Helium Ion Imaging and Patterning

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June 30, 2021

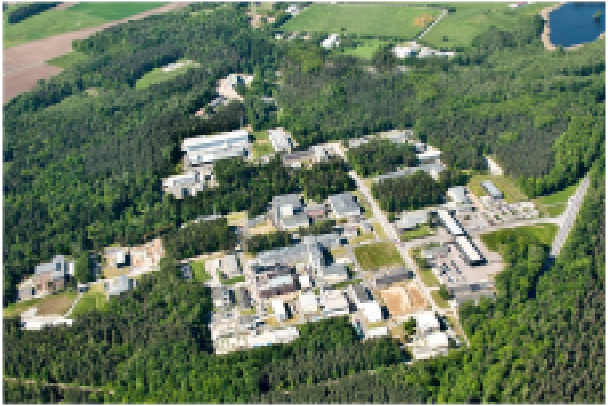
Overview

Helmholtz—Zentrum Dresden-Rossendorf

HZDR

- User facility
- Proposal based
- Free of charge
- 1000 employees
- 500 scientists
- IBC
- ELBE
- HLD
- PET Center
- Topflow

Helmholtz—Zentrum Dresden-Rossendorf



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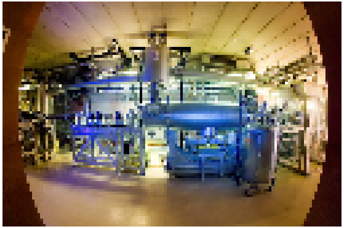
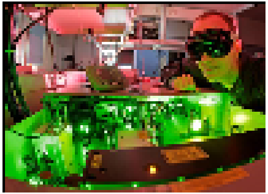
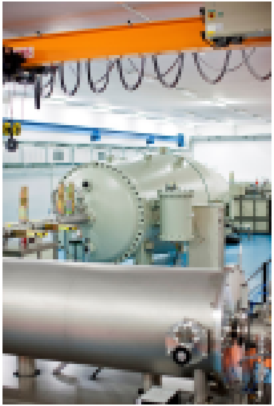
HZDR Facilities



ELBE

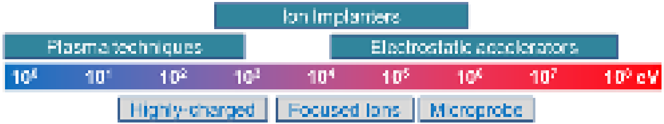


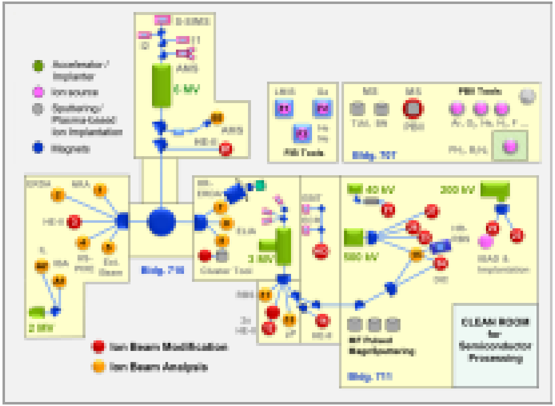


IBC

HLD

The Ion Beam Center—a user facility





0.5 nm to 8 ”

- Implantation
  - H-Bi
  - 5 eV–70 MeV
- Analysis
  - HR-RBS
  - ERDA
  - NRA
  - PIXE
  - PIGE
  - AMS
  - SIMS
  - ext. beam
- Ga-FIB
- LMAIS-FIB
  - Pb, Bi, . . .
  - Ge, Si, Li, . . .
  - Co, Fe, . . .
- HIM
  - NanoFAB
  - Orion+
  - HIM-SIMS
  - STIM

Standing on the shoulder of giants

Mental, physical, and financial Support

FWIZ-N

- **N. Klingner**
- L. Bischoff
- **W. Möller**
- **K. H. Heinig**
- D. Erb
- **X. Xu**
- **T. Prüfer**
- **E. Serralta**
- L. Zimmermann

HZDR

- **K. Lenz**
- F. Samad
- **C. Fowley**
- **R. Bali**
- O. Helwig
- S. Fascko
- A. Erbe
- **Z. Fekri**
- R. Hübner
- **H. J. Engelmann**
- A. Krasheninnikov
- **S. Kretschmer**
- S. Ghaderzadeh

Partners

- **P. Orus (UAB)**
- **J. M. de Teresa (UAB)**
- **P. Dunne (Strassbourg)**
- **M. Nord (Glasgow)**
- **G. Nanda (Delft)**
- P. Alkemade (Delft)
- **A. Gölzhäuser (Bielefeld)**
- T. Wirtz (Luxembourg)

Funding

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- npSCOPE H2020: 720964
- FIT4NANO COST: CA19140
- STHIM FNR: C16/MS/11354626
- picoFIB: Leverhulme Trust
- BMWi: Grant 03ET7016
- BMBF: Grant 03THW12F01

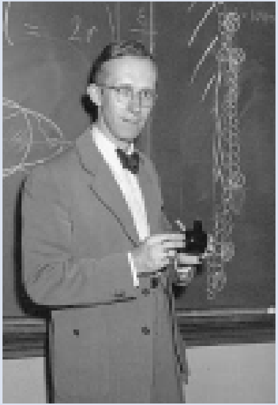
Overview

Overview

2 Helium Ion Microscopy
- The source
- Ion Beam optics and resolution
- Other subsystems

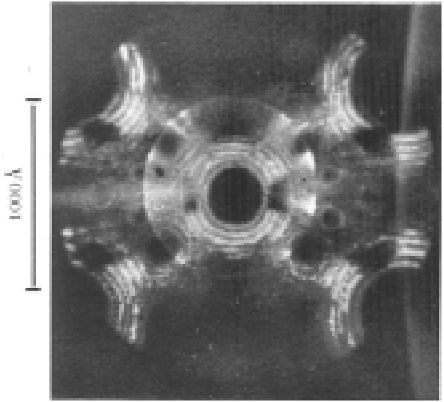
Who saw the first atoms?

Erwin Müller



E. W. Müller, Z. f. Phys., 1951

- 1951, Berlin, Germany
- First visualization of atoms



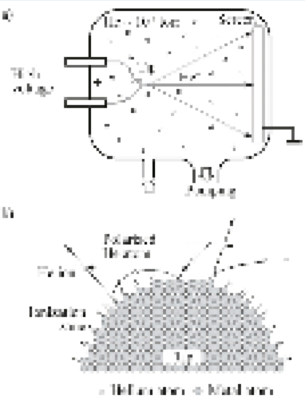
Requirements for Ultramicroscopy

Partial list for scanning beam techniques

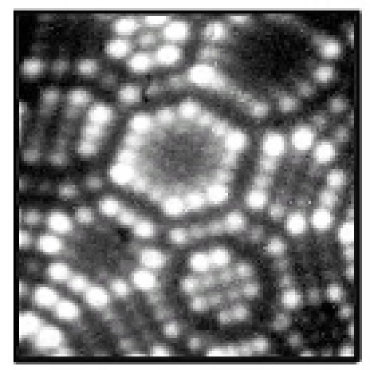
- Monochrome beam (e.g. Laser)
  - minimizes chromatic aberration
- small beam divergence
  - minimizes spherical aberration
- minimal size
  - Probe size will be folded with feature size
  - should ideally be a delta function

The source—fundamentals
Field ion microscope

Sketch of FIM



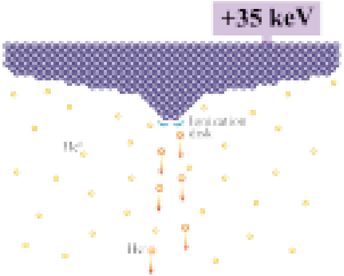
Tungsten

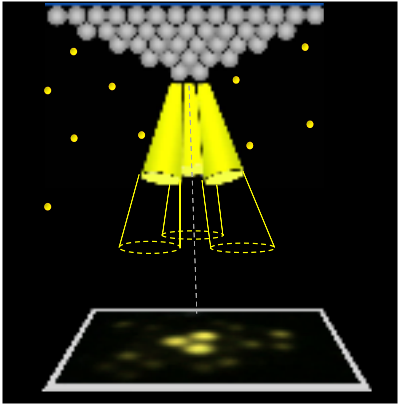


The source

Solution

- Tungsten trimer on apex
- Field ionization
- Helium (Neon) gas
- Single beamlet selected





