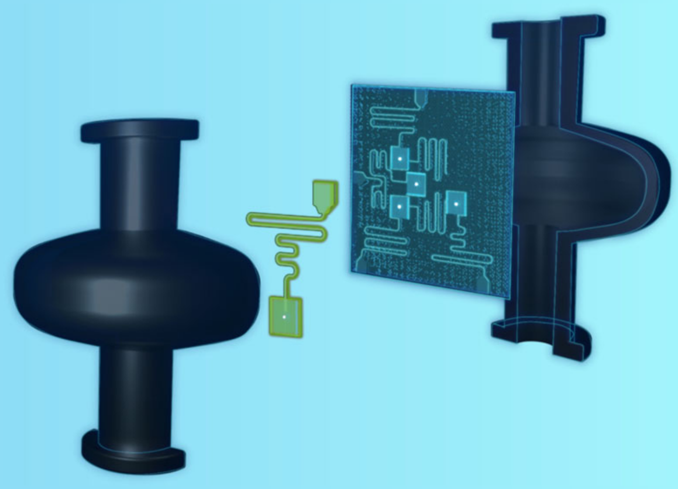


Introduction to Electron Microscopy and Associated Techniques

Lin Zhou

Department of Materials Science and Engineering, Iowa State University

& Ames National Laboratory

Outline

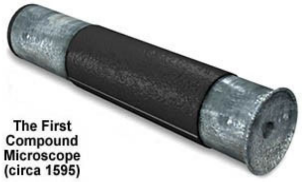
- History of electron microscope
- Types of electron microscopes and major instrument components
- Scanning electron microscopy (SEM)
- Focus ion beam (FIB)
- Transmission electron microscopy (TEM)
- Application of electron microscopy in superconducting qubits

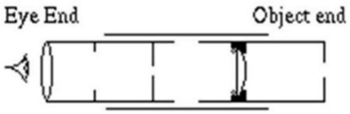
I. History of Microscopes

Microscope is an instrument for viewing what is small.

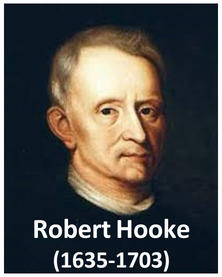
- Spectacle-makers and father-and-son team, **Hans and Zacharias Janssen** create the first microscope (~1590)

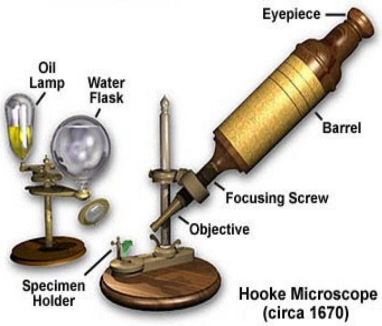
Zacharias Janssen
(1580-1638)



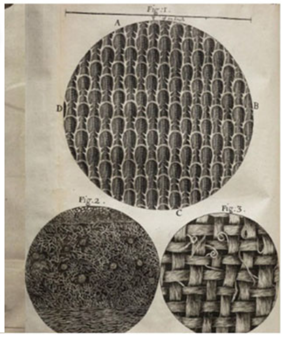


- **Robert Hooke** publishes "*Micrographia*" (~1667)





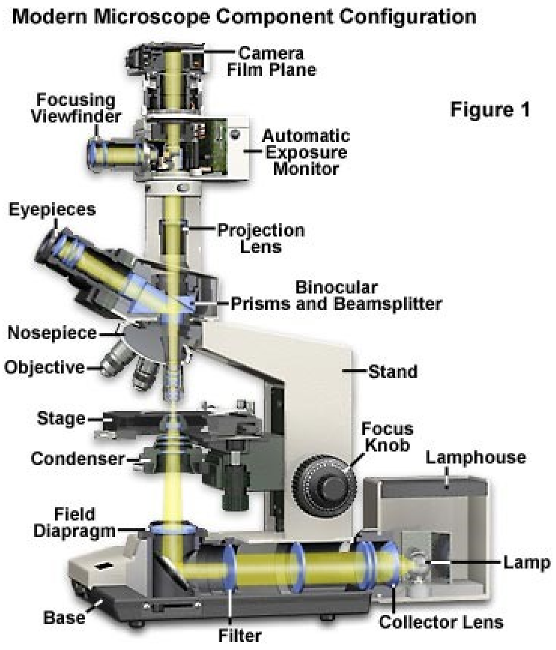
Hooke Microscope
(circa 1670)



Left, Hooke's microscope. Robert Hooke, Micrographia, London, 1665, Scheme 1, opposite sig. G2v. Right, surfaces of seaweed, rosemary leaf and fine lawn. Ibid., Scheme 14, opposite p. 141.

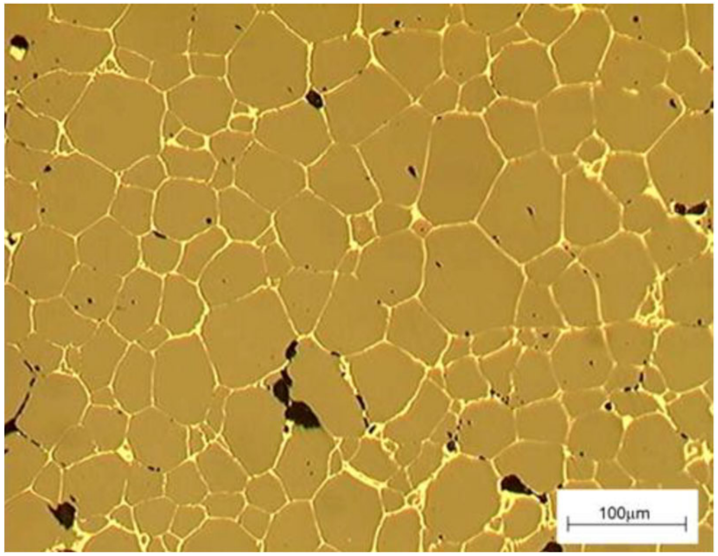
https://www.microscope.com/education-center/microscopes-101/history-of-microscopes
https://www.sciencelearn.org.nz/resources/1692-history-of-microscopy-timeline

Modern Optical Microscopes



https://micro.magnet.fsu.edu/primer/anatomy/bx51cutaway.html

W

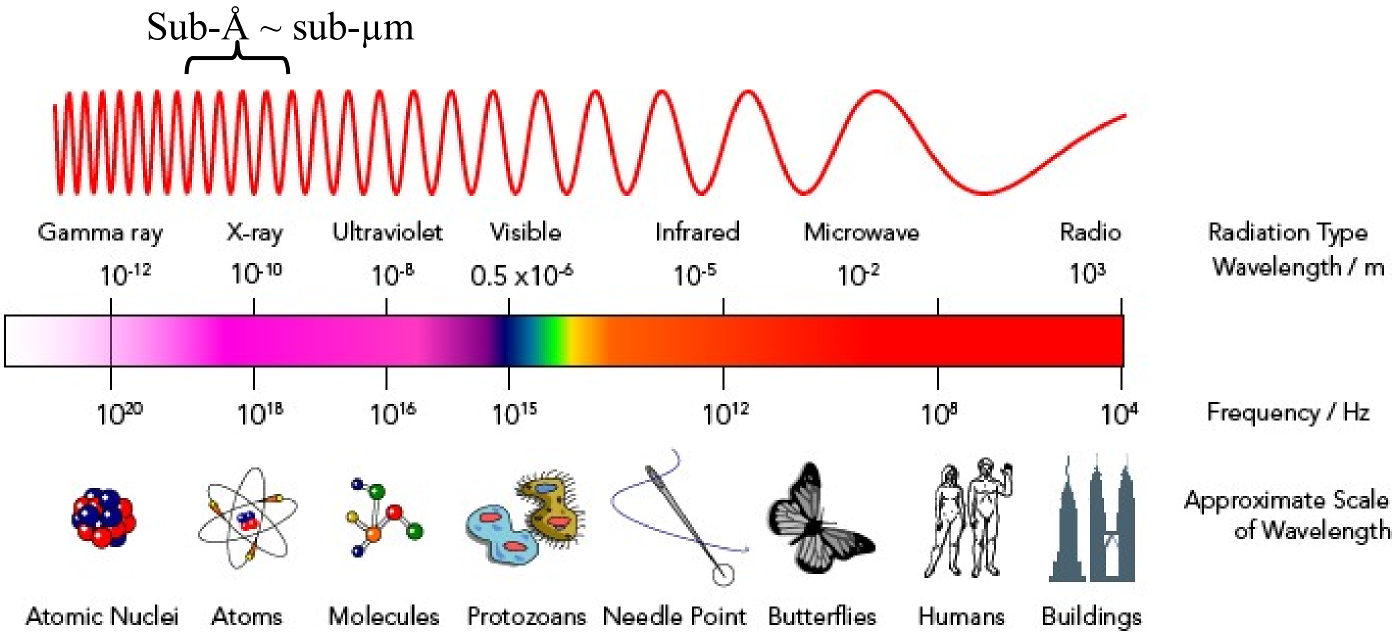


Proceedings of WRFPM 2014, Sendai, Japan, Sep. 14-17, 100092

Why Electron Microscopy

- High resolution (short wavelength, Electron: 200KV: ~ 0.025Å)
- Strong electron-matter interaction (large scattering cross section)

-> An ideal tool for imaging on the atomic to nanoscale

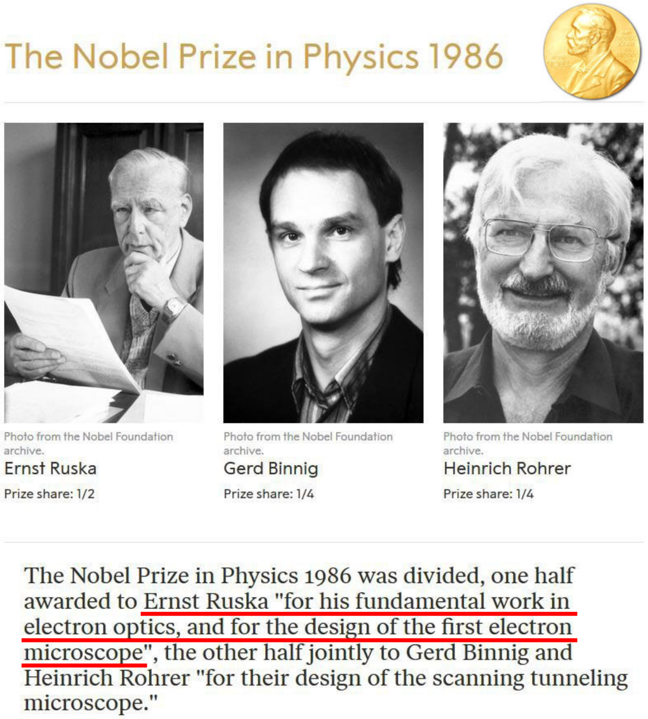


https://ozonedepletiontheory.info/ImagePages/em-spectrum-properties/

Invention of Electron Microscopes

first TEM, 1931

Ruska (in the lab coat) and Knoll Berlin in the early 1930s



The Nobel Prize in Physics 1986

Photo from the Nobel Foundation archive.
Ernst Ruska
Prize share: 1/2

Photo from the Nobel Foundation archive.
Gerd Binnig
Prize share: 1/4

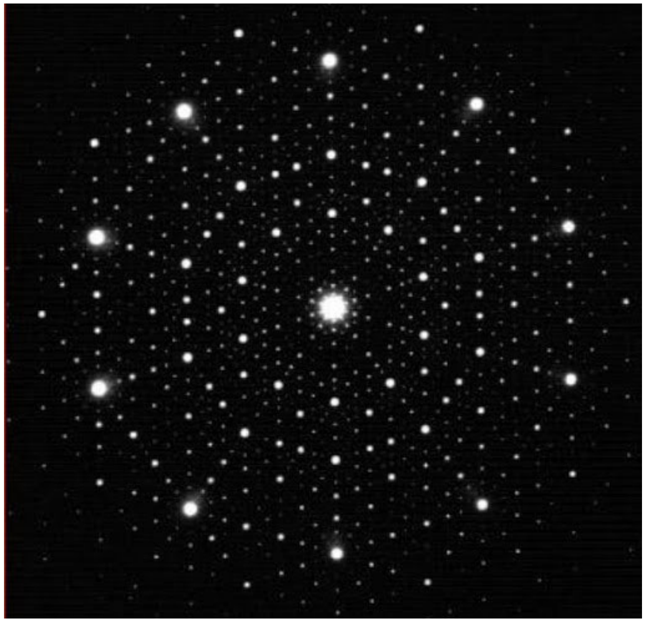
Photo from the Nobel Foundation archive.
Heinrich Rohrer
Prize share: 1/4

The Nobel Prize in Physics 1986 was divided, one half awarded to Ernst Ruska "for his fundamental work in electron optics, and for the design of the first electron microscope", the other half jointly to Gerd Binnig and Heinrich Rohrer "for their design of the scanning tunneling microscope."

Nobel Prize in Chemistry 2011: Quasicrystals

Dan Shechtman

"for the discovery of quasicrystals"



- The Nobel Prize in Chemistry 2011 – Quasicrystals: Dan Shechtman

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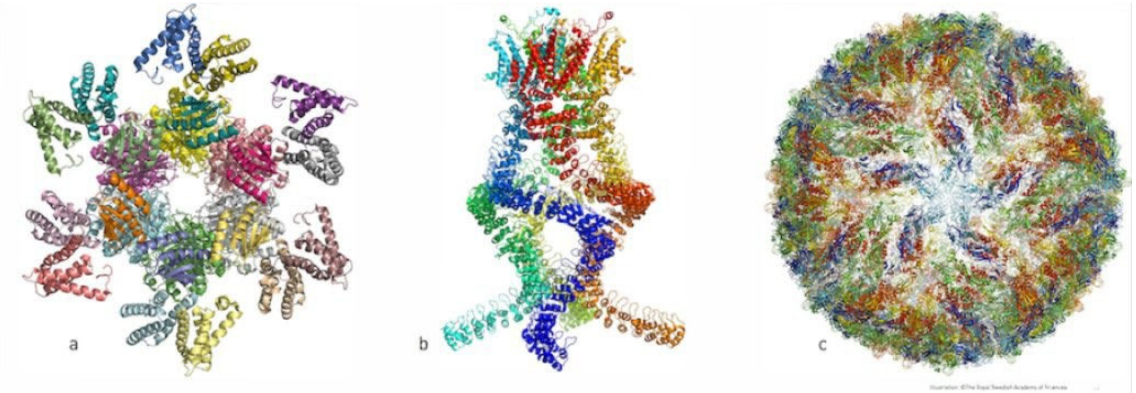
Nobel Prize in Chemistry 2017: Cryo-EM



“for developing cryo-electron microscopy for the high-resolution structure determination of biomolecules in solution”

Jacques Dubochet **Joachim Frank** **Richard Henderson**

Credit: © Johan Jarnestad/The Royal Swedish Academy of Sciences

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Outline

- History of electron microscopes
- **Types of electron microscopes and major instrument component**
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- Focus ion beam (FIB)
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SUPERCONDUCTING QUANTUM MATERIALS & SYSTEMS CENTER

Sensitive Instrument Facility

- SEM/FIB/(S)TEM: ThermoFisher (Philips, FEI.), JEOL, Tescan, Hitachi…
- SEM (typically operated at <30 kV), TEM (typically operated between 30-300 kV)

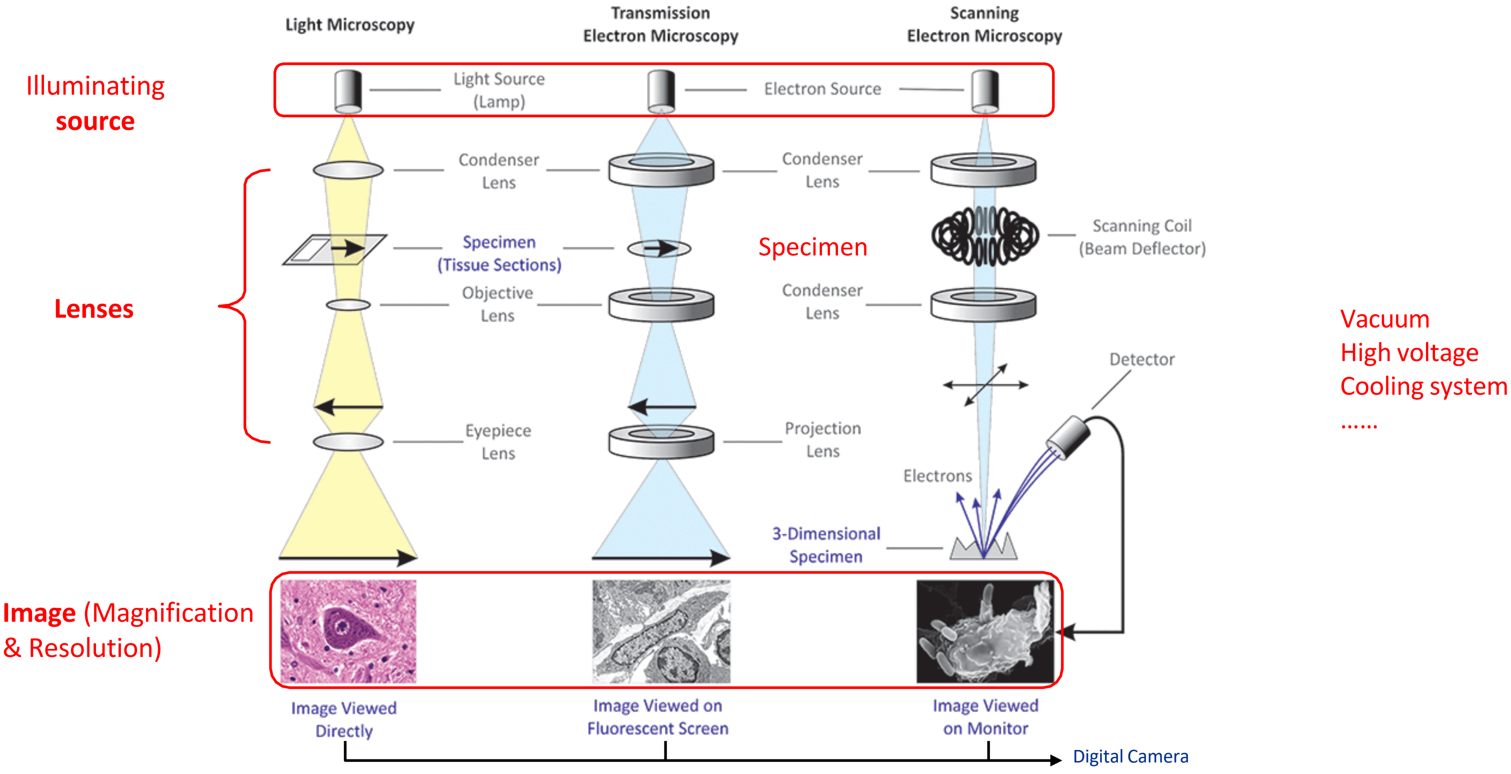
SEM-Teneo

FIB-Helios

STEM-Tecnai

STEM -Titan Themis

II. Microscopes: Optical, TEM, SEM

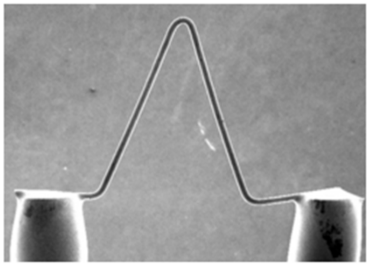


Vacuum
High voltage
Cooling system
……

https://microbiologyinfo.com/differences-between- light-microscope-and-electron-microscope/

