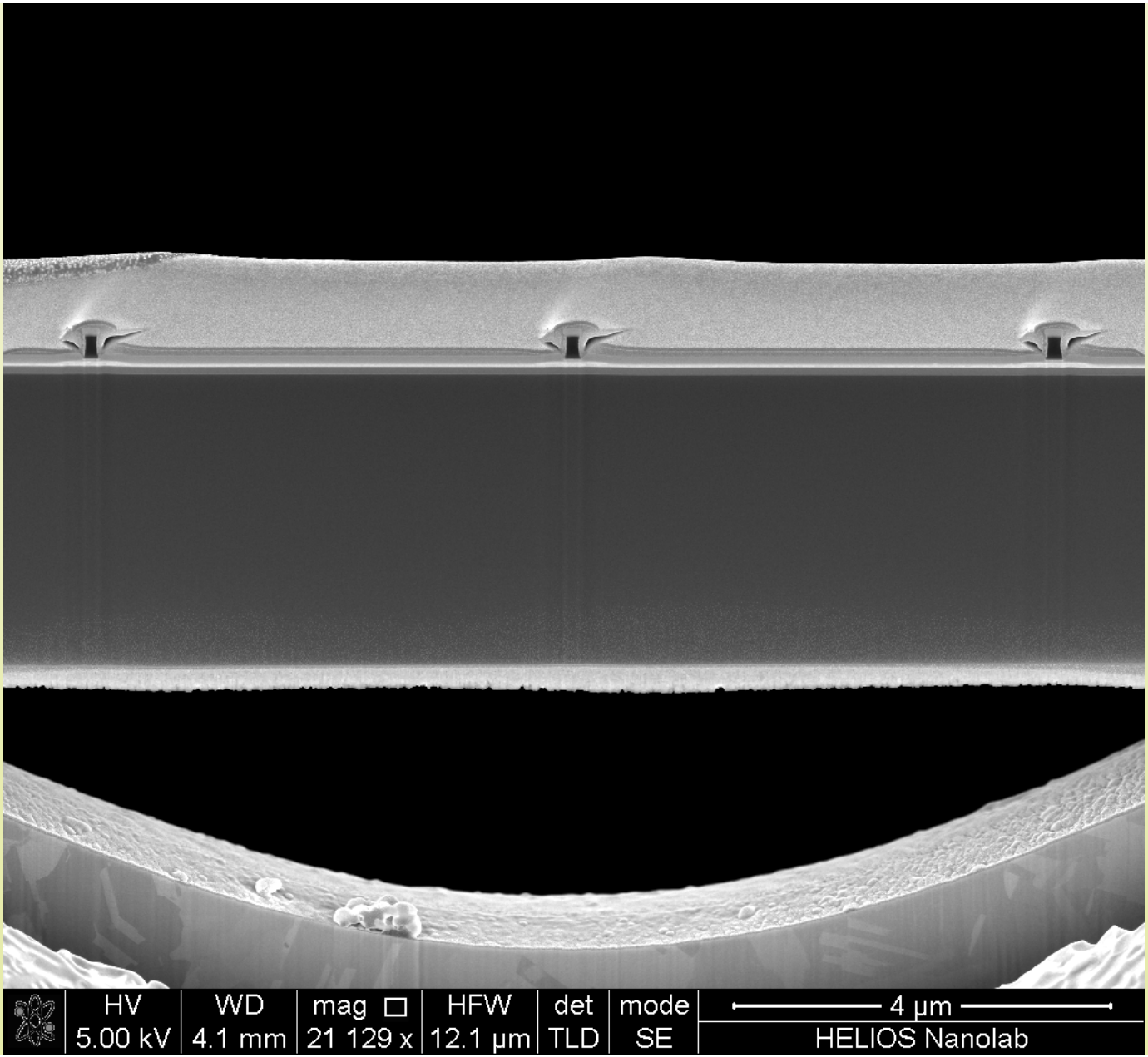

HV
5.00 kV
WD
4.0 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
curr
1.4 nA
3 µm
HELIOS Nanolab

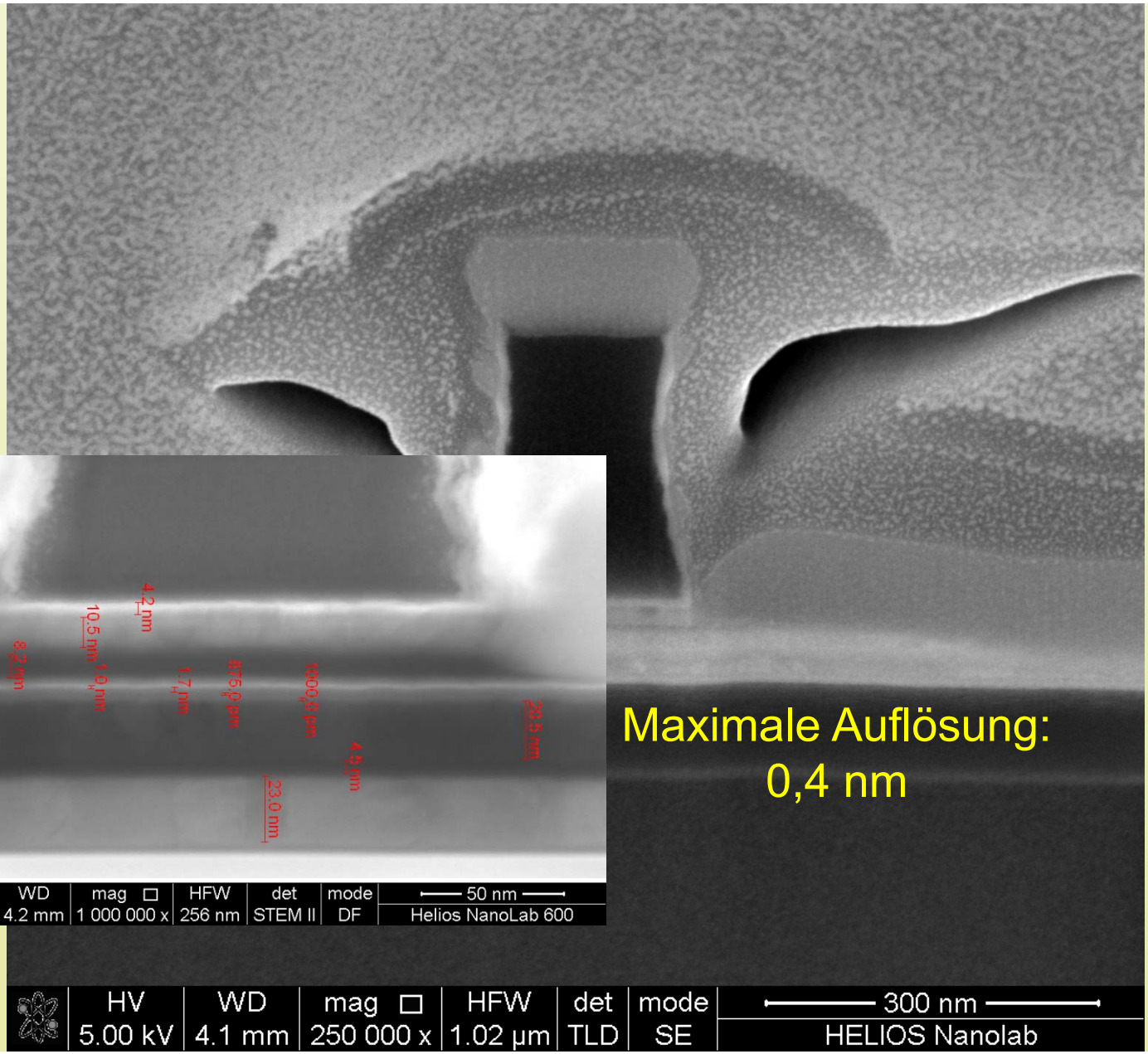

HV
5.00 kV
WD
4.0 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
curr
1.4 nA
3 µm
HELIOS Nanolab