Why He (Ne)

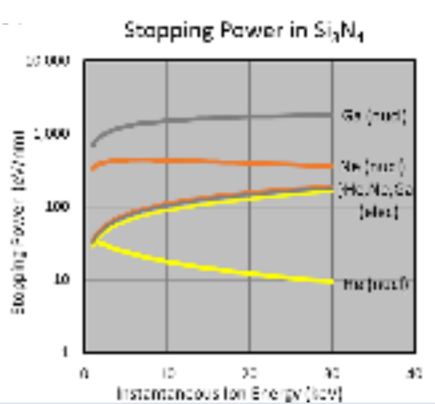
Gas requirements

- High ionization potential
  - self cleaning
- Not too high polarzibility
  - Sputter damage
- Low boiling point
  - Tip has to be cold to reduce lateral momentum
  - $T_B < 80\,\mathrm{K}$

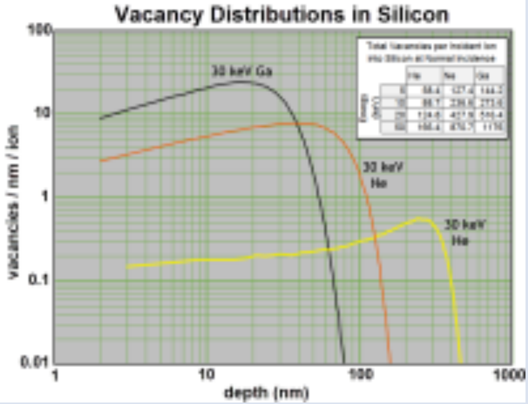
Gas	Ionization pot. eV	Polarizability $10^{-24}\,\mathrm{cm}^{-3}$	Boiling point K
Helium	24.6	0.20	4.22
Neon	21.6	0.29	27.07
Argon	15.8	1.63	87.3

When He is good and why Ne (or Ga) is better sometimes

Stopping power



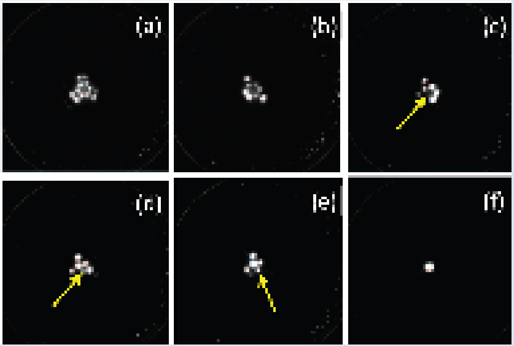
Depth distribution of defects



Tip formation

Field evaporation

- Tip shaping
- Fieldstrength $> 5\,\mathrm{V/Å}$
- removal of W atoms from the tip apex

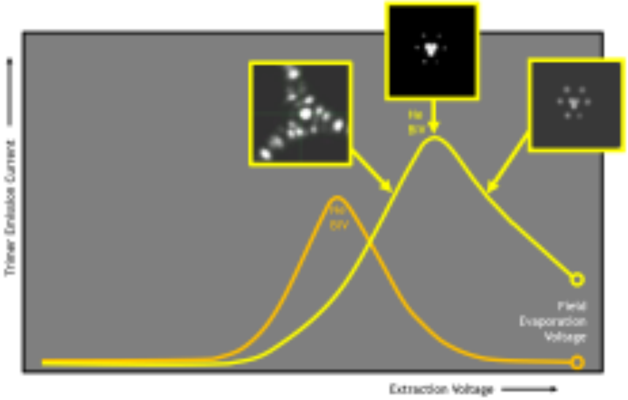


Field ionization

- normal source operation
- He: $4.5\,\mathrm{V/Å}$
- Ne: $3.4\,\mathrm{V/Å}$-$3.8\,\mathrm{V/Å}$
- current controlled by gas pressure
- p_{He}: 5×10^{-6} mbar
- p_{Ne}: 2×10^{-6} mbar
- current: 0.1 pA-10 pA (after aperture)

Optimum operation conditions

Best imaging voltage (BIV)

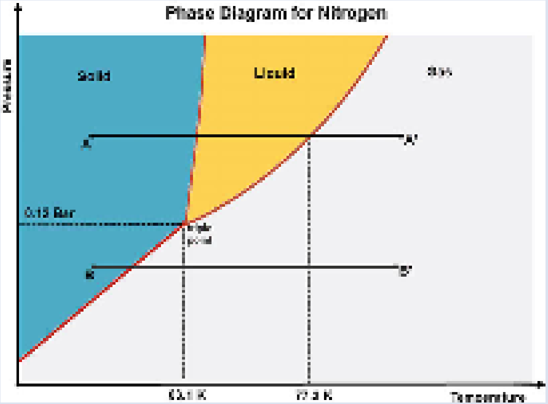


Cryogenic cooling

- Required to keep source cold
  - Better emission
  - Trimer stability
- Vibration free
- Cost efficient
  - nitrogen based

Solid nitrogen cooling

N_2 phase diagram



Overview

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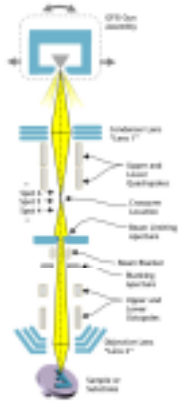
- Other subsystems

Ion optical column

GFIS column

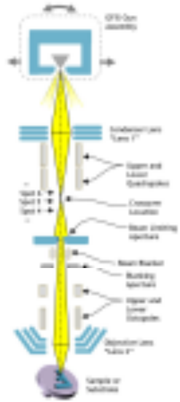


Key properties

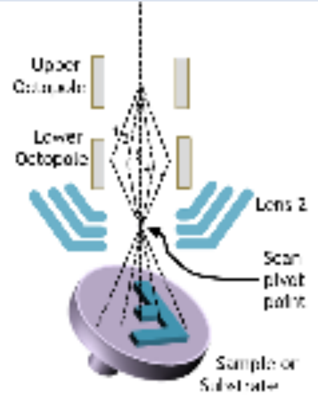
- Optical magnification ≈ 1
- 3 Cross overs points
- 14 Apertures + Big-Hole
- Upper Quadrupol
- Lower Octopol
- Two electorstatic lenses
- Acceleration voltage 3.5 keV to 40 keV
- BIV (typical): 30 keV to 35 keV
- FOV: 0.1 µm to 1000 µm