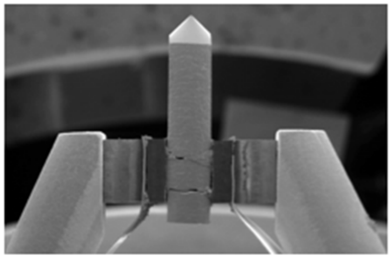
Electron Source

W



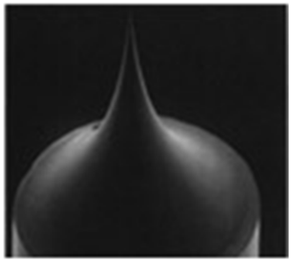
- E_w: 4.5 eV
- Temperature: 2700K
- Vacuum < 10^{-3} Pa (10^{-5} torr)
- d_0 : 30 ~ 100 mm
- Brightness: 10^4 ~ 10^5 A/cm^{2}sr
- inexpensive, but a short lifetime
- Stable high current: Suitable for X-ray analysis

LaB$_6$



- E_w ↓ : 2.5 eV
- Temperature ↓: 1800K
- Vacuum < 10^{-5} Pa (10^{-7} torr)
- Tip : d_0 = 5 ~ 50 mm
- Brightness: 10^5 ~ 10^6 A/cm^{2}sr

Field Emission Gun

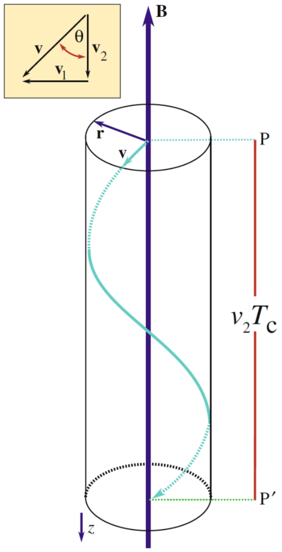


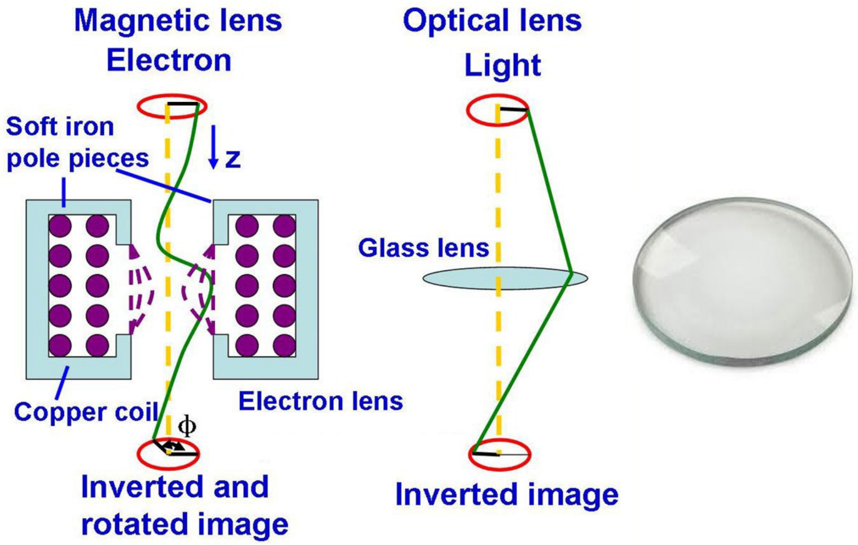
- Electric field : >10^7 V/cm
- Vacuum < 10^{-8} Pa (10^{-10} torr)
- d_0 : < 5 nm
- Brightness (b) : ~10^8 A/cm^{2}sr
- long lifetime (>1000 hr)
- Energy spread (ΔE) < 1 eV

Goldstein et al., Scanning Electron Microscopy and X-Ray Microanalysis, Third Ed. (2003)
https://www.nanoscience.com/techniques/scanning-electron-microscopy/components/

Lens

- Electromagnetic lens (for electrons)





Magnetic lens
Electron

Optical lens
Light

Soft iron
pole pieces

z

Glass lens

Copper coil

Electron lens

ϕ

Inverted and
rotated image

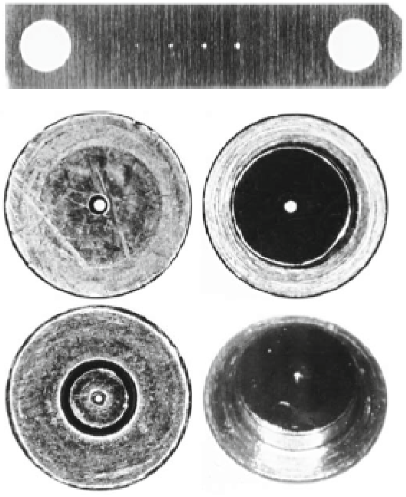
Inverted image

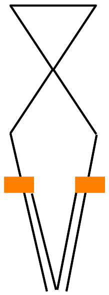
- Use a magnetic field for focusing

- Magnetic lens behaves only like a convex lens!

D. Williams & B. Carter, Transmission Electron Microscopy: A Textbook for Materials Science, Springer (2009).

Apertures





- Regulate electron/ion beam
  - Spot size
  - Convergence angle
  - Current
- Moveable (adjustable) in column

D. Williams & B. Carter, Transmission Electron Microscopy: A Textbook for Materials Science, Springer (2009).

Image Recording System

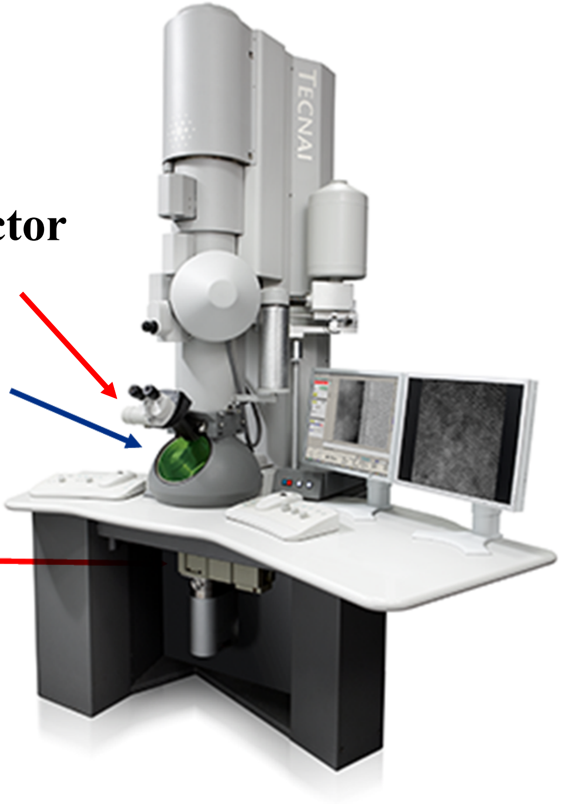
Old days…

Negative Films

Dark room: develop

STEM detector

Fluorescent screen



Nowadays: digital camera

Outline

- History of electron microscopes
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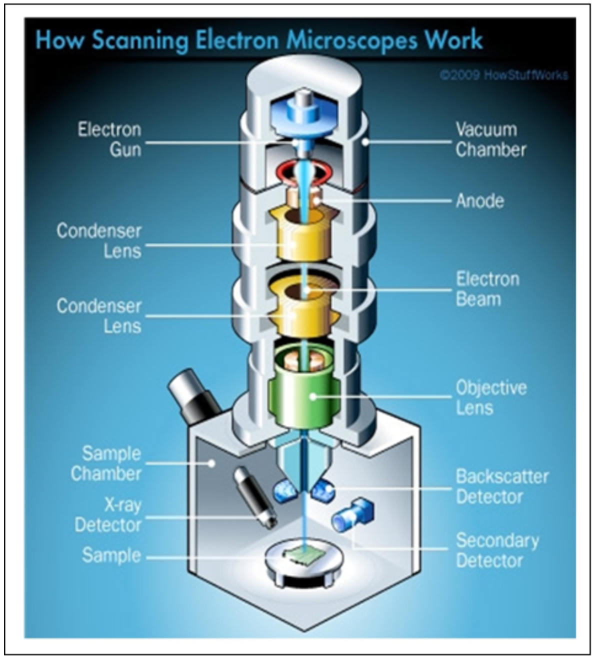
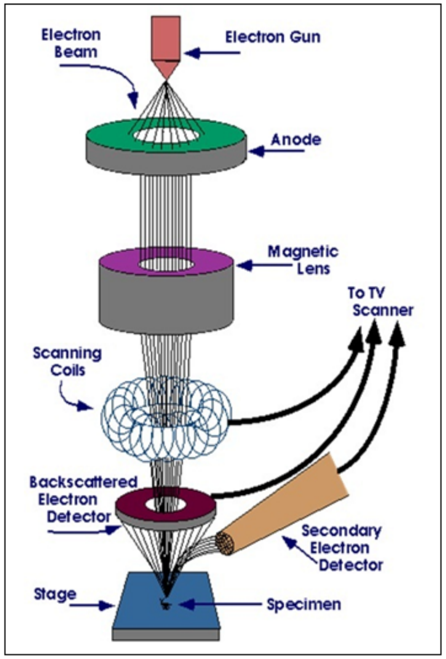
- Transmission electron microscopy (TEM)
- Application of electron microscopy in superconducting qubits

III. Scanning Electron Microscopy (SEM)

History

1942 : Ernst Ruska, Scanning electron microscope
1973 : John Venables and CJ Harland, Electron backscatter patterns observed in SEM.

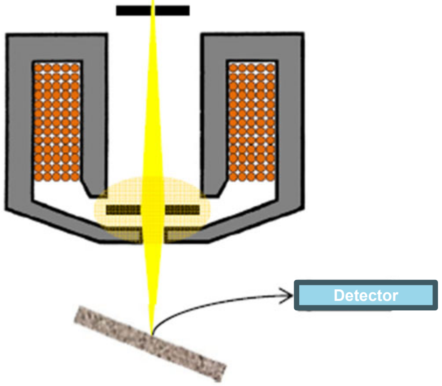




http://emicroscope.blogspot.com/2011/03/scanning-electron-microscope-sem-how-it.html

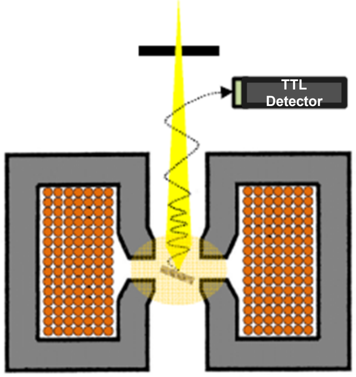
Objective Lens in SEM

Pinhole lens (conical lens)



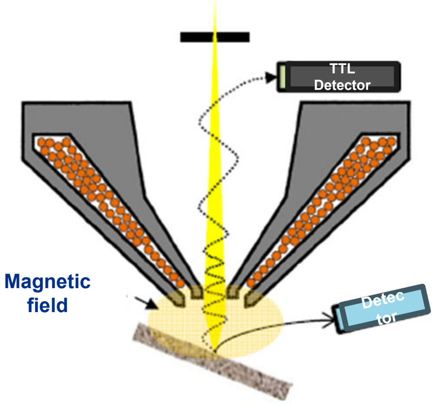
- Large specimen
- None magnetic field
- Large WD
- Large depth of field

Immersion lens (in-lens)



- Small specimen
- Lowest lens aberrations
- Smallest probe size
- Highest image resolution

Snorkel lens (semi-in-lens)

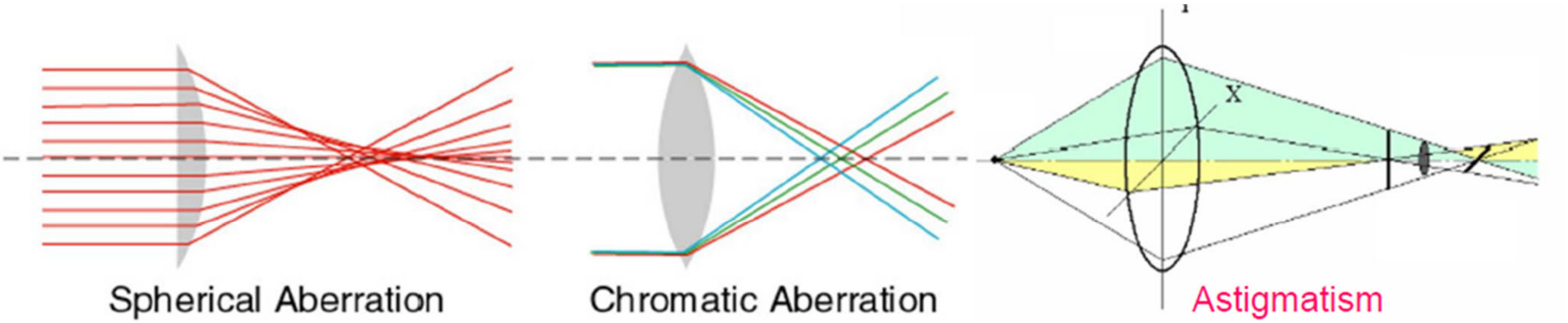


→ strong magnetic field extends out from the lens to the specimen

- Large specimen
- Small lens aberrations
- Two detectors
- High image resolution
- High imaging flexibility

Goldstein et al., Scanning Electron Microscopy and X-Ray Microanalysis, Third Ed. (2003)

Lens Aberration



Spherical Aberration

$$d_S = \frac{1}{2} C_S \alpha^3$$

Chromatic Aberration

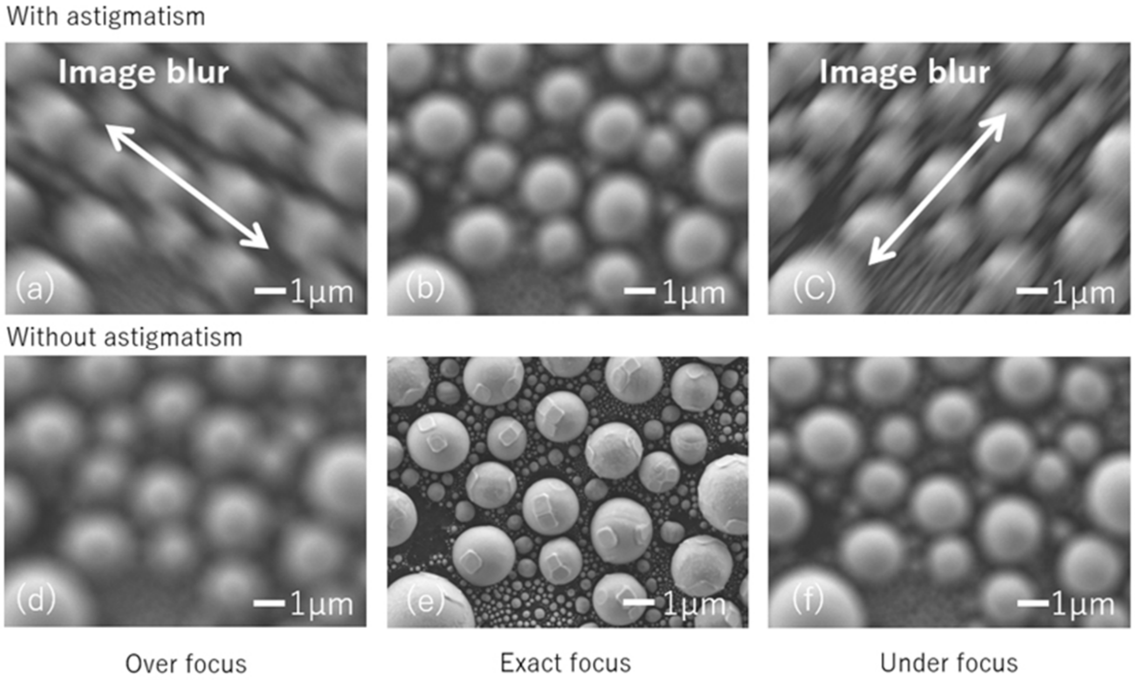
$$d_C = \frac{\Delta E}{E_0} C_C \alpha$$

Astigmatism

should be corrected !!!