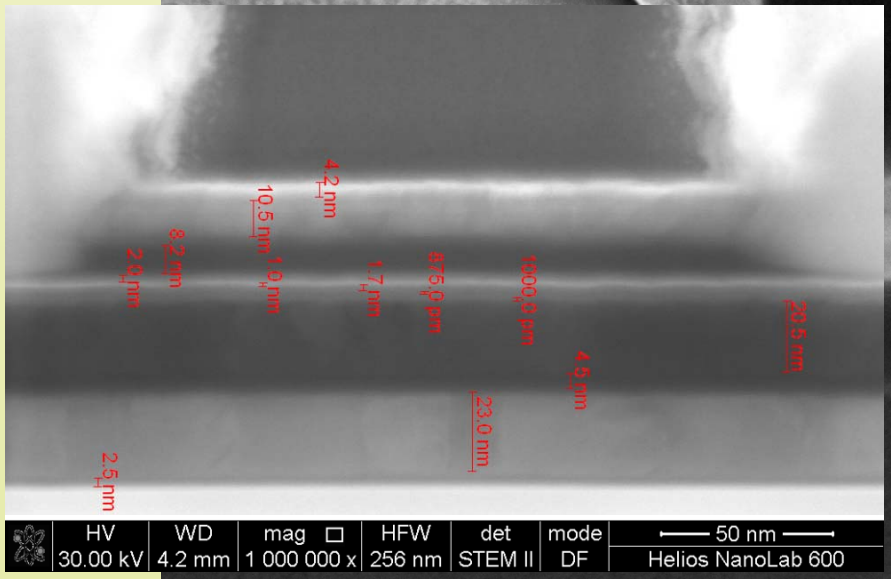

HV
5.00 kV
WD
4.0 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
curr
1.4 nA
3 µm
HELIOS Nanolab


HV
5.00 kV
WD
4.0 mm
mag
20 000 x
HFW
12.8 µm
det
ETD
mode
SE
curr
1.4 nA
3 µm
HELIOS Nanolab



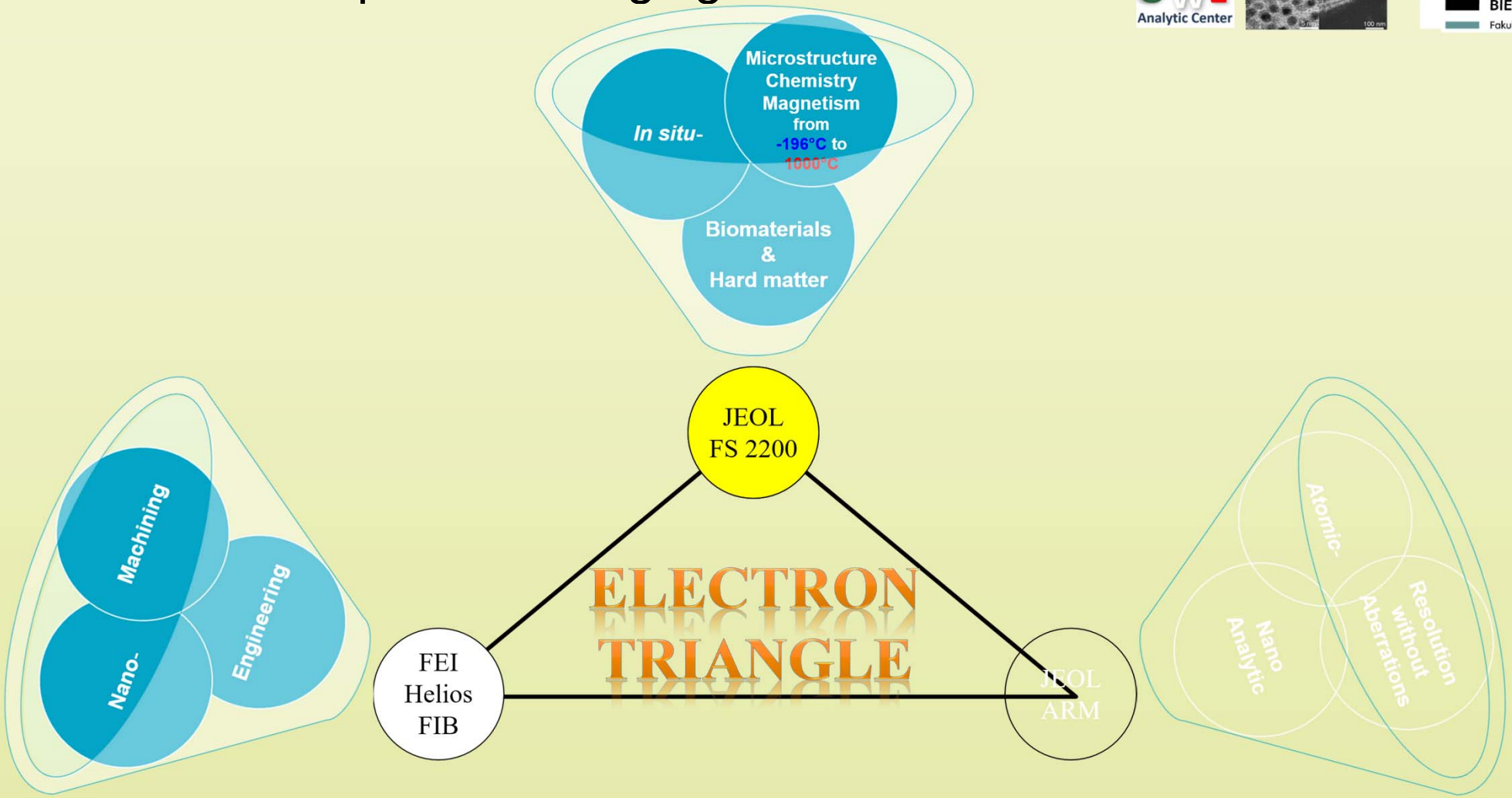

HV
5.00 kV
WD
4.1 mm
mag
21 129 x
HFW
12.1 µm
det
TLD
mode
SE
4 µm
HELIOS Nanolab