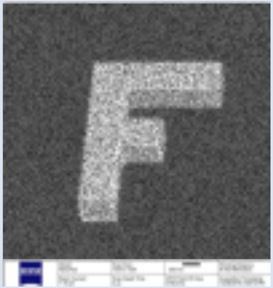
Ion optical column

GFIS column



Normal imaging

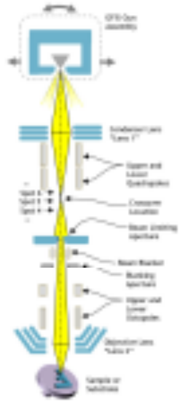




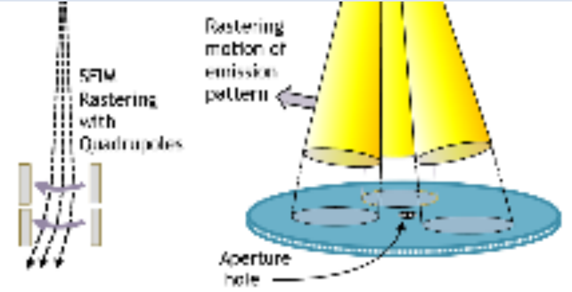
Typical (normal) imaging of a sample

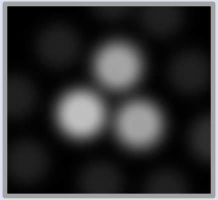
Ion optical column

GFIS column



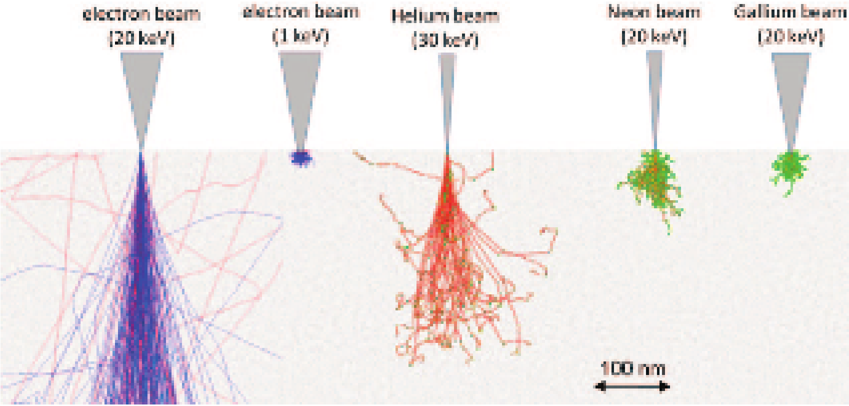
SFIM mode





Typical SFIM Image of the Source Trimer

Resolution I

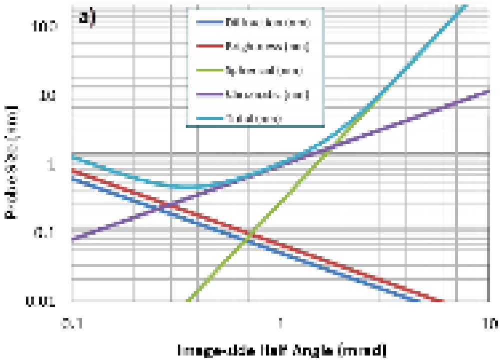


SRIM: Ga, Ne, He; CASINO: e^{-}

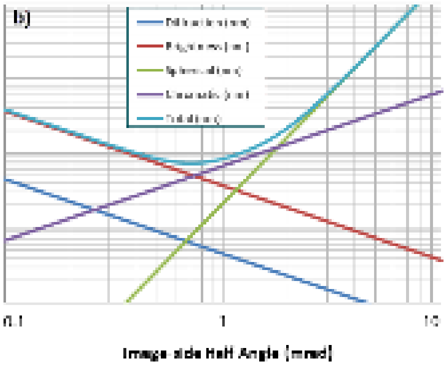
Resolution II

Beam optics

0.5 pA 30 keV He



20 pA 30 keV He



- 0.35 nm beam diameter
- 0.7 nm beam diameter

DRESDEN concept

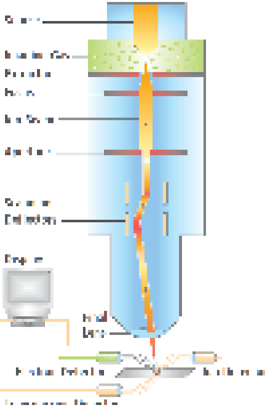
Helium Ion Microscope

HIM

- Helium ions (charged particle optics)
- Scanning beam
- High resolution
- Charge compensation

Benefits

- Smallest beam diameter
  - Probe size: 0.4 nm
- beam semi-angle: 0.8 mrad
- Energy spread: 1 eV
- Brightness: $> 10^9$ Acm^{-2}sr^{-1}
- High resolution: ≈0.35 nm (He: < 0.5 nm, Ne: 1.8 nm)



Helium Ion Microscopy, G. Hlawacek & A. Gölzhäuser, Springer 2016

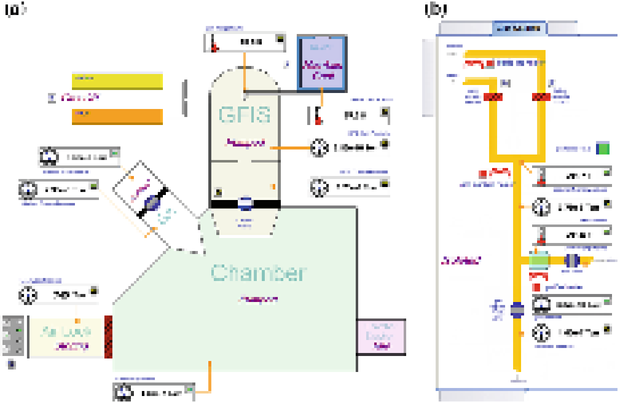
The NanoFab in Dresden

- He, Ne, 0.5 nm resolution
- 4 Kleindiek MM3A
  - electrical characterization
  - rotation axis
  - gripper
- home built heater (<770 K)
- NPVE patterning software
- GIS (Omniprobe II)
  - XeF_2
  - tungsten, SiO_x
- TOF-BS
- TOF-SIMS

Overview

Vacuum system

- source: $<1 \times 10^{-10}$ mbar
- source: 5×10^{-7} mbar to 1×10^{-5} mbar
- sample: $<5 \times 10^{-7}$ mbar



- Gun turbopump
- Column IGP
- chamber turbopump
- Gun booster (turbo)
- Gun roughing (2 stage membrane)
- Chamber booster
- Chamber roughing (2 stage membrane)
- Nitrogen dewar pump (rotary vane)

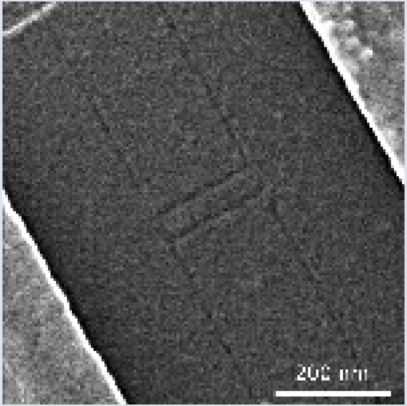
Overview

Overview

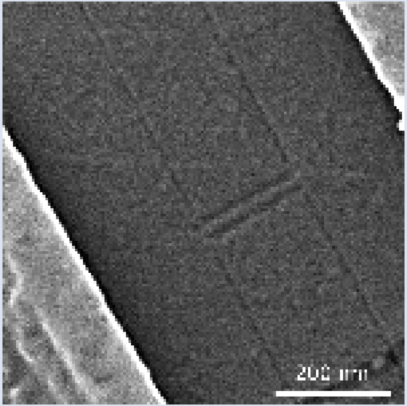
3 Low fluence metal free materials modification and in-situ probing
- 2D Materials structuring
- Creating, shaping and modification of nano magnets
- Towards a single electron transistor
- From 2D to 1D and 0D

Nanoribbons in h-BN/Gr/h-BN stacks

Width: 50 nm; Length: 200 nm

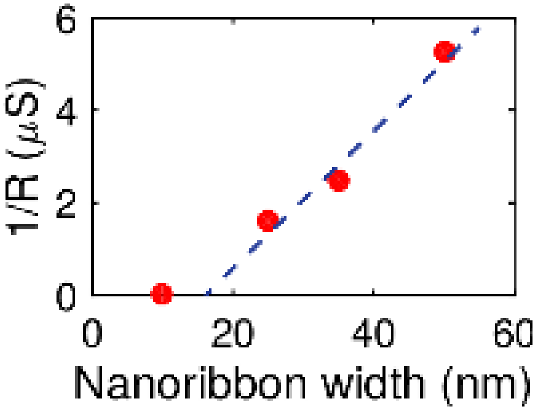


Width: 25 nm; Length: 200 nm



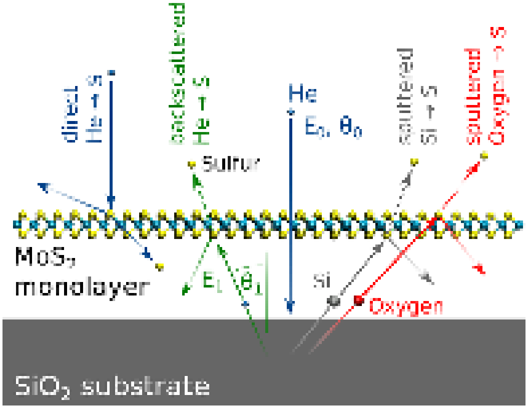
G. Nanda, et al., Carbon (2017)