- Nonuniformity in the magnetic field
- Energy spread in electrons.
- Imperfection in axial asymmetry of the lens

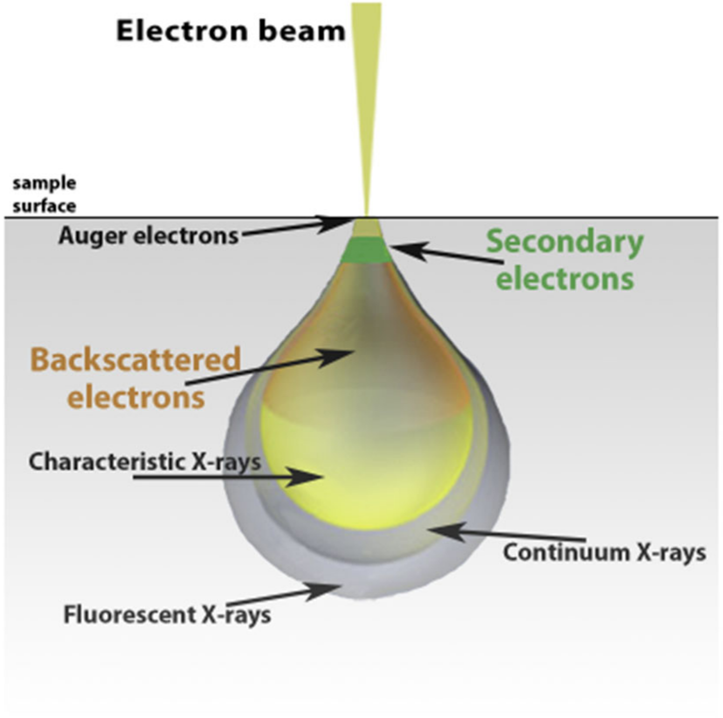
Astigmatism



- Image blur at an overfocus position (a) and an underfocus position (c) appear in the two orthogonal directions (indicated by arrows).
- At the exact focus position (b), the image blur is isotropic.

https://www.jeol.com/words/semterms/

Secondary and Backscattered Electrons



Schematic of an electron beam interaction

Secondary Electron (SE)

- Electrons from the specimen due inelastic scattering
- Weakly bound conduction-band electrons (metals) or outer-shell valence electrons (semiconductors and insulators).

Backscattered Electron (BSE)

- Re-emergent primary electron beam
- usually by multiple elastic scattering events → cumulative effect

Characteristic X-ray

- Electrons from the specimen due inelastic scattering

https://www.nanoscience.com/techniques/scanning-electron-microscopy/components/

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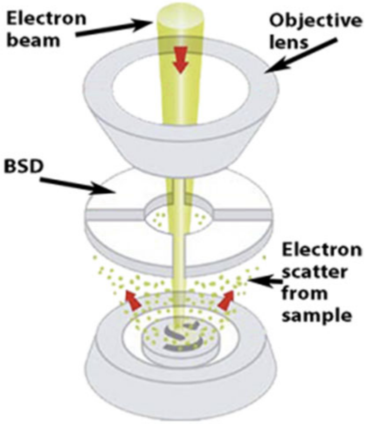
Secondary and Backscattered Electrons

Backscattered Electron (BSE)

- Sensitive to average atomic number
- A few hundred to several keV

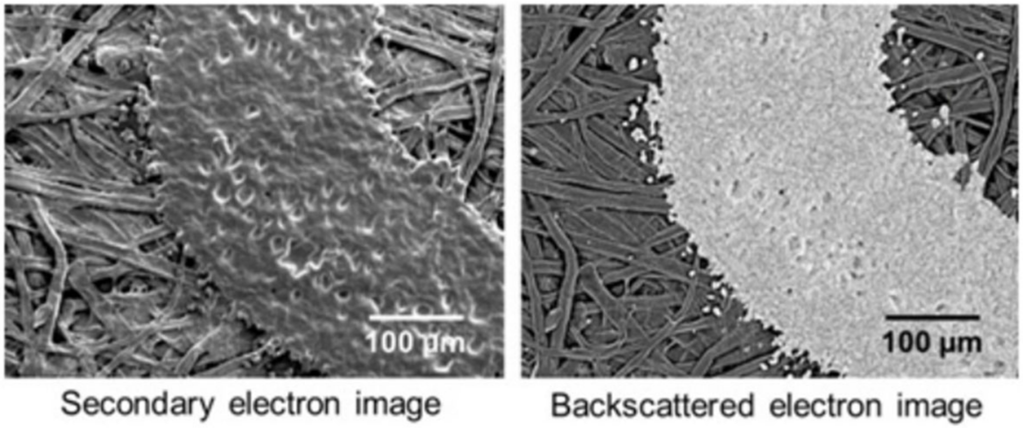
Secondary Electron (SE)

- Sensitive to surface morphology
- A few to a few hundred eV
- Strong signal



Schematic of a backscattered electron detector (BSD) for scanning electron microscopy (SEM).

https://www.nanoscience.com/techniques/scanning-electron-microscopy/components/



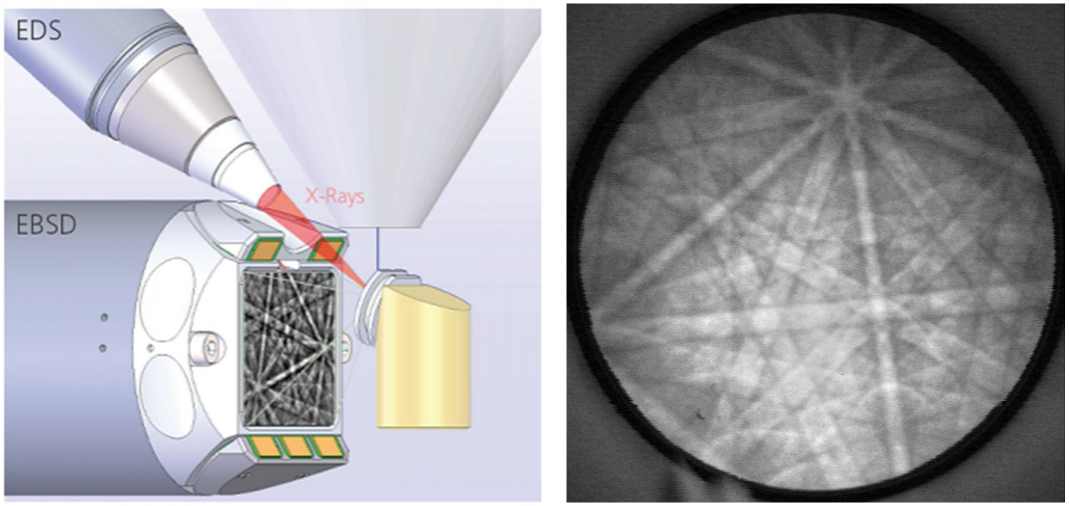
Secondary electron image
Backscattered electron image

the letter part of a carbon-coated name card

https://www.jeol.com/words/semterms/20190129.113542.php#gsc.tab=0

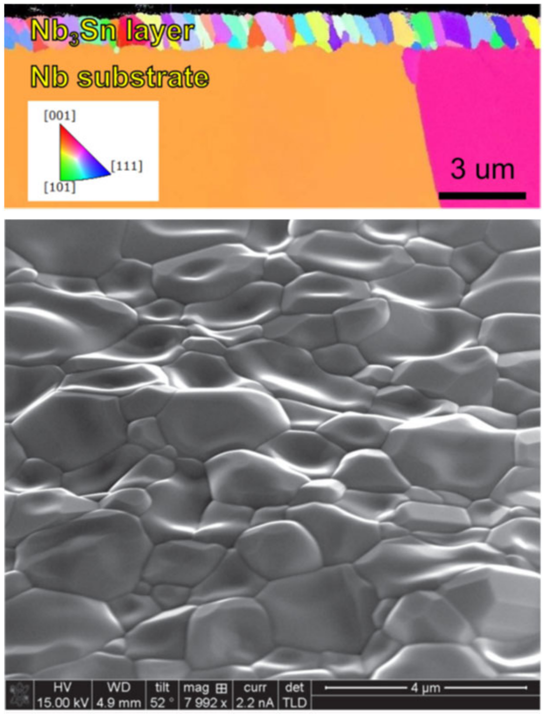
Electrons Backscattered Diffraction (EBSD)

Bragg's law: $n \lambda = 2d \sin\theta_B$



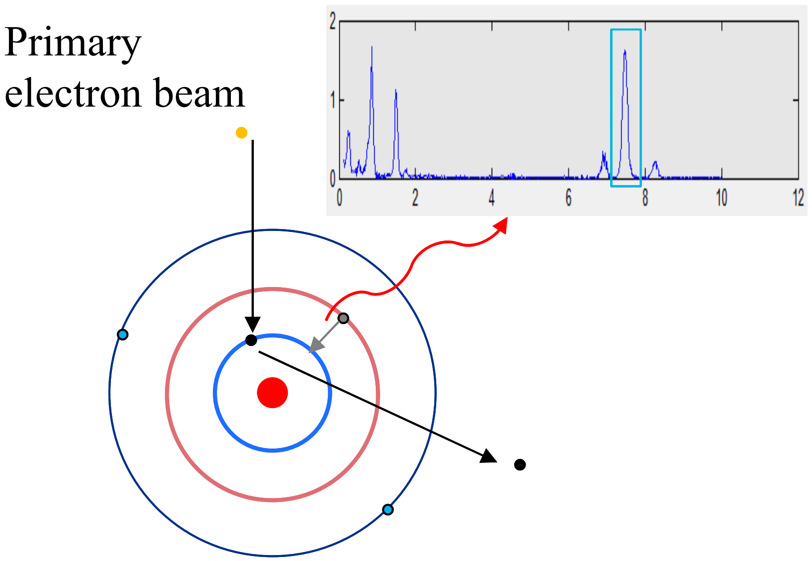
https://nano.oxinst.com/products/ebsd/

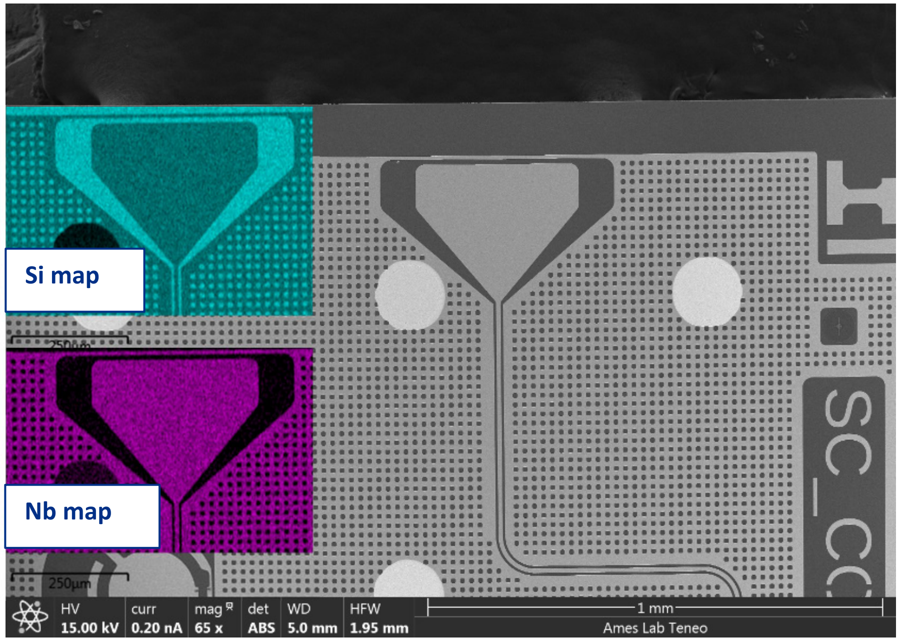
- Diffraction pattern created by Backscattered electron
- Bright bands corresponding to (hkl) plane



Posen et. al, ***Supercond. Sci. Technol.* 30** (2017) 033004.

Energy-dispersive X-ray Spectroscopy





- Micro-scale chemical composition analysis

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IV. Focus Ion Beam (FIB)

History

- **1975**, Krohn and Ringo,
  Liquid metal ion sources (LMIS)
- **1978** Seliger et. al., the first FIB based on an LMIS
  - For ion implantation without a mask on semiconductor micro device.
  - Nano-micro size material fabrication.

A high-intensity scanning ion probe with submicrometer spot size

R. L. Seliger, J. W. Ward, V. Wang, and R. L. Kubena

Hughes Research Laboratories, Malibu, California 90265
(Received 9 October 1978; accepted for publication 11 December 1978)

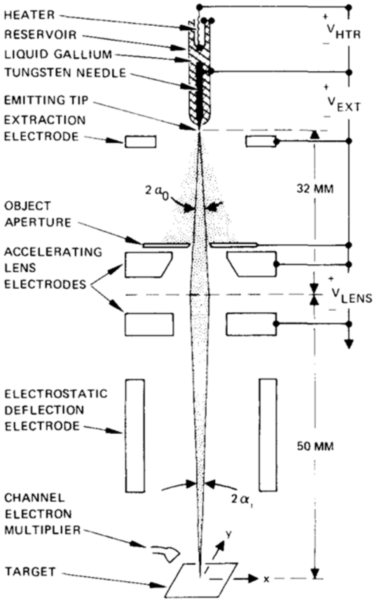


FIG. 1. Schematic of the 57-kV gallium scanning ion probe.

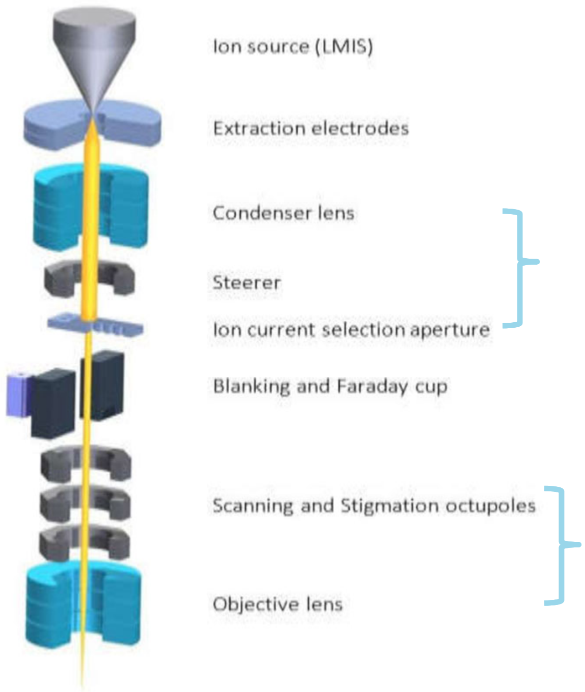
FIB-Helios

https://en.wikipedia.org/wiki/Focused_ion_beam

Electron vs. Ion

	ELECTRONS	IONS
Source size (weight)	Small	Big
Sample interaction	Mostly inner shell	Mostly outer shell
Interaction Volume	Broad	Narrow
Source speed	Fast	Slow
Charge	Negative	Positive
Lens type	electromagnetic lens	electrostatic lens

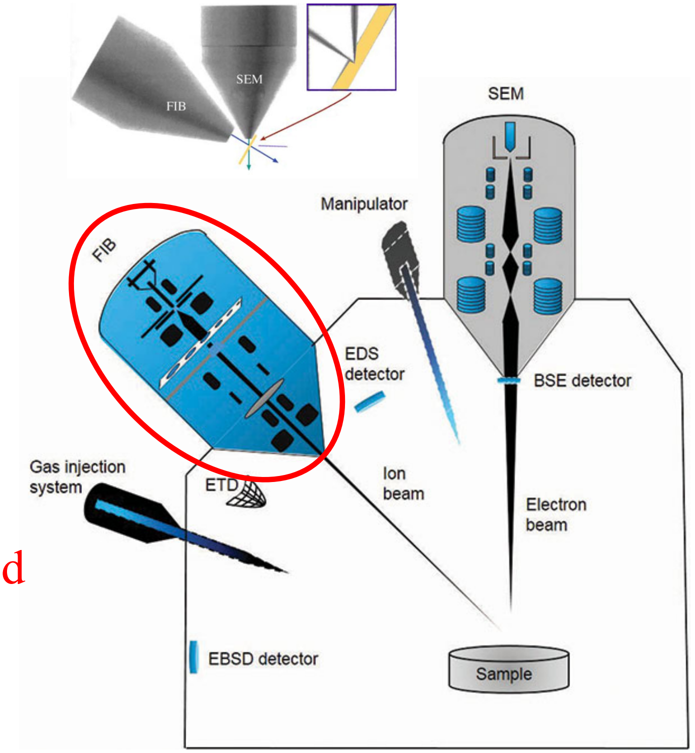
Ion Beam Column



ion beam probe formation

position to be irradiated

https://www.orsayphysics.com/what-is-fib

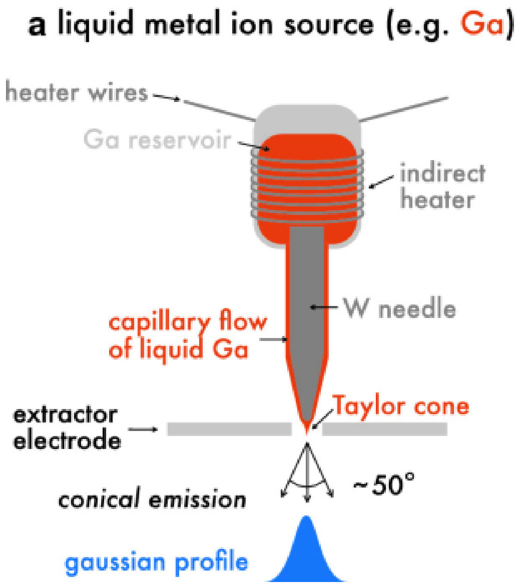


https://analyticalscience.wiley.com/do/10.1002/was.00070009/

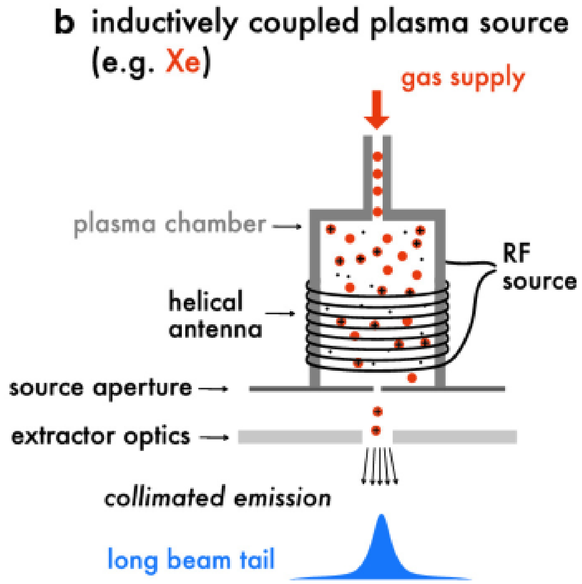
- SEM and FIB can only scan alternatively.