Maximale Auflösung:
0,4 nm

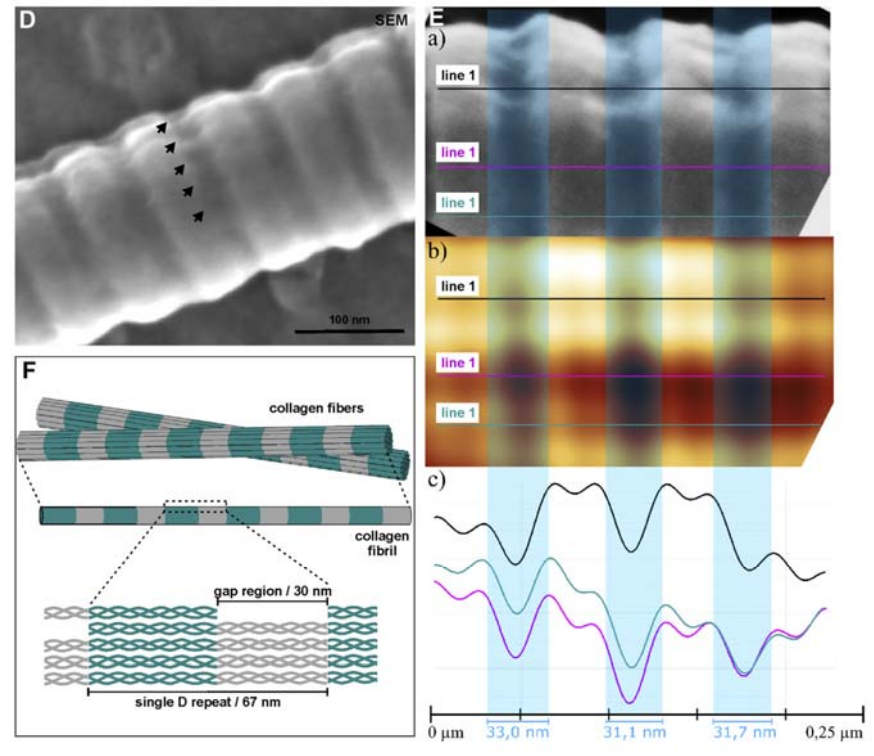
Elektronenmikroskopie: Bio-Imaging

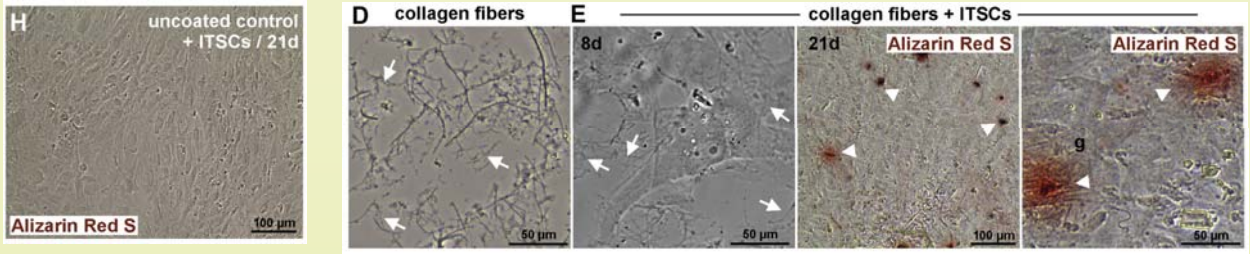


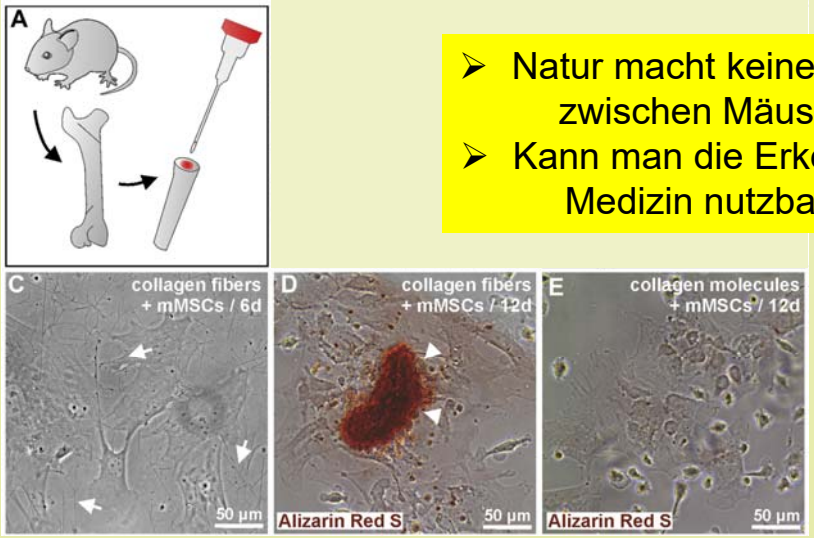
Wie bilden sich Knochen? Nanotopologie zu identifizieren, die eine osteogene Differenzierung induziert

Kooperation mit AG Kaltschmidt, Fakultät für Biologie:

Adulte Stammzellen aus der Nase oder Gewebe isoliert:

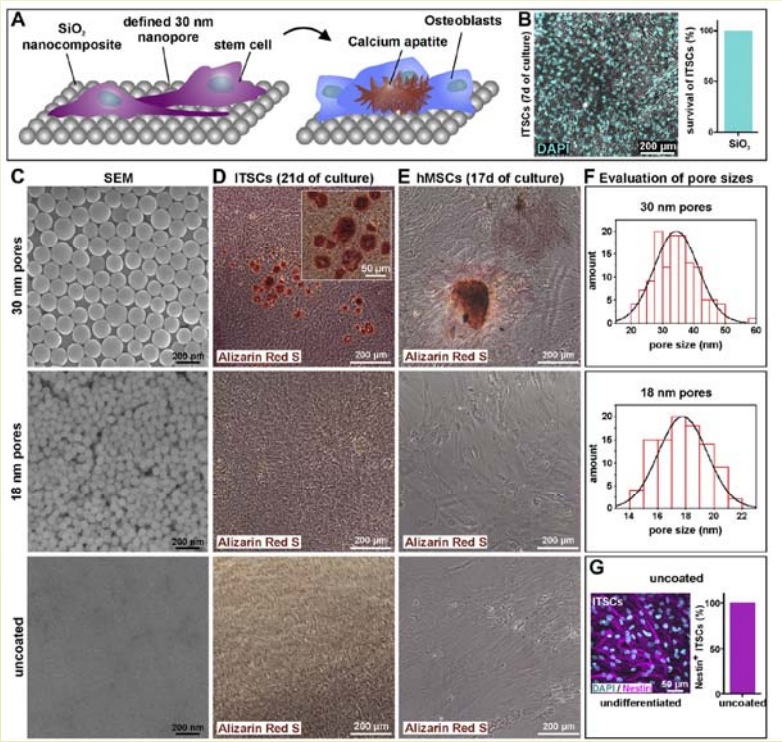




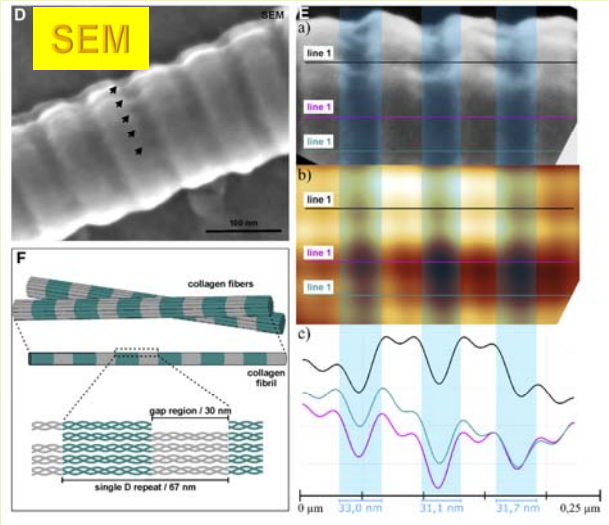


- Natur macht keinen Unterschied zwischen Mäusen und uns
- Kann man die Erkenntnis für die Medizin nutzbar machen?