Graphene nano ribbon conductivity



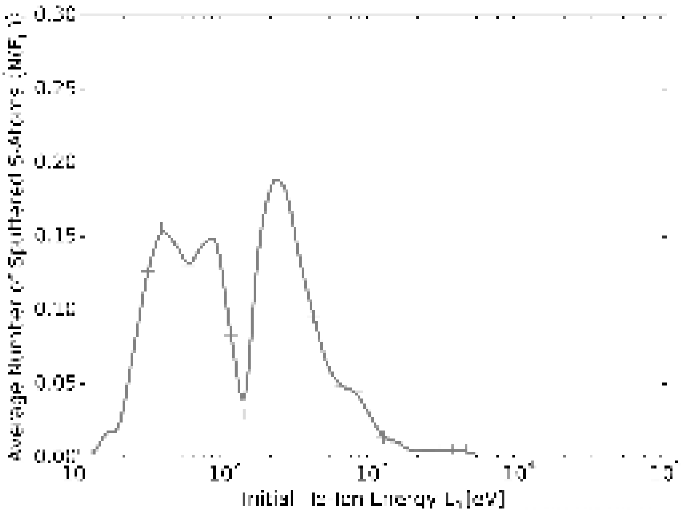
- indicates a 8 nm dead zone next to the cut
- can not be explained by the beam tails alone

Damage mechanism



Defect production

He irradiation @ MoS_2 on SiO_2



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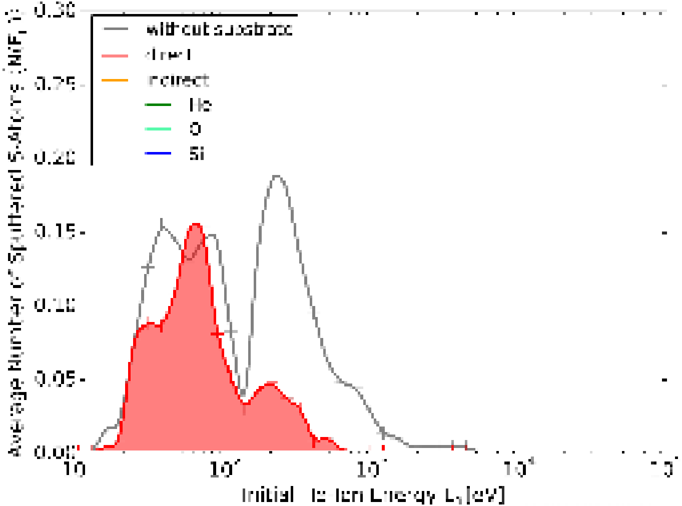

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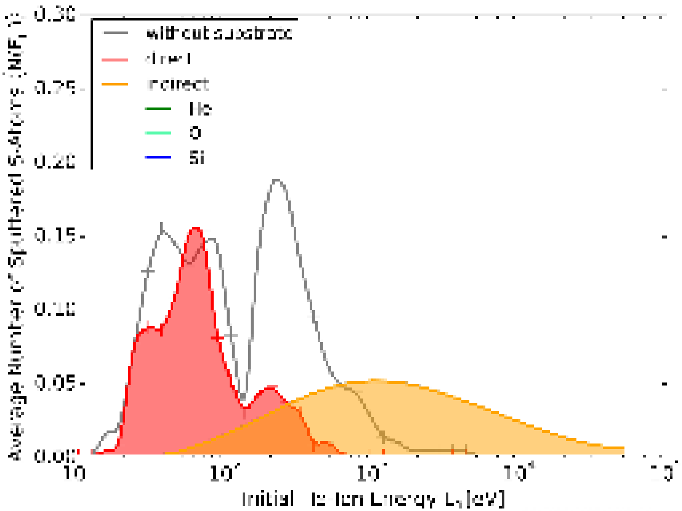

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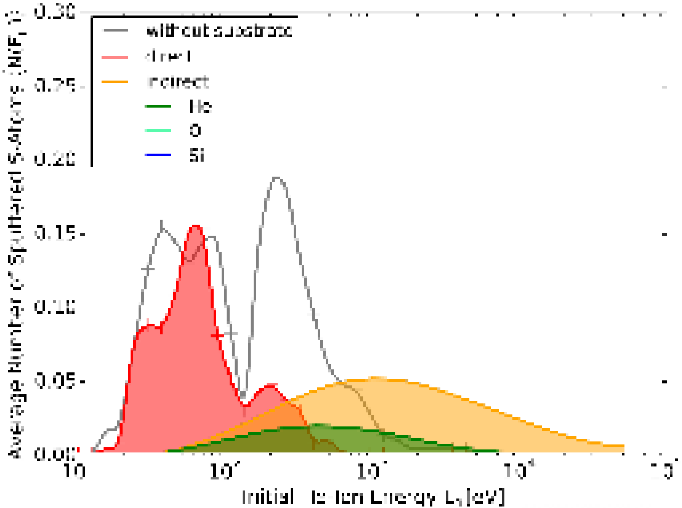

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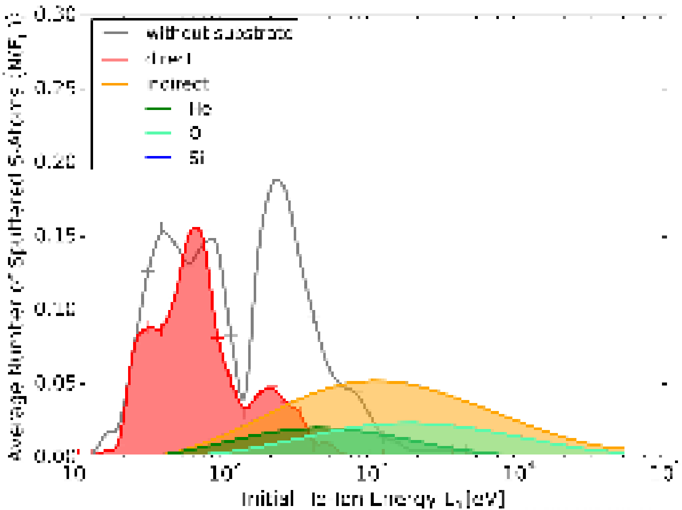

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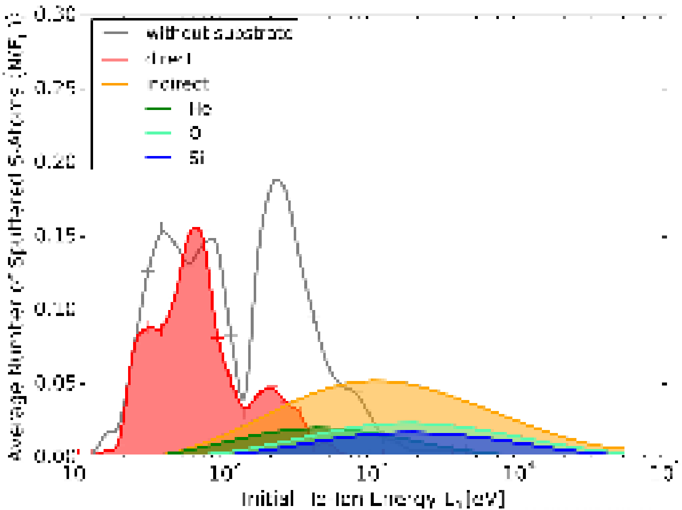
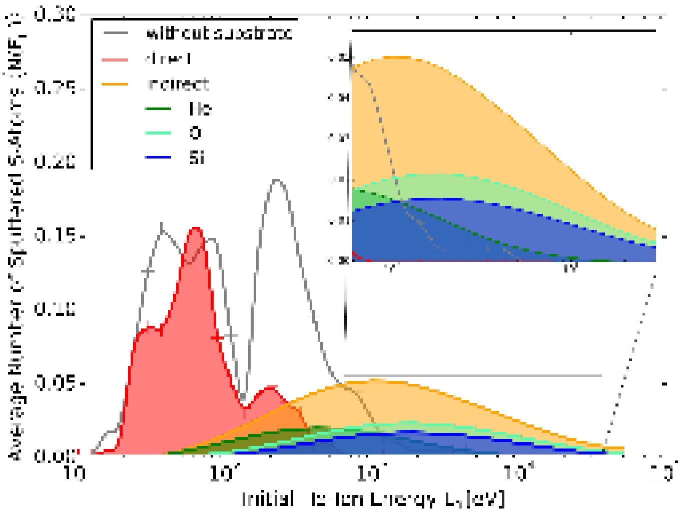
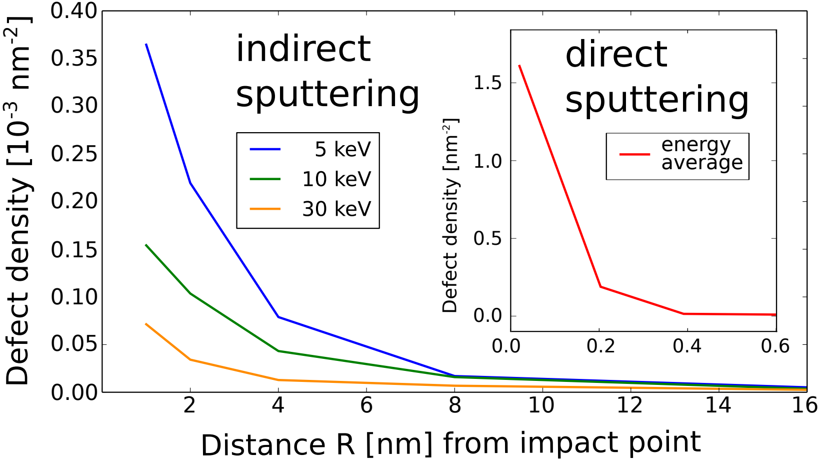