Ion Beam Column

Ga - LMIS



Xe – RF plasma



- Pico to a few nanoamperes, larger beam size, sample damage

- Nano to microamperes, smaller beam size (higher resolution), less damage, more expansive

Focused Ion Beam Micro-structuring, Maja D. Bachmann (2020)

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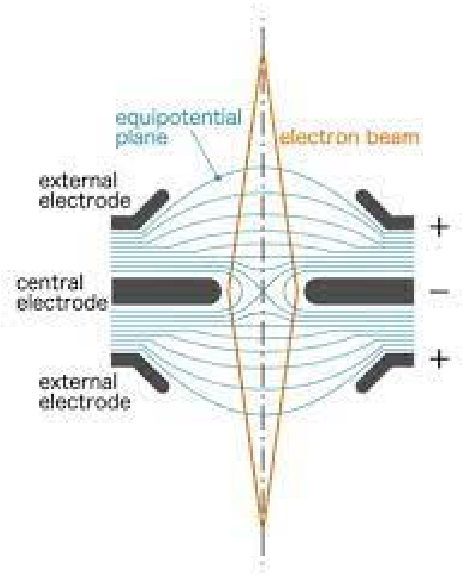
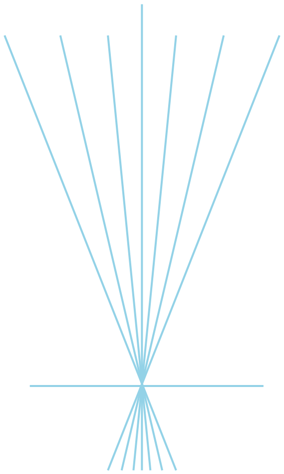
Lens

- Electrostatic lens (for ion)

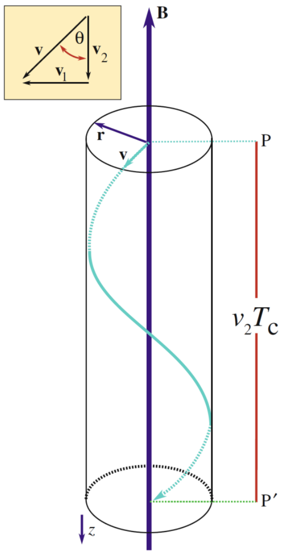




- Use an electric field to bend the trajectory of the ions

https://www.matsusada.com/column/sem-tech2.html

- Electromagnetic lens (for electrons)

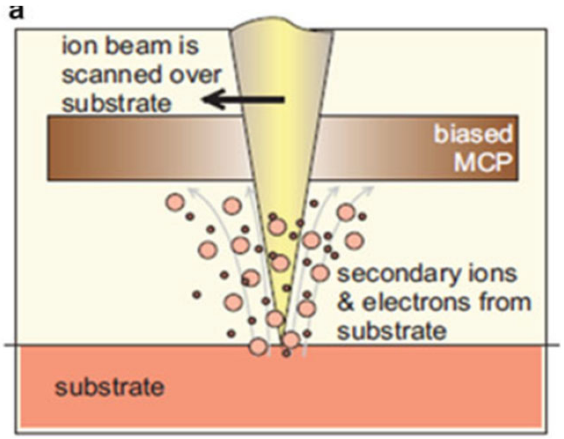


- Use a magnetic field for focusing

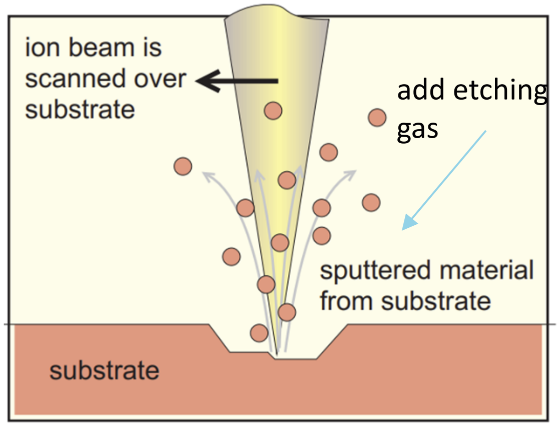
D. Williams & B. Carter, Transmission Electron Microscopy: A Textbook for Materials Science, Springer (2009).

Imaging, Milling, Deposition

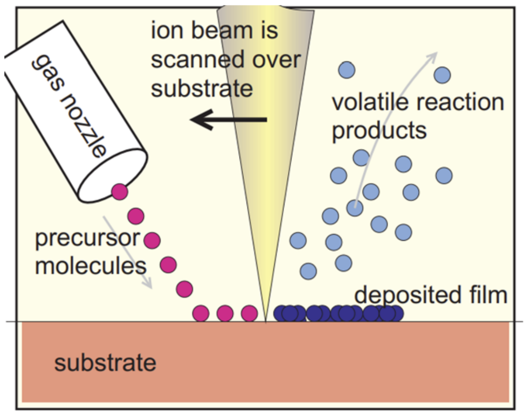
Imaging



Milling / etching



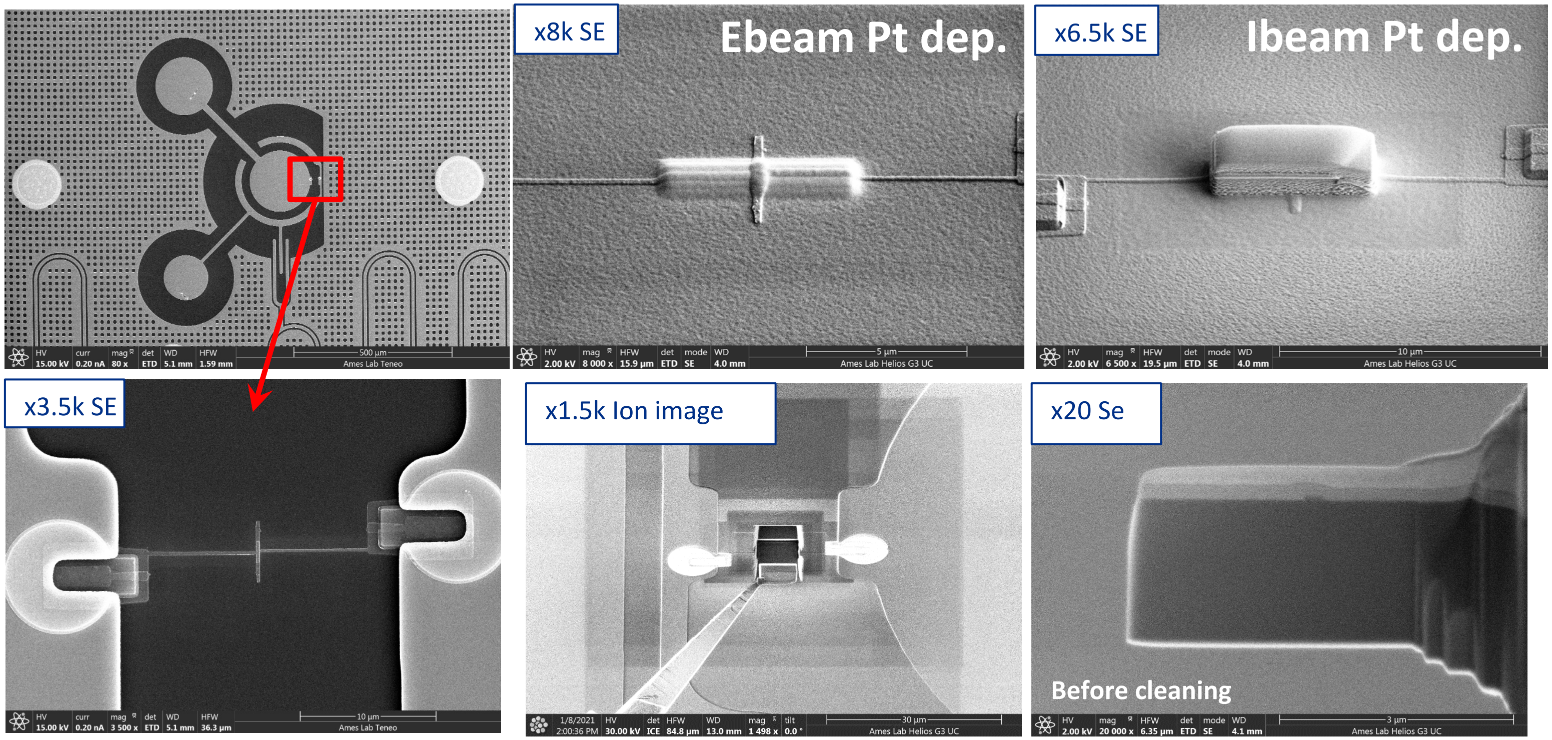
Deposition



Steve Reyntjens and Robert Puers 2001 J. Micromech. Microeng. 11 287

- Many precursors (e.g. W, Pt, Au, Al, and Cu).
- Deposited films contain both the desired metals, impurities, and source ions.

Applications: Sample Preparation



- TEM sample prepared by FIB from JJ.

SQMS
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Applications: Sample Preparation

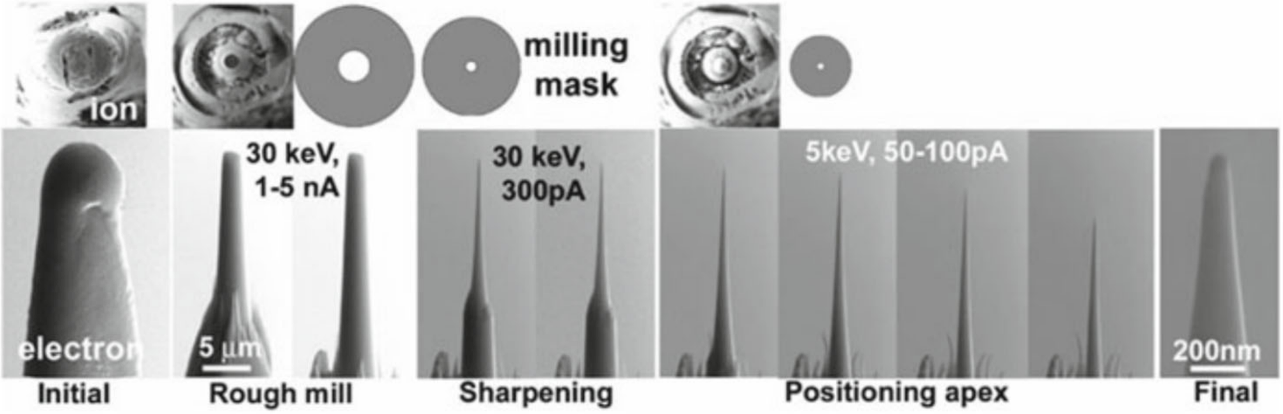
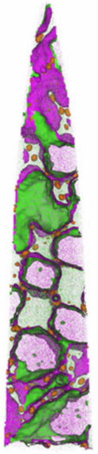


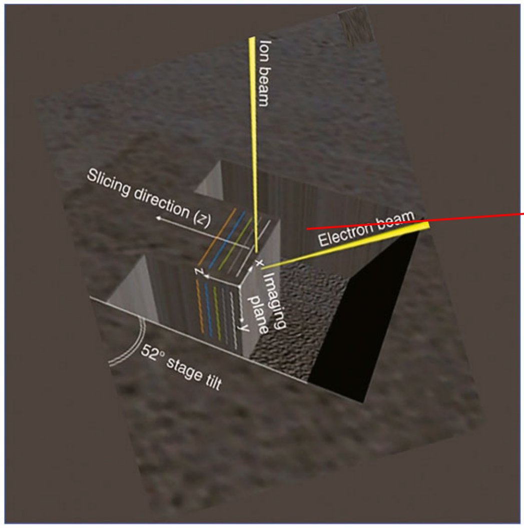
Fig. 1.8 Annular milling stage of atom probe specimen preparation. The size of the annular mask and the ion current are decreased as shown during the annular milling procedure [39]

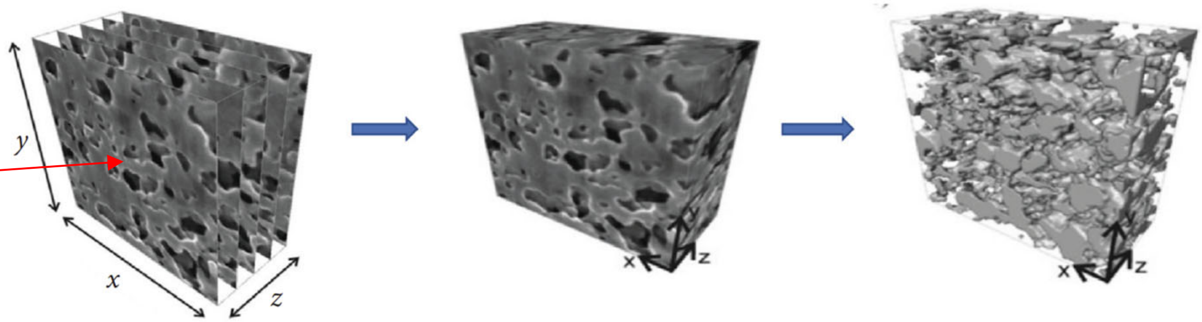
Miller, M.K., Russell, K.F., Thompson, K., Alvis, R., Larson, D.J.: Review of atom probe FIB based specimen preparation methods. Microsc. Microanal. 13 (6), 428–436 (2007)



- Atom probe tomography: needle shape sample

Applications: Tomography





(a)

(b)

(c)

Stacking sectioned SEM image

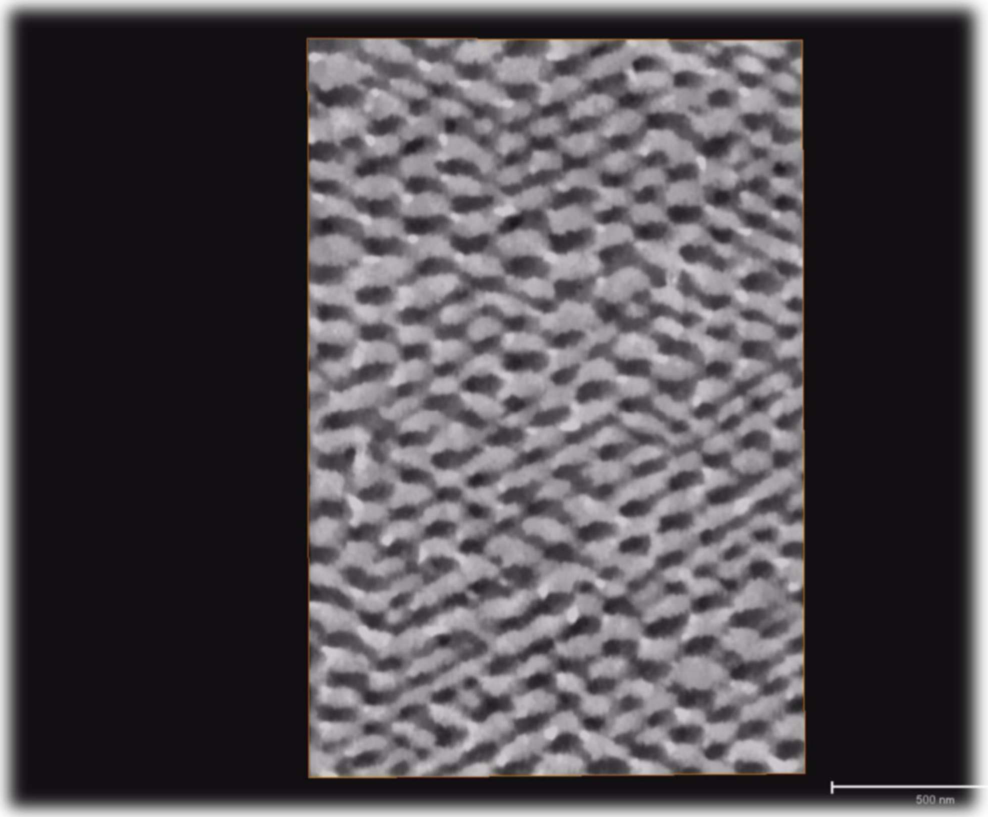
Rendering the 2D SEM image to 3D

3D reconstruction and modeling

- Taking SEM image after milling with FIB, slice by slice.