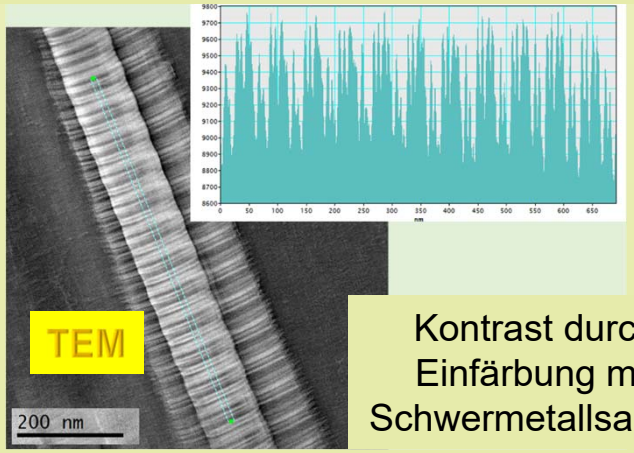
Wie bilden sich Knochen? Welche Nanotopologie gilt es zu identifizieren, die eine osteogene Differenzierung induziert?



Nanotopologie durch Beschichtung von Implantaten



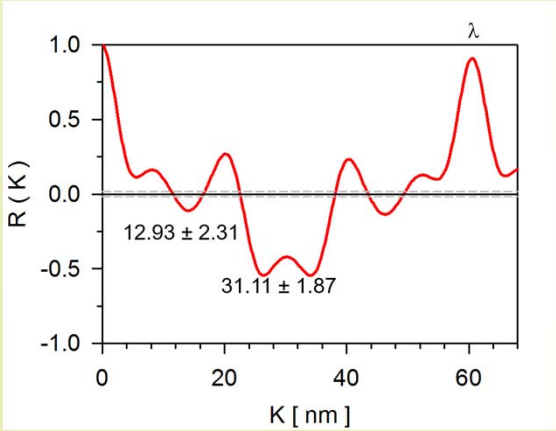
TEM: Intensität als Funktion der Längen in [nm]:



Kontrast durch Einfärbung mit Schwermetallsalzen

Autokorrelation der Mikrostruktur:

$$R(K) = \left[\frac{N}{N-K}\right] \frac{\sum_{i=1}^{N-K} (I_i - I_0)(I_{i+K} - I_0)}{\sum_{i=1}^{N} (I_i - I_0)^2}$$

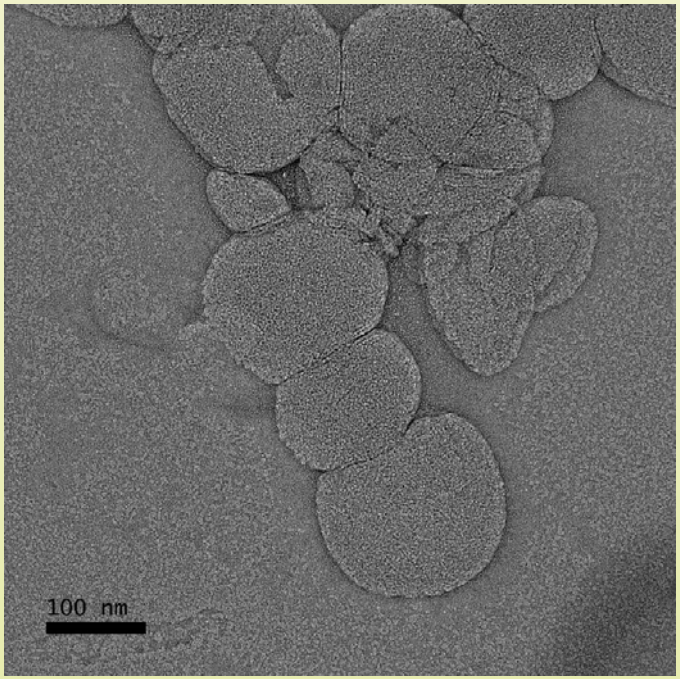


SEM ⇨ TEM:

- Strukturell viel genauer
- Daten mit statistischer Relevanz

Wie bildet man eigentlich Biomoleküle und Objekte ab?
⇨Cryo-Transmissionselektronenmikroskopie von Liposomen

Wieder kontrastiert mit Schwermetallsalzen:



Struktur im Vakuum des TEM zerstört

Einfrieren der Bio-Objekte bei T = -196,15°C

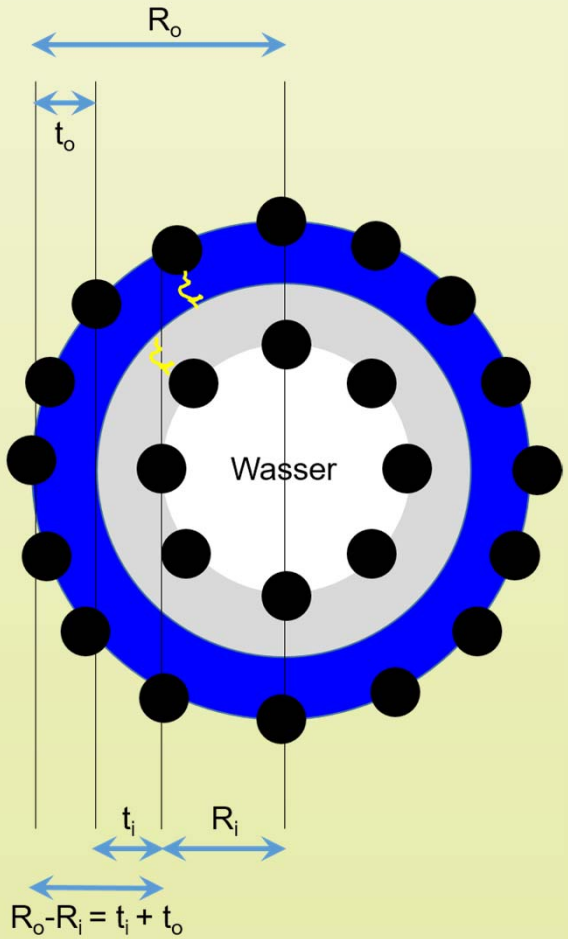
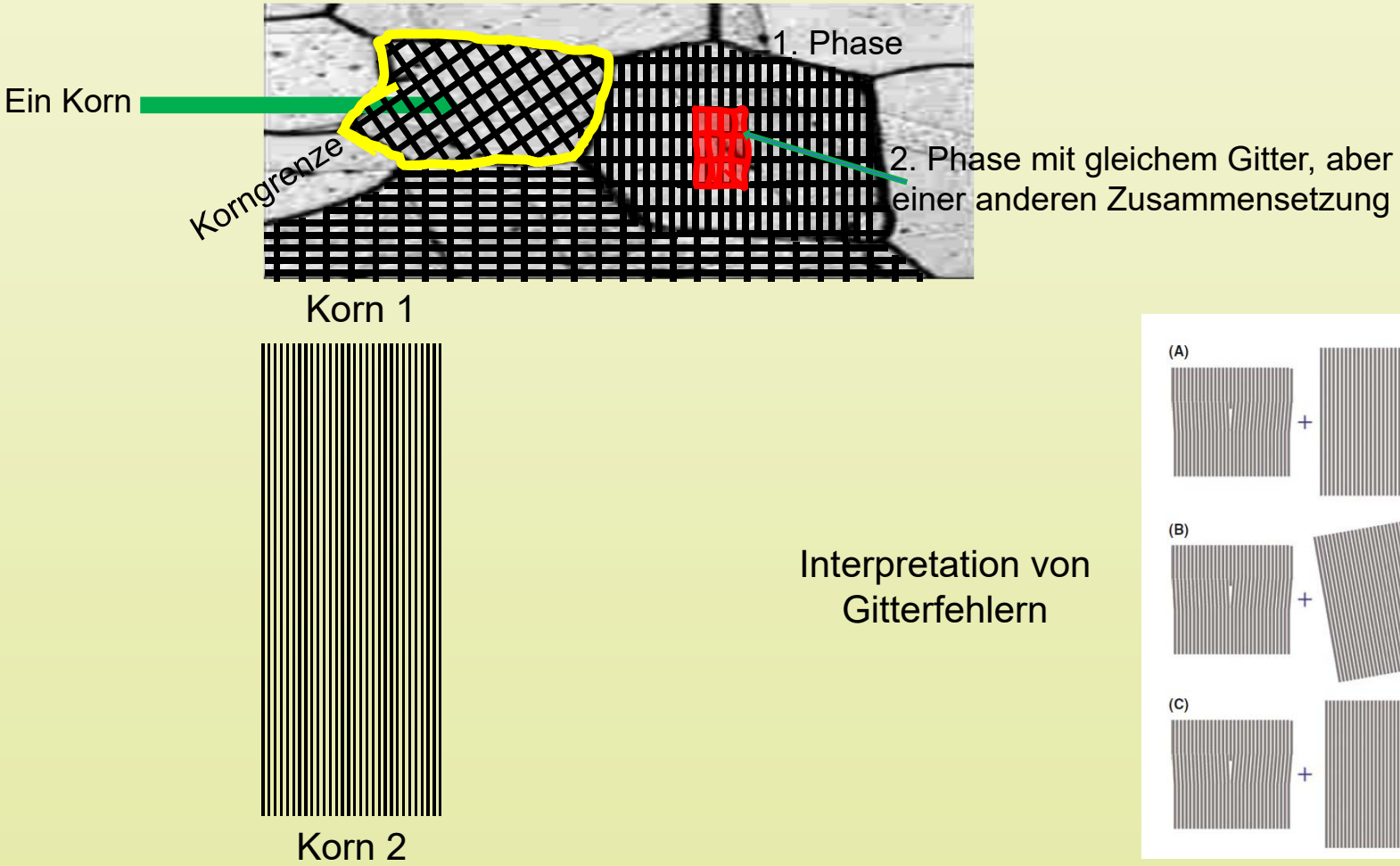
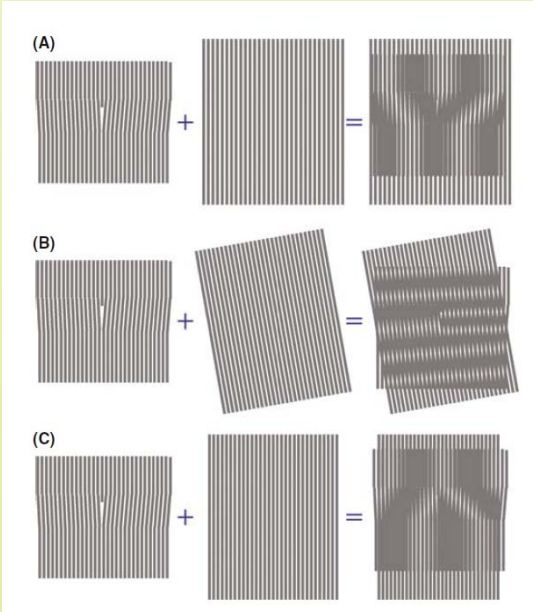


Abbildung von Atomen in Kristallgittern: Moiré Gitter

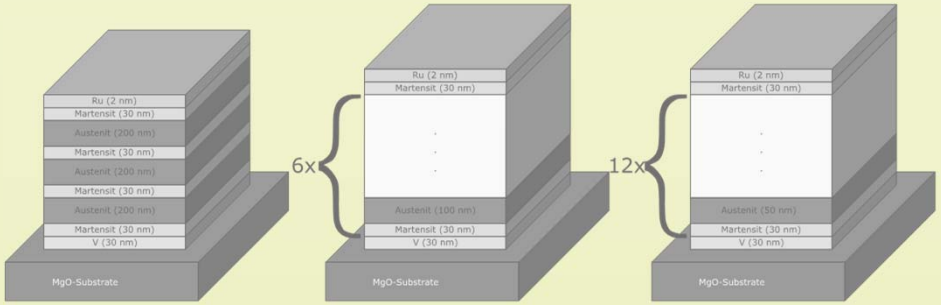
→ Ein realer Polykristall:



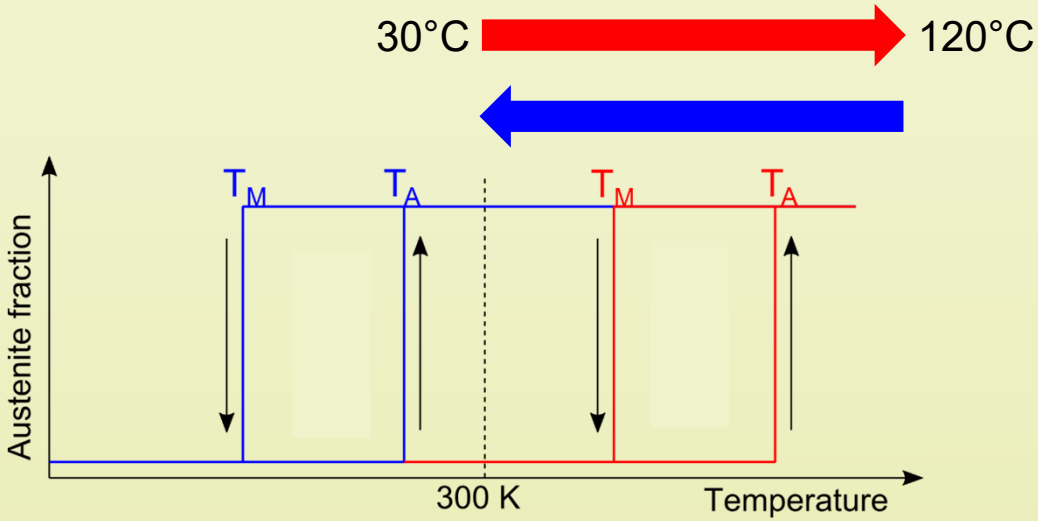
Interpretation von Gitterfehlern



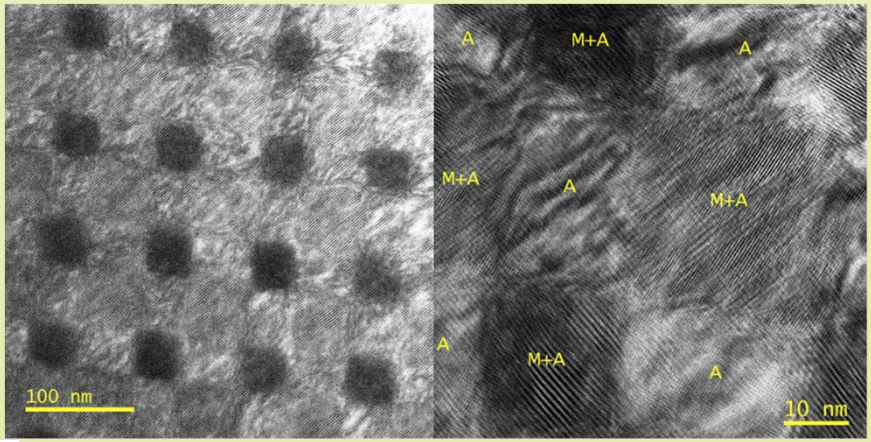
In situ Mikroskopie: heizen und kühlen

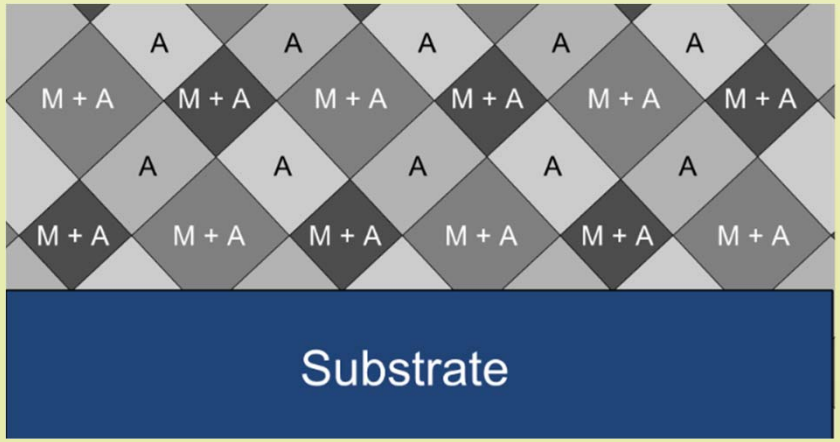


	Composition	T_M	T_A	Dicke:
Austenit	$Ni_{43}Co_7Mn_{31}Al_{19}$	232 K	248 K	50 nm
Martensit	$Ni_{47}Co_3Mn_{33}Al_{17}$	308 K	343 K	30 nm

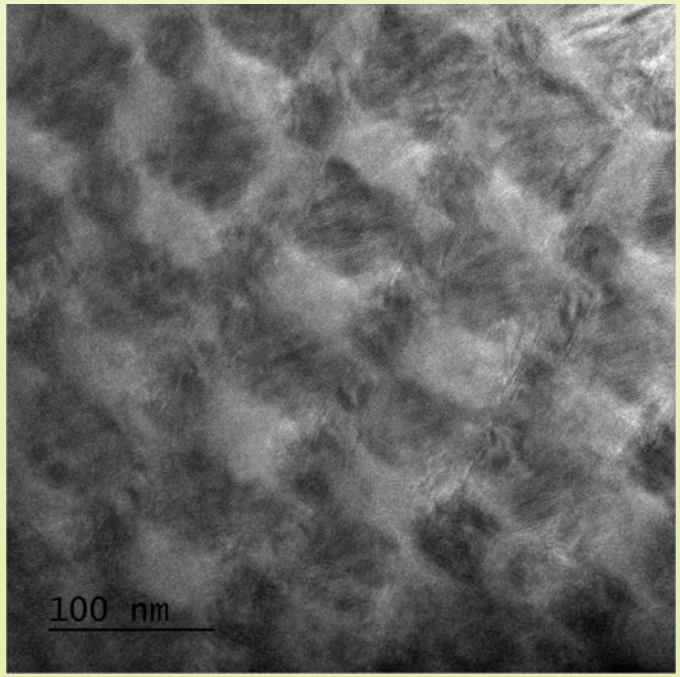


Moiré Gitter:

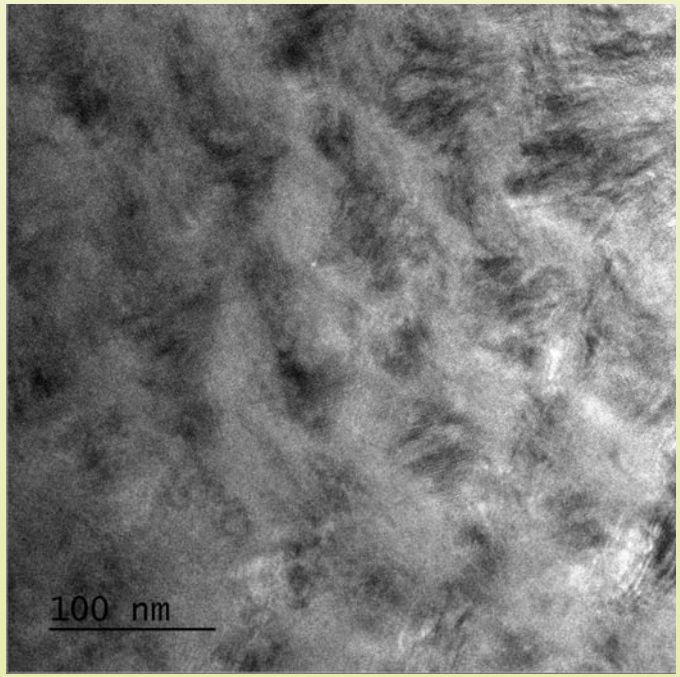




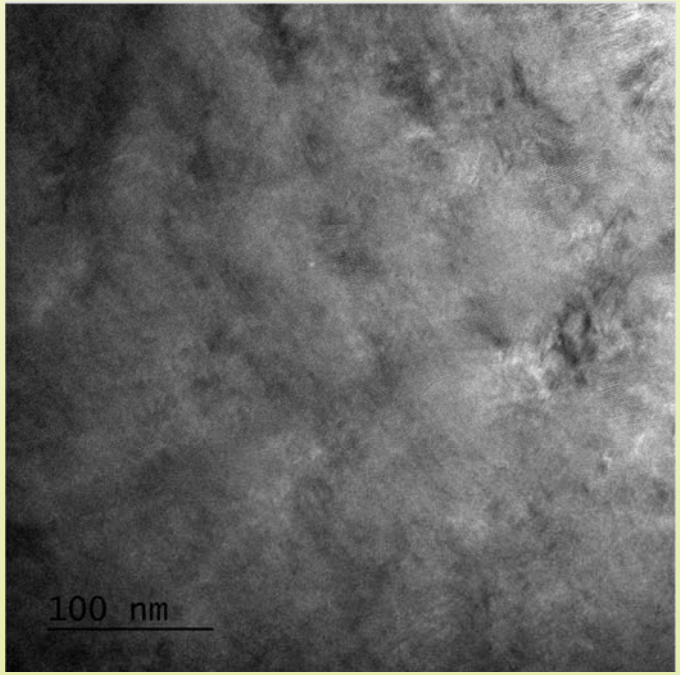
30°C



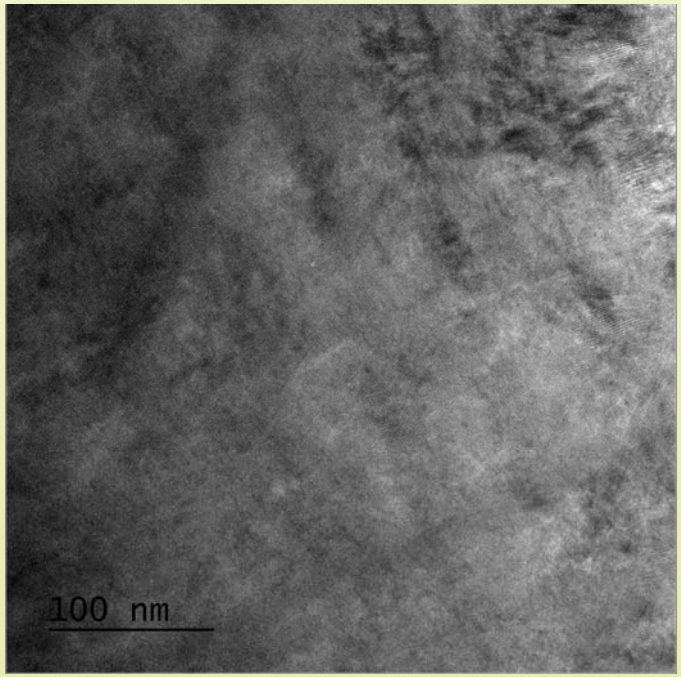
90°C



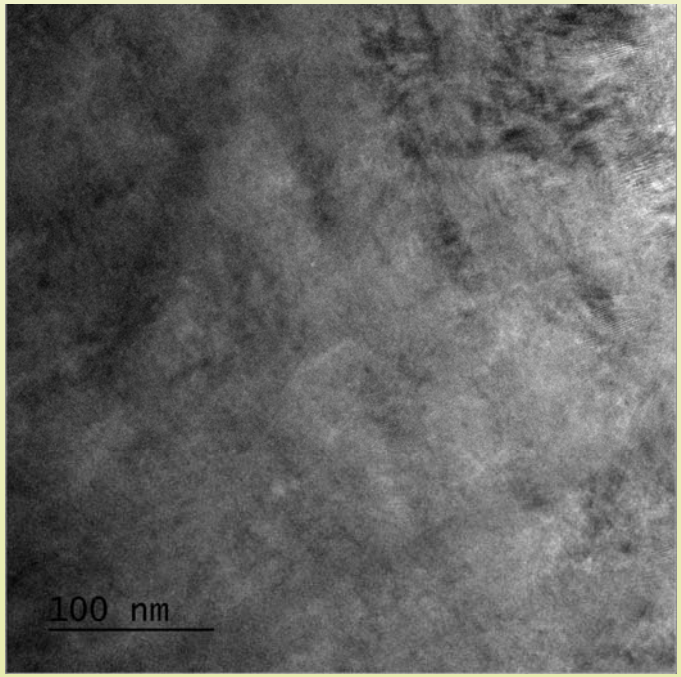
110°C



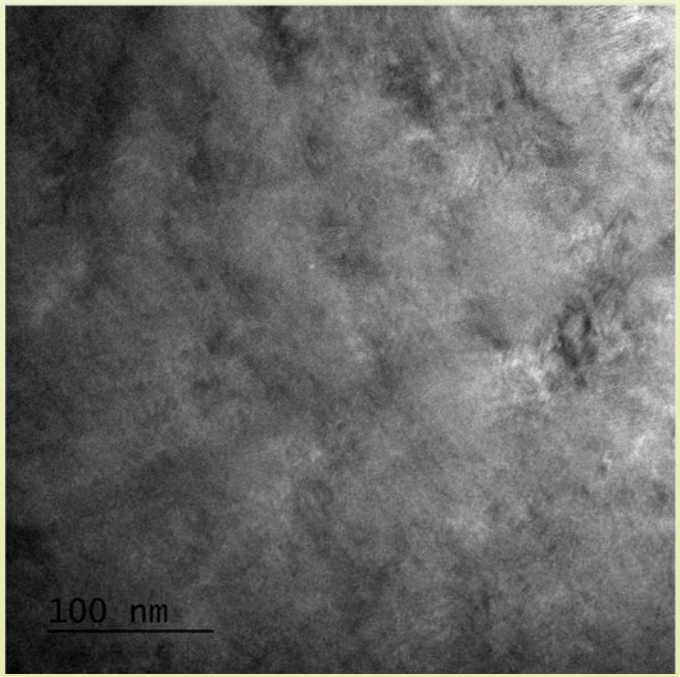
120°C



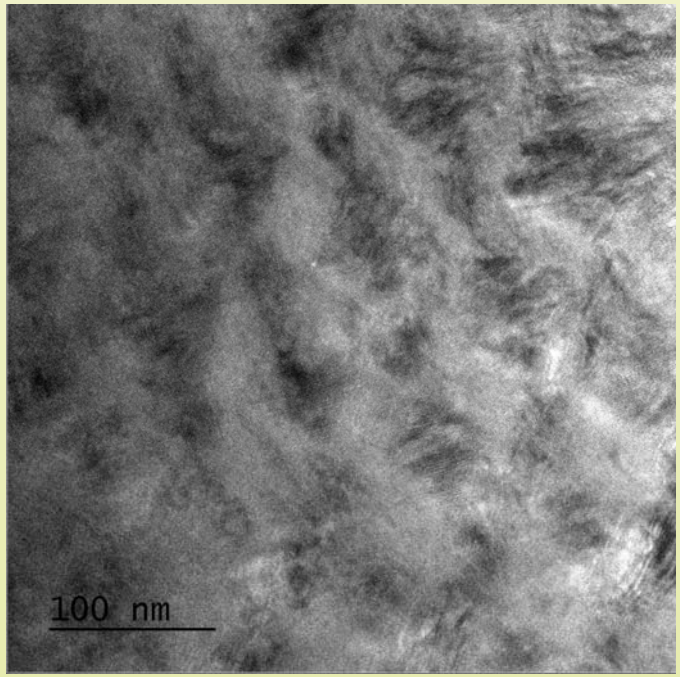
120°C



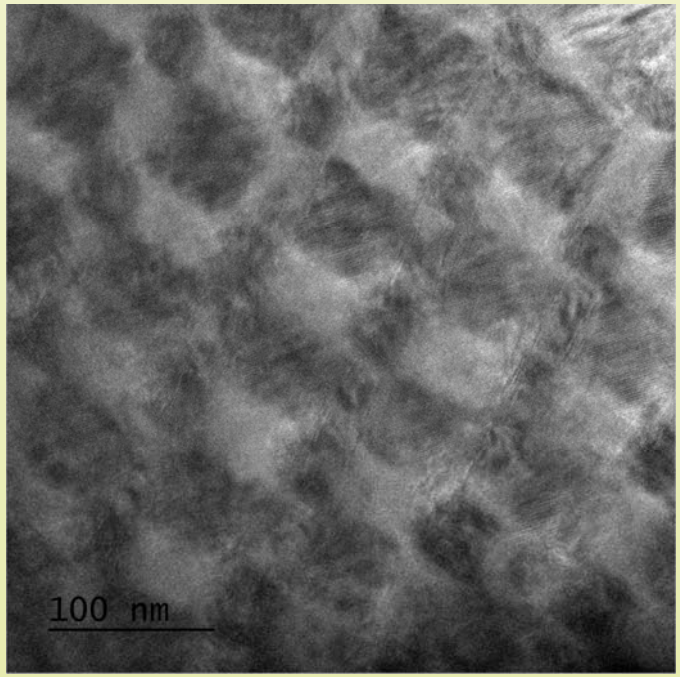
110°C



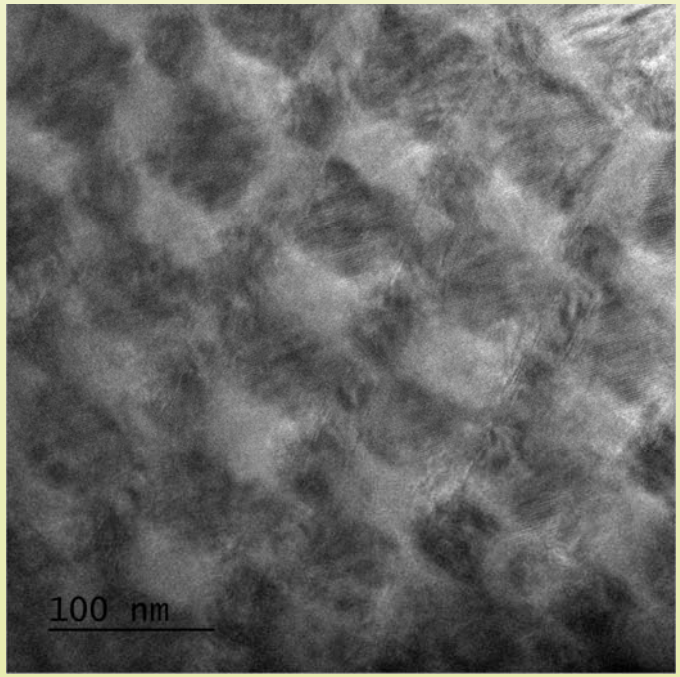
90°C



30°C



30°C

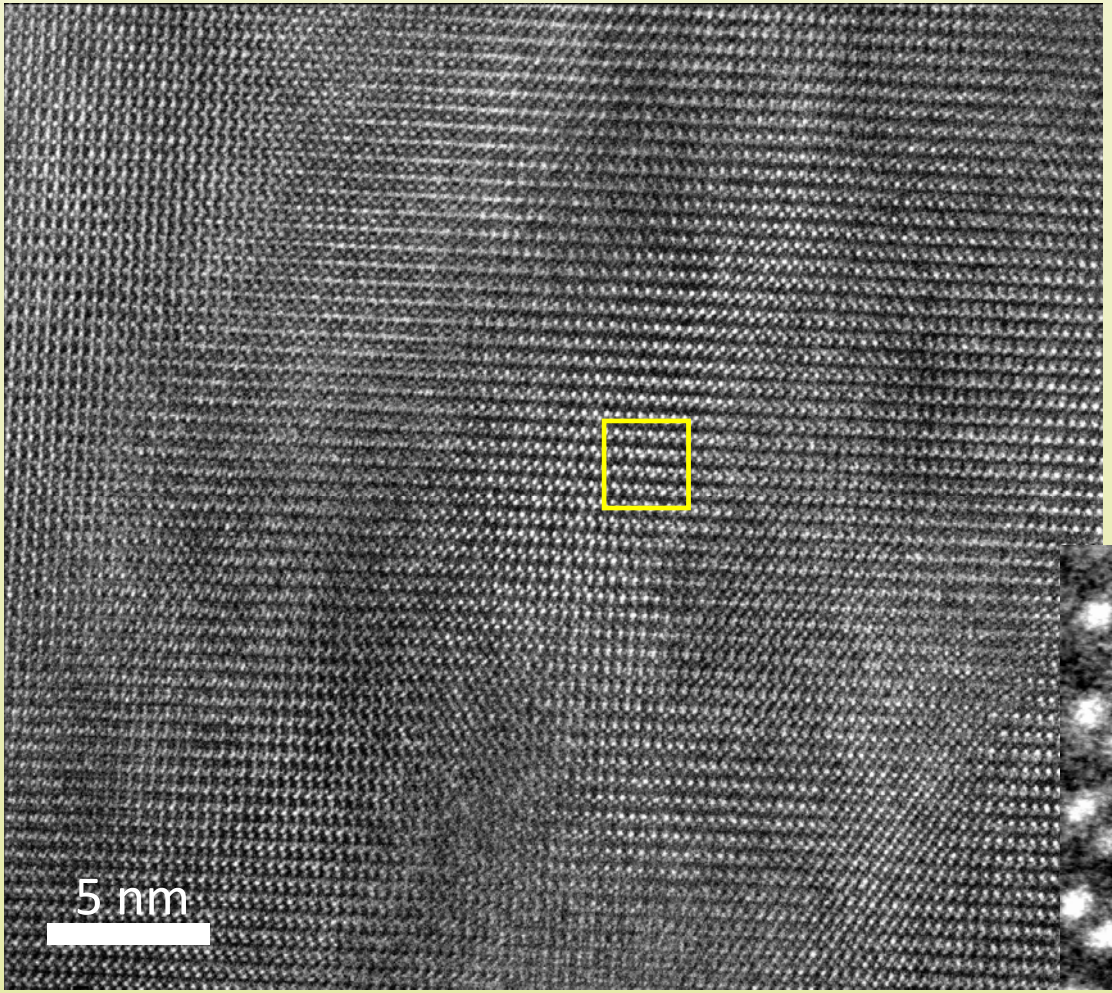