Defect production

He irradiation @ MoS_2 on SiO_2

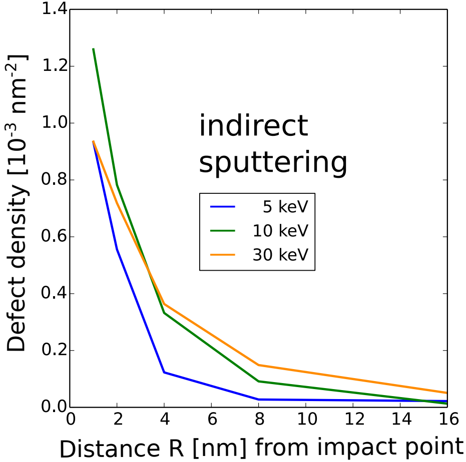


Spatial distribution of defects in MoS_2 by He



S. Kretschmer, et al., Appl. Mater. Inter. (2018)

Spatial distribution of defects in MoS_2 by Ne



S. Kretschmer, et al., Appl. Mater. Inter. (2018)

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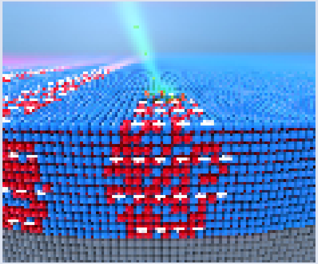
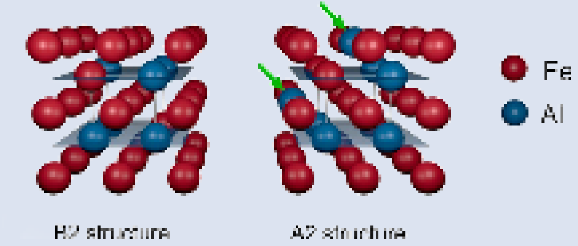
Creating nano-magnets

Creating local disorder with 25 keV He^{+}



$Fe_{60}Al_{40}$

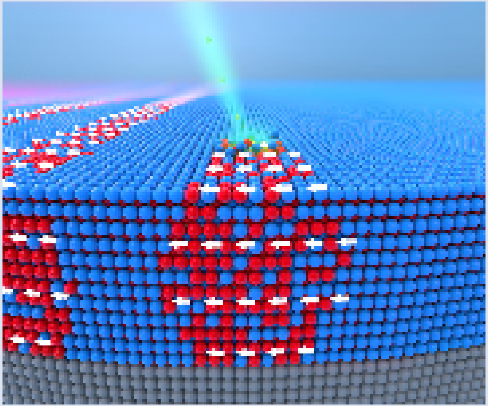


Properties

- simple cubic paramagnetic phase (B2)
- body centered cubic ferromagnetic phase (A2)
- Fe-Fe n-ns: 2.67 (B2) to 4.8 (A2)
- increased number of n-ns induces magnetism

Ion beam writing nano-magnets

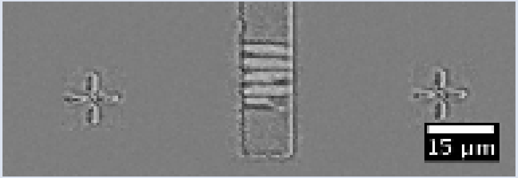
HIM at 25 keV Ne, 2 nm spot size

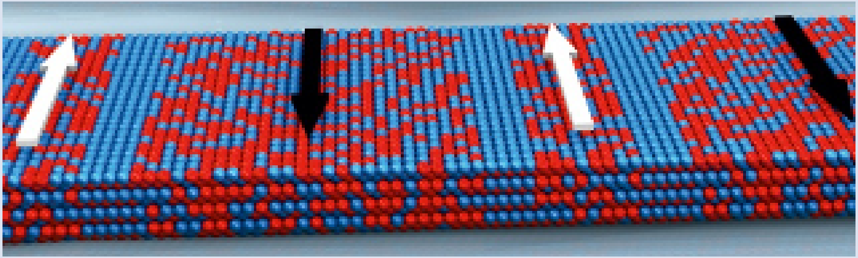


Check the results

- 6×10^{14} Ne/cm^2 (1 pA/(μs px)) @25 keV; gap: 40 nm

Kerr Microscopy

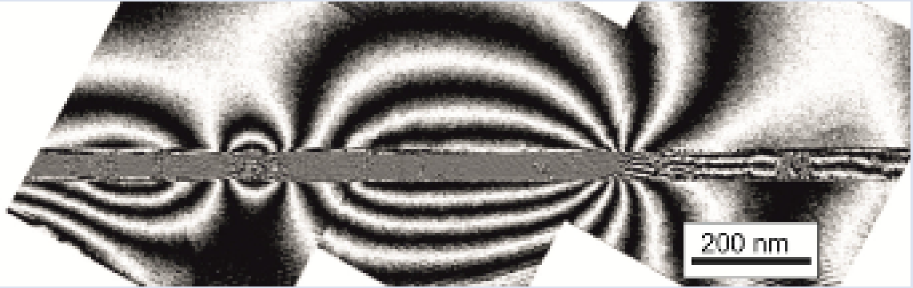




R. Bali, et al., Nano Letters 14(2), 435 (2014).

and observing it

TEM Holography



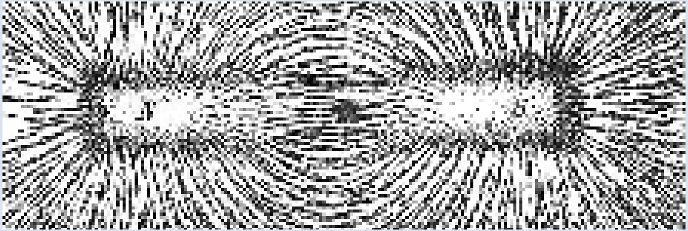
TEM off-axis holography

- 200 keV, 2 T saturation field
- ϕ_{mag} extracted by field reversal to remove ϕ_{el}
- $20 \cos \phi_{mag}$ plotted for a 76 nm thick lamella

F. Röder, et al., Scientific Rep. (2015).

and observing it

TEM Holography



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- ϕ_{mag} extracted by field reversal to remove ϕ_{el}
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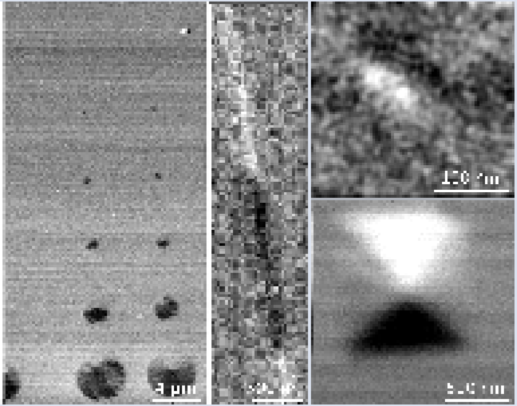
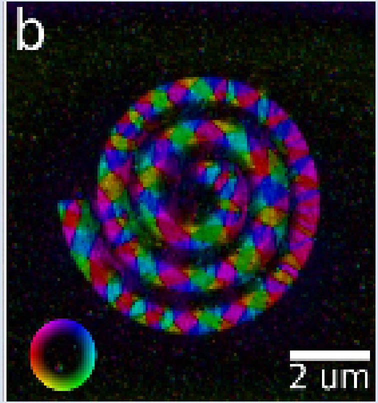
F. Röder, et al., Scientific Rep. (2015).

Arbitrary shaped nano-magnets

XMCD

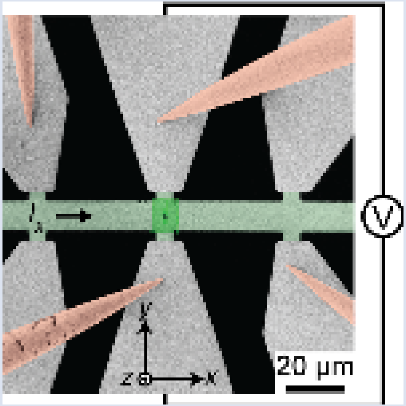


STEM-DPC



In-situ Control of Anisotropy for Spin Torque Switching

4 probe geometry



- 25 keV local He irradiation
- 3x[Pt/Co/Ta] SOT stack

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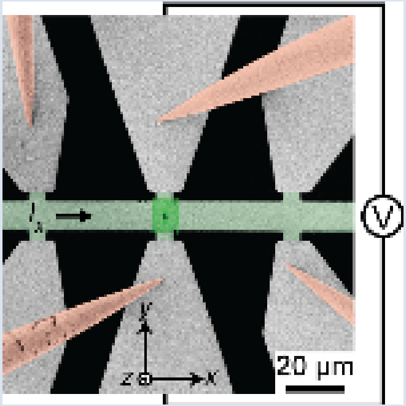
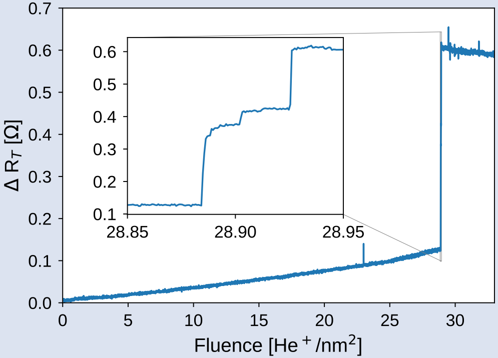


- 25 keV local He irradiation

Out of plane to in-plane magnetization



- 3x[Pt/Co/Ta] SOT stack

Overview

3 Low fluence metal free materials modification and in-situ probing

- 2D Materials structuring
- Creating, shaping and modification of nano magnets
- Towards a single electron transistor
- From 2D to 1D and 0D

What we will build

Targeted benefits

- low power single electron transistor

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- room temperature operation

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What we will build

Targeted benefits

- low power single electron transistor
- room temperature operation
- CMOS compatible

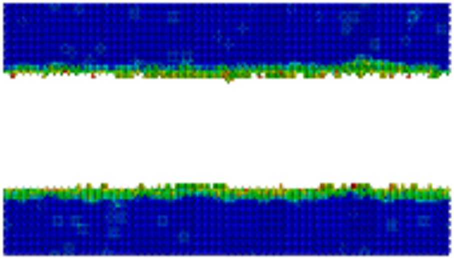
Requirements

- nanocrystal size $\approx 2 \ldots 3\,\mathrm{nm}$
- separation from source and drain $\leq 2\,\mathrm{nm}$
- single nanocrystal
- massively parallel site specific nanocrystal formation

A possible solution

Solution

- ion beam mixing of $Si/SiO_2/Si$ into $Si/SiO_x/Si$
- thermally stimulated self organization (RTA)



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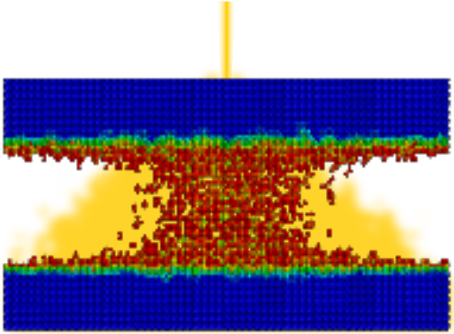

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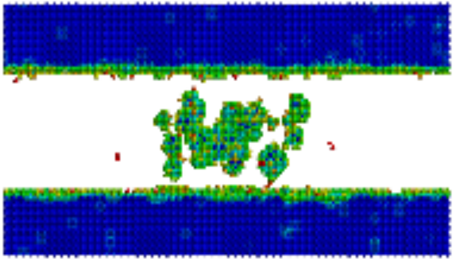

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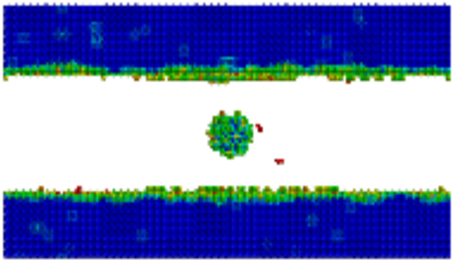

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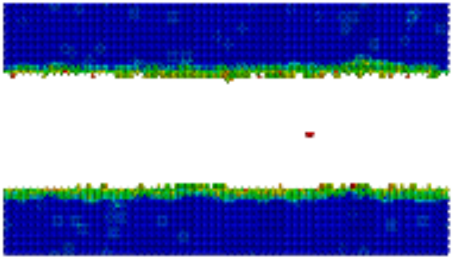
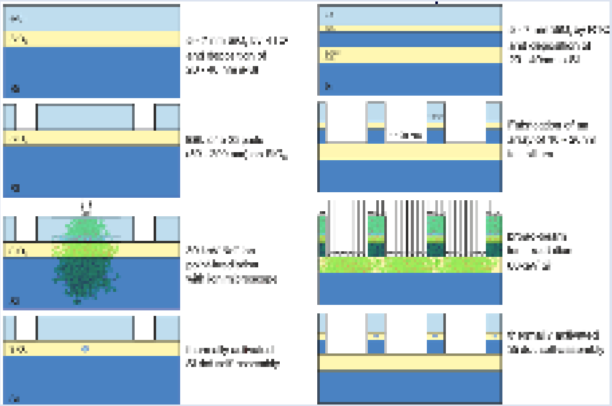


How to create a single Si nanocrystal—in reality

Two routes

Academic **Industry**



Overview

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- 2D Materials structuring
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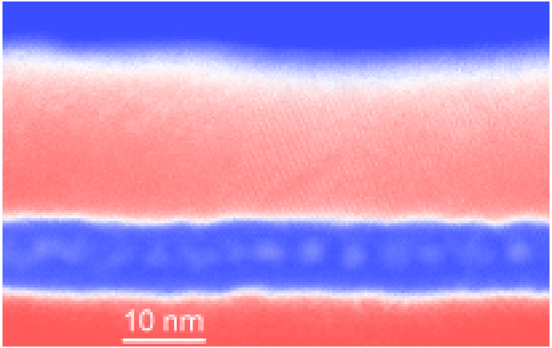
A 2D layer of nanocrystals using broad beam irradiation



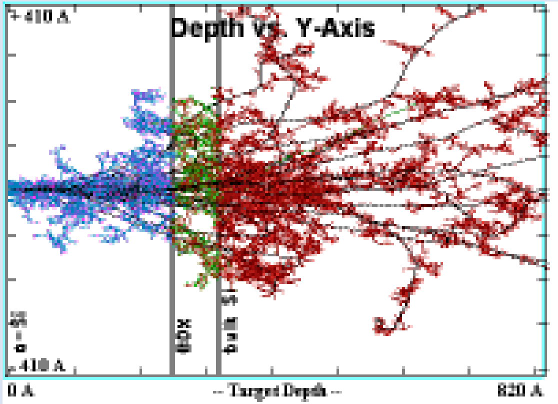
- diameter: 2.3 nm$\pm$0.6 nm
- spacing $\approx$12 nm
- tunneling gap: 2.1 nm

Parameters

- Ion fluence: $\approx 170\,\mathrm{Si}^+/\mathrm{nm}^2$
- Ion energy: 50 keV
- Thermal treatment: 1323°C, 60 s

Rescaling of ion beam fluence

From Broad beam to point like irradiation



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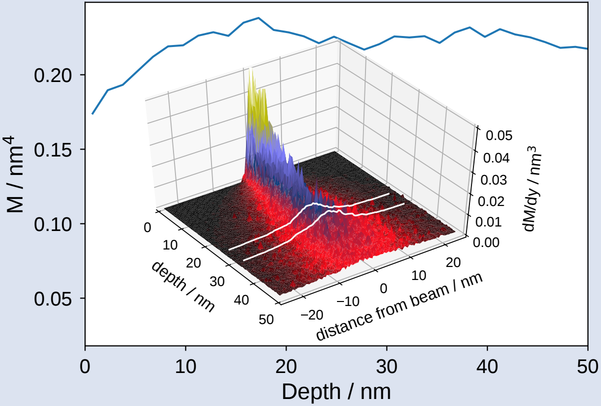
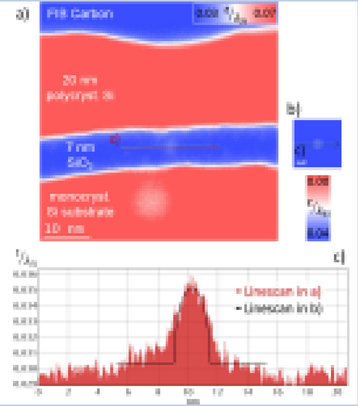


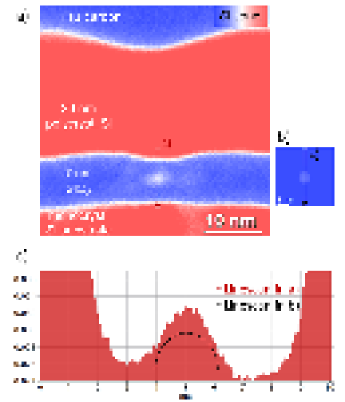
1D line irradiation

3000 Ne/nm^2 at 25 keV into a single line

0 nm line width

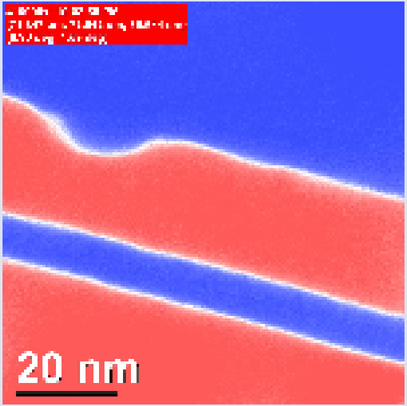


4 nm line width



0D point irradiation

Increasing the fluence from 1250 Ne/nm^2 to 20000 Ne/nm^2



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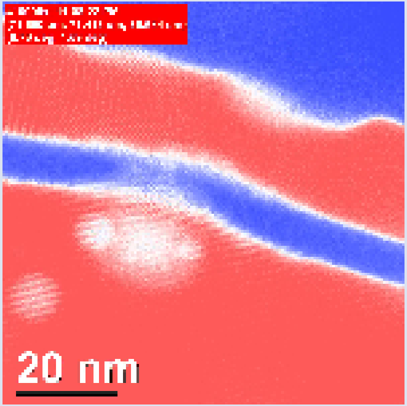

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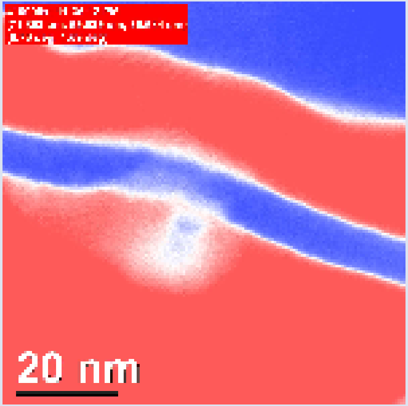

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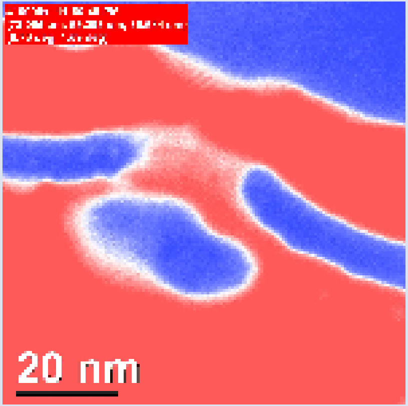
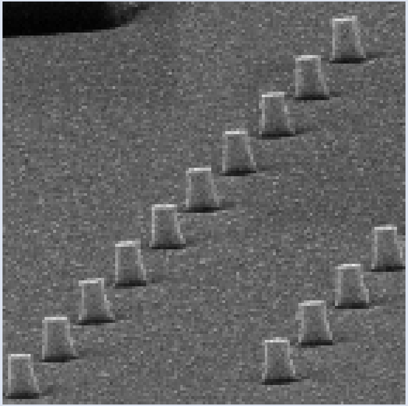


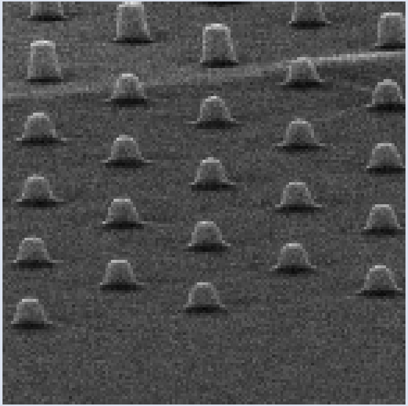
Irradiating individual pillars

Si pillar unirradiated



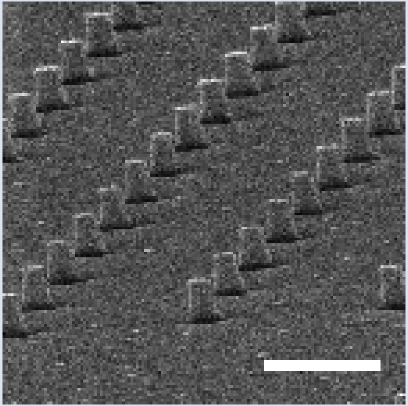
Irradiating individual pillars

Si pillar 2×10^{16} Ne$^{+}/cm^{2}$ at RT



Irradiating individual pillars

Si pillar 2×10^{16} Ne^{+}/cm^{2} at 400°C



Irradiating individual pillars

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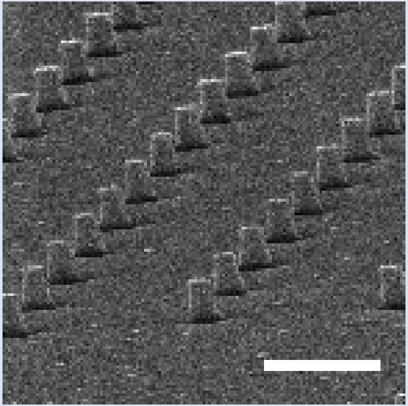
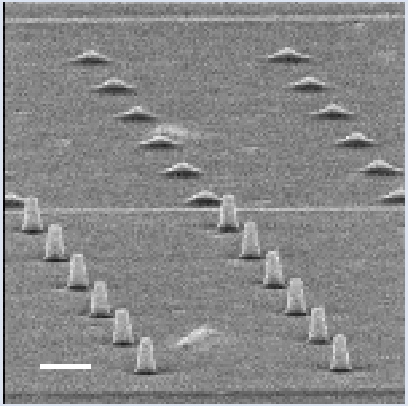


Direct comparision

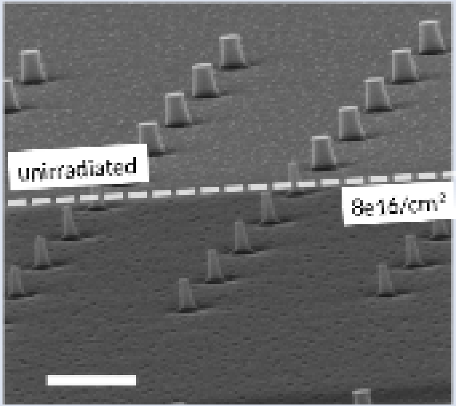
RT $(2 \times 10^{15}\,\mathrm{cm}^{-2})$ + HT $(1.8 \times 10^{16}\,\mathrm{cm}^{-2})$

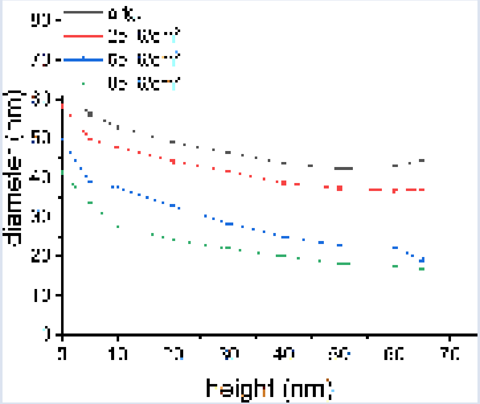


HT $(1.8 \times 10^{16}\,\mathrm{cm}^{-2})$ + RT $(2 \times 10^{15}\,\mathrm{cm}^{-2})$

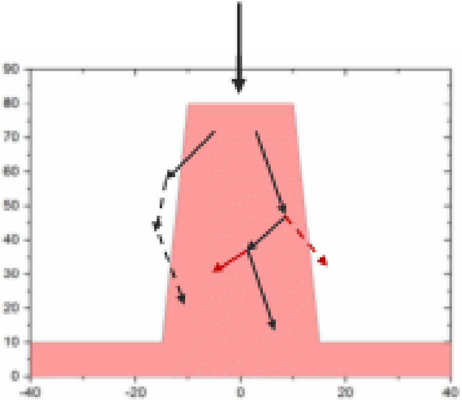
Homogeneous diameter reduction during ion irradiation

8×10^{16} Ne^{+} at 400°C into 50 nm Si pillars

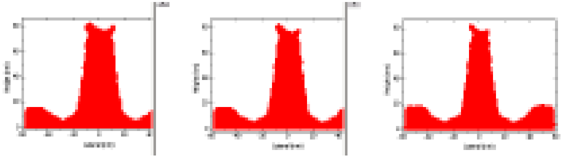




Why is this happening

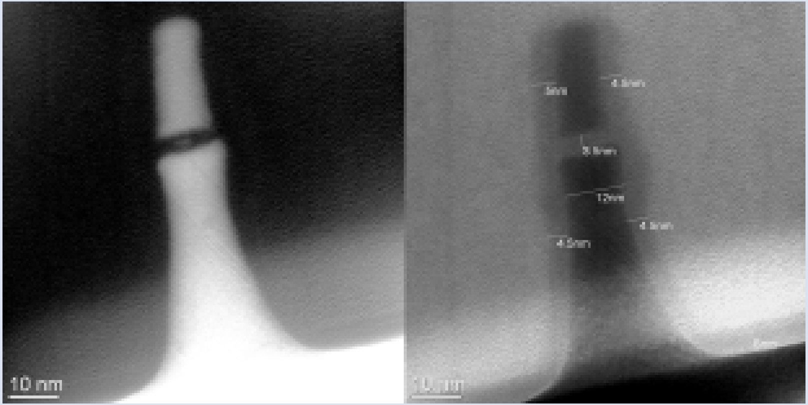


- small temperature increase keeps structure crystalline
- viscous flow of amorphous Si suppressed
- shape is preserved at HT
- highly efficient forward sputtering on the side walls



Broad beam irradiation followed by pillar and cluster fabrication

Forming a single addressable ≈2 nm Si cluster

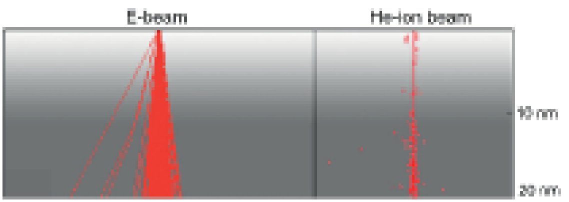


X. Xu, et al. Beilstein Journal Nanotech., 2019

HIBL—Helium ion beam lithography

Resist exposure without proximity effect

Properties of SEs



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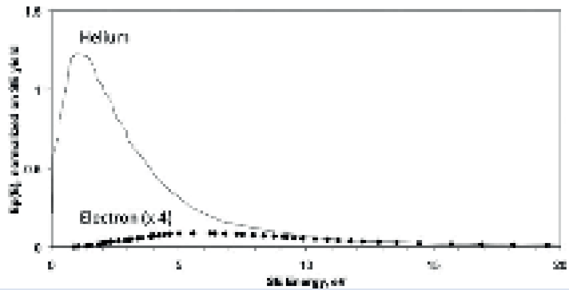

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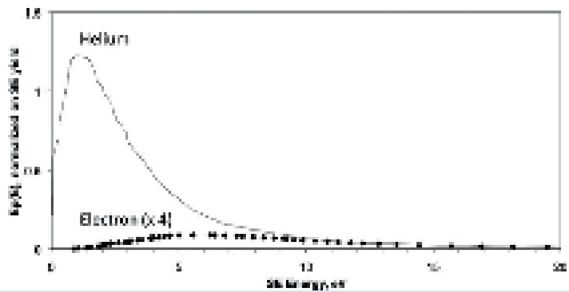
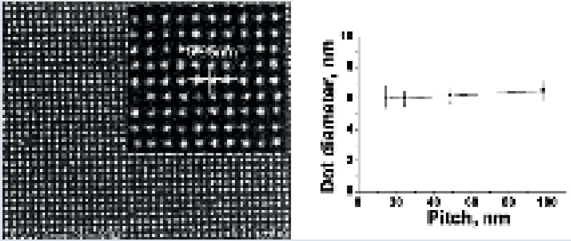


Results



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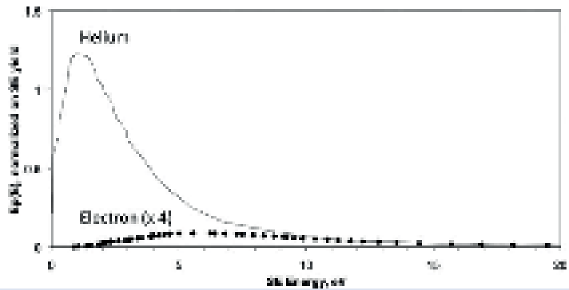

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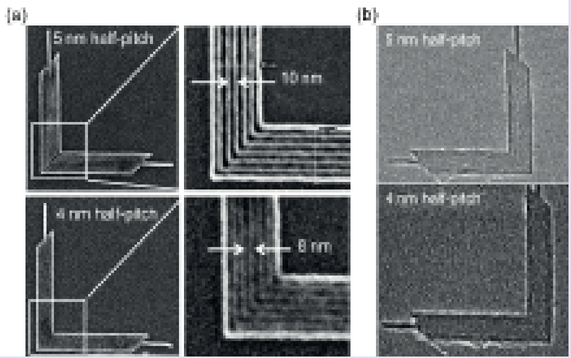

Overview

Summary
Conclusion

Results

- HIM (FIB) is more than just TEM sample prep
- High resolution imaging
- Nanoscale ion beam analysis
- high resolution milling
- Low fluence materials modification aka. defect engineering
- Apply for beamtime

IBC.

How to get beamtime at the IBC

1. Read the general information
   - https://www.hzdr.de/ibc Ion Beam Center
   - https://www.ionbeamcenters.eu transnational access also to other centers (travel grants!)
   - https://www.hzdr.de/fwiz-n information and contacts to HIM/FIB experts
2. Register at https://gate.hzdr.de
3. Use one! of the following proposal templates (IBC and RADIATE have slightly different templates)
   - https://www.ionbeamcenters.eu/radiate_scientific_case_form/
   - Take the direct templates from the IBC homepage
4. This is the latest point to discuss your proposal with us.
5. I mean it. Talk to us!!
6. Submit the proposal at https://gate.hzdr.de (selected either IBC or RADIATE as the infrastructure)
7. Wait up to 6 weeks for the proposal to be reviewed by an international panel.
8. Welcome at the IBC!