Advances in Materials Science and Engineering, Volume 2019, Article ID 8680715, 8 pages
https://doi.org/10.1155/2019/8680715

Applications: Tomography



SUPERCONDUCTING QUANTUM MATERIALS & SYSTEMS CENTER

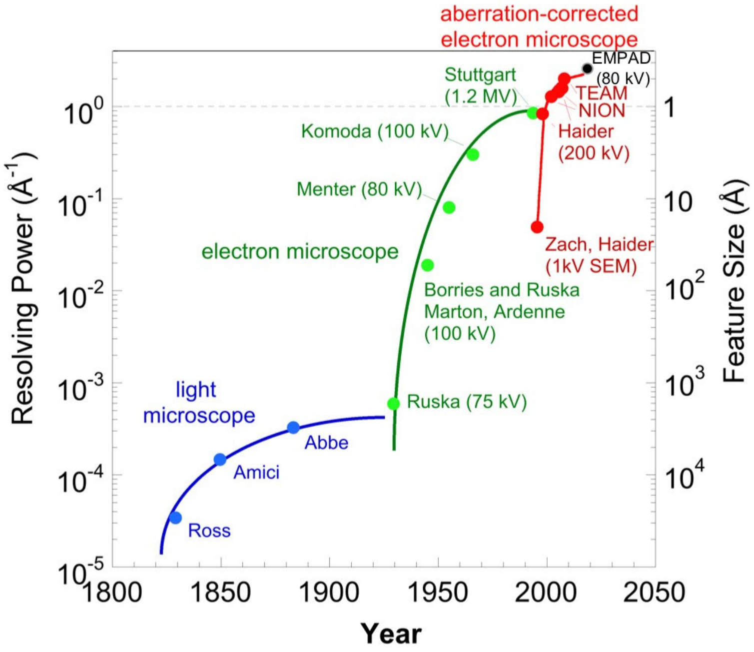
Outline

- History of electron microscopes
- Types of electron microscopes and major instrument component
- Scanning electron microscopy (SEM)
- Focus ion beam (FIB)
- **Transmission electron microscopy (TEM)**
- Application of electron microscopy in superconducting qubits

Improvement of Resolution

- Increasing beam voltage
- Correcting electron-optical aberrations

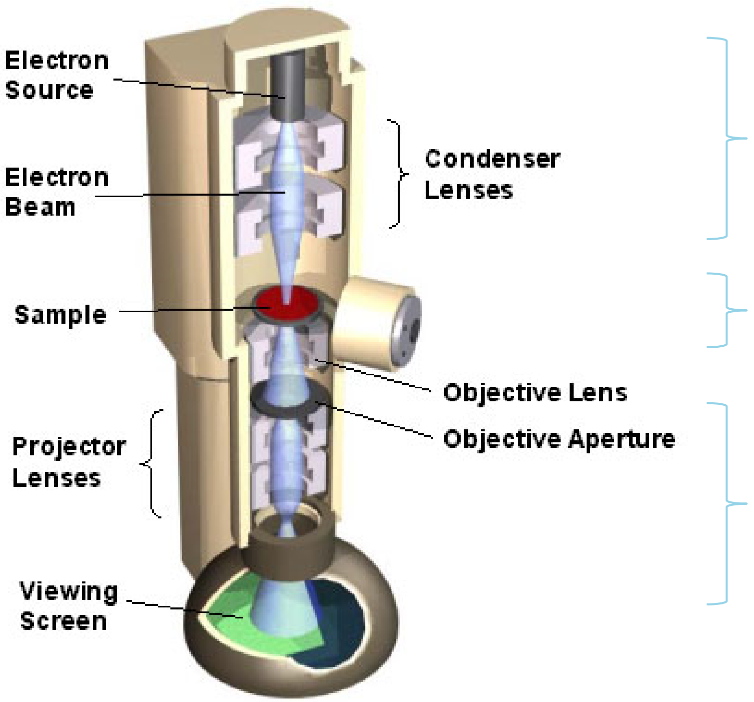
Ruska's 1930s



D. A. Muller, Nature Mater. 8, 263-270 (2009). Adapted.

Modern-day

TEM: Column



Illumination system

Objective lens/stage

Imaging system
(CTEM/STEM)

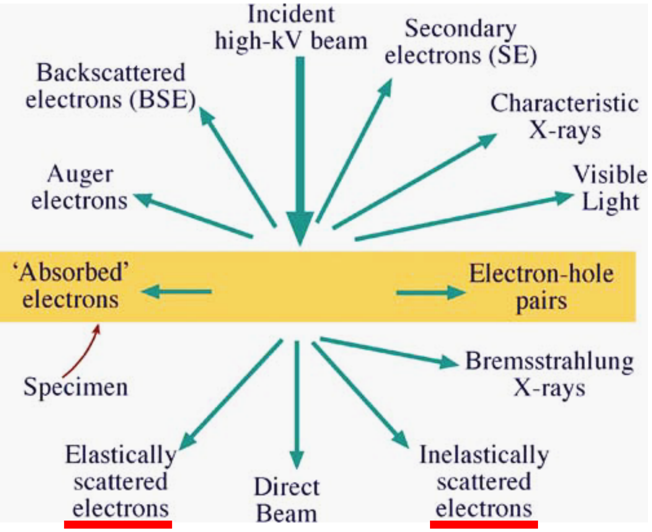
TEM: Beam and Sample Interaction

$$d \sim \lambda$$

- Eyes: ~ 0.1-0.2 mm
- Visible light microscope: ~**300 nm**
- Electron: 200KV ~ **0.025Å**

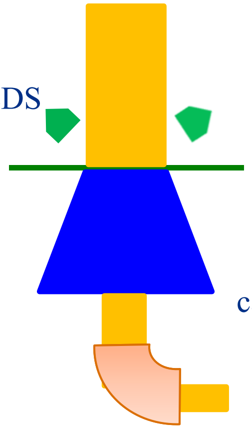
- **resolution, chemistry, strain, magnetic/electric fields, bandgap, …**

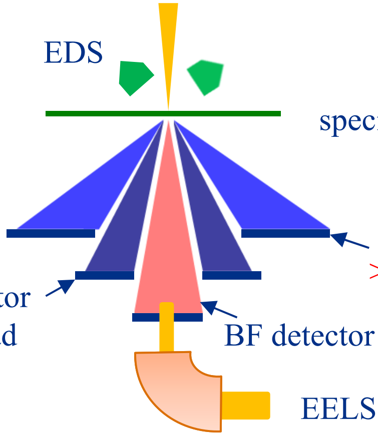
$$\Psi_i(\boldsymbol{r}) = A_i(\boldsymbol{r})exp[i\varphi_i(\boldsymbol{r})]$$



D. Williams & B. Carter, ***Transmission Electron Microscopy: A Textbook for Materials Science,*** Springer (2009).

TEM *vs* STEM





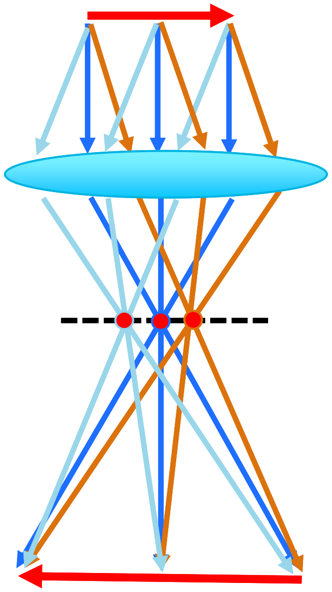
Conventional TEM

- Planar illumination,
- Multi-beam scattering.

Scanning TEM

- Scans a fine probe;
- Electrons scattered to annular detectors.

Diffraction and Imaging



Object plane

Sample
(real space)

Objective lens

FFT

Back focal plane

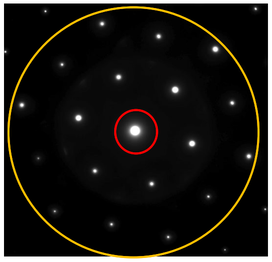
Diffraction pattern
(reciprocal space)

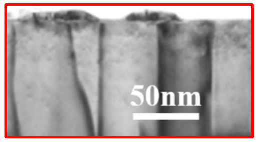
FFT^{-1}

First image plane

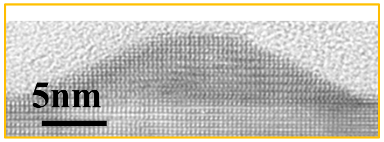
ImageReal space

Bragg's law:
$n \lambda = 2d \sin\theta_B$



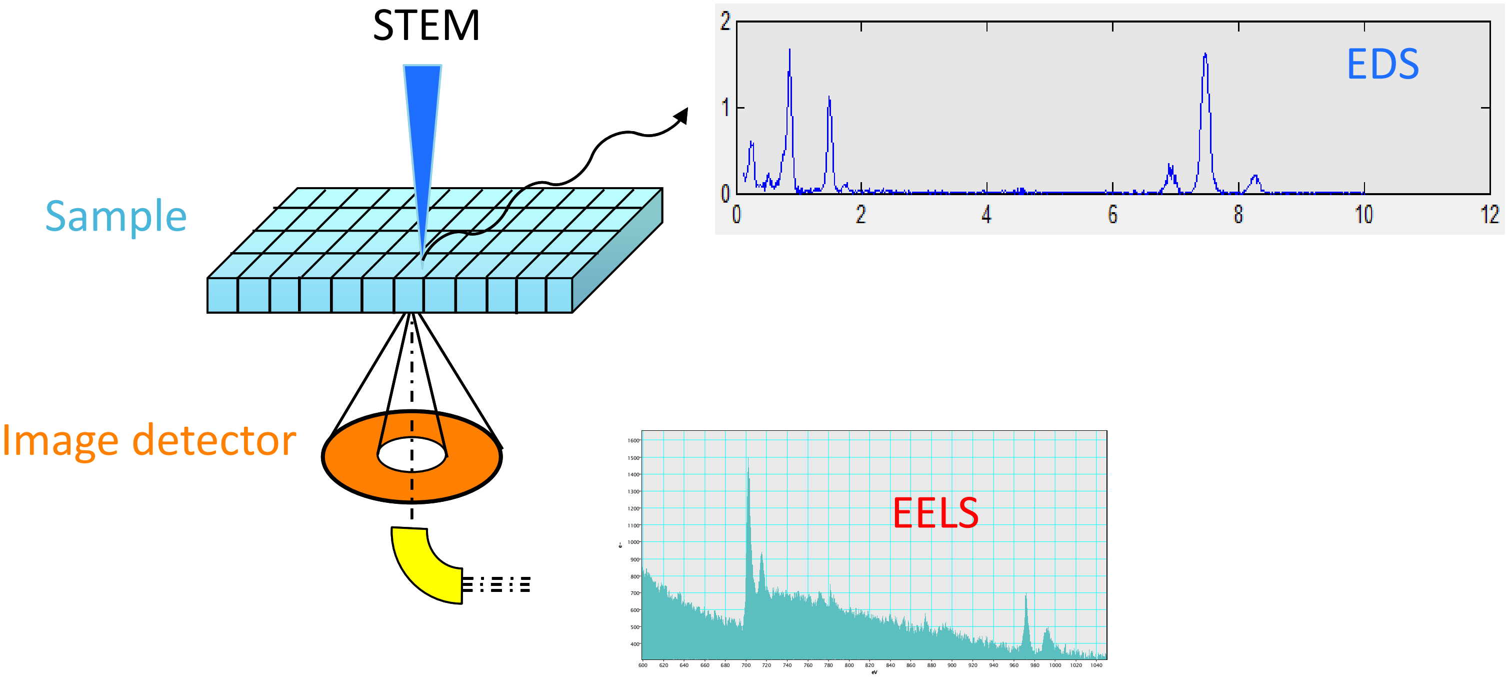


diffraction
contrast imaging



high-resolution
TEM imaging

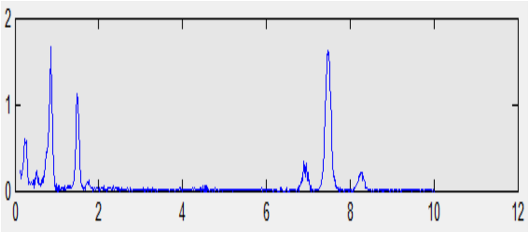
Z-contrast Imaging with EELS and EDS

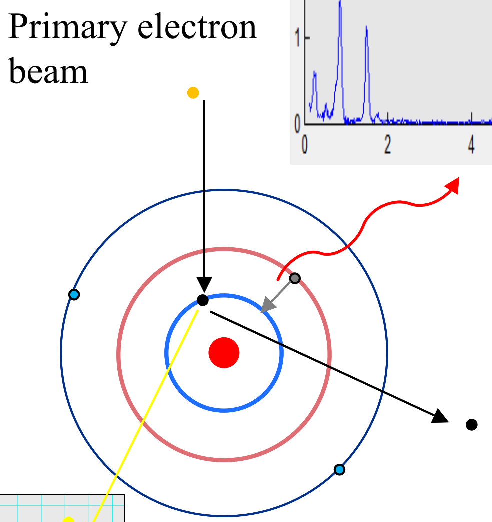


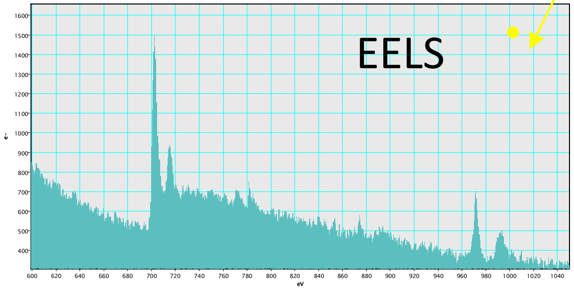
- Combination of high-resolution imaging with subnanometer chemical analysis.

EDS and EELS

- Ionization process (two steps): an inner-shell electron is ejected and replaced by an outer-shell electron.
- Energy difference between the different shell levels is released and X-ray is generated.
- The primary electron is inelastically scattered by sample elements and loss some energy.
- The energy loss is measured and analyzed to obtain information about the sample's electronic properties



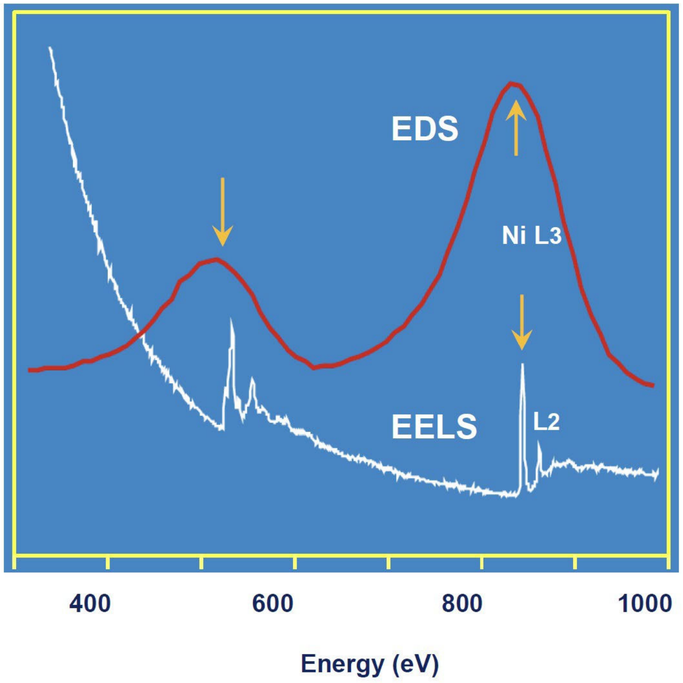




Comparison of EELS and EDS

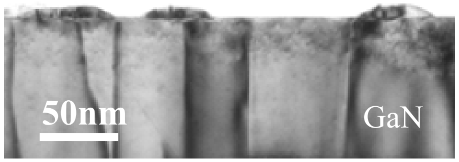
- **EDS**
  - Low collection efficiency (small solid angle)
  - Good peak/background ratio
  - Good for high-Z elements
  - No banding information
- **EELS**
  - High collection efficiency (>90%)
  - Poor peak/background ratio
  - Good for low-Z elements
  - Banding information
  - High spatial resolution (<1 nm)
  - ***Only for TEM & thin samples***

NiO: EDS vs. EELS



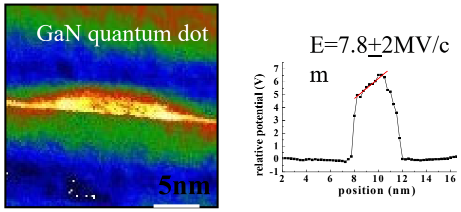
Transmission Electron Microscopy (TEM)

- **Diffraction contrast imaging**
  overall microstructure, defects



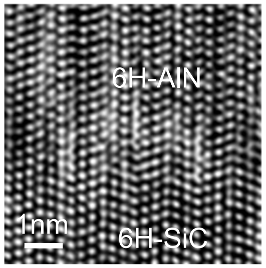
*Zhou, et. al., **Appl. Phys Lett, 88*** (2006) 231906.

- **Electron holography**
  nanoscale electric/magnetic fields



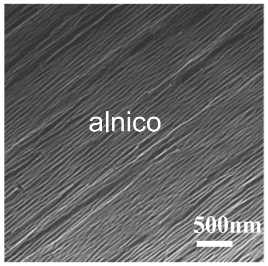
*Zhou, et. al., **Appl. Phys. Lett, 99,** (2011) 101905.*

- **High resolution TEM**
  atomic arrangement



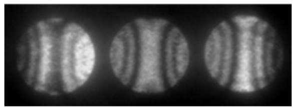
*Zhou, et. al., **J. Crystal Growth**, **311** (2009) 1456.*

- **Lorentz microscopy**
  magnetic domain imaging



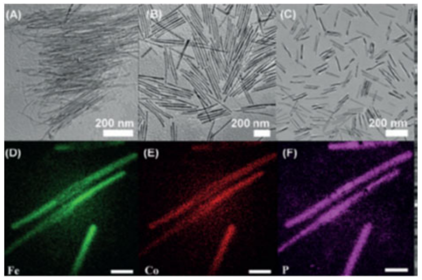
*Zhou, et. al., **Acta Materialia***, **74**, (2014) 224.

- **Convergent beam electron diffraction**
  crystallographic analysis;
  lattice parameter determination



*Zhou, et. al., **J. Crystal Growth**, **311** (2009) 4162.*

- **Energy-filtered imaging (EFTEM)**
  diffraction, composition mapping



*Mendoza-Garcia, et. al, **Angew. Chem., 127** (2015), 9778.*

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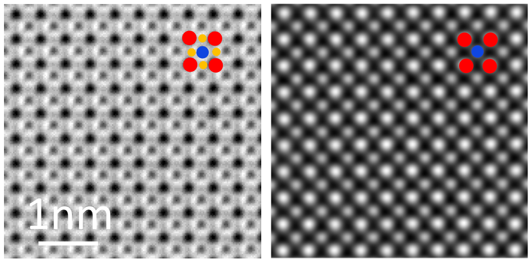
Scanning Transmission Electron Microscopy

- **(aberration-corrected) (Scanning) Transmission electron microscopy (STEM)**

  atomic arrangement imaging, orientation/strain/ordering mapping, electric/magnetic field, …
  - High-angle annular dark field (HAADF, "Z"- contrast) / annular bright filed imaging (ABF)…
  - 4D-STEM: 2D probe image + 2D probe position
- **Nanospectroscopy (EELS, EDS)**

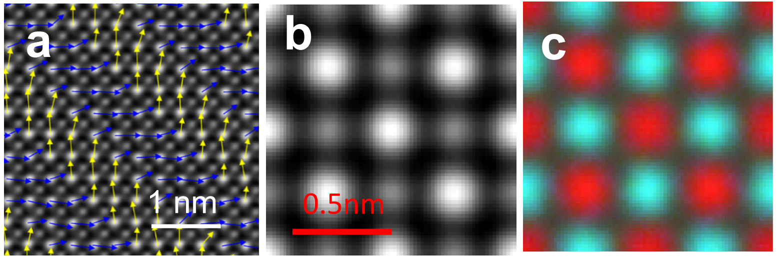
  determination of local composition, bonding states, bandgap …
  - energy dispersive X-ray spectroscopy
  - electron energy loss spectroscopy

$BaTiO_3$



ABF HAADF

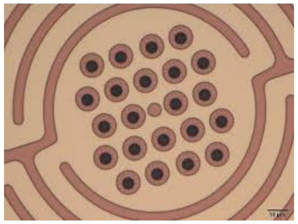
$Pb_{0.99}\{Nb_{0.02}[(Zr_{0.57}Sn_{0.43})_{1-y}Ti_y]_{0.98}\}O_3$

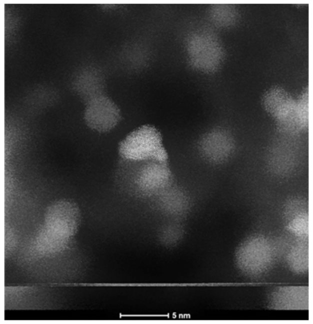


T. Ma,…L. Zhou, ***Physical Review Letters***, **123** (2019) 217602.

In-situ/operando Electron Microscopy

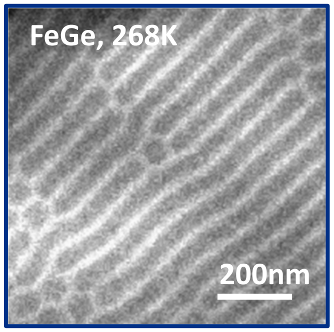
heating and/or biasing





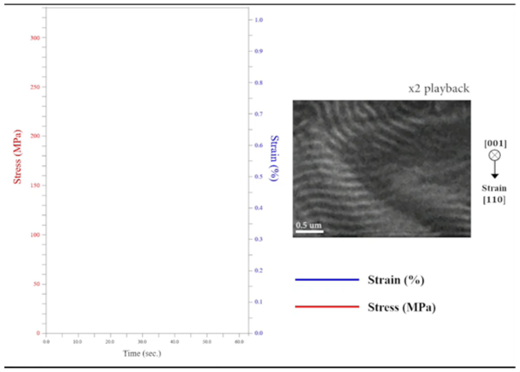
T. Ma, …L. Zhou, ***CHEM***, **5** (2019) 1235.

cryo/cooling



Peng, …Zhou, ***Nano Letters***, 18 (2018) 7777.

mechanical force



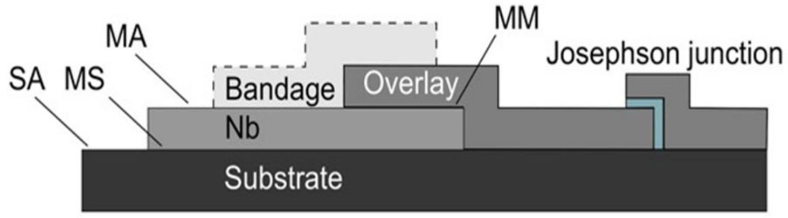
Kim, …Zhou, manuscript ***in preparation***.

Outline

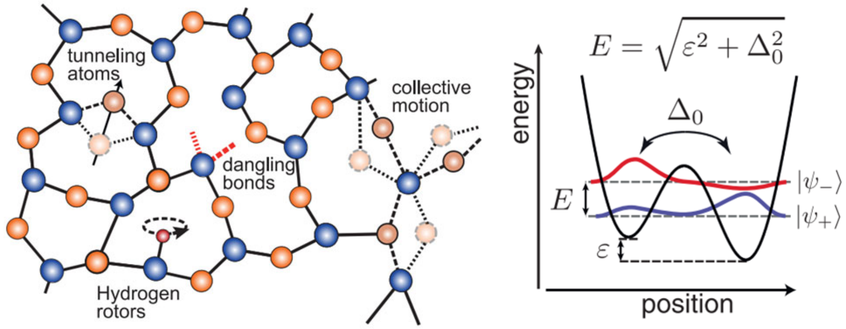
- History of electron microscopes
- Types of electron microscopes and major instrument component
- Scanning electron microscopy (SEM)
- Focus ion beam (FIB)
- Transmission electron microscopy (TEM)
- **Application of electron microscopy in superconducting qubits**

Coherence Limiting Factors

- Major coherence loss source: microwave dissipation by two-level system (TLS) defects at interfaces, such as the metal/air, metal/substrate, and substrate/air interface.
- TLS: individual atoms tunneling between two local energy minimum within the amorphous part of the device.



Nersisyan et al, ***2019 IEEE International Electron Devices Meeting (IEDM),*** doi: 10.1109/IEDM19573.2019.8993458.



C. Müller et al., ***Rep. Prog. Phys.*** *82 (2019)*

- Structure origin of decoherence ⟷ processing conditions

Nb Resonator: Formation of NbOx

- Niobium: cavity, readout resonators, capacitors, and interconnects.
- Nb surface oxide: increase surface resistance, ~5-6 nm thick NbOx.
- Formation of Nb-hydride (non-superconducting at T>1.3K): high field Q slope

Nb
metal, bcc
Tc: ~ 8-9K

→

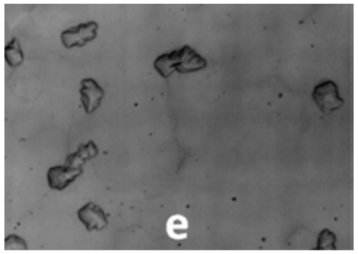
NbO
n-type
conductor
Tc:~ 1.4K

→

NbO_2
n-type
semiconductor

→

Nb_2O_5
Insulator

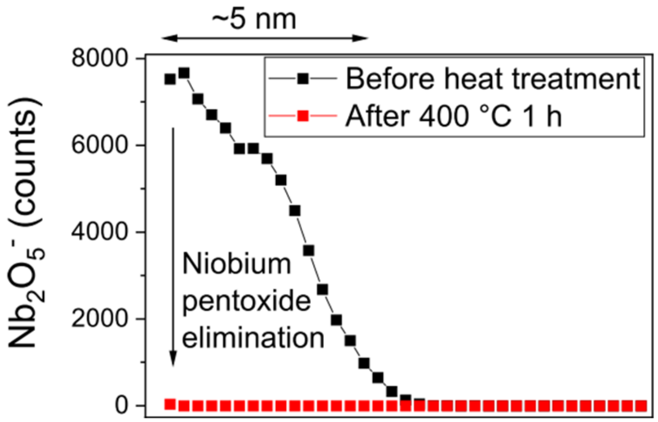


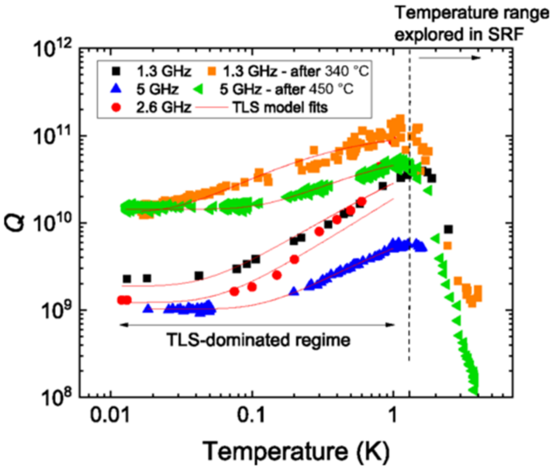
Barkov et al., **J. Appl. Phys.** 114, 164904 (2013)

- What is the structure and formation mechanism of NbO_x?
- Why can we mitigate the impact of NbO_x and/or hydride by
  - annealing?
  - N-doping?

Nb Resonator: Annealing

- Heating under UHV eliminates Nb_2O_5, improves the quality factor of cavities.
  - reduce surface resistance and TLS by eliminating the surface oxidation layer.

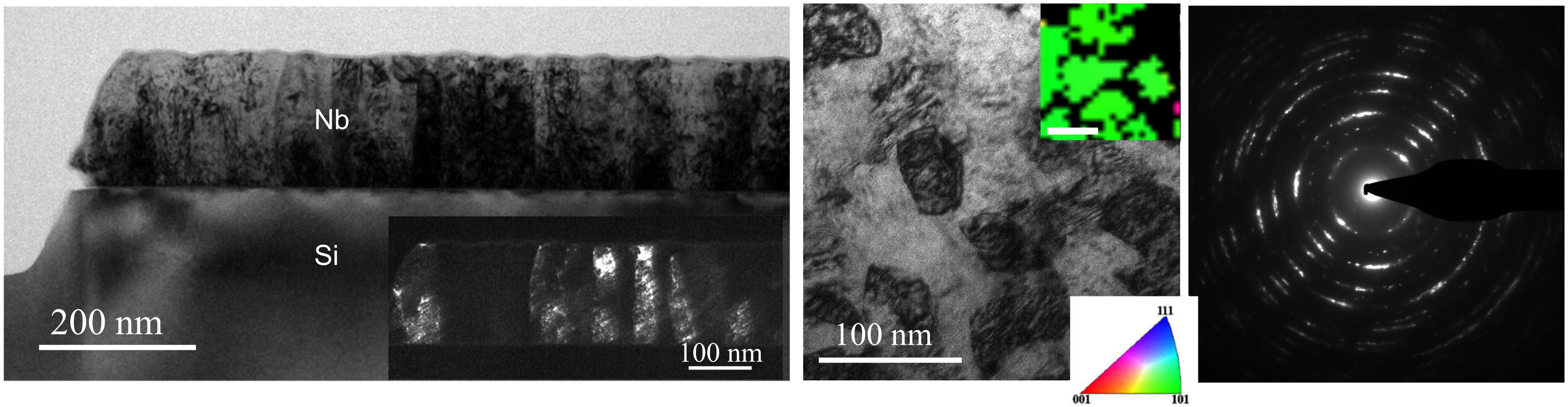




A. Romanenko et al., ***Phys. Rev. Applied*** 13 (2020)

- Microstructural evolution of the oxide layer? ↔ *In-situ* heating in TEM

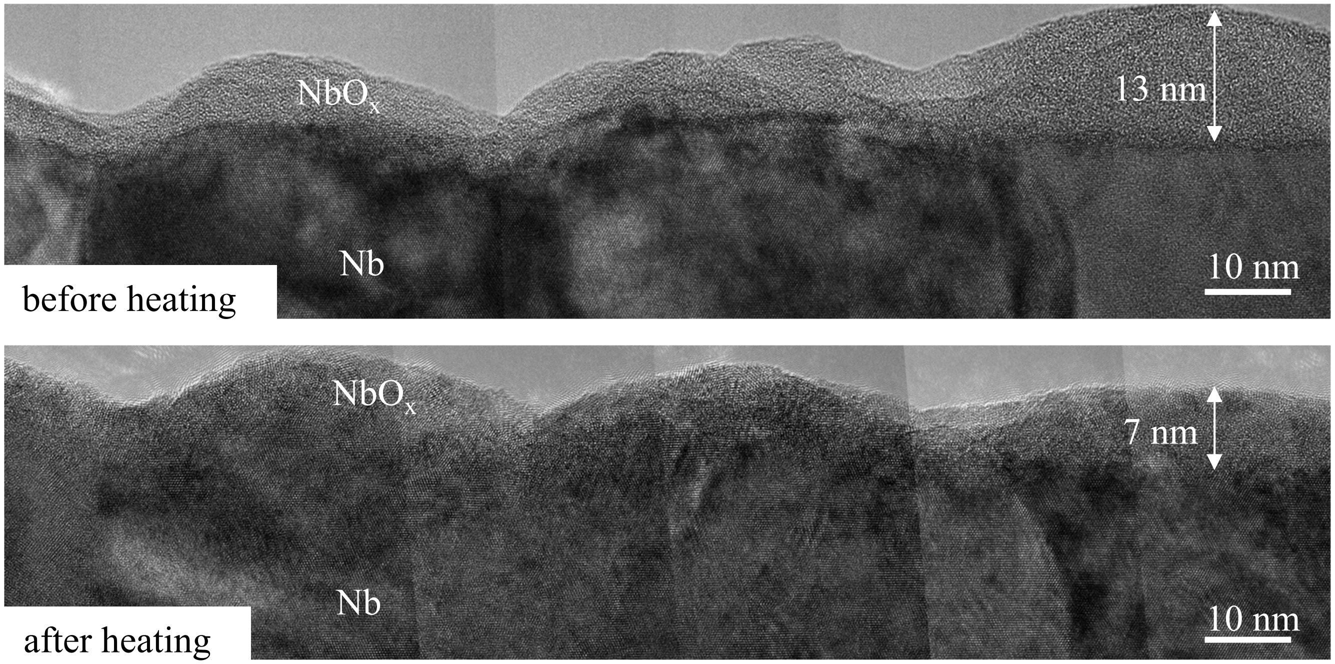
Microstructures of Nb Film



- Nb film deposited on Si [100] substrate by HiPIMS.
- Nb film has thickness of ~160 nm, lateral grain size of ~50 nm.
- Columnar structures with [110] texture.

Jin-Su Oh, et.al., ***Applied Surface Science***, (2023), 157297.

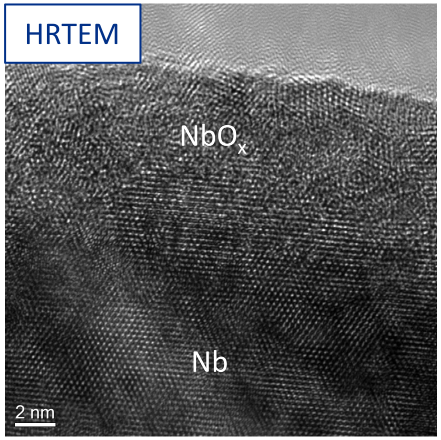
Decomposition of Nb_2O_5 During Heating

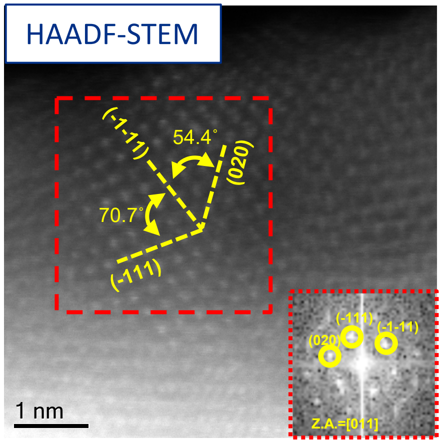


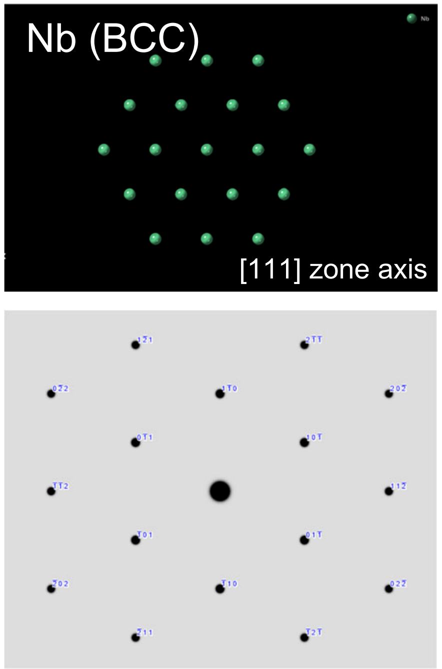
- *In-situ* heating up to 360 ˚C for 30 min at 5 x 10^{-5} mTorr.
- NbO_x layer decomposed after heating.

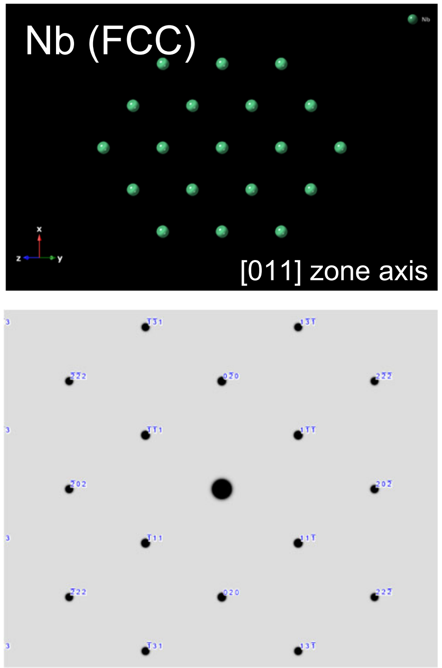
Jin-Su Oh, et.al., ***Applied Surface Science***, (2023), 157297.

Structure of NbO_x After Heating





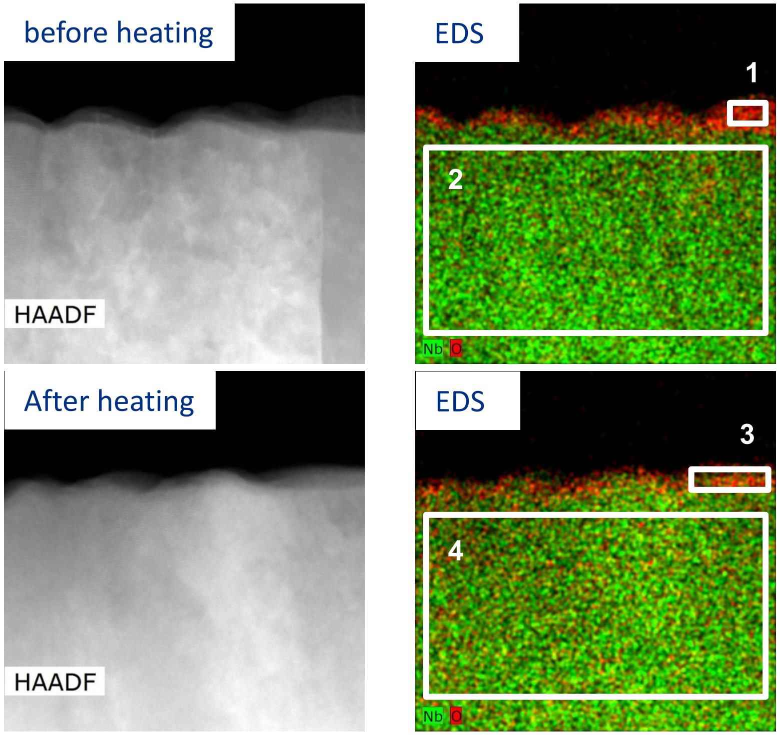




- Nanograins in the NbO_x region after heating is indexed as ***fcc***-Nb.

Jin-Su Oh, et.al., ***Applied Surface Science***, (2023), 157297.

Decomposition of Nb_2O_5 During Heating



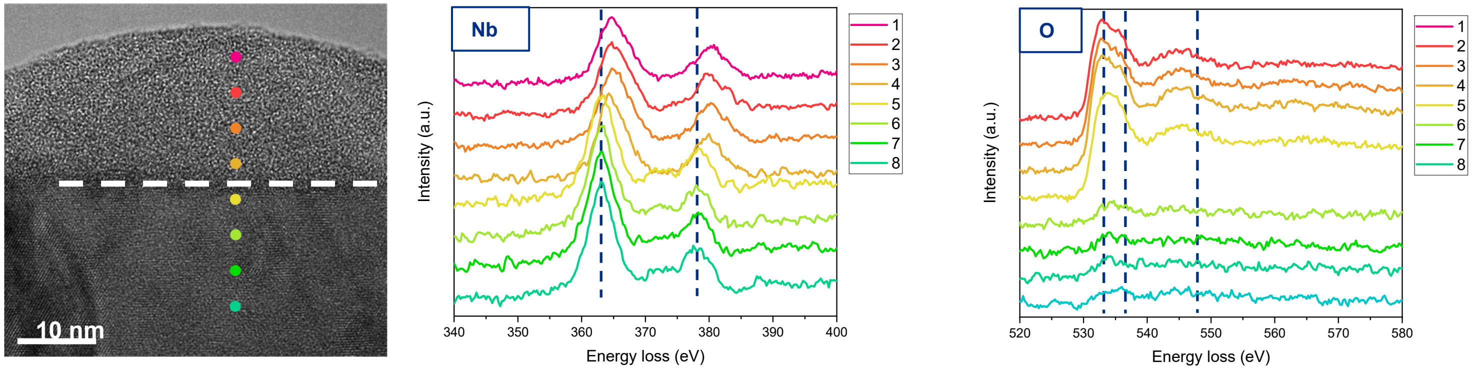
at%	Nb	O
Region 1	35.37	64.63
Region 2	91.52	8.48
Region 3	60.98	39.02
Region 4	98.34	1.66

- Oxygen content significantly decreased after heating.
- Thickness of oxide layer decreased.
- Original interface remains almost unchanged.

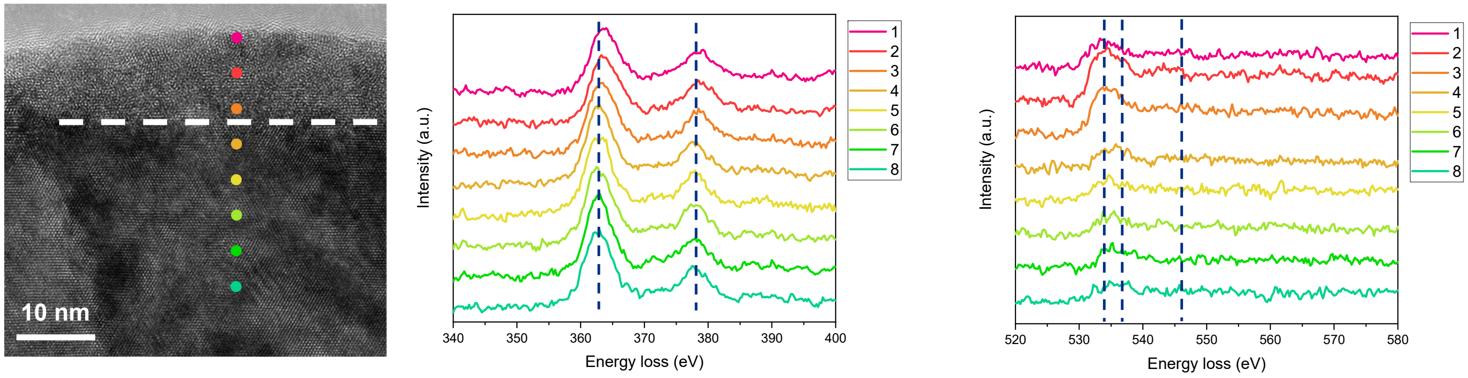
Jin-Su Oh, et.al., ***Applied Surface Science***, (2023), 157297.

Structure of NbO_x After Heating

before heating



after heating



- Nb_2O_5 decomposed into NbO after heating.

J. Oh, et al., arXiv:2204.06041.

Jin-Su Oh, et.al., ***Applied Surface Science***, (2023), 157297.

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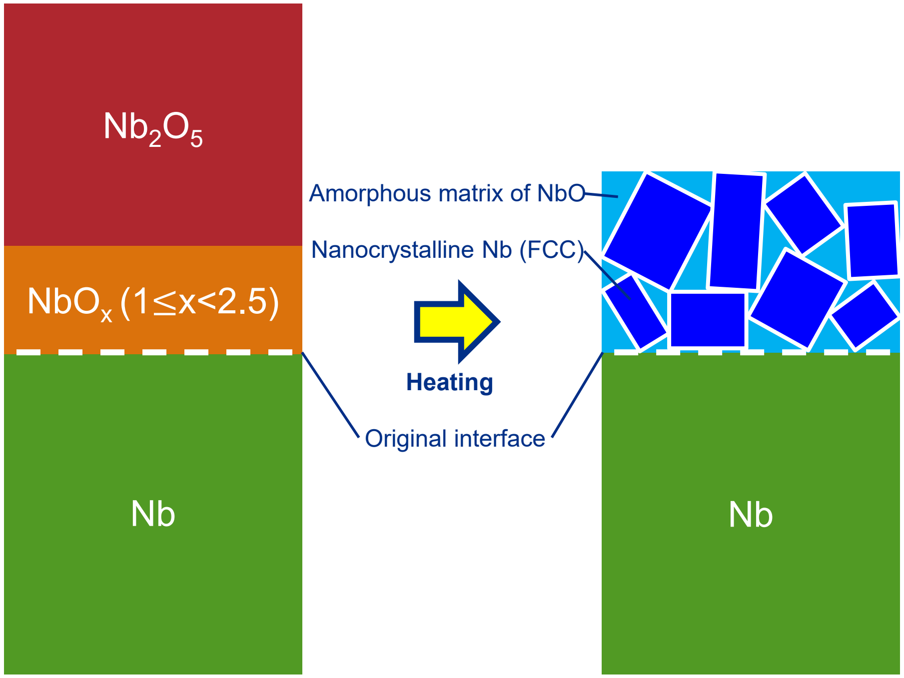
Structure of NbO_x After Heating



- The amorphous niobium oxide layer decomposed into nanocrystalline FCC Nb in amorphous NbO matrix.

Jin-Su Oh, et.al., ***Applied Surface Science***, (2023), 157297.

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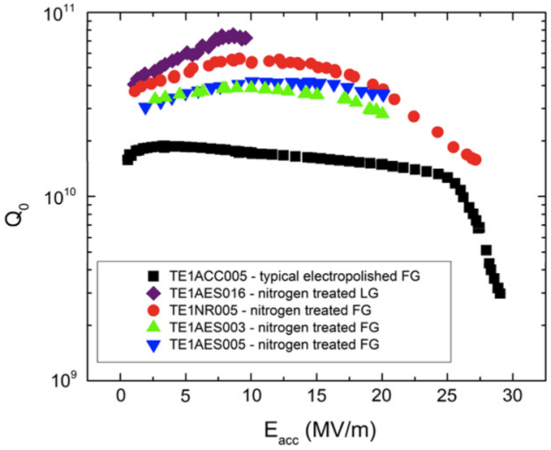
Nb Resonator: Nitrogen-doping

- Oxides and hydrides still formed after baking or polishing.
- Nitrogen surface treatment improves the quality factor of niobium radio frequency cavities beyond the expected limit for niobium.



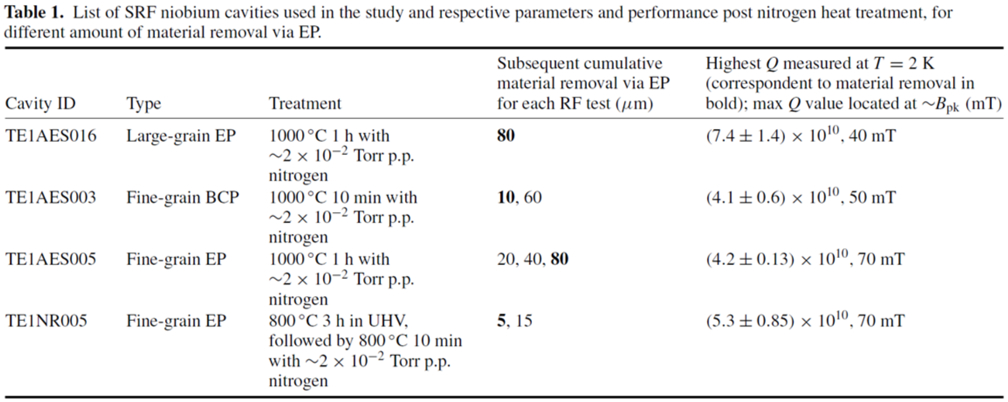


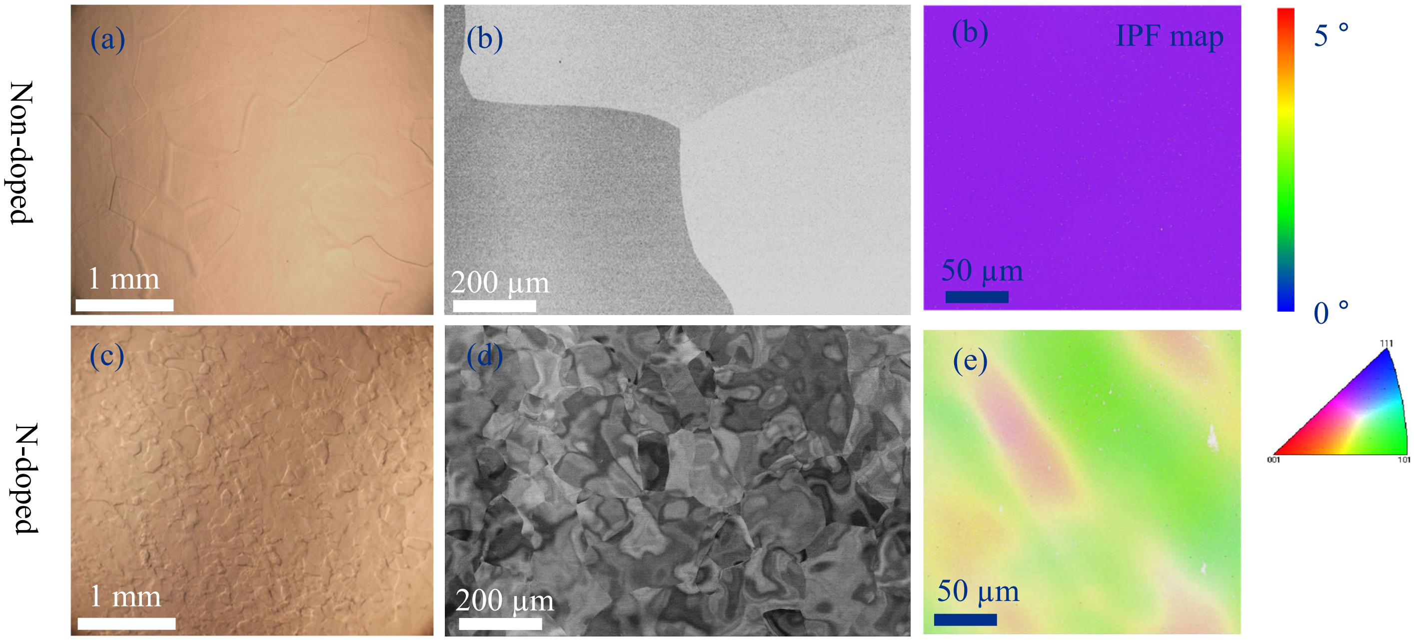
Table 1. List of SRF niobium cavities used in the study and respective parameters and performance post nitrogen heat treatment, for different amount of material removal via EP.

Cavity ID	Type	Treatment	Subsequent cumulative material removal via EP for each RF test (μm)	Highest Q measured at $T = 2$ K (correspondent to material removal in bold); max Q value located at $\sim B_{pk}$ (mT)
TE1AES016	Large-grain EP	1000 °C 1 h with $\sim 2 \times 10^{-2}$ Torr p.p. nitrogen	**80**	$(7.4 \pm 1.4) \times 10^{10}$, 40 mT
TE1AES003	Fine-grain BCP	1000 °C 10 min with $\sim 2 \times 10^{-2}$ Torr p.p. nitrogen	**10**, 60	$(4.1 \pm 0.6) \times 10^{10}$, 50 mT
TE1AES005	Fine-grain EP	1000 °C 1 h with $\sim 2 \times 10^{-2}$ Torr p.p. nitrogen	20, 40, **80**	$(4.2 \pm 0.13) \times 10^{10}$, 70 mT
TE1NR005	Fine-grain EP	800 °C 3 h in UHV, followed by 800 °C 10 min with $\sim 2 \times 10^{-2}$ Torr p.p. nitrogen	**5**, 15	$(5.3 \pm 0.85) \times 10^{10}$, 70 mT

A. Grassellino et al., Supercond. Sci. Technol. 26 (2013) 102001.

- Structural changes that bring Q improvement?

Nb Resonator: Nitrogen-doping



- Nitrogen doping introduces a compressive strain close to the Nb/air interface.
- The strain impedes the diffusion of oxygen and hydrogen atoms.

Xiaotian Fang, Jin-Su Oh, Matt Kramer, A. Romanenko, A. Grassellino, John Zasadzinski, Lin Zhou, Materials Research Letter, 11, 108 (2023)

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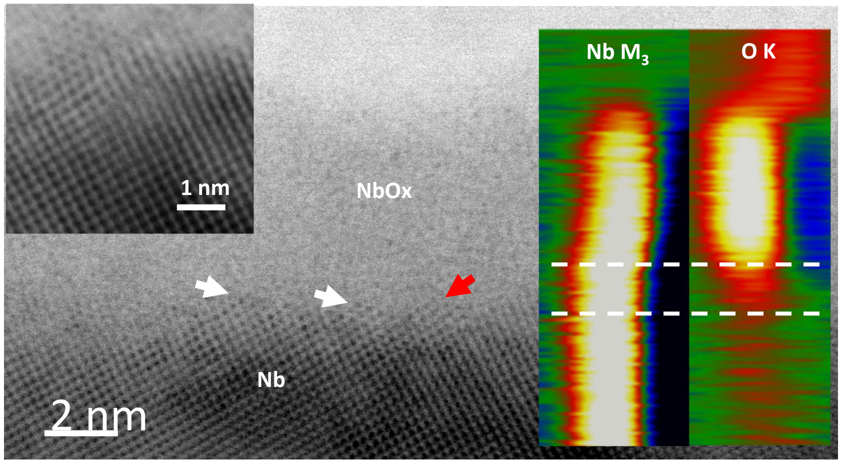
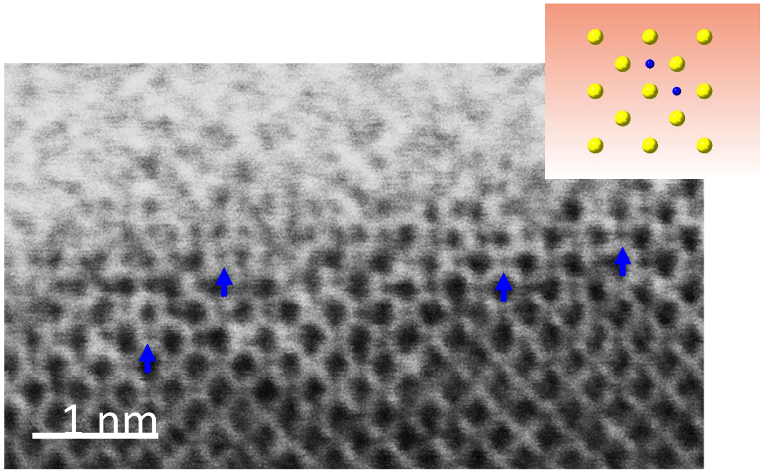
Formation of NbOx

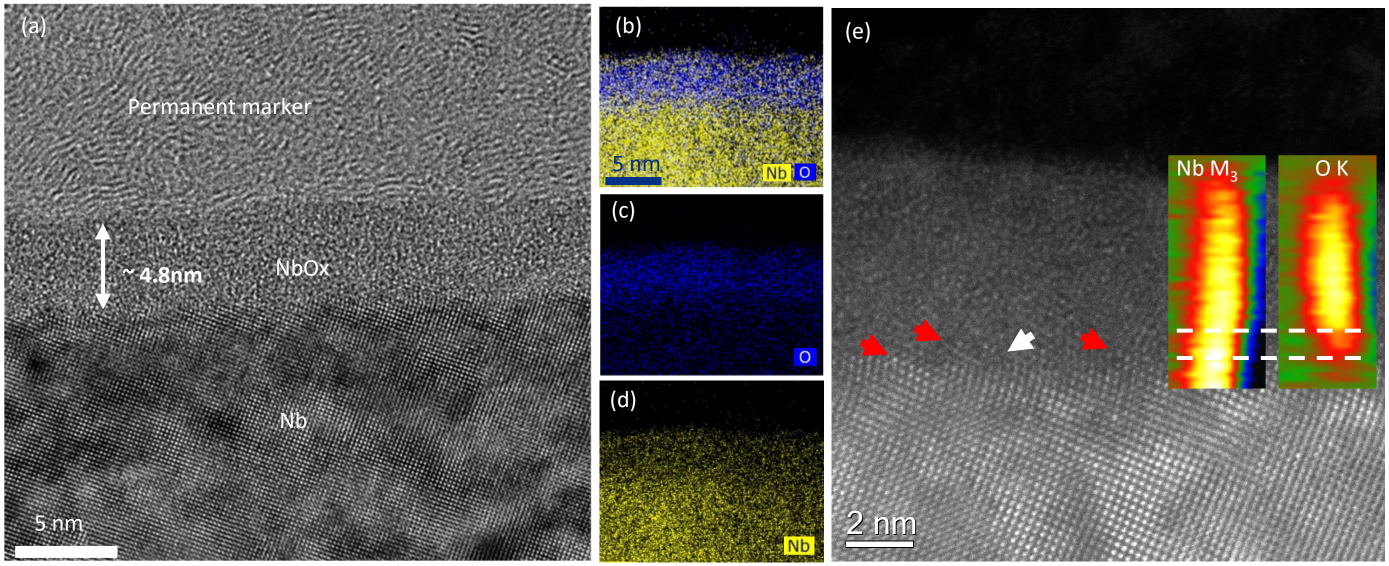




- The NbOx/Nb interface is faceted on {100} and {110} planes.
- Nb lattice is distorted close to the NbOx/Nb interface.
- Interstitial oxygen at octahedral center of Nb bcc lattice(indicated by blue arrows).
- Suboxide(s) at NbOx/Nb interface has the potential of magnetic moments as a possible two-level-system and pairbreaking source.

Xiaotian Fang, Jin-Su Oh, Matt Kramer, A. Romanenko, A. Grassellino, John Zasadzinski, Lin Zhou,
Materials Research Letter, 11, 108 (2023)

Nb Resonator: Nitrogen-doping

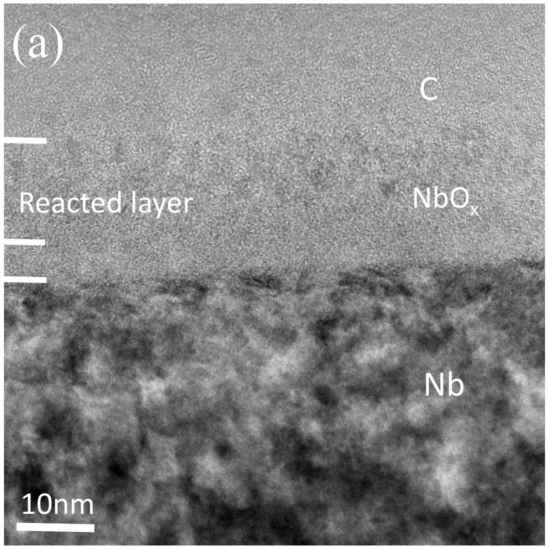


- **The NbOx layer is ~4.8nm thick, ~20% thinner than non-N-doped sample.**
- NbOx/Nb interface is faceted on {100} and {110} planes.
- Suboxide(s) at NbOx/Nb interface has the potential of magnetic moments as possible two-level-system and pairbreaking source.

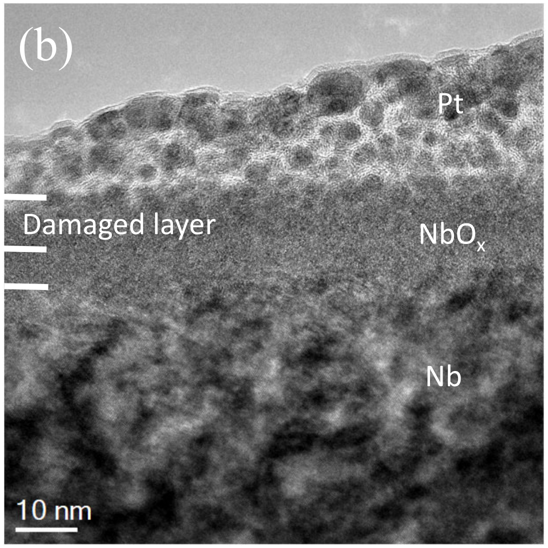
Xiaotian Fang, Jin-Su Oh, Matt Kramer, A. Romanenko, A. Grassellino, John Zasadzinski,
Lin Zhou, Materials Research Letter, 11, 108 (2023)

Nb resonator: TEM Sample Preparation

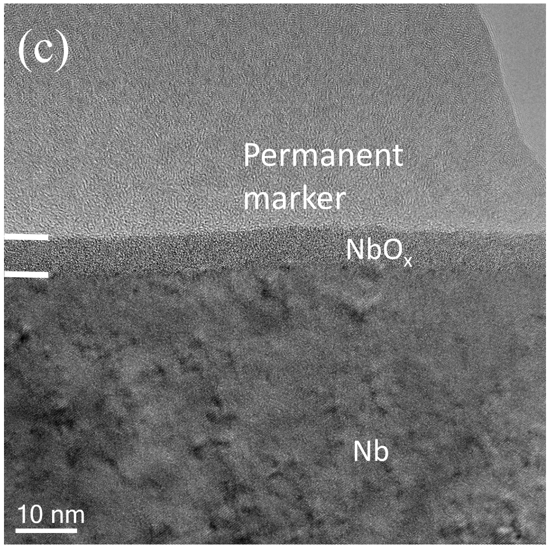
e-beam carbon



e-beam Pt

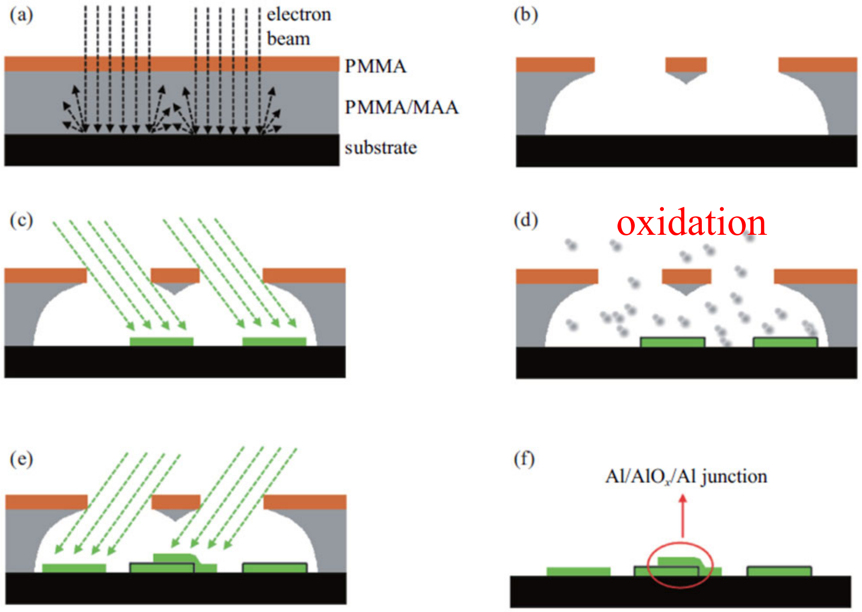


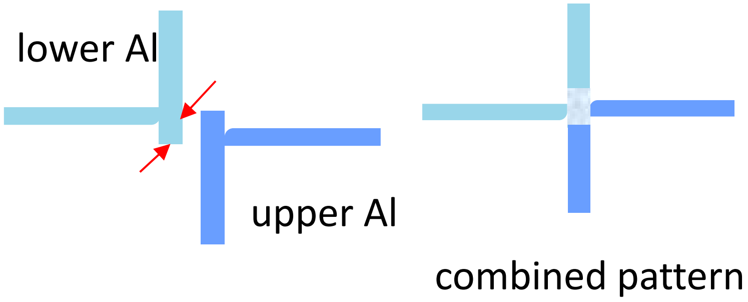
Permanent marker

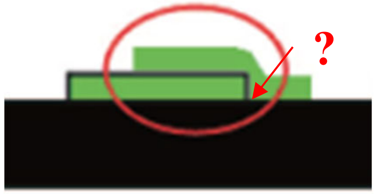


- Surface protection is required before FIB TEM sample preparation for Nb.

Josephson Junction: Two-angled Al Evaporated Layers



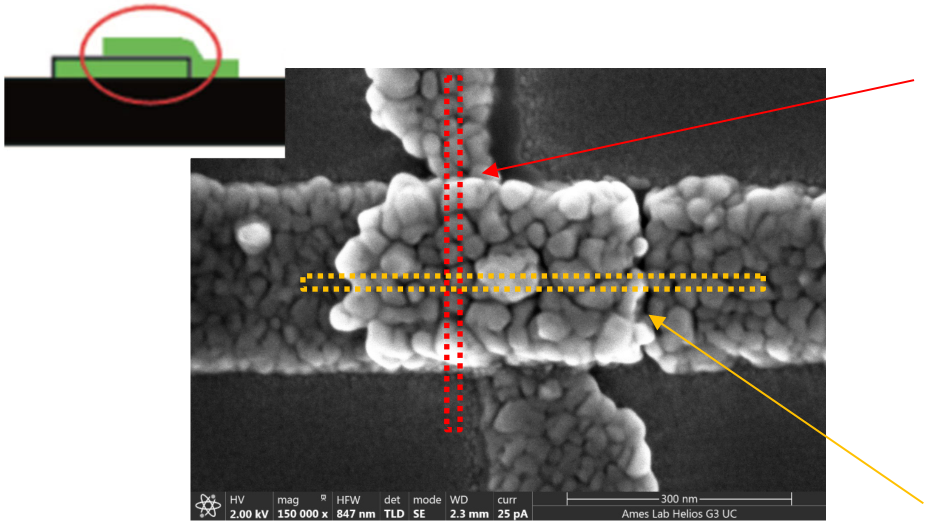


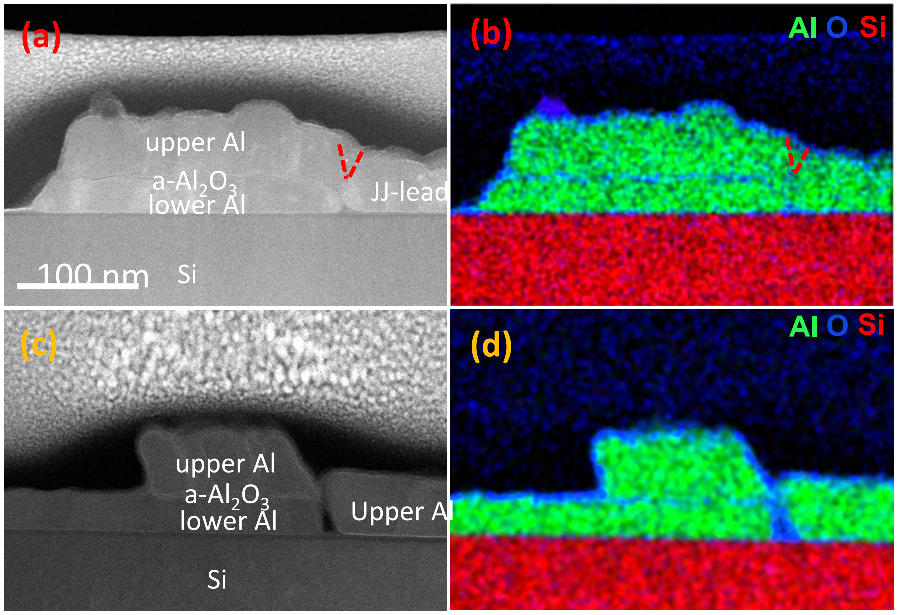


Y. Wu, et. al., Chin. Phys. B Vol. 22, No. 6 (2013) 060309

- Aluminum is a common choice:
  ease of creating a high-quality oxide, a low melting point (660 °C), making it versatile.
- Two angled Al evaporated layers on Si with an oxidation step in between.

Josephson Junction: Fabrication Defects





R. Kim, et al, Communications Physics, (2023) 6, 147.

- A notch and small gap is formed between the upper Al and upper JJ lead.
- A gap partially filled with oxides is on one side of the JJ.
- The gap and notch structures are potential superconducting weak link that may impact coherence.

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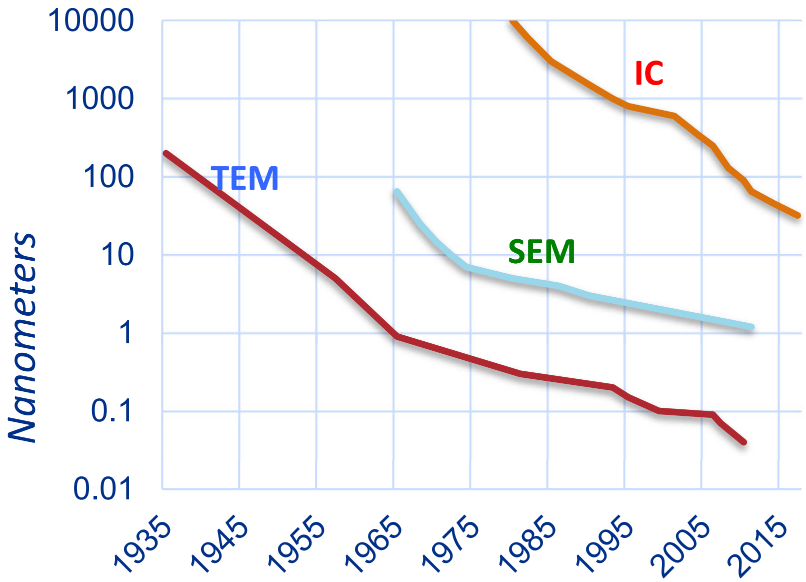
Conclusion

- Electron microcopy and associated techniques provide critical information and guidance for material improvement, fabrication optimization, and device design modification for qubit.



Courtesy of Prof. A. King

Acknowledgements

This work was supported by the U.S. Department of Energy, Office of Science, National Quantum Information Science Research Centers, Superconducting Quantum Materials and Systems Center (SQMS) under the contract No. DE-AC02-07CH11359 and performed at Ames National Laboratory.

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