- In situ Monitoring von Phasenumwandlungen
- Test auf Materialintegrität
- Validierung der Herstellungsparameter

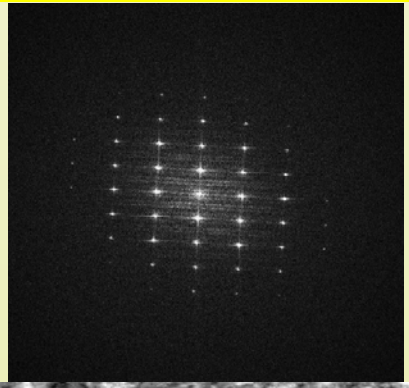
Die atomare Auflösung:

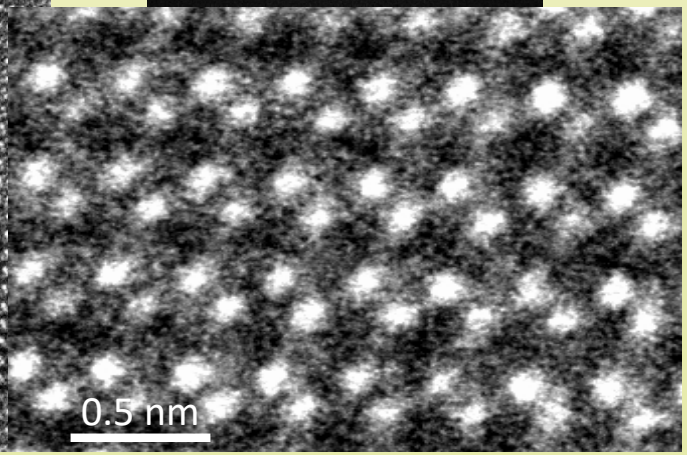
$Mn_{1.25}Fe_{0.7}P_{0.55}Si_{0.45}$:

- single crystal
- Ball milled
- FIB lamella



➢ Punktauflösung: 0,22 nm





➢ Nächster Nachbarabstand
➢ Atomare Ordnung
➢ Vergleich mit DFT-Simulationen

Als Dank für Ihre Aufmerksamkeit

Besuch der Labore: