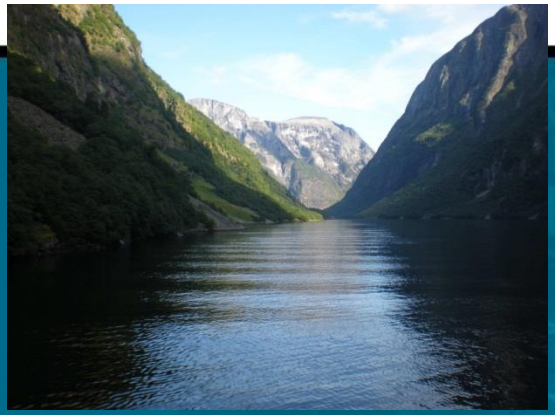
Nanoimaging

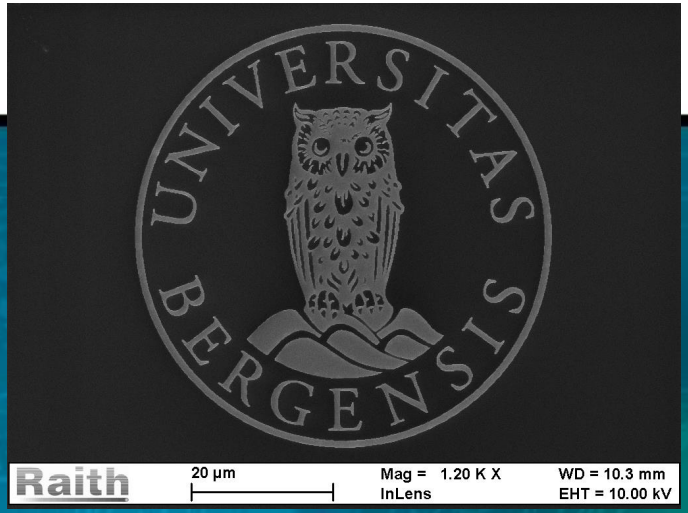
"If you cannot measure it, you cannot improve it."

Bodil Holst,

Department of Physics and Technology, University of Bergen

07.08.2019





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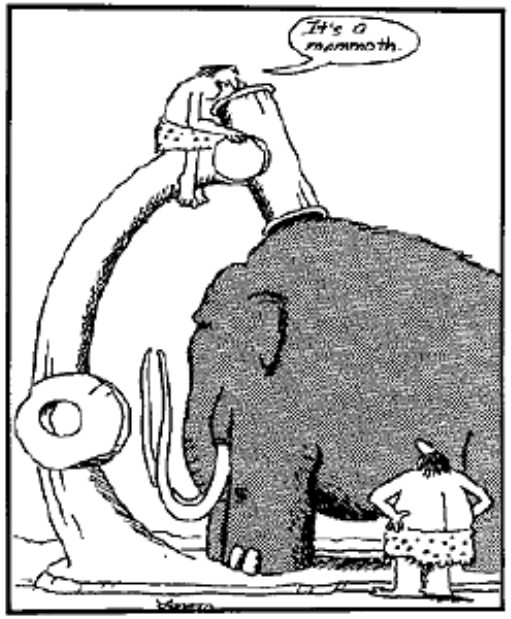
Talk Overview

- Brief introduction to optical microscopy
- Breaking the resolution limit - Atoms as “in built” light sources (Hell, Betzig, Mörner Nobel Prize 2014 and for Hell also Kavli Prize 2014)
- Using needles (Binning and Röhrer, Nobel prize 1989 Scanning Tunneling Microscope, Binning, Kavli Prize 2016 Atomic Force microscope
- Matter Waves: Electron Microscope (Nobel prize 1989) and (Helium ion microscope)
- Seeing with neutral atoms (Me and others, No Prize yet)
- Outlook – nanoimaging and nanolithography
- A fun example – microscopy meets Archaeology



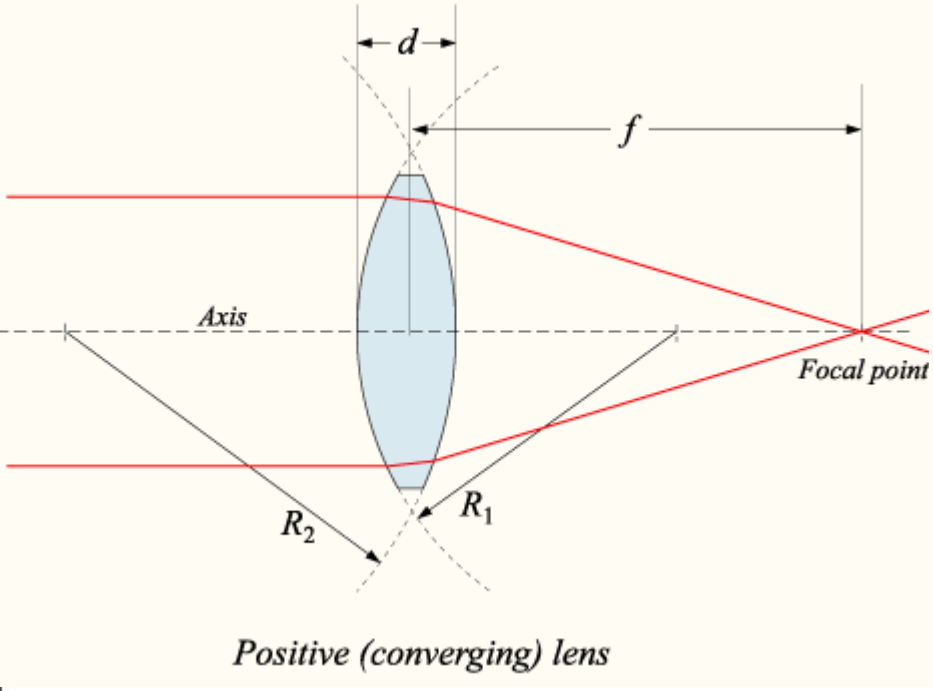
How does it work

A standard optical microscope is basically a magnifying glass

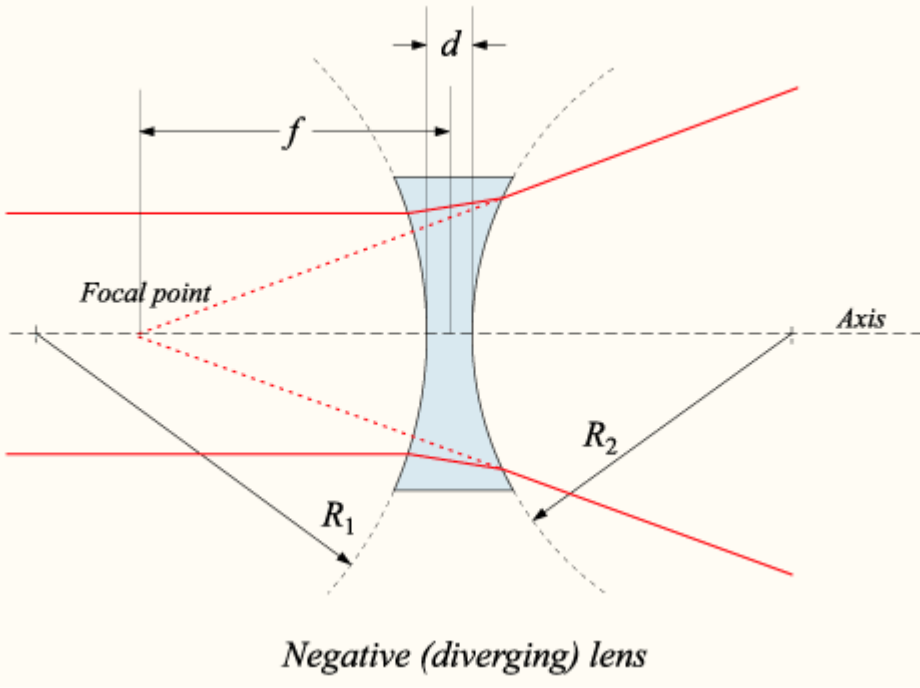
Any further lenses in the microscope is just an attempt to improve performance (reduce abberations).

In the simple mathematical threatment (Gaussian Optics, Ray optics) the performance can be reduced to one numer: The magnification

Hugens Principle in the form of refraction is exploited in lenses to focus light



Positive (converging) lens

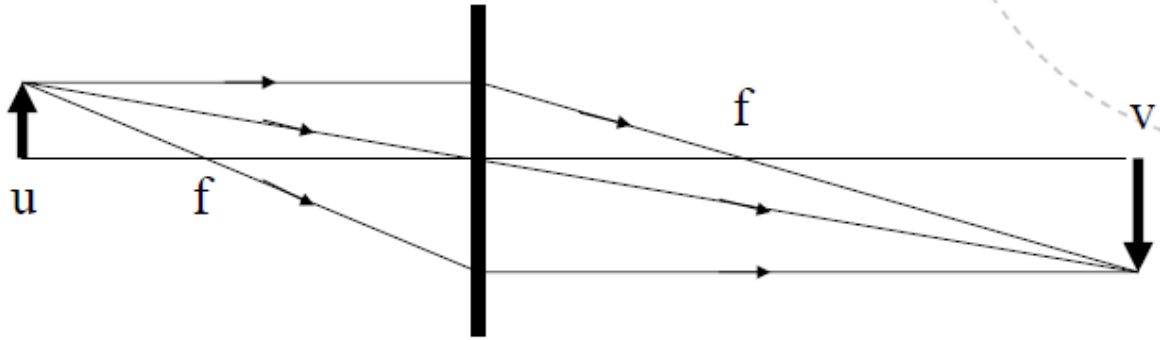


Negative (diverging) lens

The difference in refractive index between two media leads to the wave being bent at a certain angle. Knowing the difference in refractiv index between a material and a medium we can calculate a shape which leads to focussing of a plane incident wave at a particular point.

The focal point of a lens is the point where a parallel beam of light entering perpendicular to the lense wo come to focus. For diverging lenses we speak of a virtual focal point.

Lensemakers Formula



- Lens formula: $1/f = 1/u + 1/v$

If you know the focal length of a lens you can always work out the size of an image – The magnification factor: v/u, if v<u then we have a demagnification.

The pathway of the beam can be sketched following simple rules.

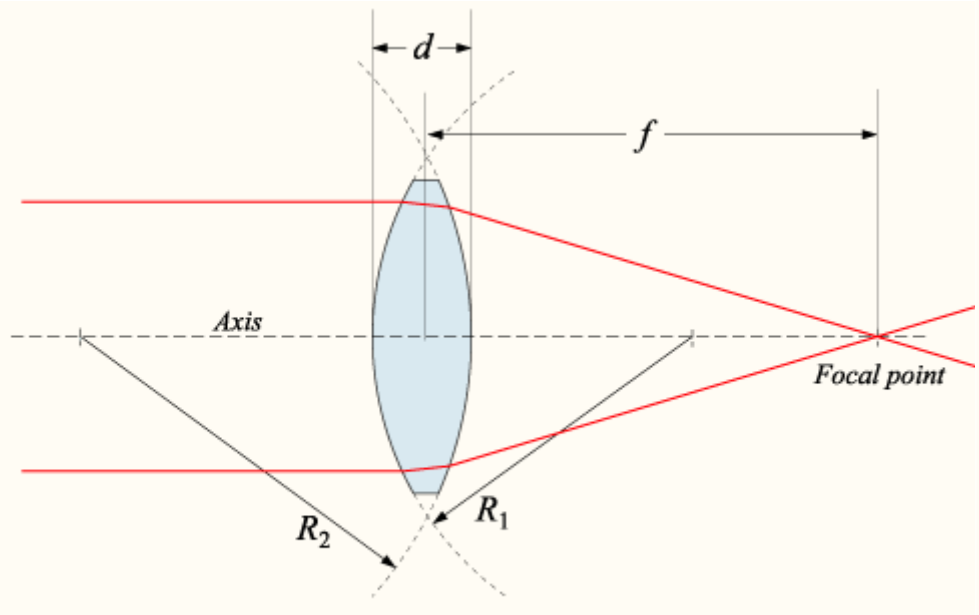
i) The object at diatance u is illuminated with a diffuse light source and emits spherical waves at all points according to Huygens principle.

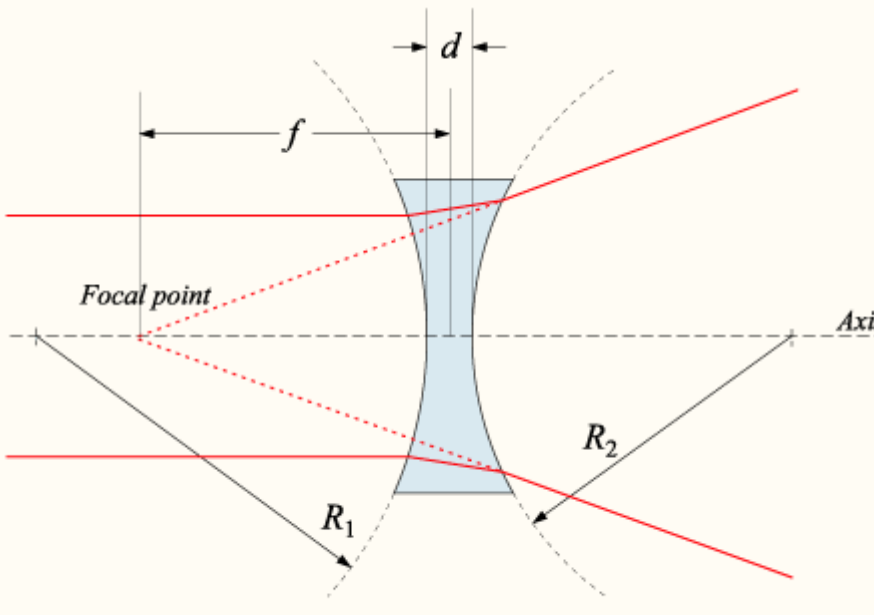
ii) All waves going through the centre of the lense are not bent

iii) All waves going thorugh the focal point on one side come out as parallel waves on the other side.

iv) Question: So why do you not see the image upside down when you look in a magnifying glass?

Hugens Principle in the form of refraction is exploited in lenses to focus light





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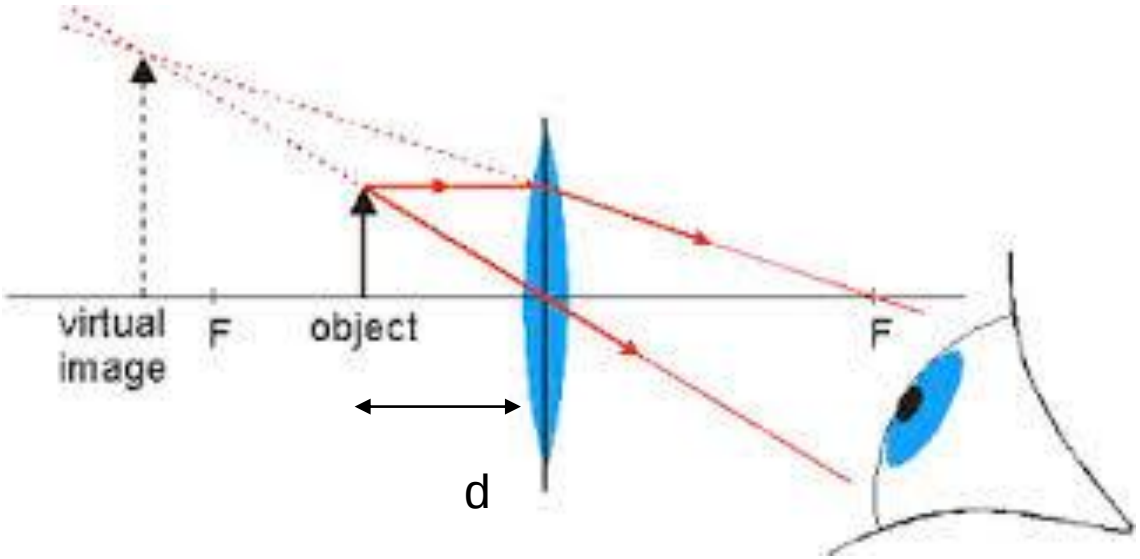
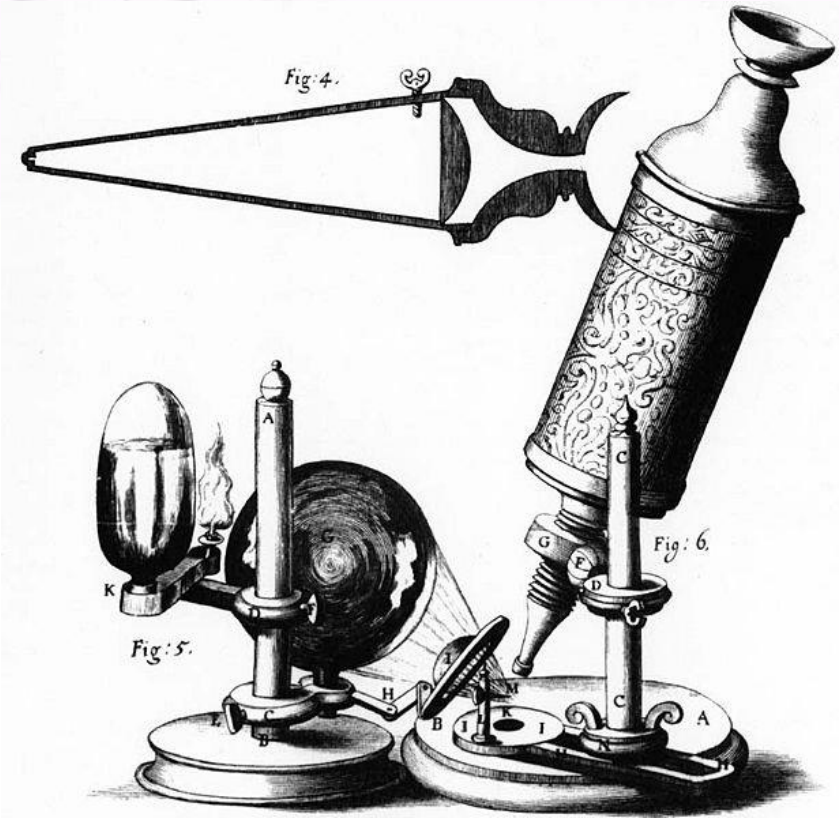
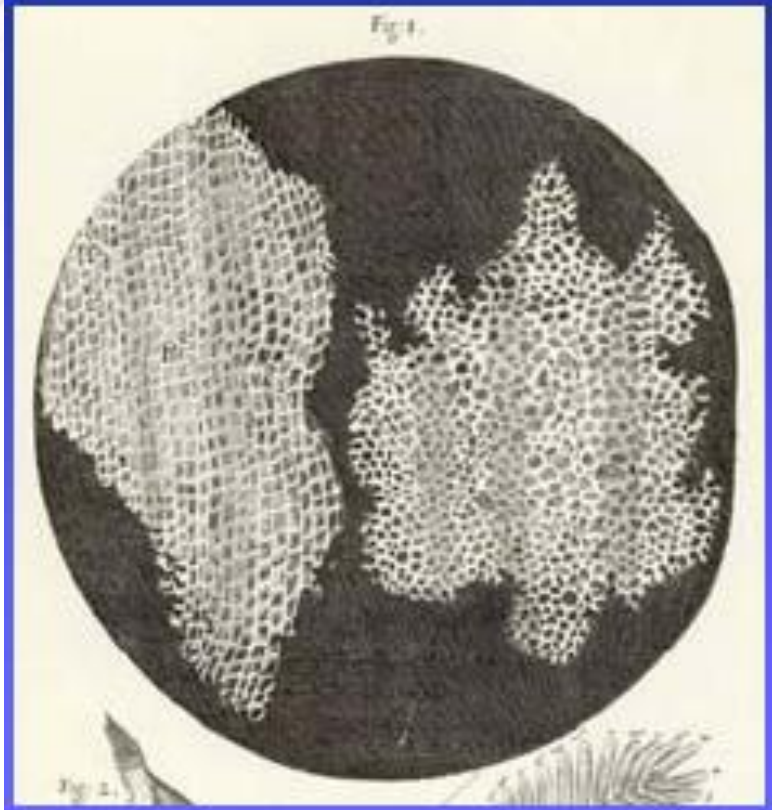


Optical Microscope History



- Already the romans used glass bowls filled with water for magnification
- 1665 Robert Hook used a home built microscope (about 50x magnification)
- The object is imaged in reflection and has a point like illumination created from a candle, a diffuser, a condenser lense and a limitng aperture.

Hooke saw 'cells' in cork

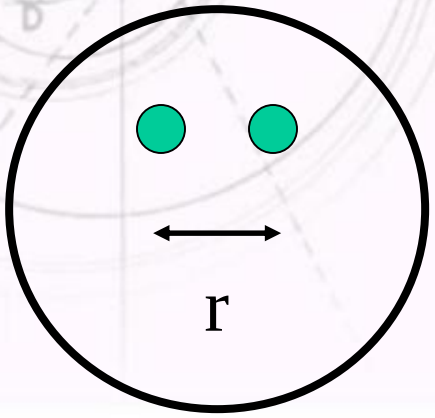


Thin slice of Cork

I could exceedingly plainly perceive it to be all perforated and porous, much like a Honey-comb…

…these pores, or cells…

Resolution (r) – the most important microsocopy parameter



The resolution of a microscope is the minimum spacing between two objects, which still allows them to be viewed as separate objects.

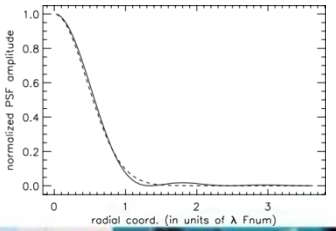
NO UNIQUE DEFINITION, but depends crucially on:

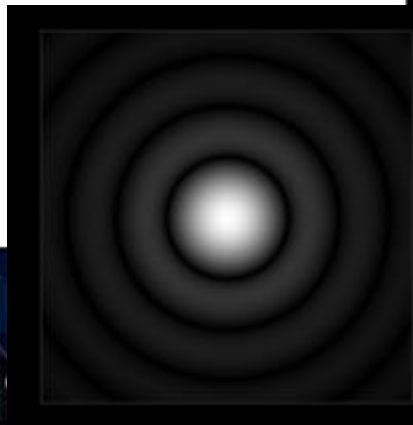
- Size of lens
- Wavelength of light
- Type of illumination (coherent/incoherent)

Resolution, continued: Airy Disk – Diffraction at the edge of the lense

- In optics, the **Airy disk** (or **Airy disc**) and **Airy pattern** are descriptions of the best focused spot of light that a perfect lens with a circular aperture can make, limited by the diffraction of light
  - The light diffracts at the edge of the lense. Imagine that you have a range of spherical waves being emitted around the edge of the lense.
  - This means that a point source will not be focused to a perfect point, but to a central spot with circular rings around
- You can calculate, using the Frauenhofer diffraction formula that far away from the lens (by far away we mean at a distance several orders of magnitude larger than the wavelength and several orders of magnitude larger than the diameter of the lens), the angle at which the first minimum occurs, measured from the direction of incoming light, is given by the formula (λ=wavelength, D=diameter of lens):

$$\sin\theta = 1.220\lambda/D$$





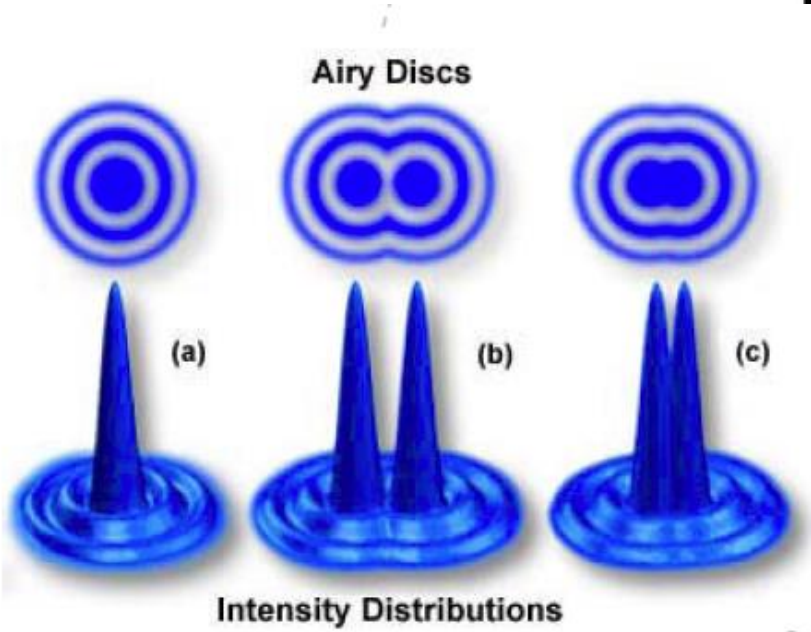
Rayleigh criterion of resolution

- The Rayleigh criterion: If two points are separated by an angle small enough that their Airy disks start overlapping, the objects can not be clearly separated any more in the image, and they start blurring together. Two points are said to be *just resolved* when the maximum of the first Airy pattern falls on top of the first minimum of the second Airy pattern (the Rayleigh criterion).
- We see from the picture that this criterion is fulfilled when separation is precicely the diameter of one airy disc. So in angle this gives us again the formula

  $\sin\theta = 1.220\lambda/D$

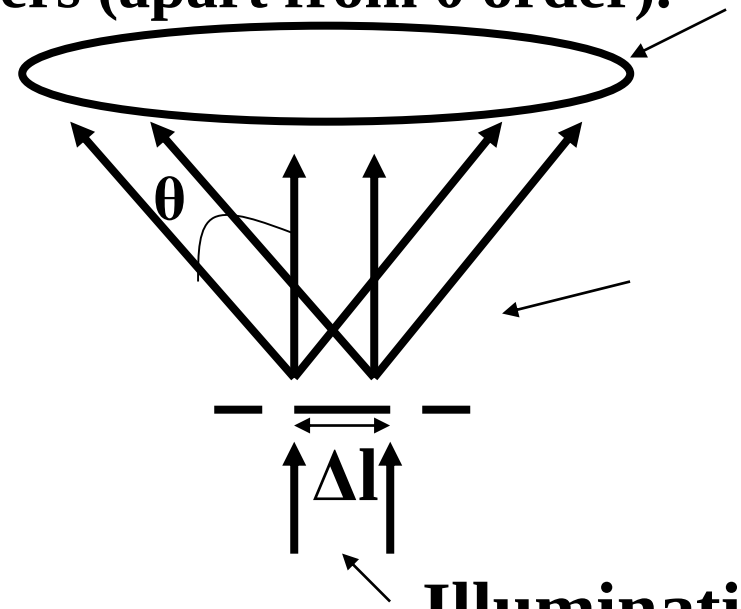
We can convert this to a distance, Δl, when we have the focal length of the lens, f:

$$\Delta l = 1.220\frac{f\lambda}{D} \qquad \textbf{(}\Delta\textbf{l/f=sin}\theta\textbf{)}$$



1873: The Simple Abbe-Criterion for resolution

To resolve a feature, the lens must catch at least two diffraction orders (apart from 0 order).



Lens span the angle θ

Visibel Light:
λ in the range:
400 – 700 nm

Illuminating Beam

Second diffracted peak in a double slit occurs at
$2n\sin\theta=\lambda/\Delta l \Rightarrow \Delta l=\lambda/2n\sin\theta$ (n is the refractive index)
$n\sin\theta$ is also called NA (numerical aperture) of the lens.
The higher NA, the higher the resolution.)

Ernst Abbe Monument

Fürstengraben, Jena

d — Minimum resolved distance

λ — Wavelength of imaging radiation

α — Half-aperture angle

n — Refractive index of medium

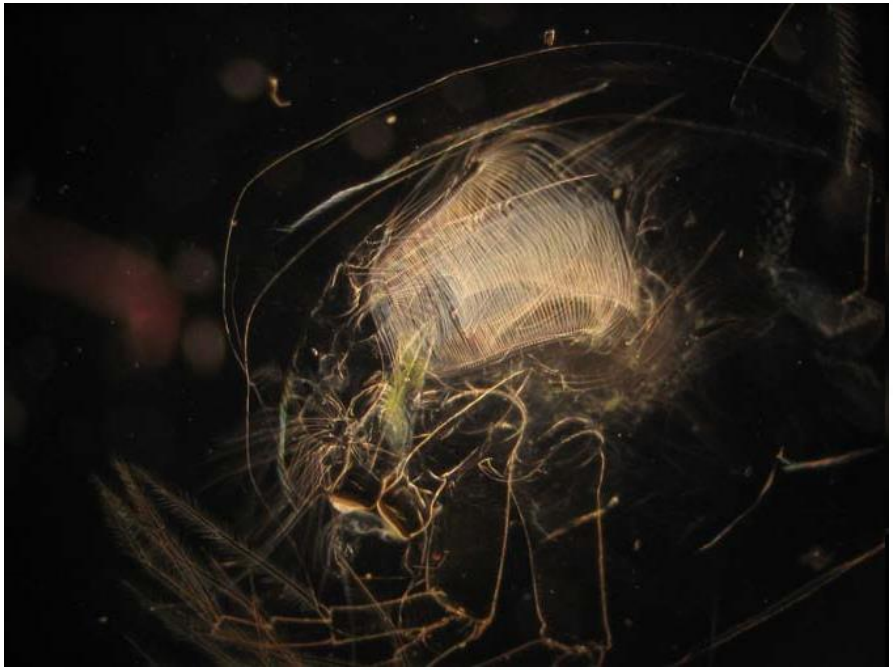
Numerical Aperture

Minimum resolved distance is also often expressed as

$d = 0.61 \lambda / NA$

Bright Field

Dark Field – 0-order beam Filtered out

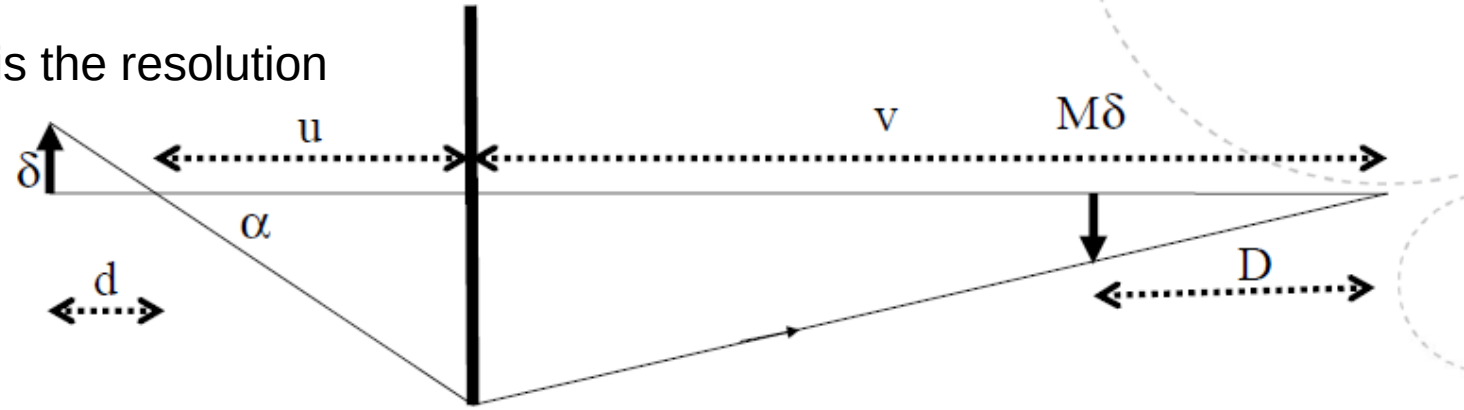


Shed exo-skeleton of a *Daphnia magna*, main body, antenna and legs

Important Definition

- **Depth of Field – This is important if you want to image 3D objects (hills and valleys)**
  - Depth of field is the axial depth of the space on both sides of the **object plane** within which the object can be moved without detectable loss of sharpness in the image, and within which features of the object appear acceptably sharp in the image while the position of the image plane is maintained.
- Depth of Focus
  - Depth of focus is the axial depth of the space on both sides of the **image plane** within which the image appears acceptably sharp while the positions of the object plane and of the objective are

Where δ is the resolution

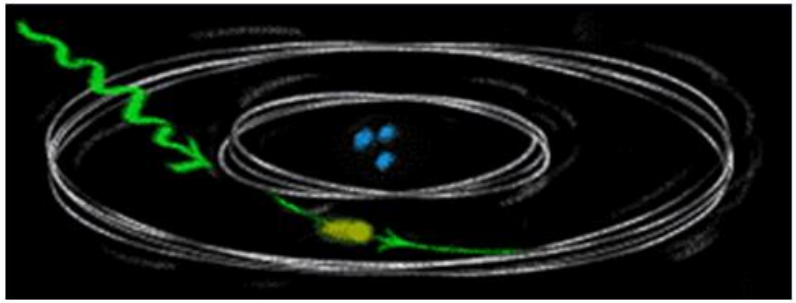


- Depth of field: $d = \delta / \tan\alpha = \mu\lambda / (NA)^2 = 0.61\lambda / \mu \sin\alpha \tan\alpha$
- Depth of focus: $D = M^2 d$
- (Field of view: area of sample on image)

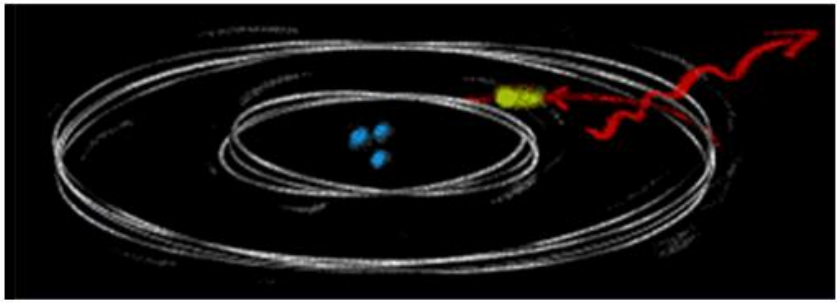
Surpassing the limitations of the light microscope:

For a long time optical microscopy was held back by a presumed limitation: that it would never obtain a better resolution than the wavelength of light. Helped by fluorescent molecules the Nobel Laureates in Chemistry 2014 ingeniously circumvented this limitation. Their ground-braking work has brought optical microscopy into the nanodimension.

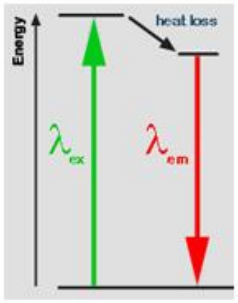
Flourescence



Fluorescence absorbtion - excitation



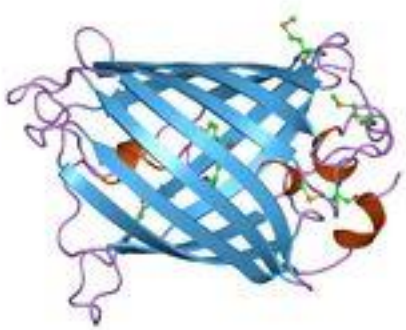
Fluorescence emission



Adapted from Internet GIF, source unknown

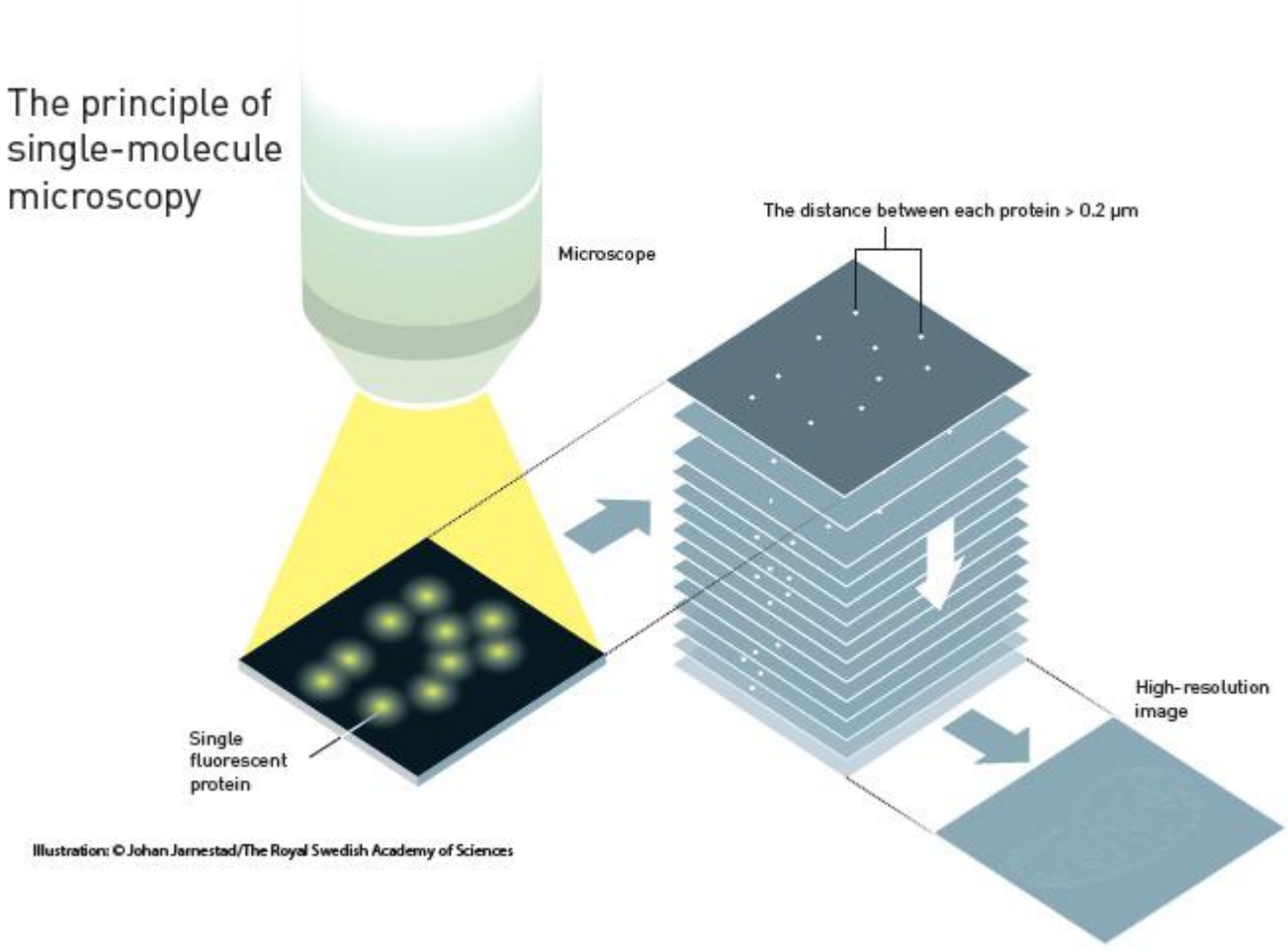
Moerner – The Light Switch Molecule

Modified Green Flourescent Protein



When you illuminate it with a given wavelength, it flourescence, But when
It has flourescenced, you cannot
get it to flourescenced again before you have illuminated it with another wavelength - It is so to say switched off

Betzig
Exploiting the “Switch Molecule”



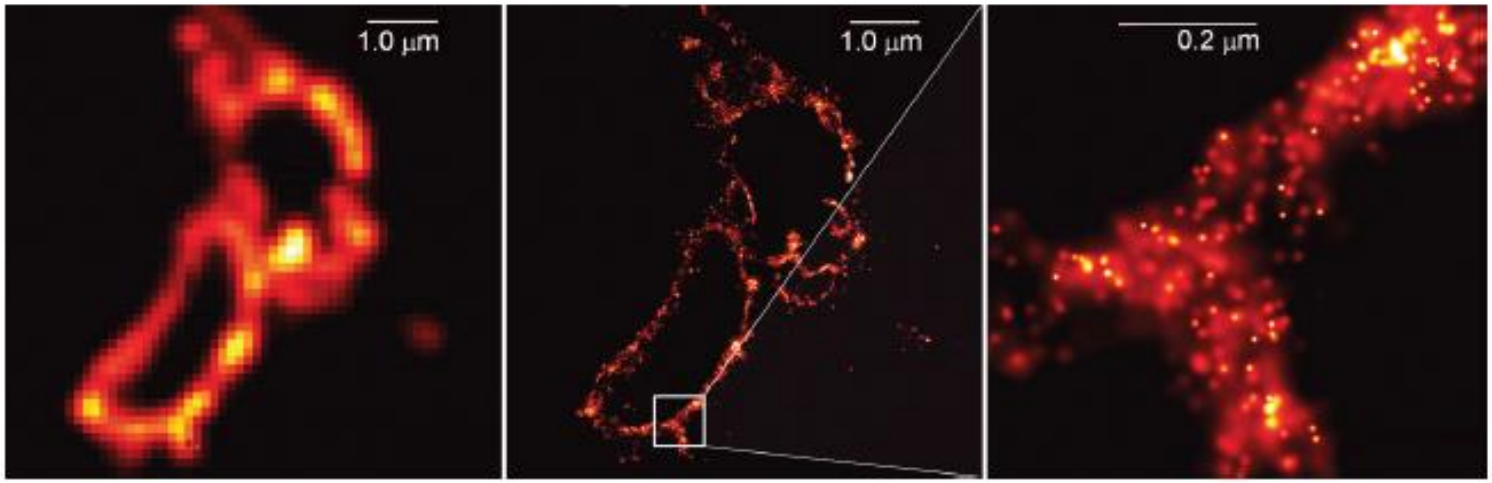


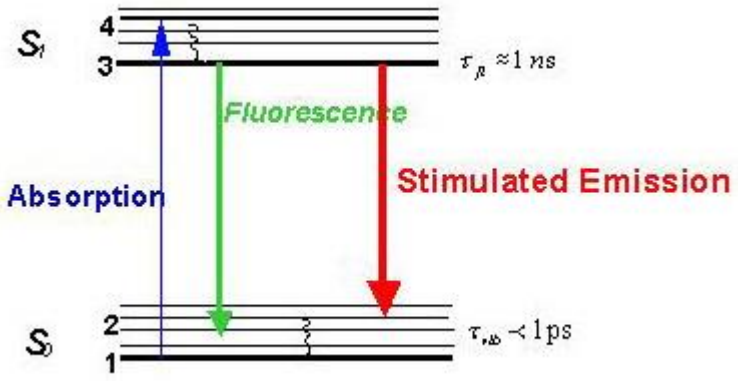
Figure 5. The centre image shows lysosome membranes and is one of the first ones taken by Betzig using single-molecule microscopy. To the left, the same image taken using conventional microscopy. To the right, the image of the membranes has been enlarged. Note the scale division of 0.2 micrometres, equivalent to Abbe's diffraction limit. The resolution is many times improved. Image from *Science* 313:1642–1645.

The STED Microscope

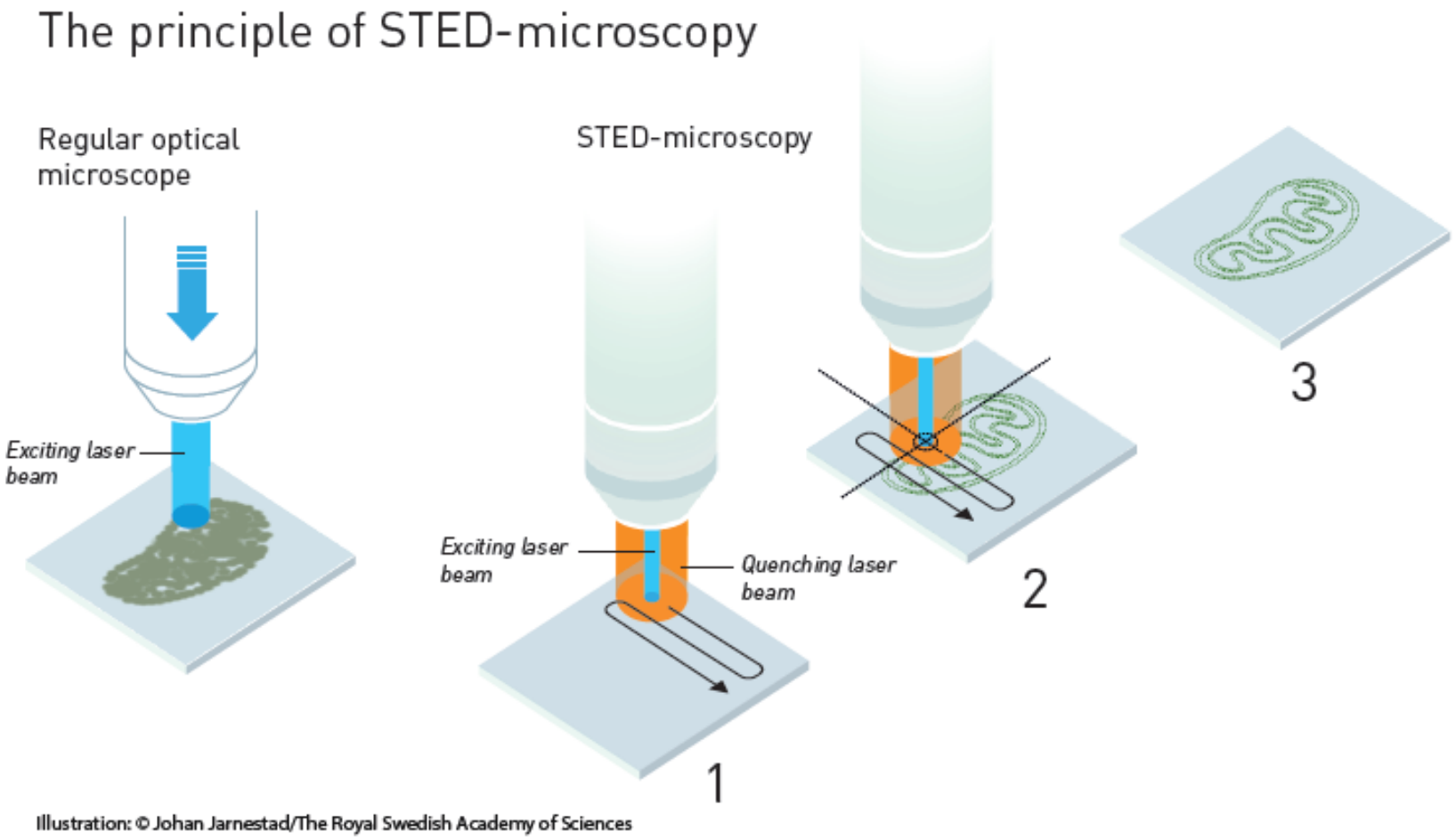
Einstein predicted the phenomenon of Stimulated Emission:

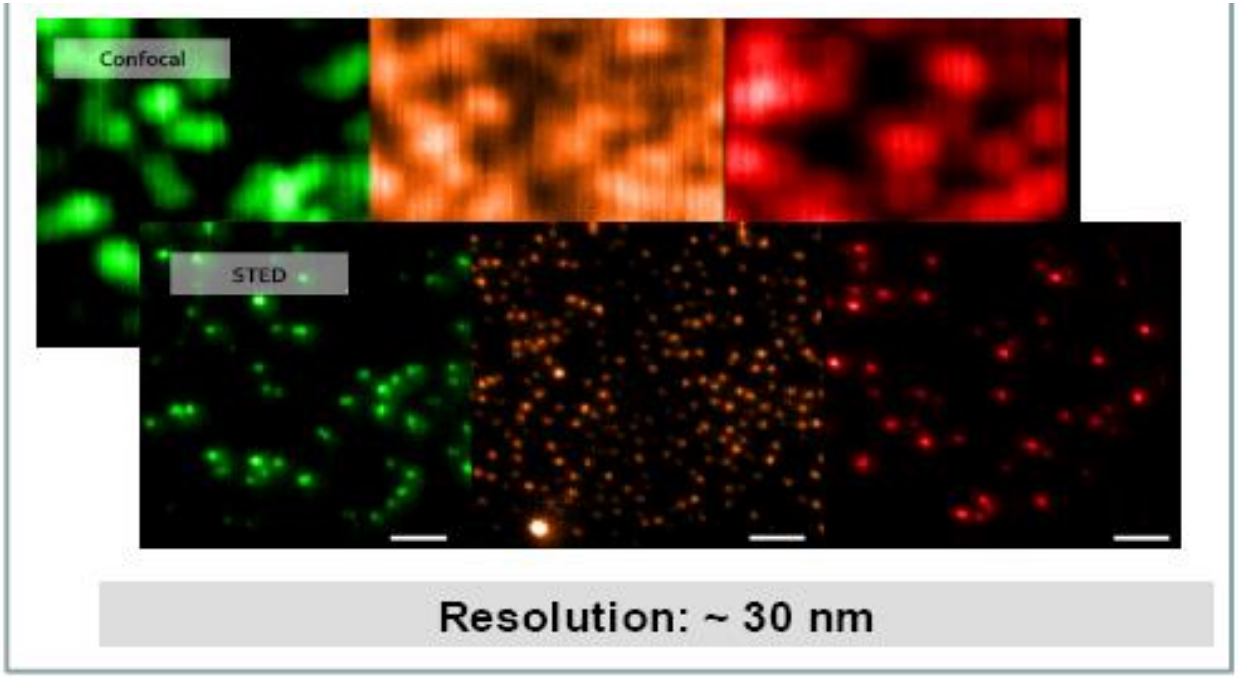
By placing a particular excited molecule in a particular light field,

the molecule is forced to de-excite at the wavelength of the light field, instead of flouresing.



Stimulated Transmission Emission Deplition



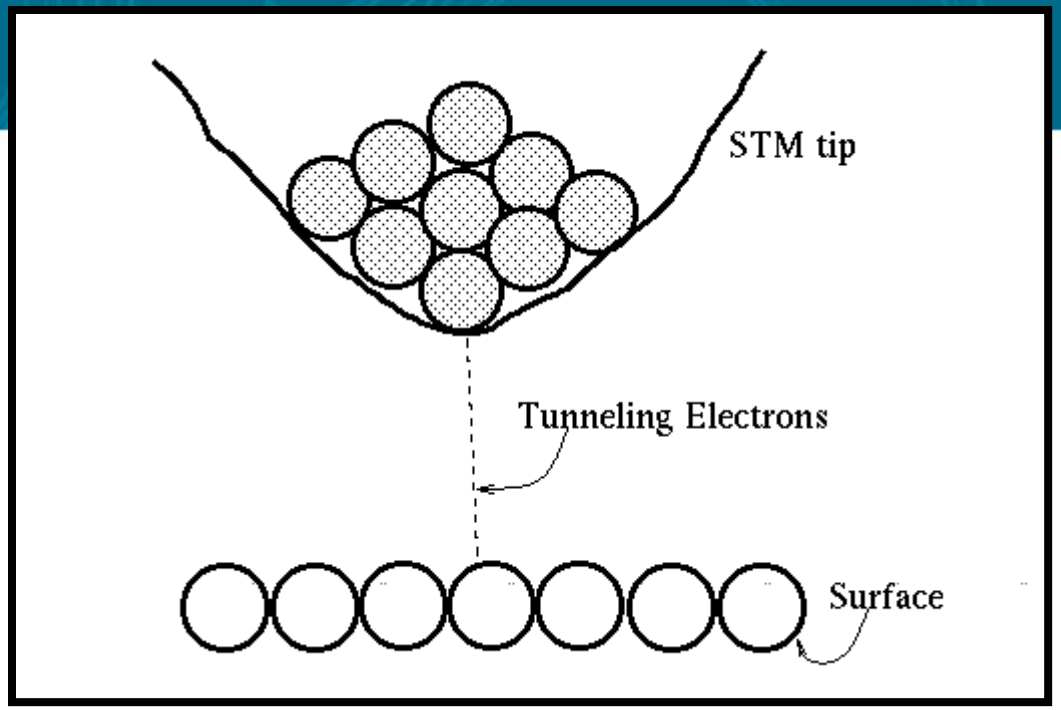


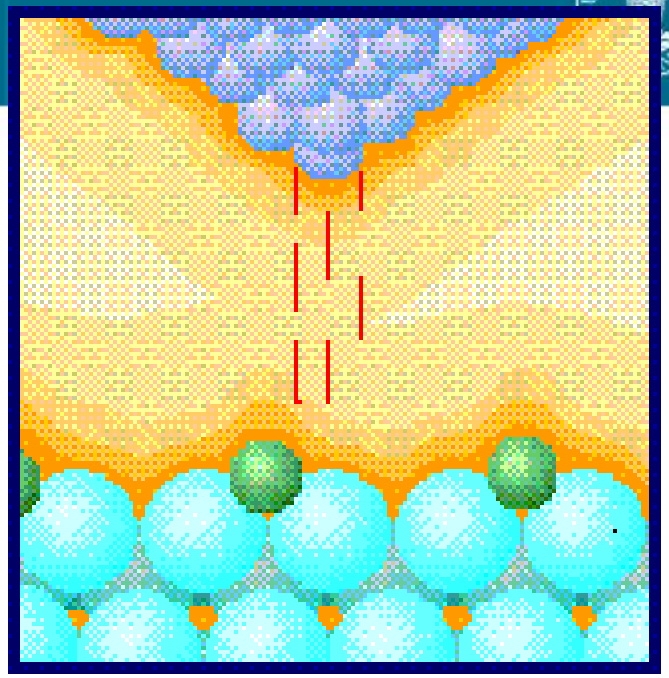
Wildanger *et al.*, *Opt. Expr.* **16** (13), 9614 (2008)

Imaging of bio-functionalised nanoparticles using STED

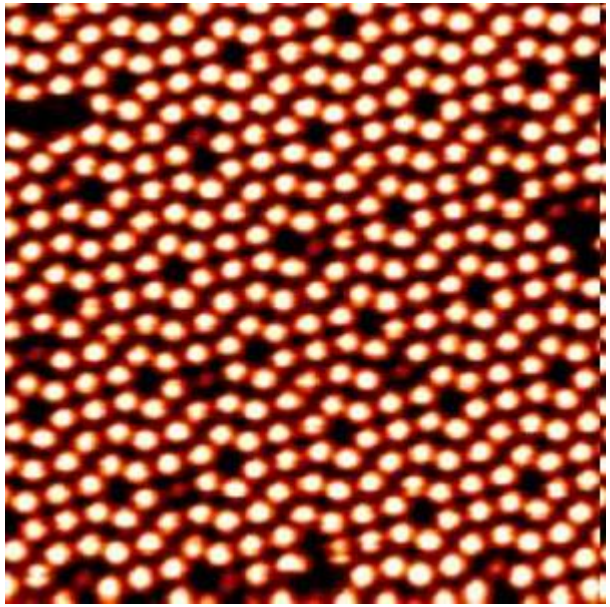
Scanning Tunneling Microscopy (STM)

- I 1980 HEINRICH ROHRER og GERD BINNIG got a great idea: To exploit the tunnel effect to see atoms and molecules.





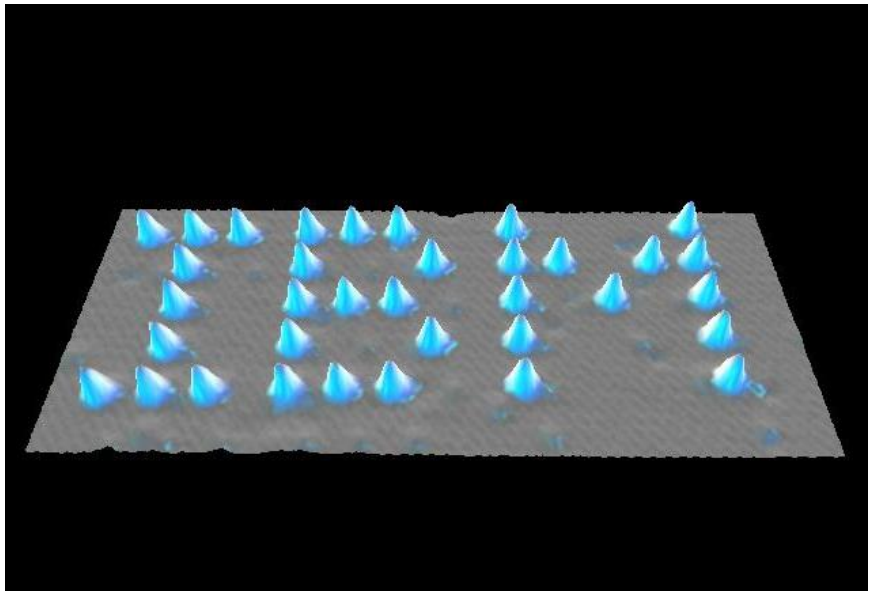
In the scanning tunneling microscope (STM) a small voltage (typically around 5 V) is applied between a needle and the sample. If the distance between needle and sample is very small (around 1 nm) then electrons can tunnel skanning from the sample to the tip (or the other way depending on the bias). This current can be measured and used to «see» the surface.



One of the first important questions to be solved using STM was the structure of the silicon surface: Si(111)-7x7 structure.

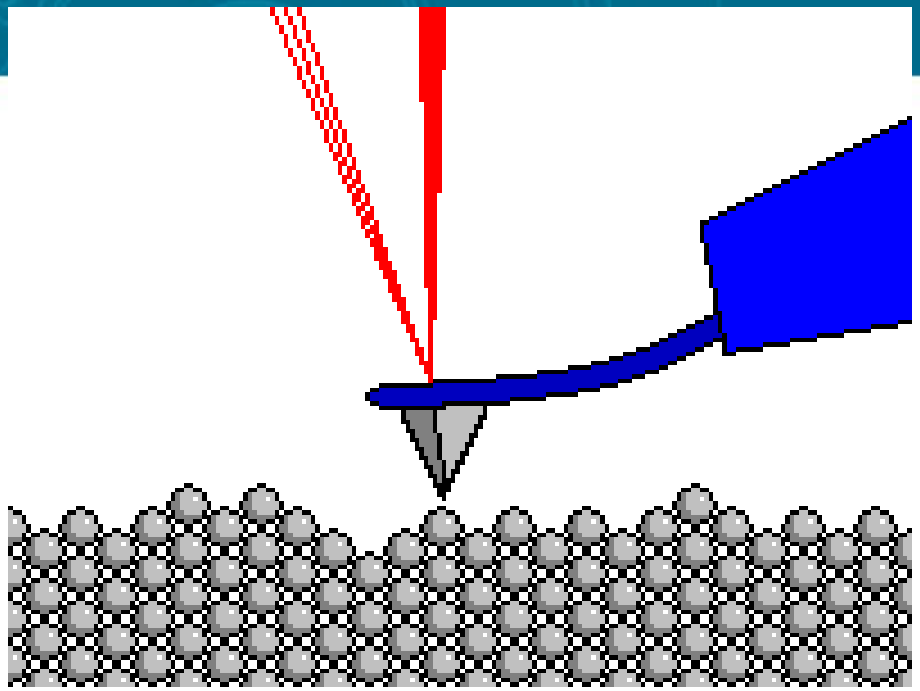
Using STM to write with Atoms

- Don Eigler, IBM –Kavli Prize in 2010
- Very slow, but used to make model devices on the nanoscale (spintronics)



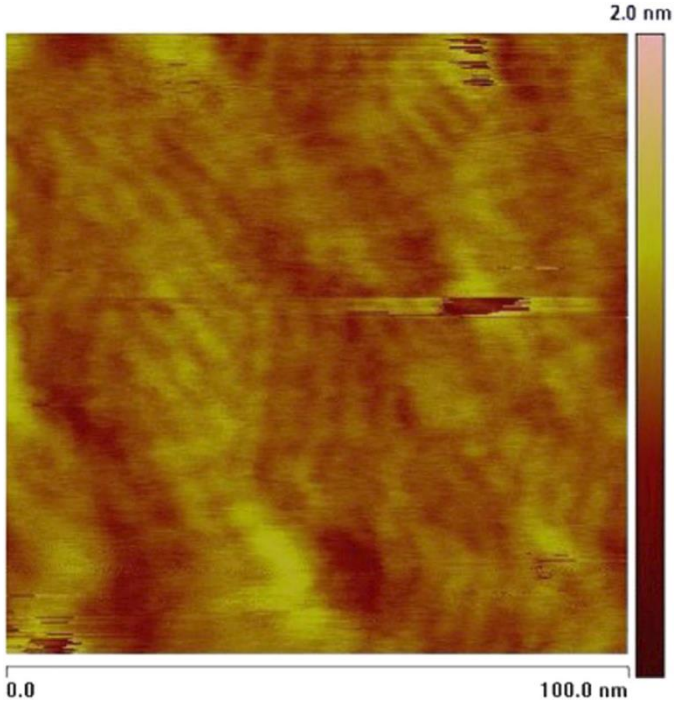
Atomic Force Microscopy (AFM)

- **As the name says – AFM measures forces at the atomic level.**



AFM: The tip moves up and down due to the inter-molecular forces acting between the atoms on the tip and the atoms on the surface

MORE DETAILS TOMORROW



AFM image of an α-quartz (0001) surface. Three domains of “ribbons”rotated 120° relative to each other are clearly visible.

S. D. Eder, K. Fladischer, S. R. Yeandel, A. Lelarge, S. C. Parker, E. Søndergård, B. Holst, Scientific Reports,5:14545 (2015)

Matter Waves

- **Matter Waves**
  - **The concept was introduced in 1924 in the Ph.D theses of Louis de Broglie (1892-1987). Noble price 1929.**
  - **All matter (any object) has a wave-like nature (wave-particle duality). The** de Broglie relations **show that the wavelength is inversely proportional to the momentum of a particle and that the frequency is directly proportional to the particle's kineti energy. The wavelength of matter is also called** de Broglie wavelength**.**
  - **Experimentally verified in 1927 by electron diffraction from nickel ($\lambda=h/p=h/mv$)**
    - **(simple form, ignoring relativity effects)**
  - **$E=1/2mv^2 \rightarrow E = h^2/(2\lambda)$**
  - **Note: For a given wavelength: The higher the mass the smaller the energy of the particle matter wave.**

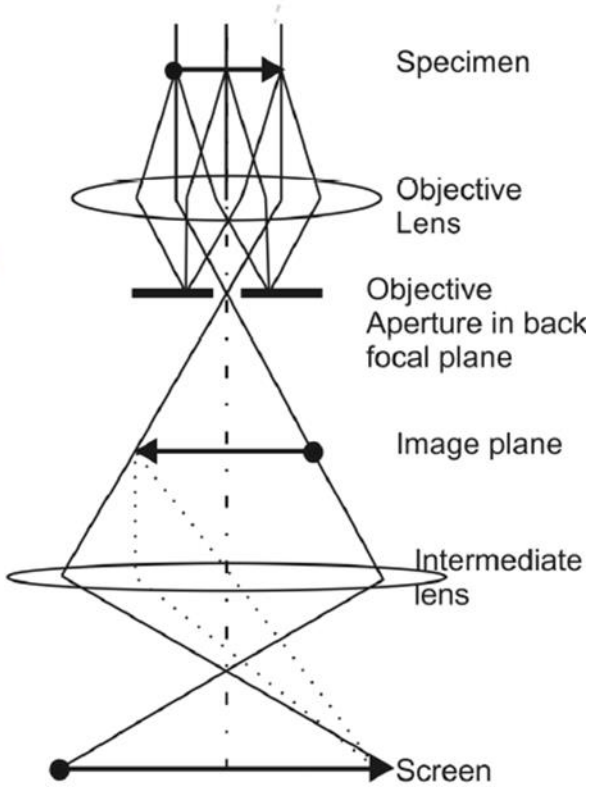
Prince Louis de Broglie

Microscopy with matter waves

- The electron microscope was invented in 1932. Used in a transmission and a reflection version.
- The optics used exploits the electron charge. Due to some fundamental problems with lens aberrations the resolution was limited to a few nm until ..
- 1989 Harald Rosen fixes the Mathematics
- 1991-1997 K. Urban and others build an aberration corrected microscope with support from the Volkswagenstiftung
- 200? – First commercially available atomic resolution TEM

Ray diagram TEM imaging

- Image is determined by what is selected by objective aperture!
- Same general principles electron, light and X-ray microscopy



Scattering

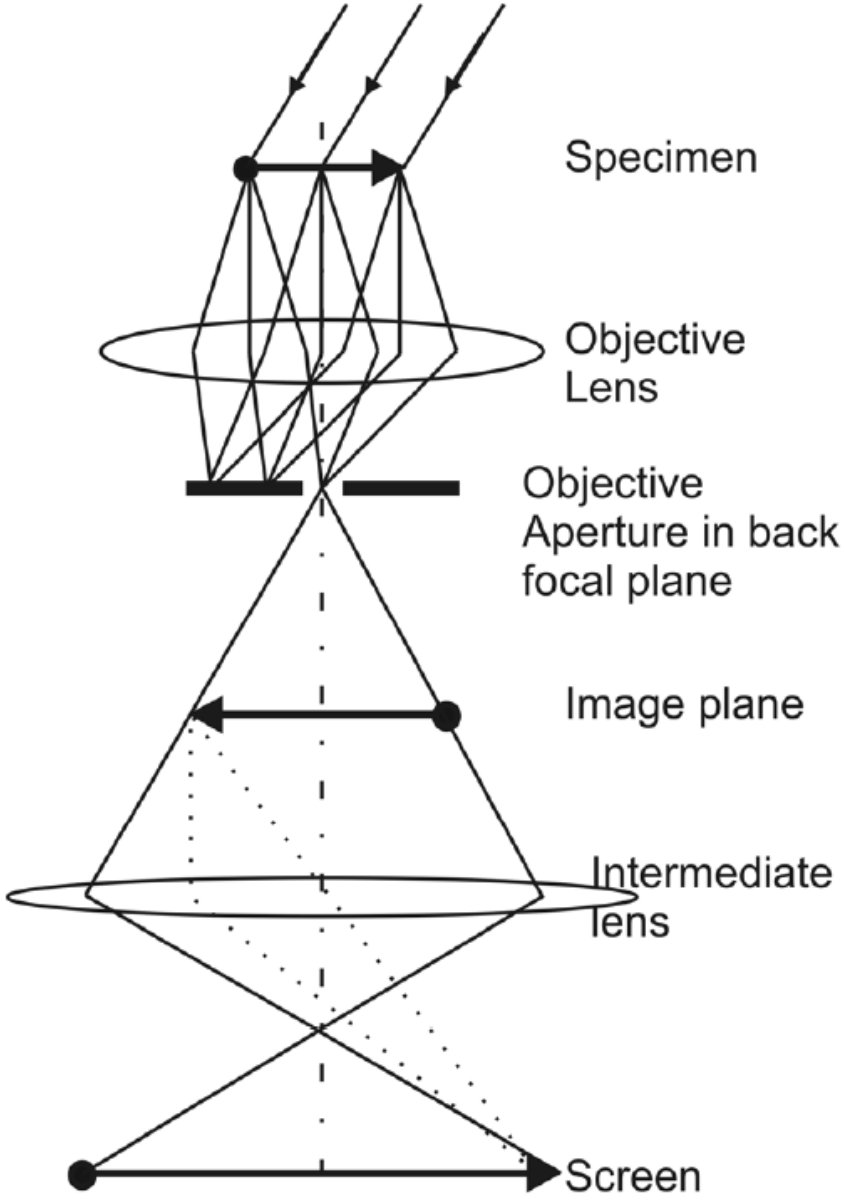
- Electrons are charged particles!
- Scatter on electrons and nuclei
- Large angle (incoherent) after scattering on nuclei: Rutherford scattering approach (BSE): strong Z dependence
- Low angle (< 3°, wave approach): collective scattering on arranged electrons: diffraction: dominates scattering in TEM
- Atomic scattering factor $f(\theta)$ depends on λ, θ and Z
- $|f(\theta)|^2$ is proportional to scattered intensity (a measure for it like the differential cross section)
- Structure factor:

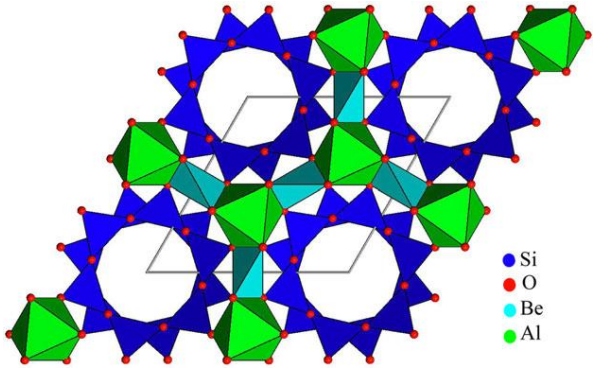
$$F_{hkl} = A_0 \sum_{n}^{unitcell} f_n e^{2\pi i (hx_n + ky_n + lz_n)}$$

First Atomic Resolution Transmission Electron Microscopy images of natural minerals - Beryls

Dark field (DF)

- Use a diffracted beam for forming the image
- Improved contrast (like in optical) but lower signal.
- "dirty" or central DF
- Very powerful technique as orientation info is in image (DF_{hkl})

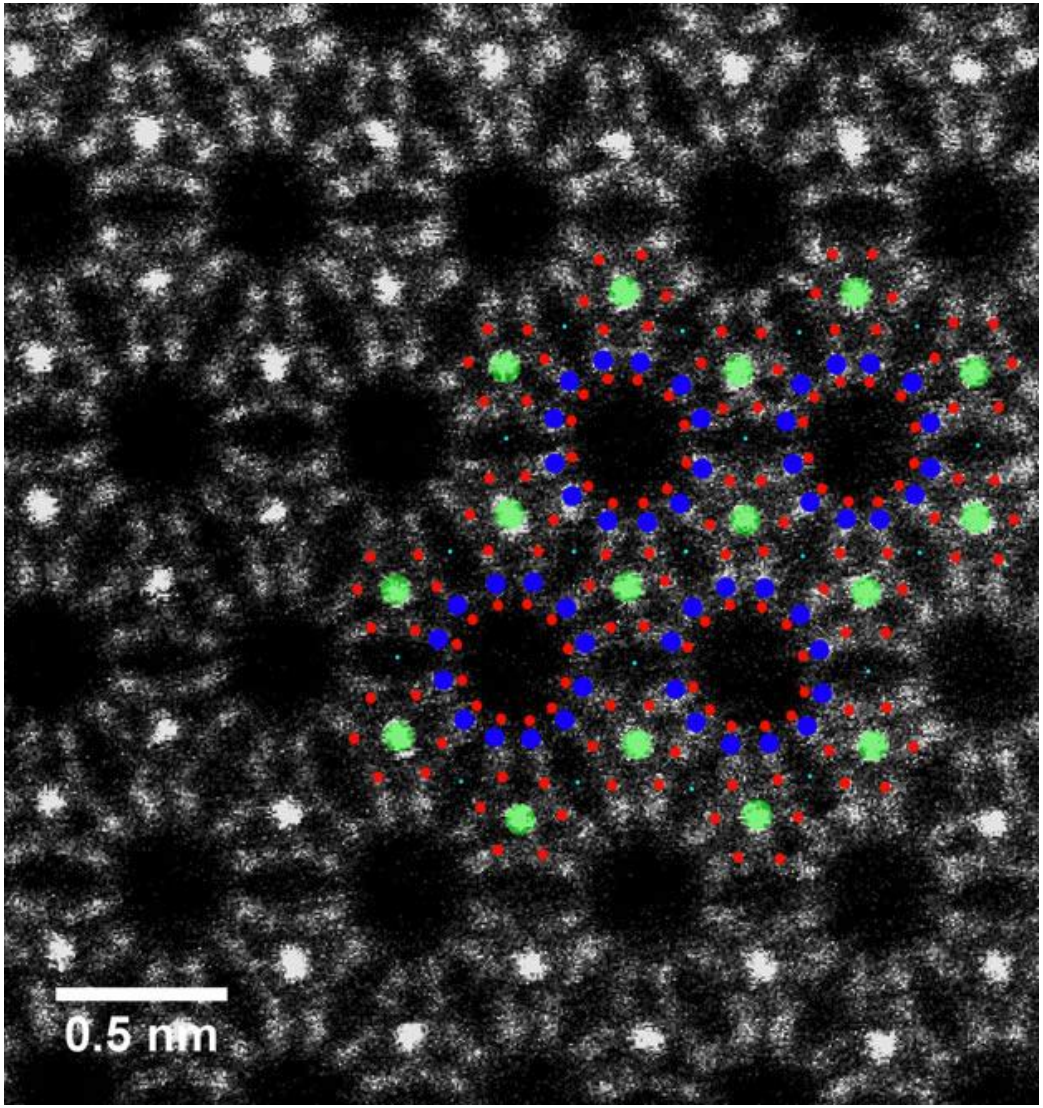




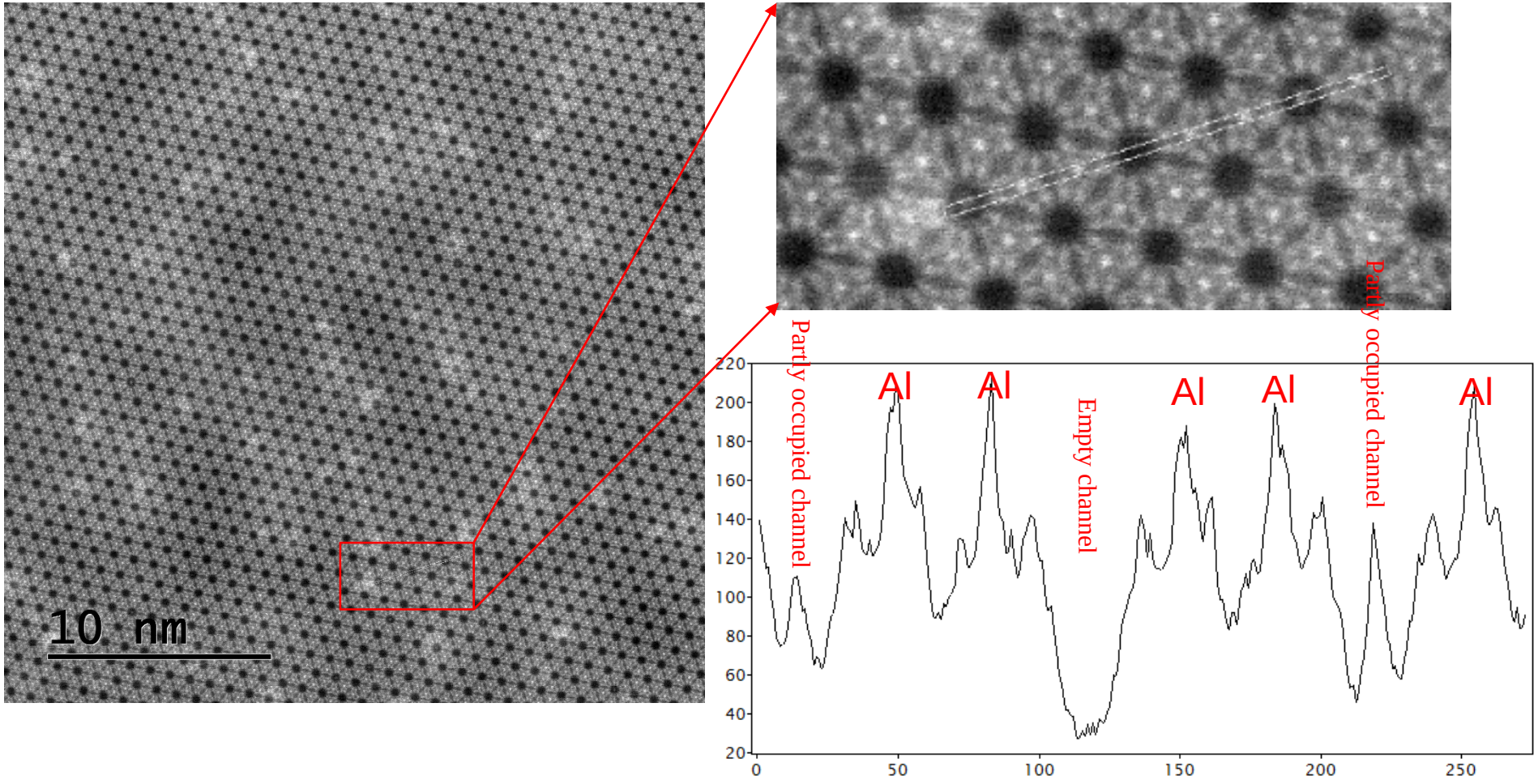
Al (Z=13), Si (Z=14), Be (Z=4), O (Z=8)

$Be_3Al_2(Si_2O_6)_3$

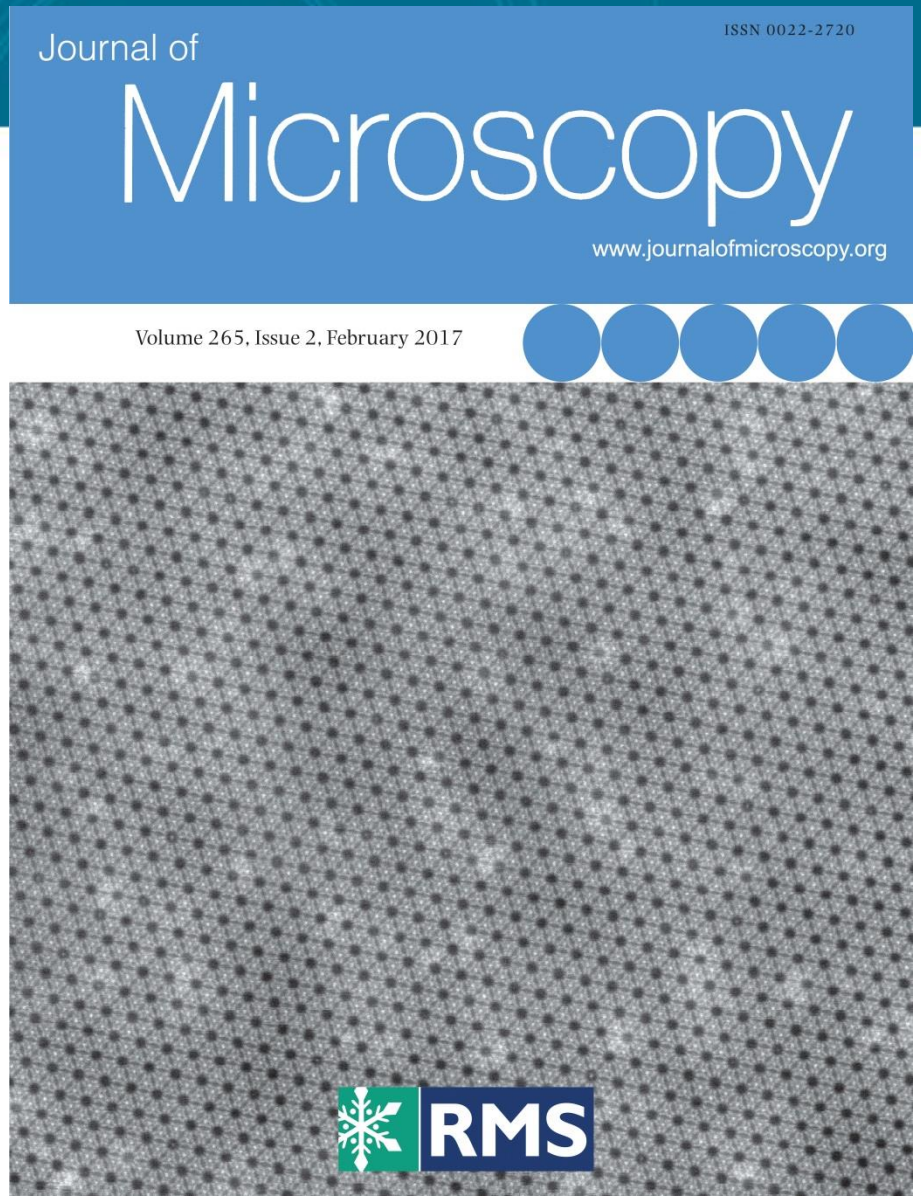
Why do we see Al most clearly –
Because there are twice as many Aluminium atoms i a column as Silicon atoms



Direct imaging of occupied Channels

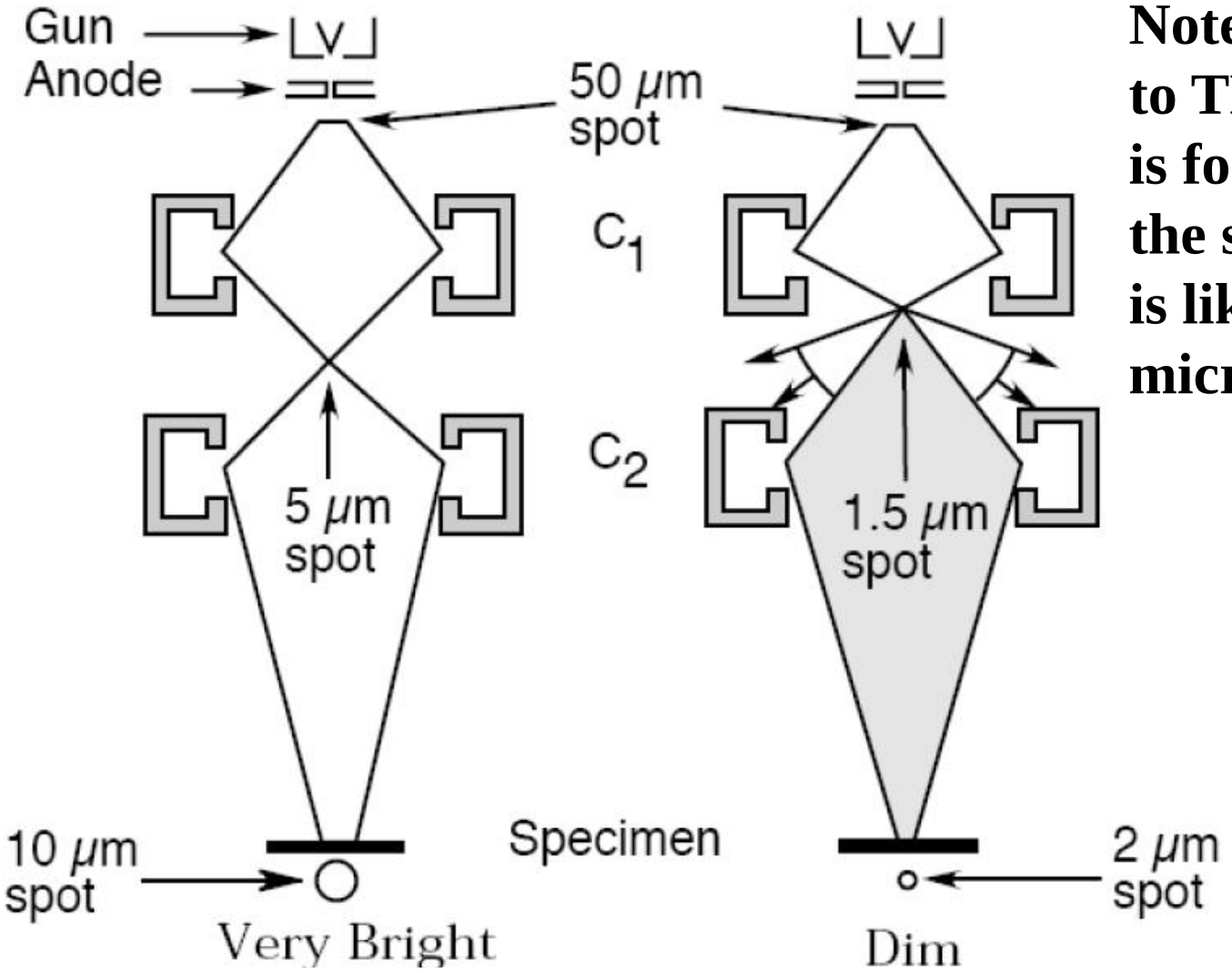


Intensity profile along a line covering 2 filled and one empty channels, in addition to 5 Al columns and parts of several Si columns.



V. Arivazhagan, F. D. Smith, P. E. Vullum, A. T. J. van Helvoort, B. Holst, Atomic resolution imaging of beryl: an investigation of the nano-channel occupation, p. 245

Ray diagrams: Probe (Spot) size

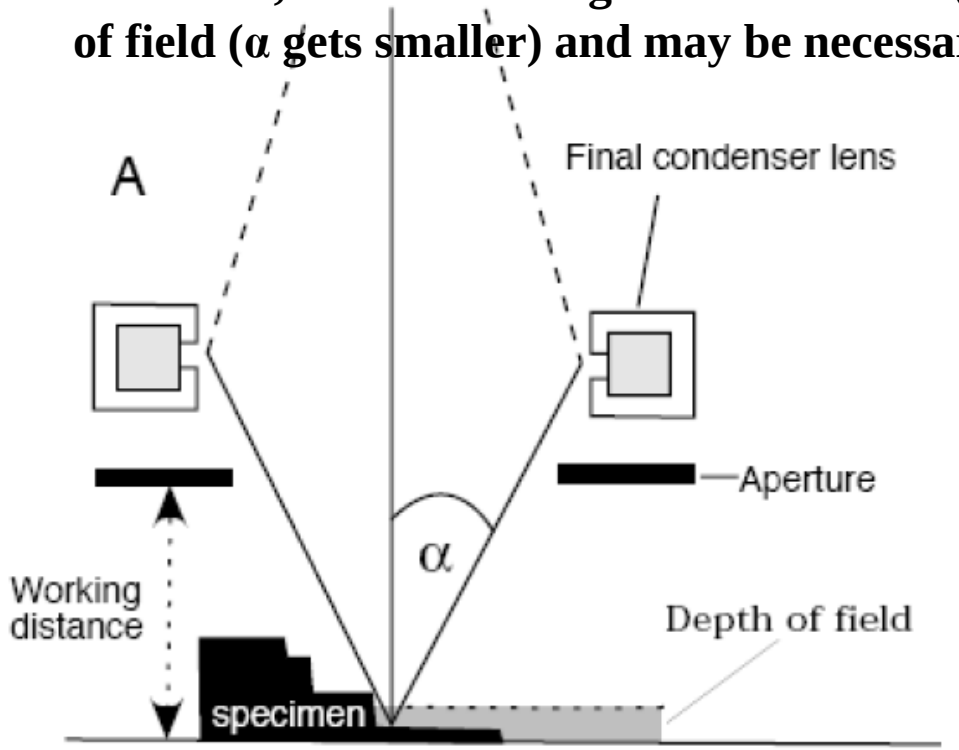


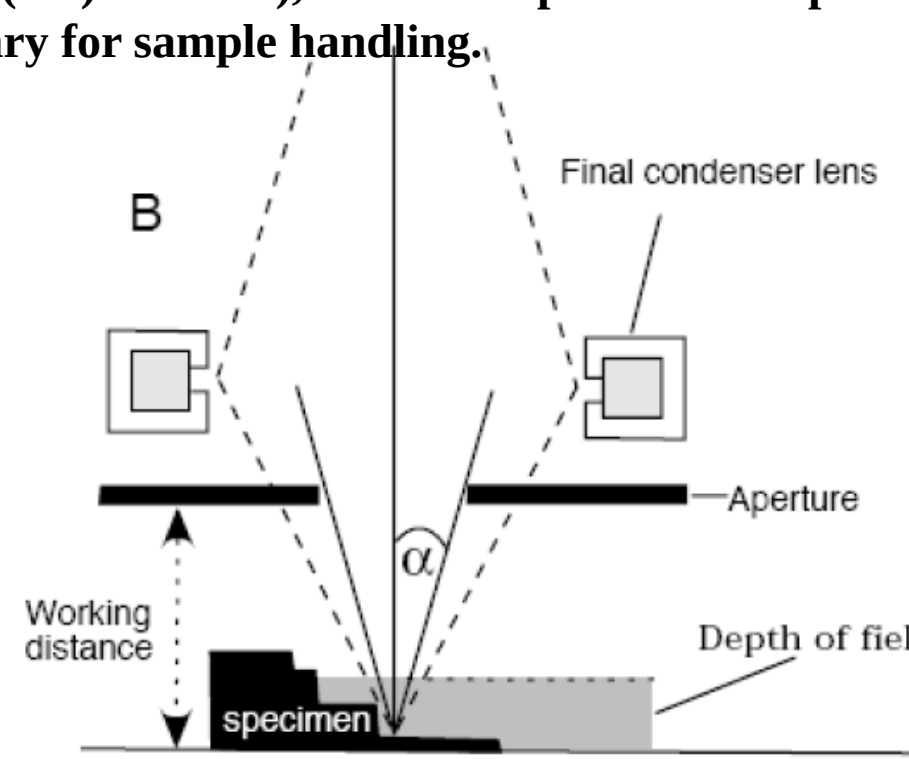
Note difference to TEM, beam is focussed onto the sample (this is like a confoca microscope)

Focussed beam scanned across sample. NB! Resolution determined partly by the spot size (more later). The smaller the spot size the fewer electrons in the focussed spot as illustrated in the diagram. Spot size down to a few nanometers possible. Limited by the optics.

Ray diagrams: Aperture size

Smaller aperture decreases the beam intensity, but improves the depth of field. Resolution is decreased (spotsize is increased) but this is a small effect. Increasing the working distance decreases the resolution (u+v is constant, so if you increase v, then the demagnification factor (v/u) increase), but this improves the depth of field (α gets smaller) and may be necessary for sample handling.





Depth of Field (d)

- **Optical Microscopy**, assuming greenish light (λ=500 nm) and NA=0.65, no oil, →sinα=0.65→ α = 40° og resolution **δ=0.47 µm**

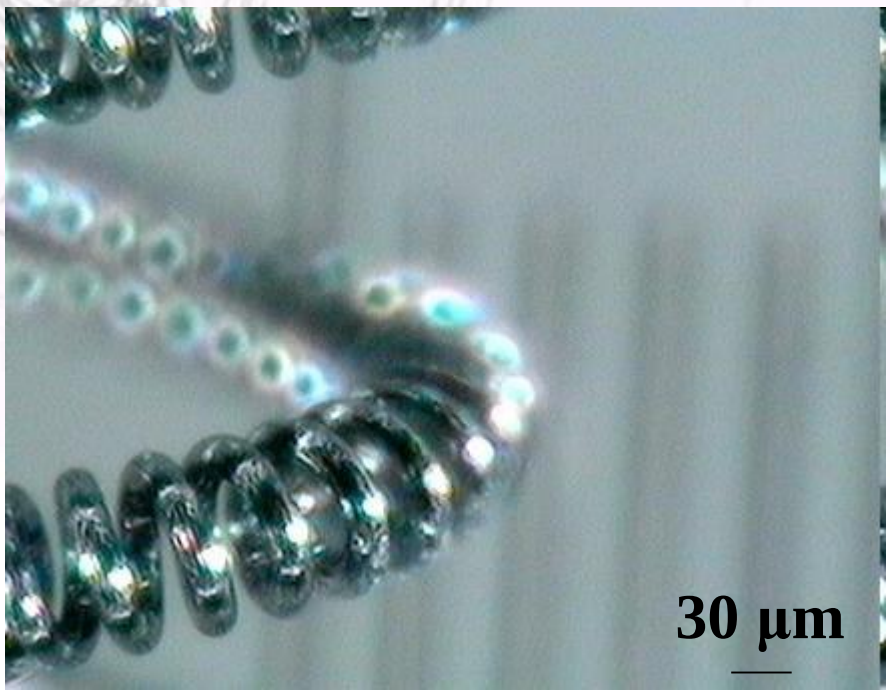
  Depth of field: **0.65 µm**
- **TEM** (100 kV, λ=0.0037 nm), : α ~0.01 rad → d~0.61λ/α² = 20 nm = **0.02µm**
- **SEM** (1 kV, λ=0.037 nm), WD=10 mm, Aperture diameter=200µm → α~0.01 rad → d~0.61λ/α² = 20 nm = **0.2 µm**
- **SEM not diffraction limited:** If we want the resolution to be similar to that of the optical microscope (δ=0.61 × 500 nm/0.65=470 nm), then we get:

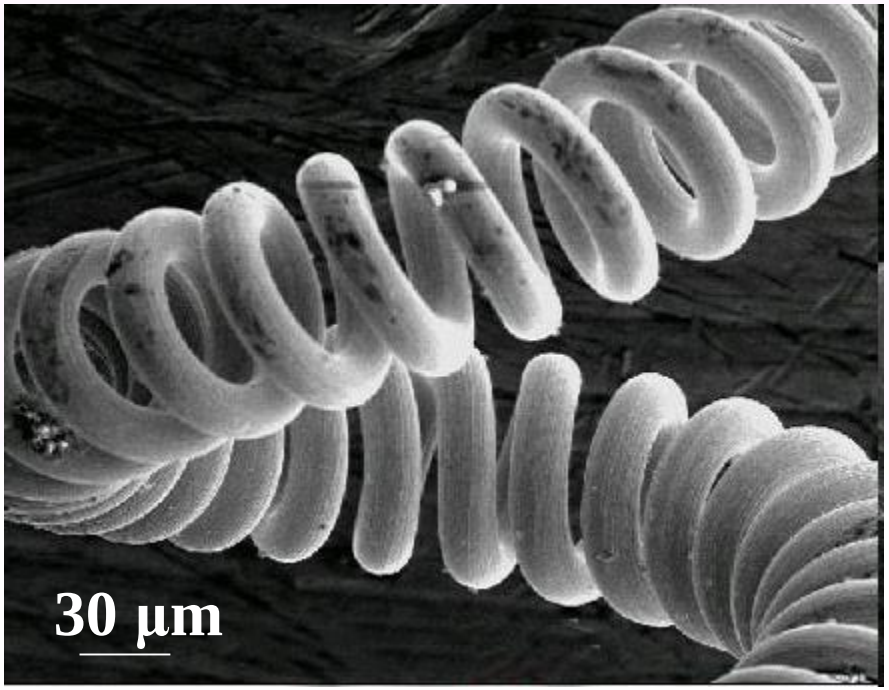
  d~δ/α= **65 µm**

SO YOU MAY WANT TO USE AN SEM EVEN THOUGH YOU ONLY NEED THE RESOLUTION OF AN OPTICAL MICROSCOPE – TO INCREASE THE DEPTH OF FIELD

d= δ/tanα (where δ is the resolution of the optical system)

=0.61λ/nsinαtanα=0.61λ/NAtanα, when the resolution, δ, is diffraction limited

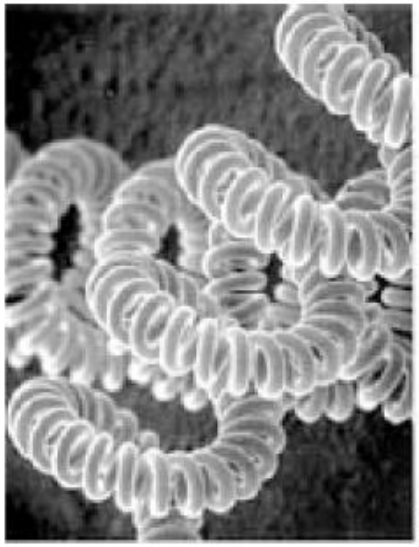


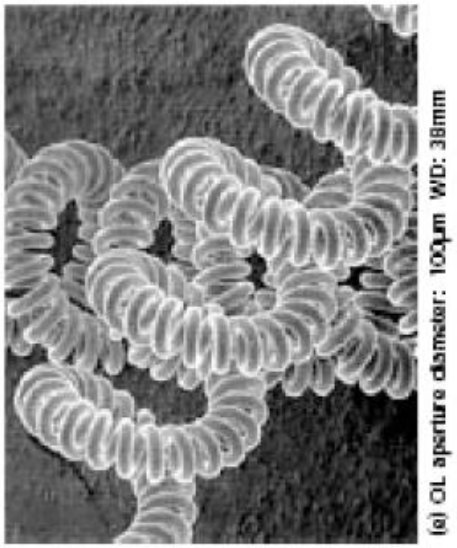


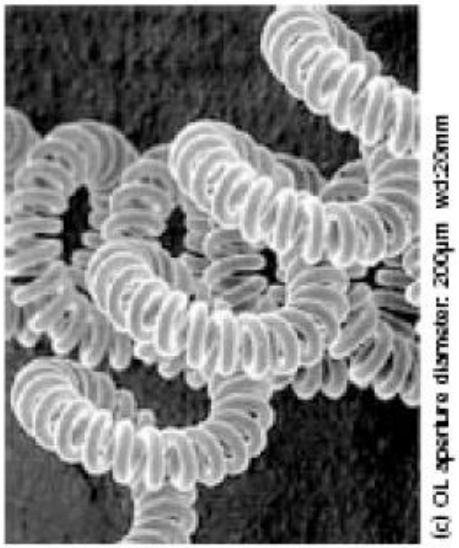
Tungsten Filament, imaged with Optical Microscopy and with SEM.

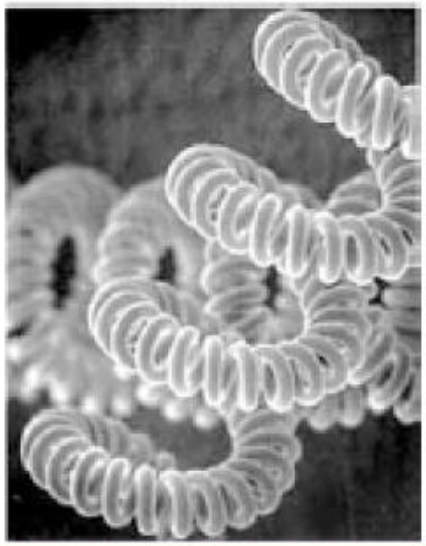
Image: WD and aperture

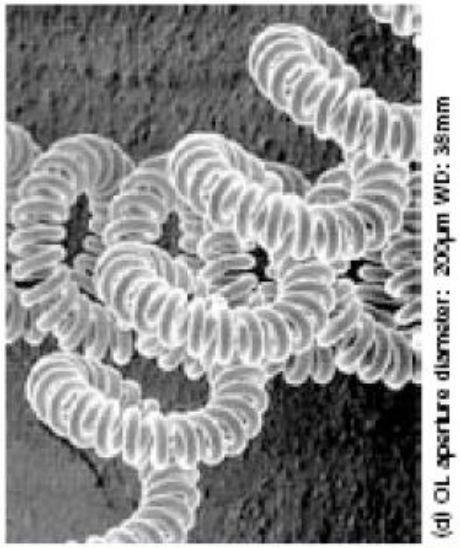
Images of Tungsten Filaments







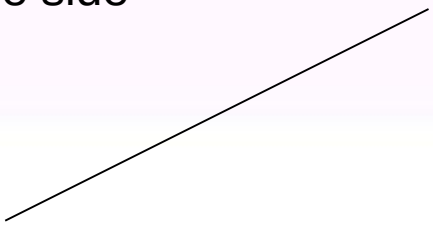




WD = Working Distance

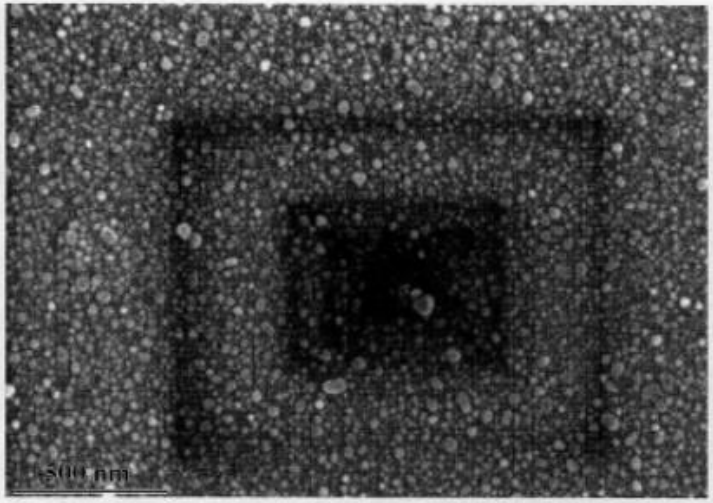
BUT REMEMBER NO TRUE 3 D INFORMATION:

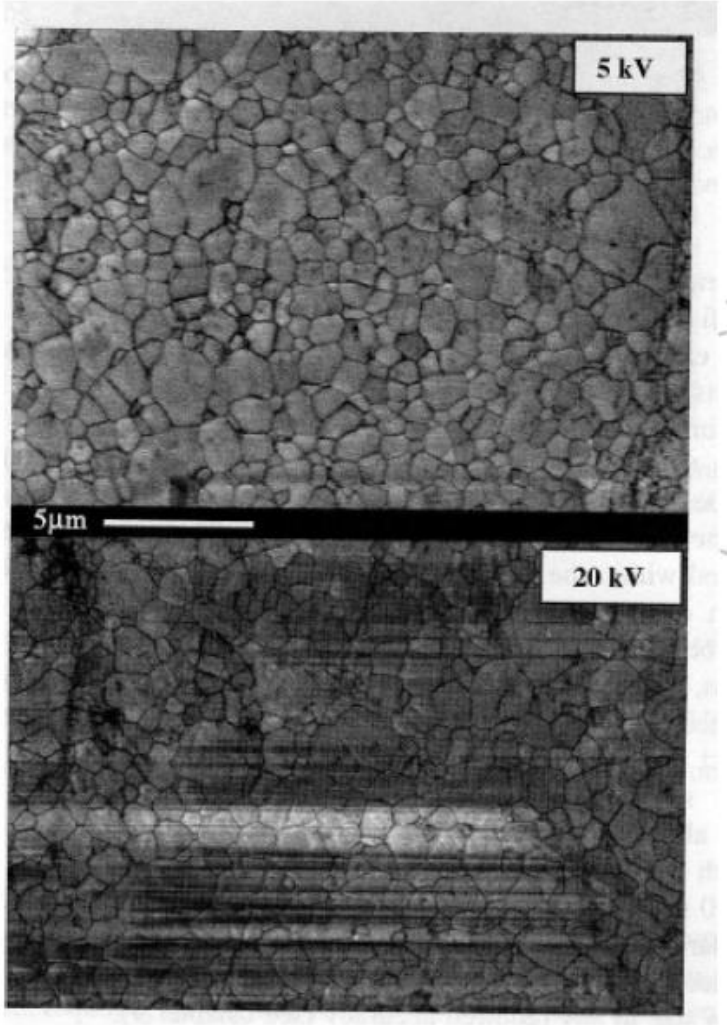
Feature seen from the side



Feature seen from above - it appears shareverywhere, but you do not know the true length

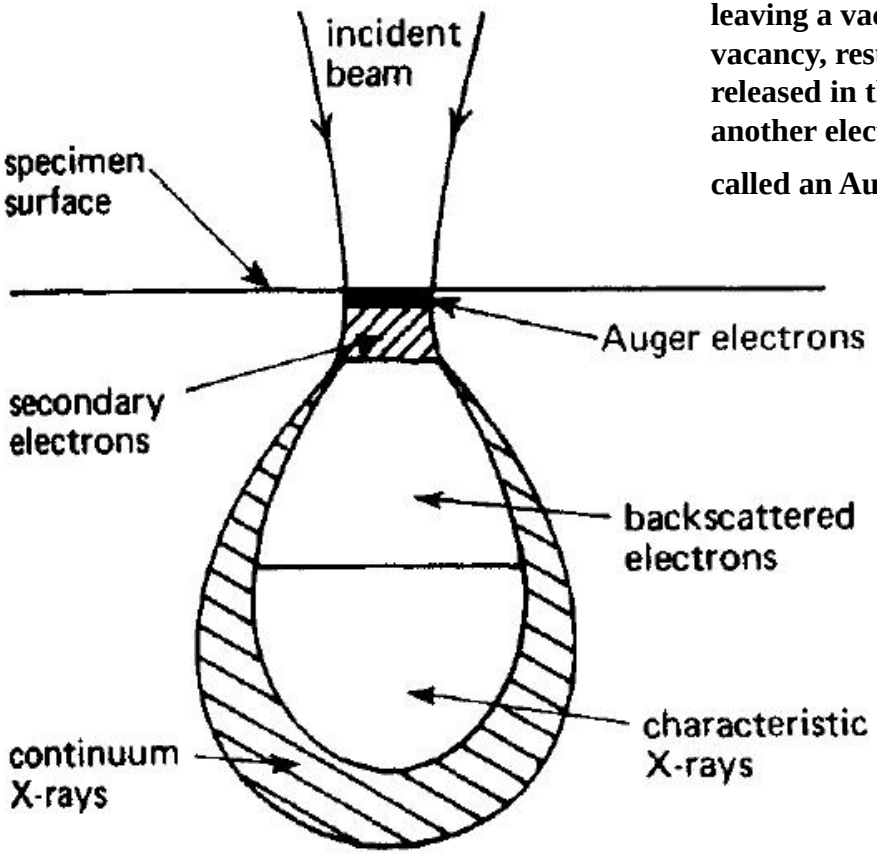
Charging, contamination and resolution





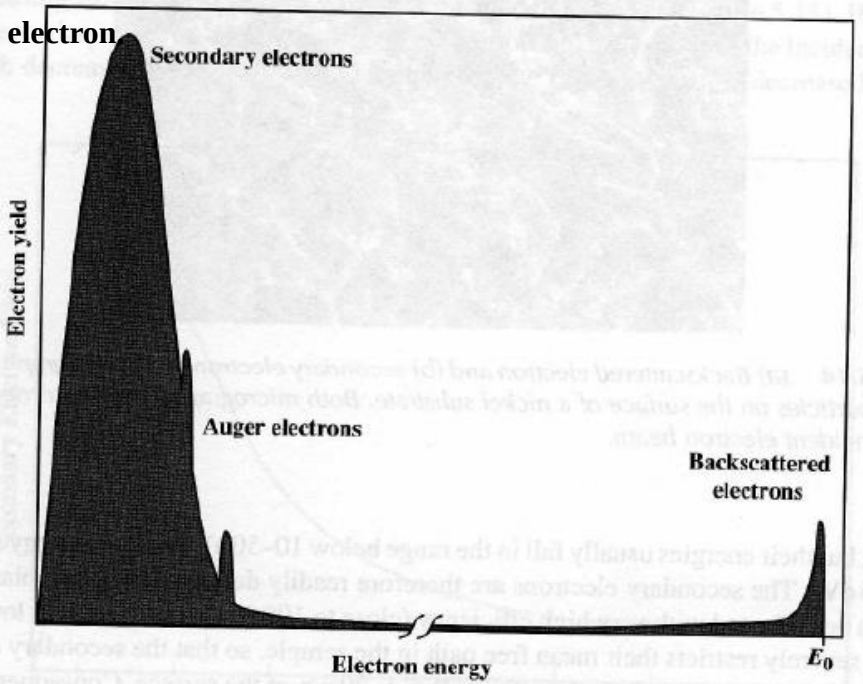
- Contamination (carbon) problem in long scans with high electron density
- None-conducting: use suitable coating (Au for imaging, C for analytical work) or lower the HT.

Interactions:



How does this affect resolution? Secondary electrons emitted from smallest volume, so gives the highest resolution (in principle it would be Auger electrons giving the highest resolution, but they are difficult to spearate from the secondary electron peak because the energy is roughly the same (see below).

Auger Electron: When an electron is removed from a core level of an atom, leaving a vacancy, an electron from a higher energy level may fall into the vacancy, resulting in a release of energy. Although sometimes this energy is released in the form of an emitted photon, the energy can also be transferred to another electron, which is ejected from the atom. This second ejected electron is called an Auger electron.



Elastic/inelastic scattering, Z dependence, thickness dependence, E_0 dependence, diffusion depth X_D and penetration depth X_R. Fluorescence

Secondary electrons

- SE: created (complex) + escape from surface region (1-20 nm, depending on mean free path L_s)
- Electron emission coefficient > 1
- E_{SE} = 10-50eV: small bias to collect with high efficiency
- SE_1: Created by primary electron interaction, from surface, resolution determined by probe size.

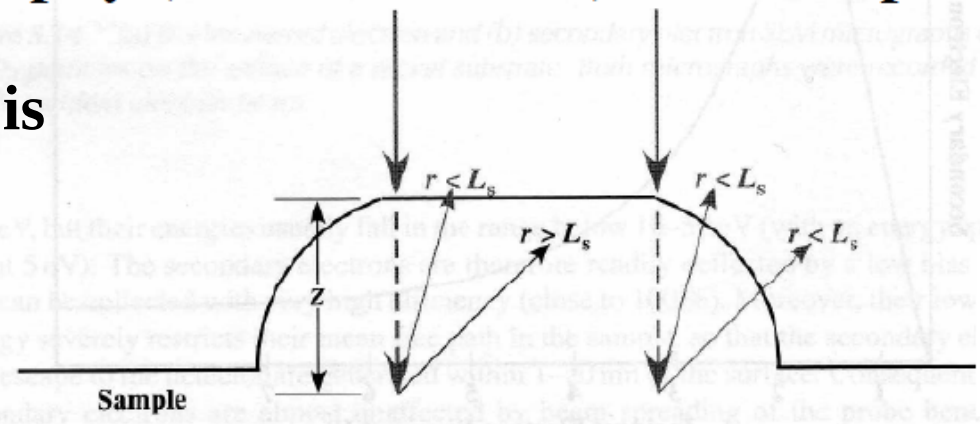
  SE_2: Created by BSE

  SE_3: Indirectly created, originating from column, not specimen

SE signal affected by:

1. Work function of surface: depending on composition and crystal orientation, few eV, affected by surface cleanness. Flat specimen only.
2. Beam energy (E_0) and beam current (I_{beam}): max. at low E_0
3. Density (Z): effect size is unclear: Z up results in more BSE, but also more inelastic and reduced x_D. Most pronounced at low E_0.
4. **Topography (local curvation): most important effect.**

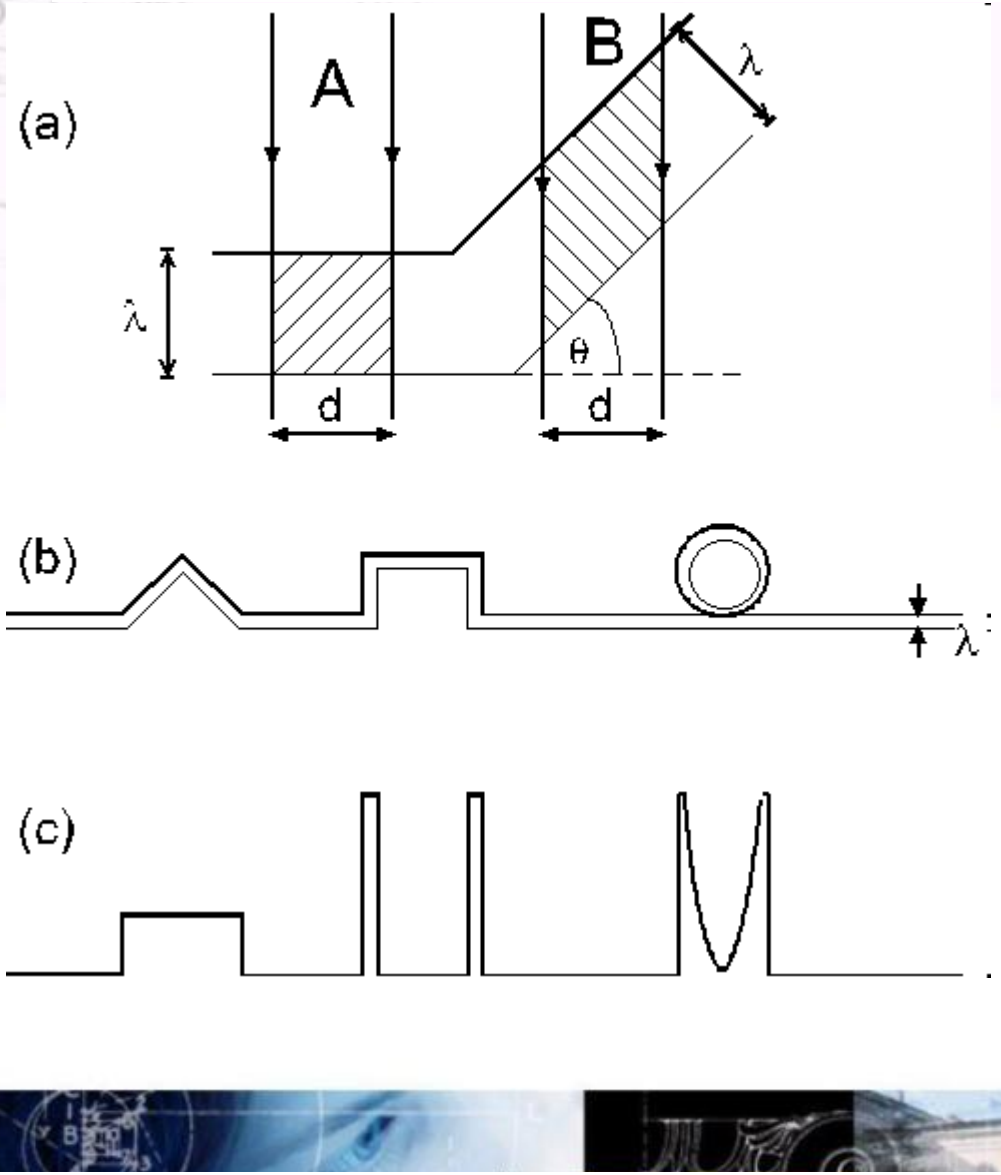
This is what is exploited.

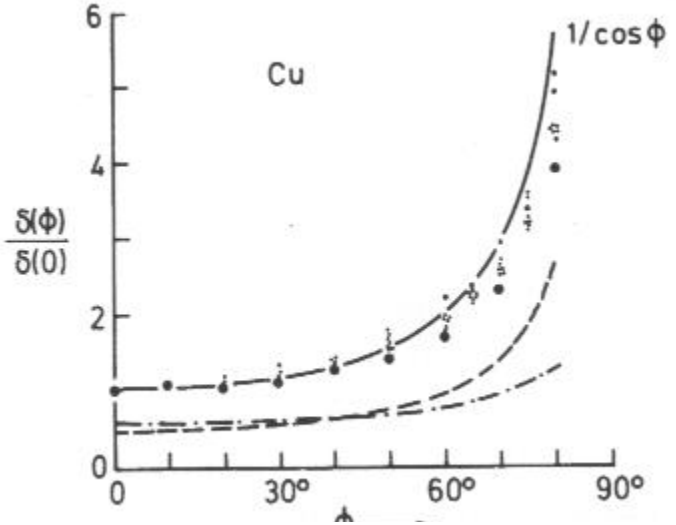


Secondary Electrons

- Because the energy of the Secondary electrons is relatively low (typically less than 50 eV), they can be extracted effectively by a detector with a small positive bias (up to about 200 V). The high energy back scattered electrons are not trapped.
- Secondary Electron Imaging is by far the most common imaging method in SEM.

Effect on Sample Tilt on SE Image





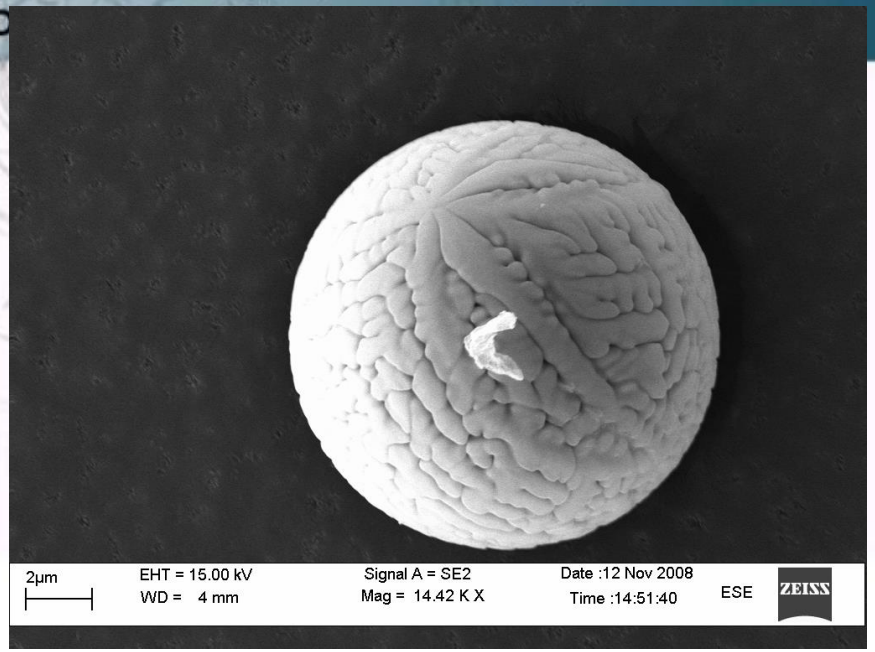
As the figure above shows the secondary-electron yield depends on the angle between the incoming primary electron and the surface. Its value is lowest for normal (perpendicular) incidence and increases with increasing angle between the primary beam and the surface-normal. The reason for this is illustrated in fig. a to the left, which shows a focused parallel beam of primary electrons incident at two locations on a specimen, where the surface is normal (at A) and inclined (at B) to the incident beam. The volume from which secondary electrons can escape is that which lies within the escape depth l of the surface, measured *perpendicular* to the surface. This escape volume, and therefore the SE yield d , is greater at point B.

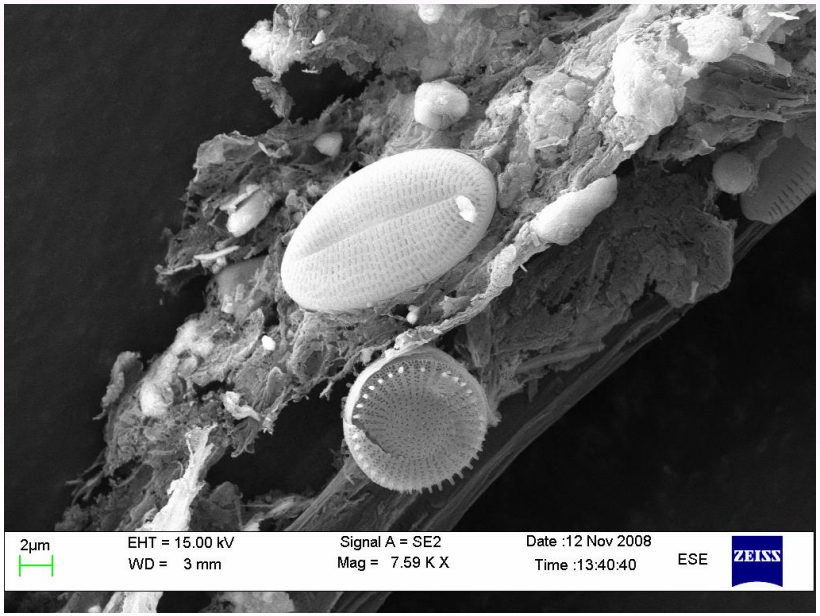
(b) Specimen surface containing triangular and square protrusions, a square-shaped well and a round particle.
(c) Corresponding secondary-electron signal (from a line-scan along the surface),

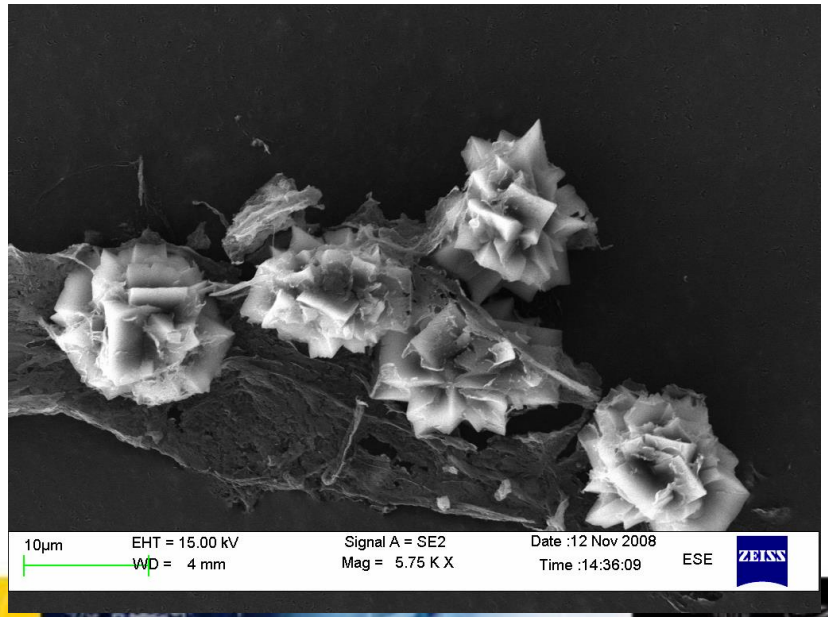
Effect of Detector Position on SE Image

SEM image of the ball from a ball-point pen; the SE detector is located towards the top of the image. The scale bar is of length 0.5 mm.

There is usually some asymmetry in an SE picture due to the fact that the SE detector is located to one side of the column rather than directly above. Surface features that are tilted *towards* the detector appear *particularly* bright because electrons emitted from these regions have a greater chance of being collected; It gives a characteristic three-dimensional appearance to the SE image, and makes the topographical contrast relatively easy to interpret, from analogy with a rough surface which is obliquely illuminated by light.

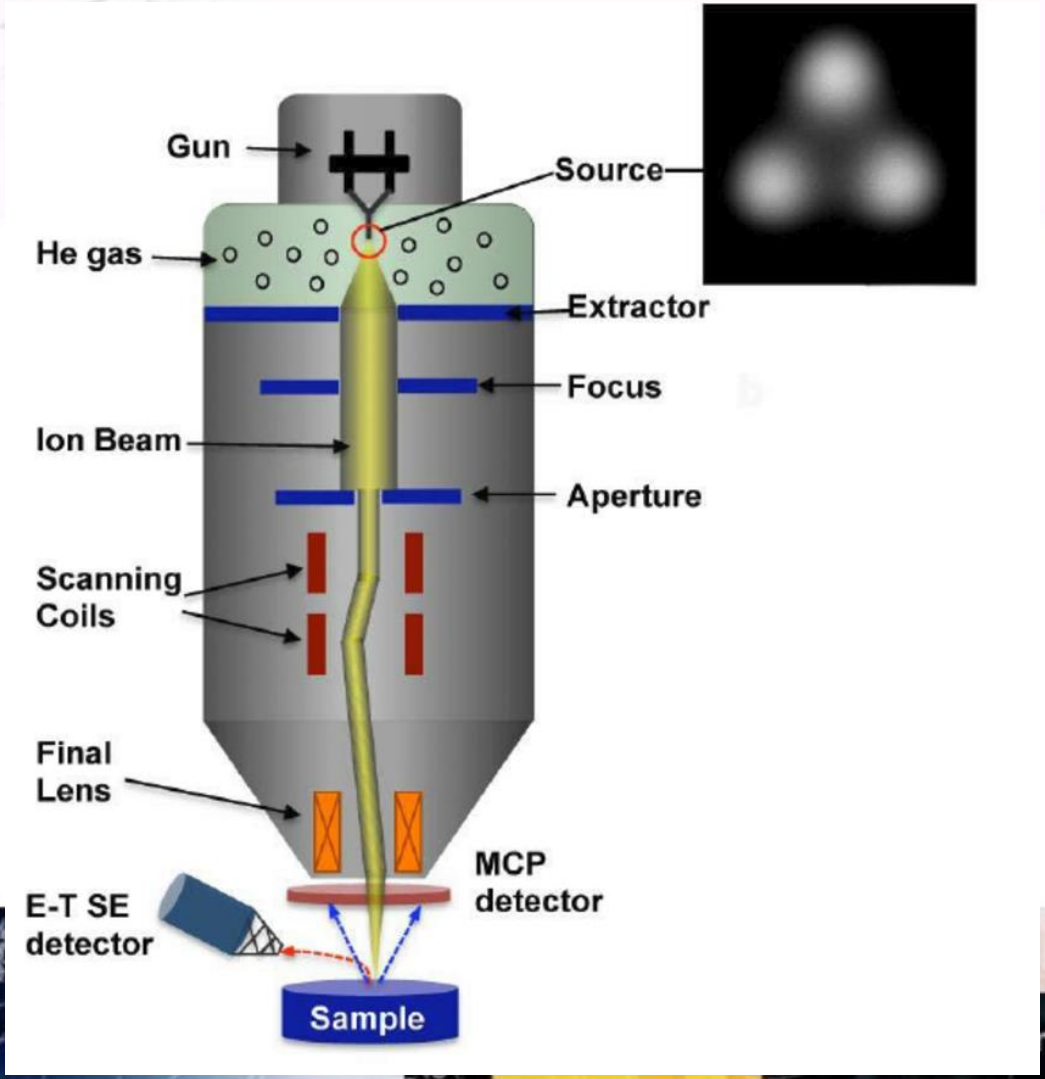






Images by Christian Bergfjord
Top left: Pollen
Top right: Algea (diatomes)
Botton left: Calcium oxalate crystals in a burnt nettle sample

Helium Ion microscopy – Since≈ 2010



Advantages:
Imaging also with secondary emitted electrons but lower penetration depth
Samples can be imaged without coating – positively charged helium ions neutralize the sample.
Large depth of field (even larger than electron beam microscoy.

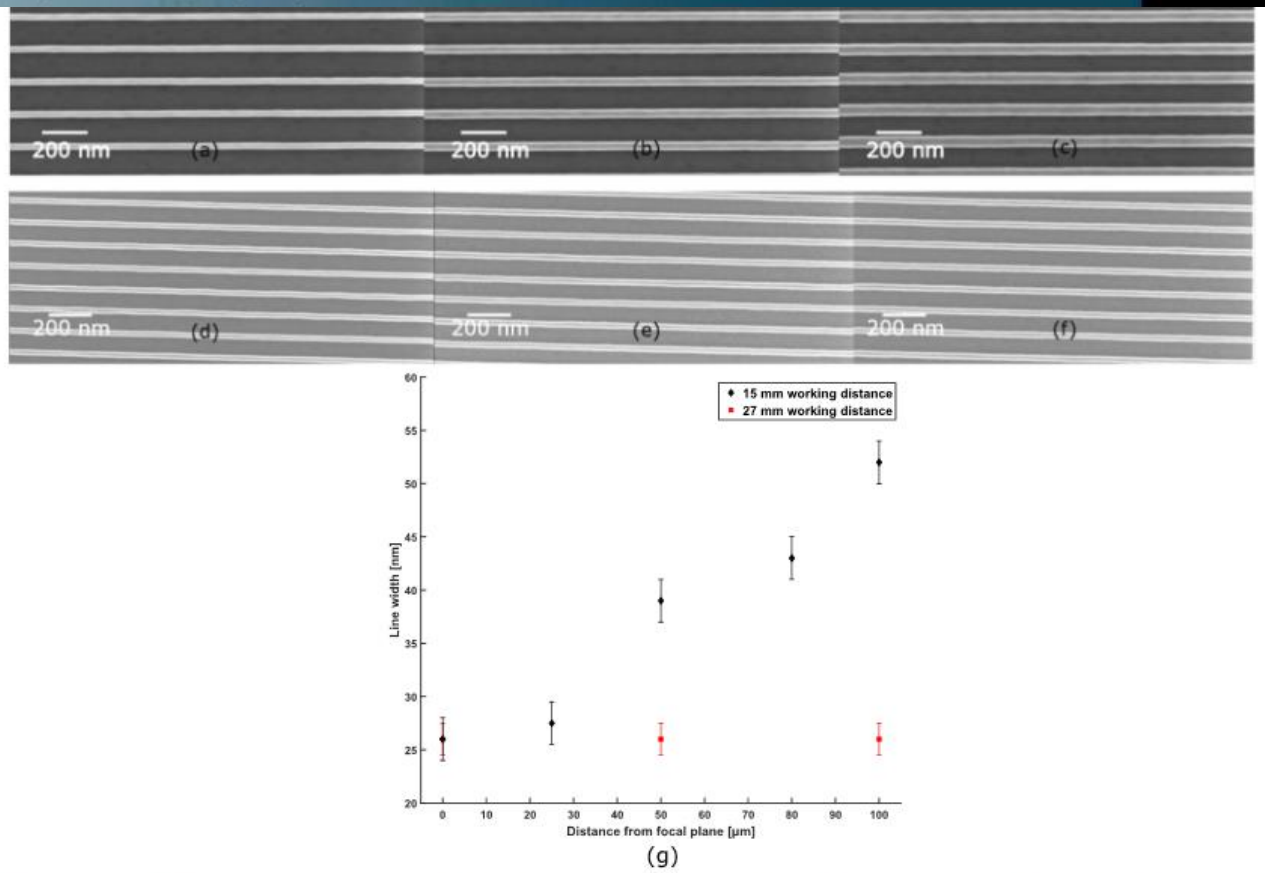


Figure 5. (a)–(f) SEM image along the diagonal of the 100 μm grating written in HSQ while having the sample tilted 45°. In (a)–(c) the working distance was 15 mm. (a) Lower left sample area linewidth 26 nm to (b) middle sample area linewidth 39 nm to (c) upper right sample area linewidth 52 nm. As the width of the lines has already changed noticeably for the middle sample area, the depth of field is smaller than 50 μm. (d) Lower left sample area (e) middle sample area (f) upper right sample area. The working distance used to exposure the line (d)–(f) was 27 mm. The width of the lines remains the same across the hole patterned area, 26 $\pm$ 1.5 nm, and therefore the depth of field for this resolution must be greater than 100 μm. (g) Plot of the linewidth versus the distance from the focal plane for a working distance of 15 mm (black) and 27 mm (red).

Exploring proximity effects and large depth of field in helium ion beam lithography: large-area dense patterns and tilted surface exposure

To cite this article: Ranveig Flatabø *et al* 2018 *Nanotechnology* **29** 275301

Why use Helium to Investigate Materials?

- Lower energy for a given wavelength than most other "particle" probes

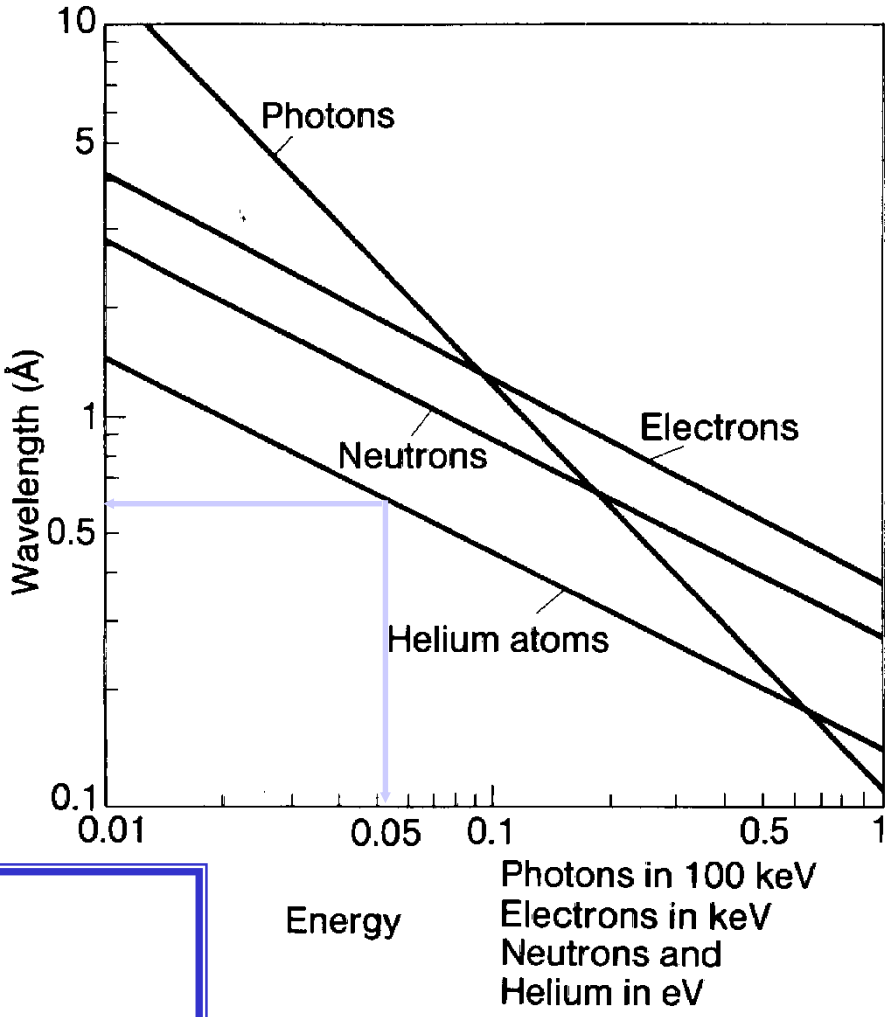
  $\lambda = h/mv$ (de Broglie matter wave)

$\lambda \sim 0.6$ Å => E ~60 meV

Atoms are uncharged and unreactive

No penetration into the material:

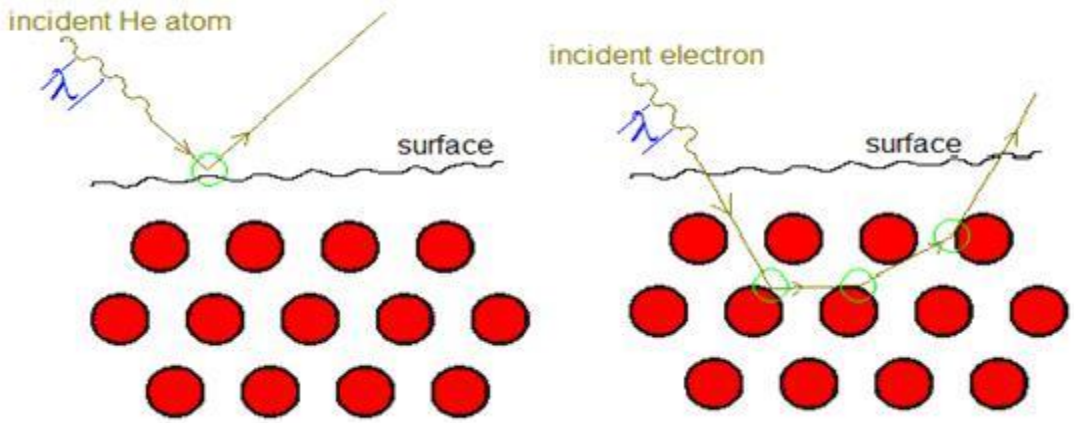
He-atoms probe the surface electronic structure



No sample damage even for fragile, insulating materials.

Surface structures and porous materials can be investigated

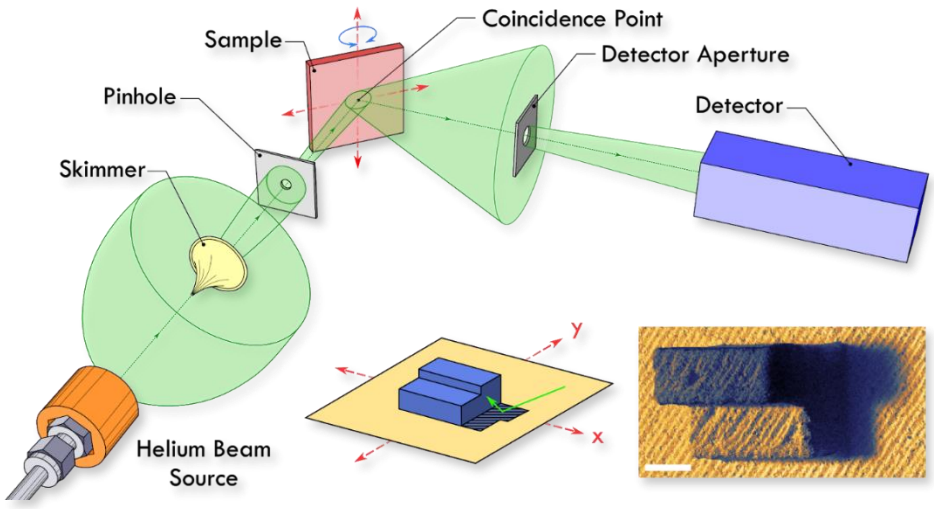
Strictly Surface Sensitive



Current Instruments

Pinhole apertured SCANNING NEUTRAL He MICROSCOPE

Idea: apertured He- pencil beam scans in a 90° beam line setup over the sample surface.



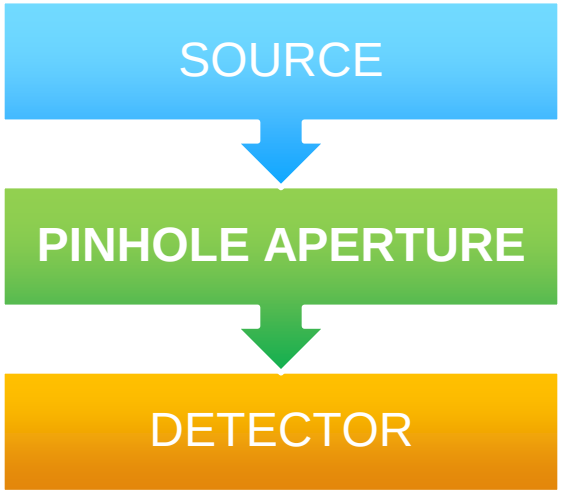
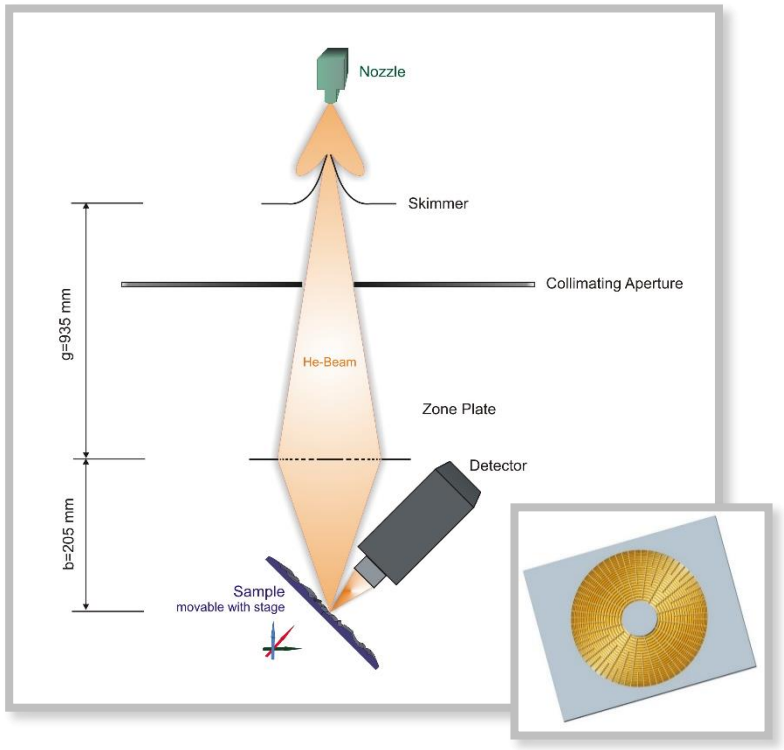


Image taken from 'Taxonomy through the lens of neutral helium microscopy' S.D. Eder, T.A. Myles, M.G.Barr, A. Fahy, J. Martens & P.C. Dastoor. Nature Scientific Reports **9:2148**, 2019

Current Instruments

Focused - zone plate SCANNING NEUTRAL He MICROSCOPE

Idea: a focused neutral He- beam scans over the sample surface in reflection mode.



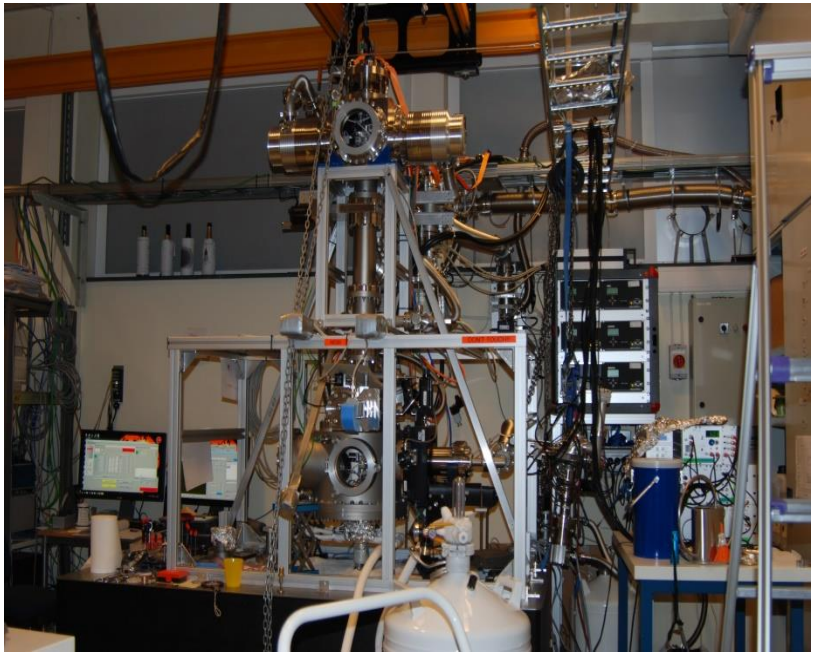
SOURCE

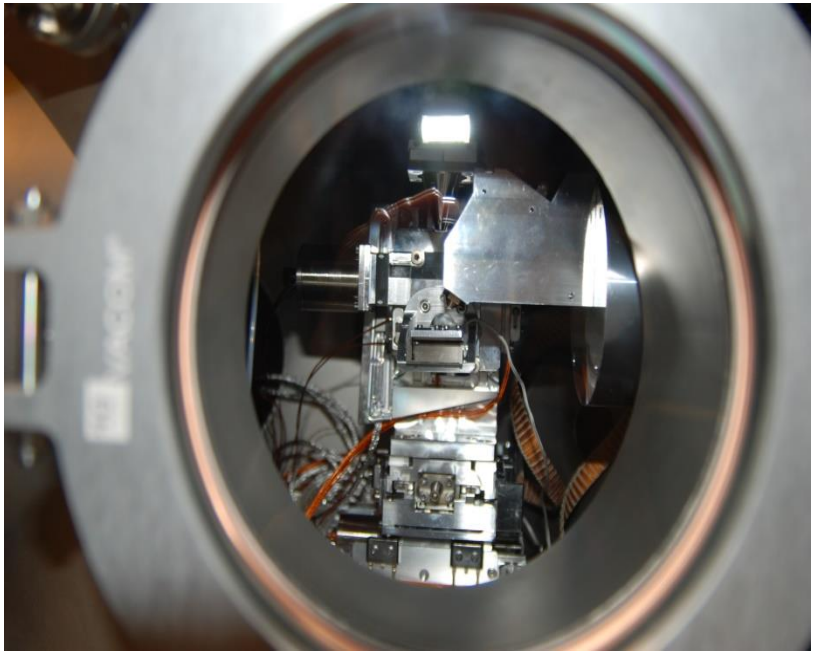
FOCUSING ELEMENT (ZP)

DETECTOR

Real Life

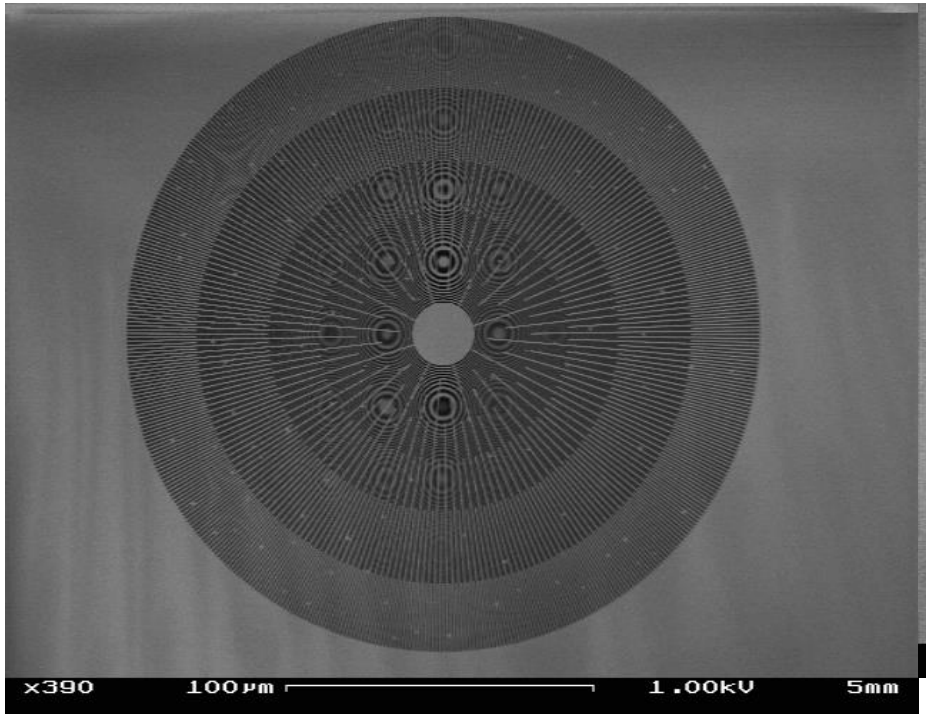
Current Nemi Setup Pictures

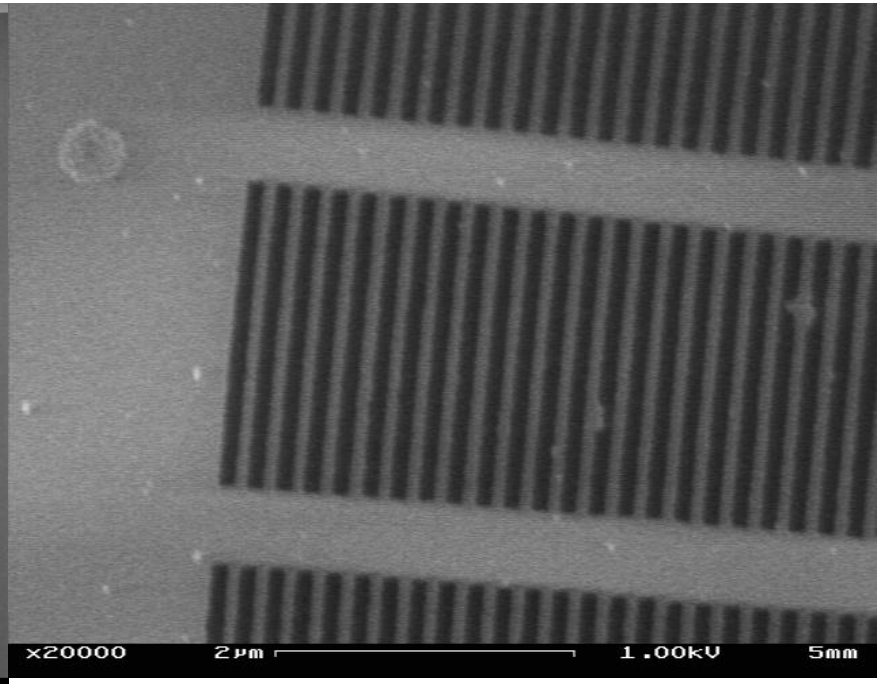




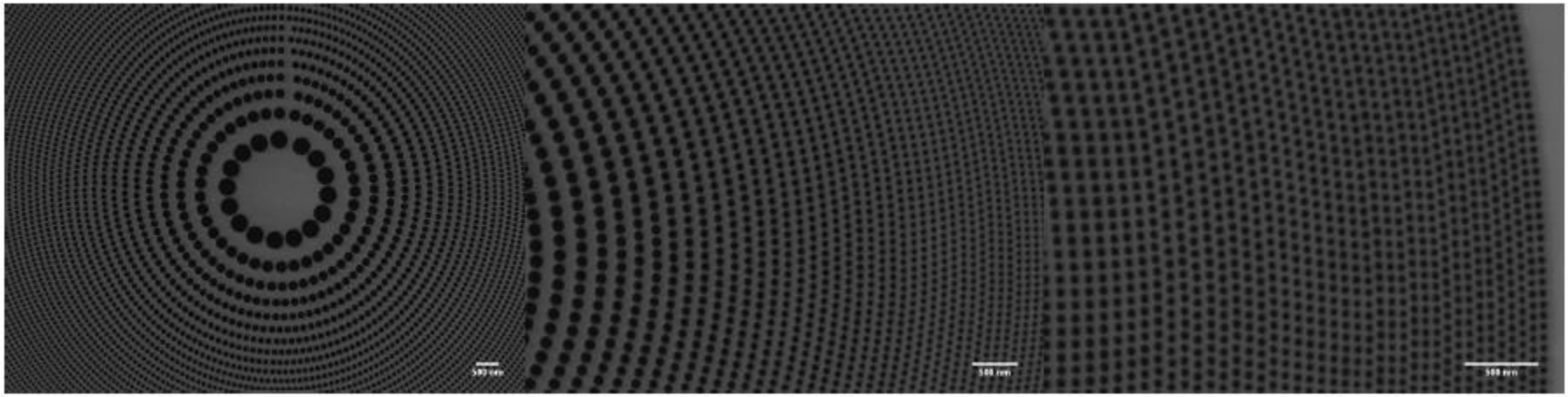
Zone Plates for atom focussing

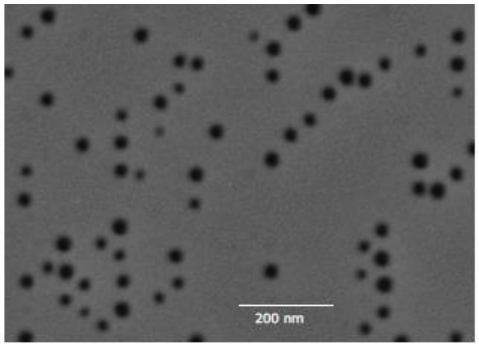
Outermost zone: 46 nm, 360 micron diameter





The Atom Sieve





Free-standing holes with a diameter ranging from 25 nm to 15 nm.

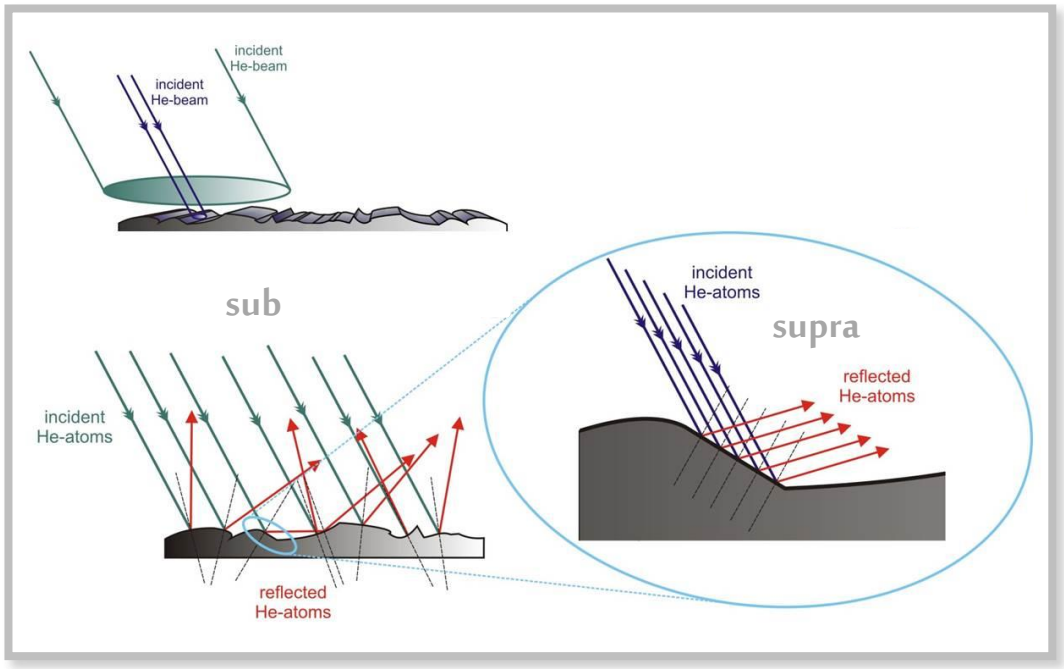
Inspired by the photon sieve developed by Lutz Kipp et al. Our sieve has 47011 holes with a diameter varying from 376 down to 15 nm.

S. D. Eder, X. Guo, T. Kaltenbacher, M. M. Greve, M. Kalläne, L. Kipp, B. Holst, Focusing of a neutral helium beam with a photon-sieve structure, Phys Rev A 91 043608 (2015)

R. Flatabø, M. M. Greve, S. D. Eder, M. Kalläne, A. Salvador Palau, K. K. Berggren, B. Holst, Atom sieve for nanometer resolution neutral helium microscopy, J. Vac. Sci T. B, 35 06G502 (2017)

Contrast in Helium Microscopy

TWO variants OF contrast



1. Supra - resolution

Surface topography of features bigger or equal to He-beam diameter

1. Sub - resolution

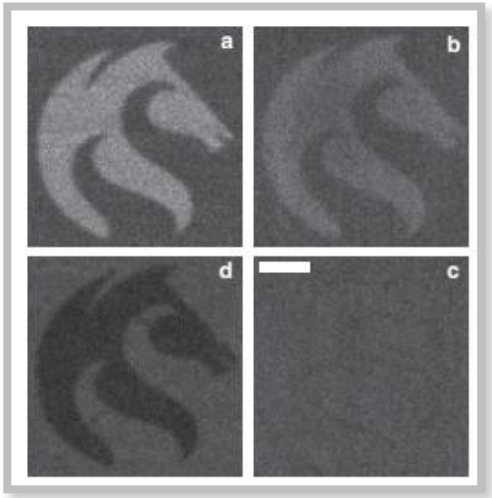
Surface roughness features much smaller than He-beam diameter.

Topological Contrast

Sub resolution

Gold

Nickel



Chromium

Platinum

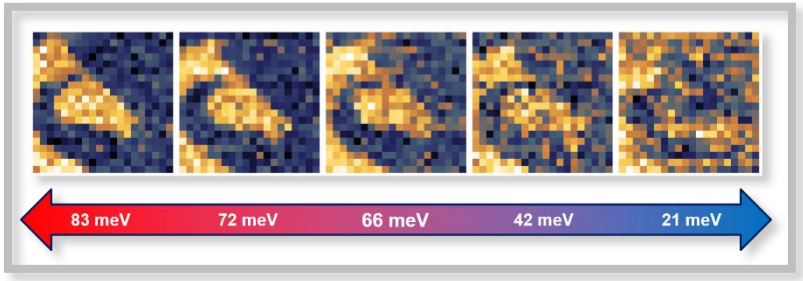
SHeM micrographs of four different metal layers (40 nm thick) laid down on a silicon substrate. Scale bar is 50 um.

Beam cooling

Cooling of the stagnation volume allows for control of the energy of the helium probe particle.

Beam energy

Reducing the beam energy (longer wavelength) resulted in a loss of contrast.

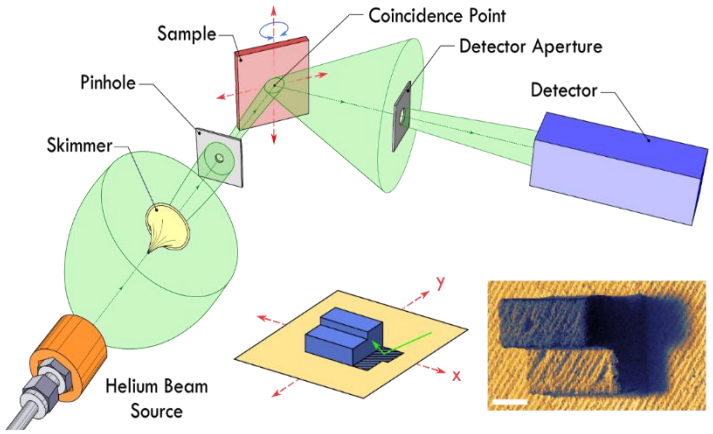


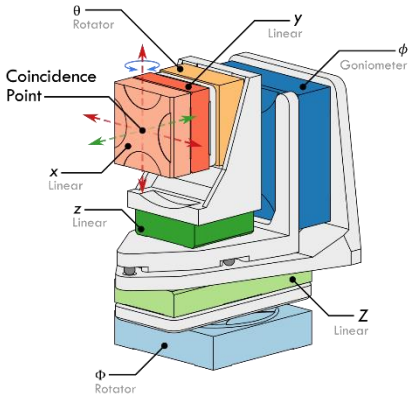
'Unlocking new contrast in a scanning helium microscope' M. Barr, A. Fahy, J. Martens, A.P. Jardine, D.J. Ward, J. Ellis, W. Allison & P.C. Dastoor. Nature Communications **7,** 10189 **(2016)**

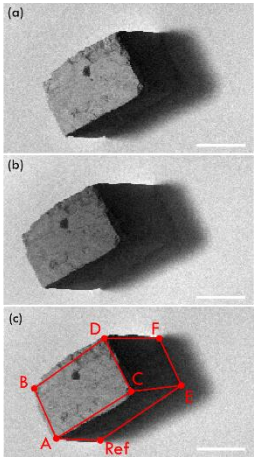
Topological Contrast

Supra - resolution scattering

Stereophotogrammetry on the SHeM allows us to also extract height information !



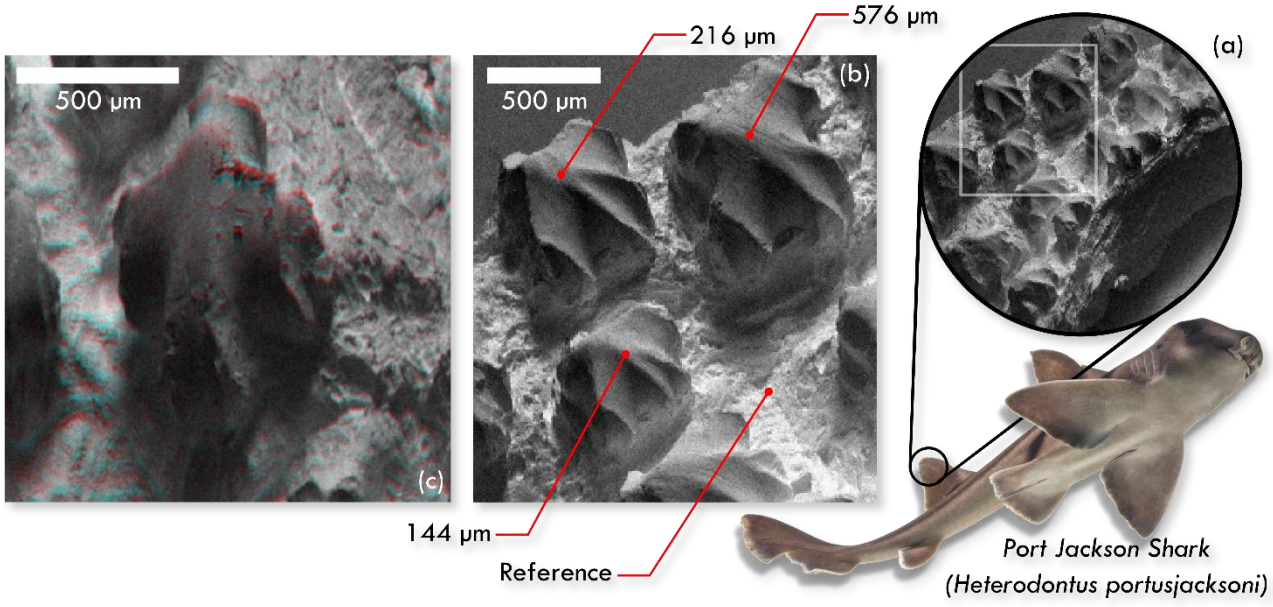




'Taxonomy through the lens of neutral helium microscopy' S.D. Eder, T.A. Myles, M.G.Barr, A. Fahy, J. Martens & P.C. Dastoor. Scientific Reports **9:2148**, 2019

Topological Contrast – True 3D contrast

Supra - resolution scattering



3D SHeM technique

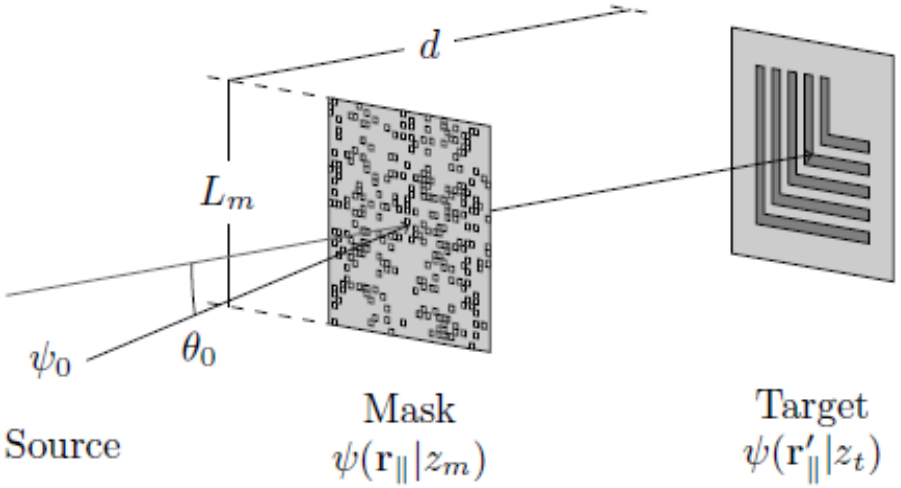
Quantitative taxonomical classification through stereophotogrammetry imaging with the apertured neutral helium microscope.

'Taxonomy through the lens of neutral helium microscopy' S.D. Eder, T.A. Myles, M.G.Barr, A. Fahy, J. Martens & P.C. Dastoor. Scientific Reports **9:2148**, 2019

Dom Eigler Revisited: Mask Based Matter Wave Lithography

Imaging and writing goes hand in hand.

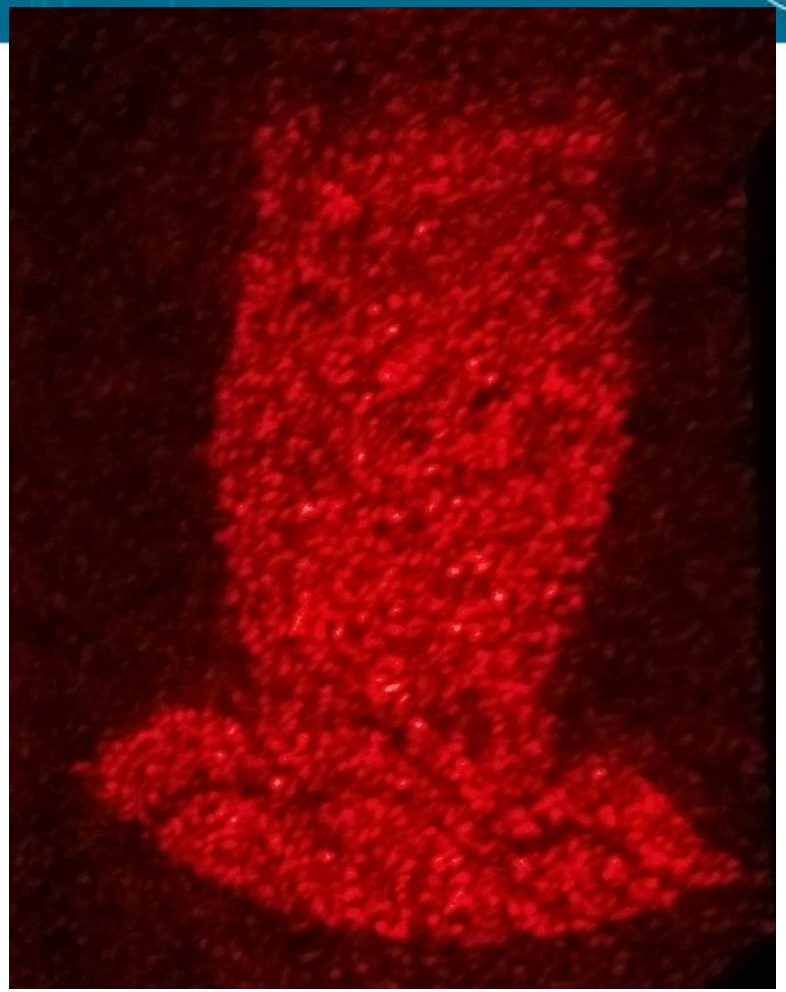
Idea – Use porous samples like the beryl you saw before as 2D template for near field binary holography lithography with metastable helium atoms



Advantage: Wavelength 0.1 nm, means patterns to the nanoscale can in principle be easily made. Present state of the art lithography uses a 193 nm wavelength light source. Proposed EUV is 13.5 nm – fundamental resolution limit around 6-8 nm due to problem with resist.

Binary holography – demonstrated with metastable neon in the far field (mm resolution) M. Yasuda, S. Matsui and F. Shimizu, Nature 380, 691 (1996)

Nanometer-Resolution Mask Lithography with Matter Waves: Near-Field Binary Holography Torstein Nesse, Ingve Simonsen, and Bodil Holst, Phys. Rev. Applied 11, 024009 (2019)



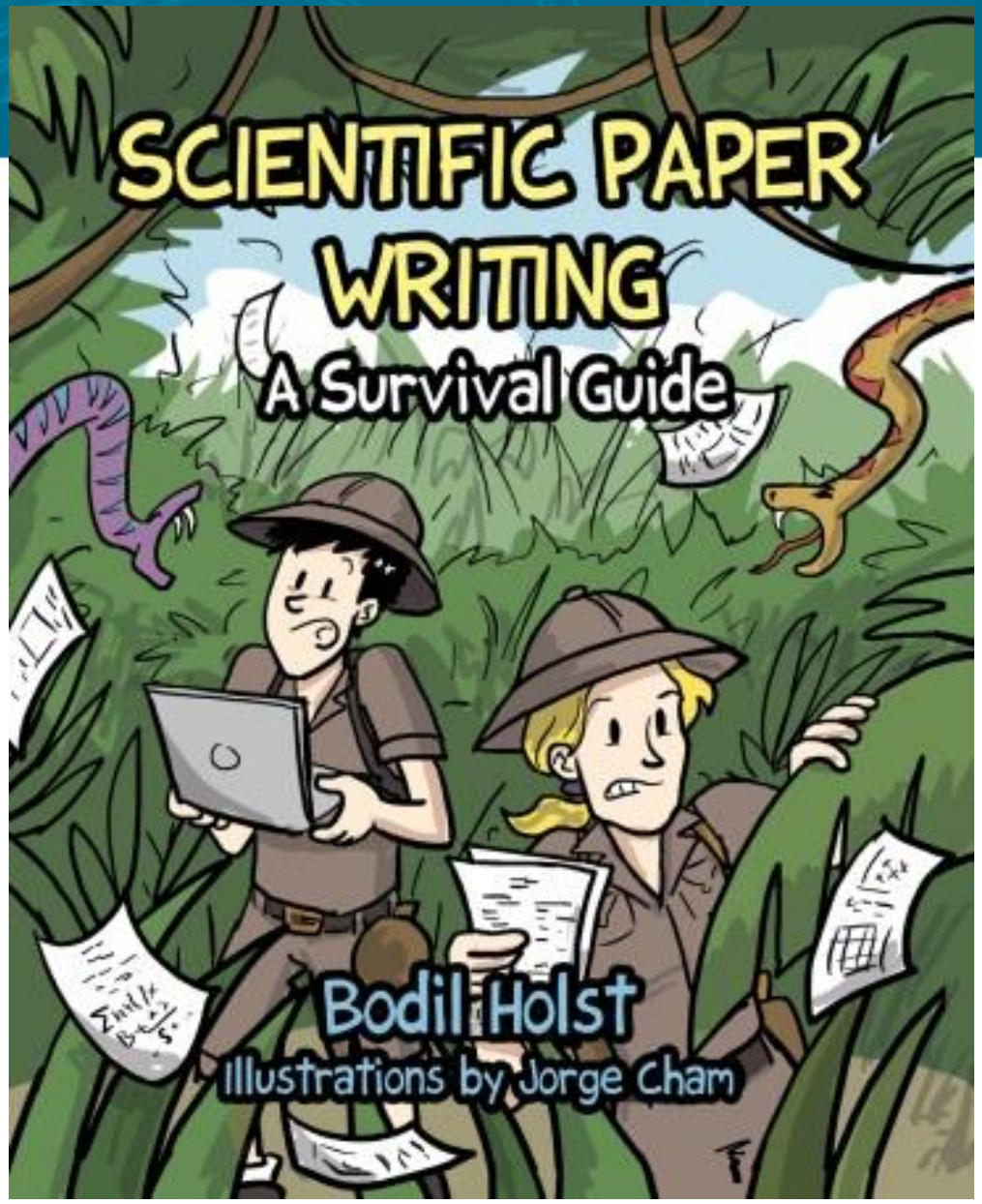
Thanks to
Gianangelo Bracco
and Torstein Nesse

We just got the news that we have received ≈3.5 Million Euro from the EU for The Future and Emerging Technology project Nanolace to demonstrate this Idea in praxis. Project is coordinated by UiB

Dot over the i = 100 nm diameter

Acknowledgements

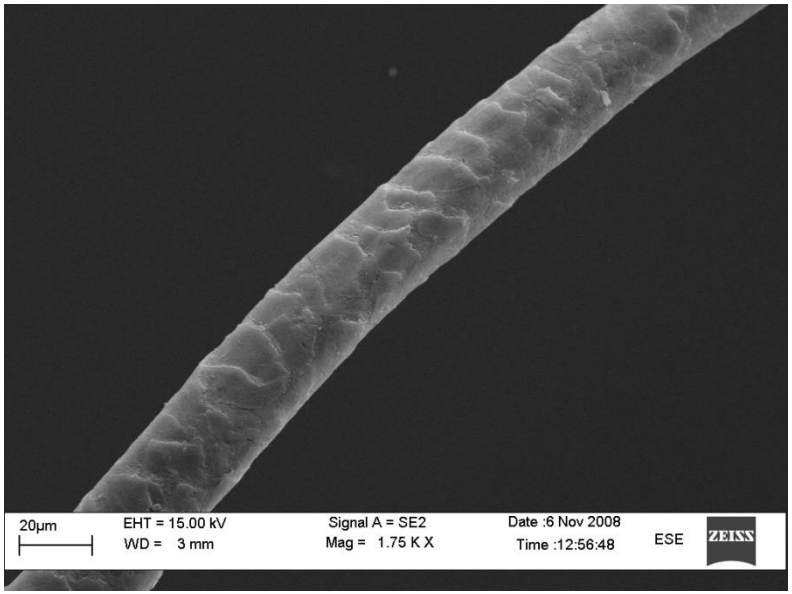
S. Eder, B. Samelin, Ranveig Flatab (UiB)
P. E. Vullum, A. T. J. van Helvoort *NORTEM
Ingve Simonsen, Torstein Nesse *NTNU
Karl Berggren MIT
NFR
European Commission Project NEMI



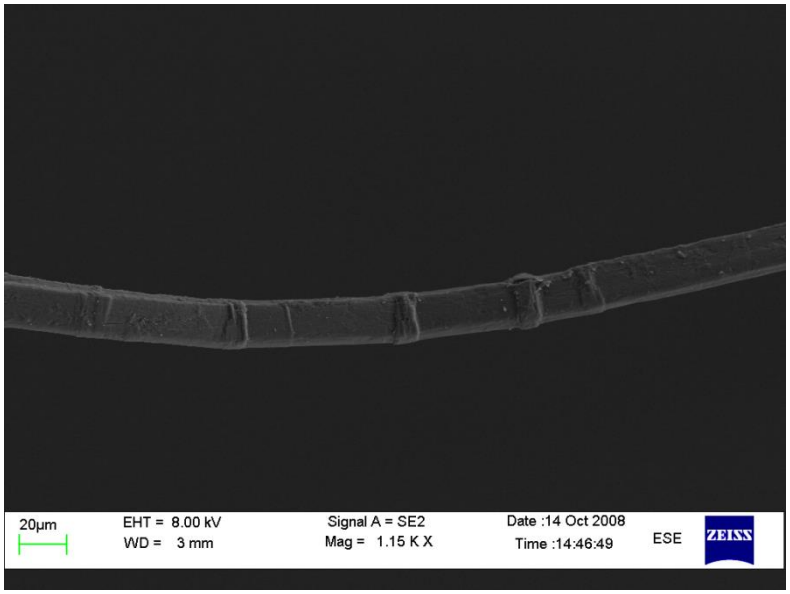
Available on Amazon, Adlibris and bokus, See review on Chemistry World, Royal Society of Chemistry

Plant Fibre Identification with Microscopy

- Distinguishing between animal and plant fibres is normally relatively easy.

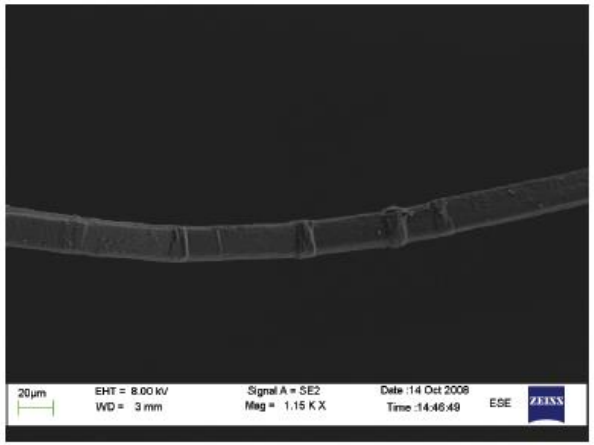


Wool

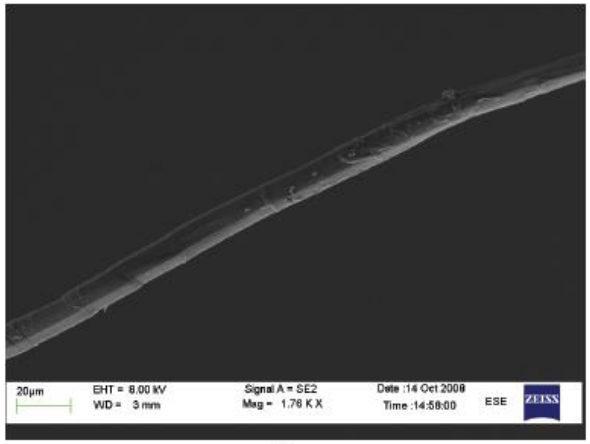


Flax

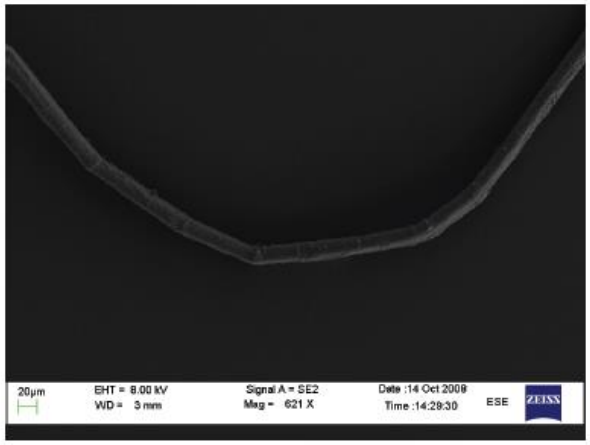
Distinguishing between different bast fibres is much more difficult, in particular if only small amount of material is available.



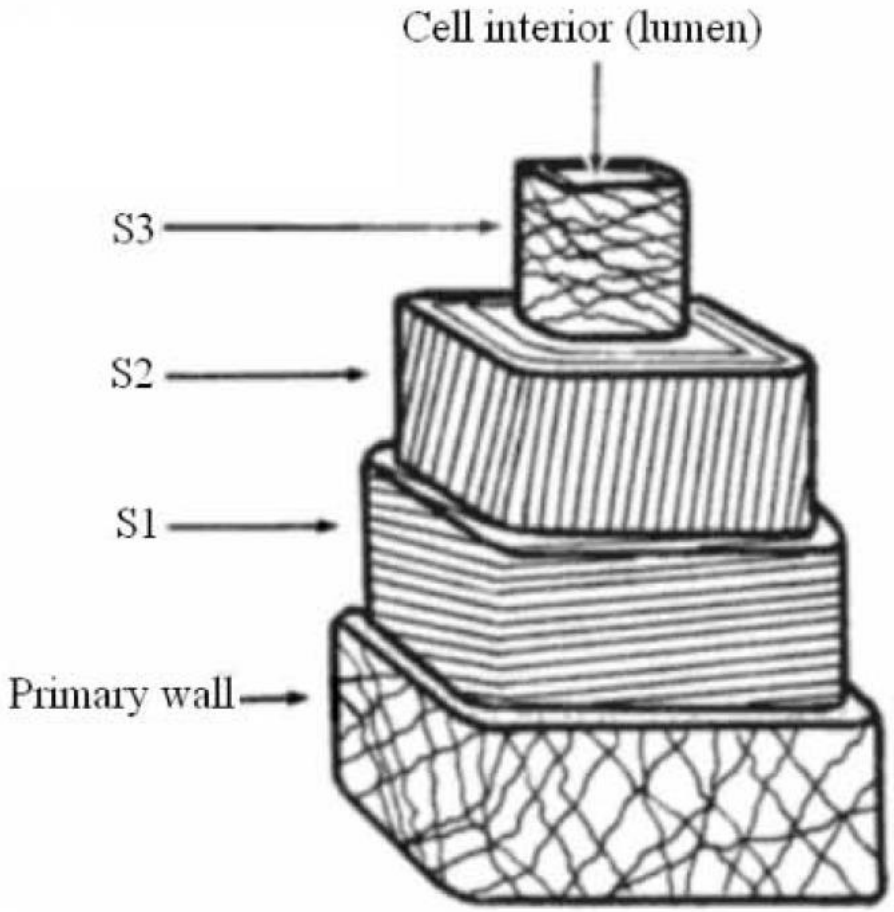
Flax



Hemp



Nettle



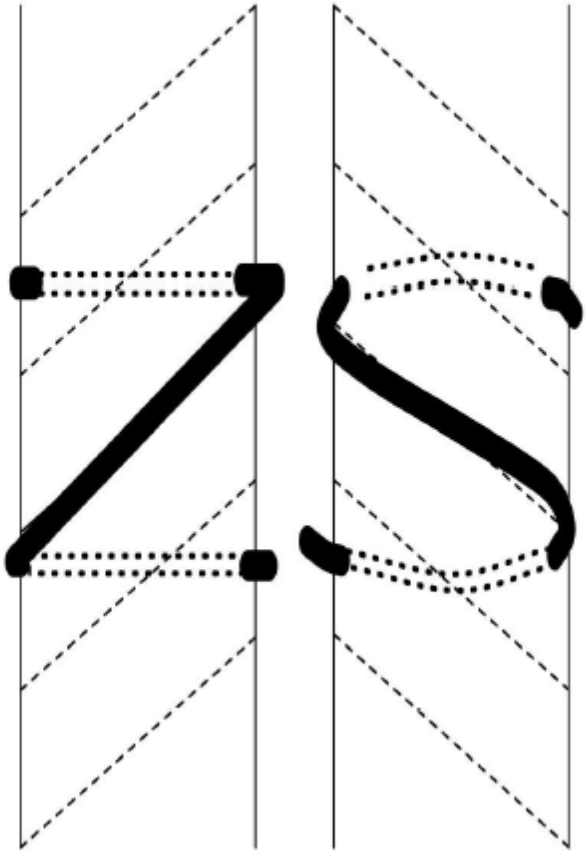
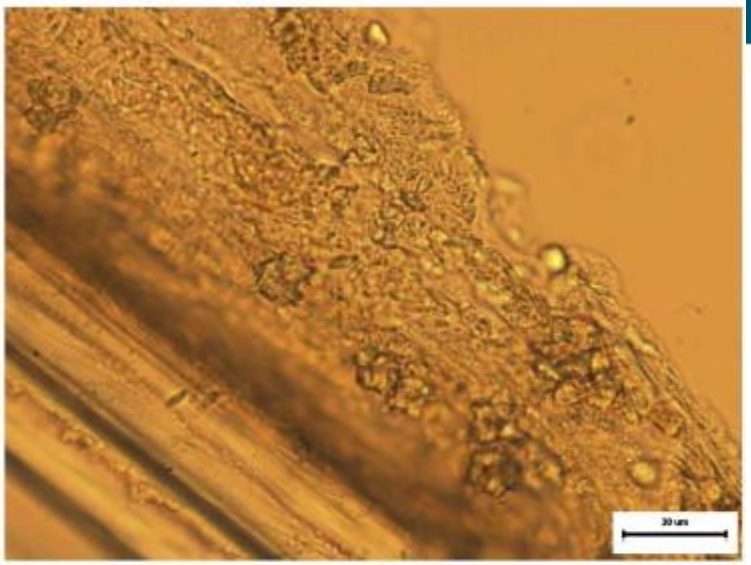
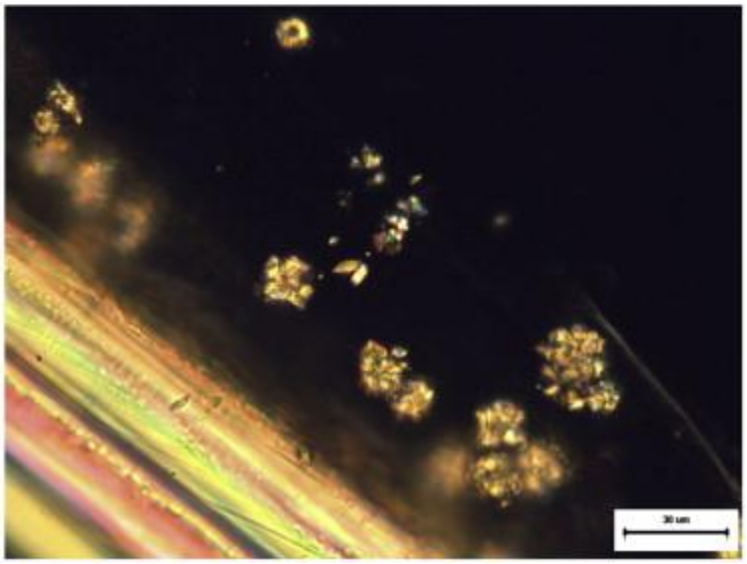


Fig. 1. Diagram of a typical textile bast fibre cell showing the fibrillar orientations of the sublayers. S2 is here shown with Z-twist. Edited from Burgess (1985).

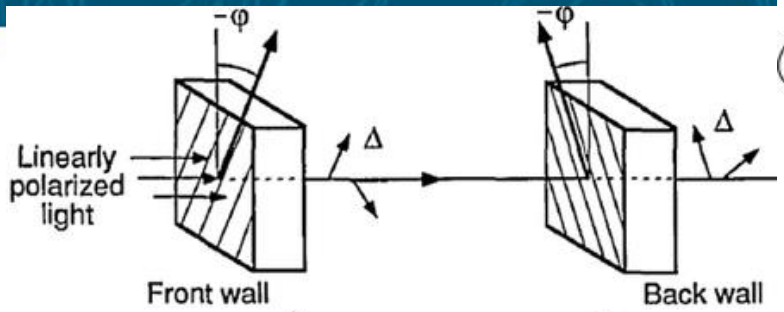
a

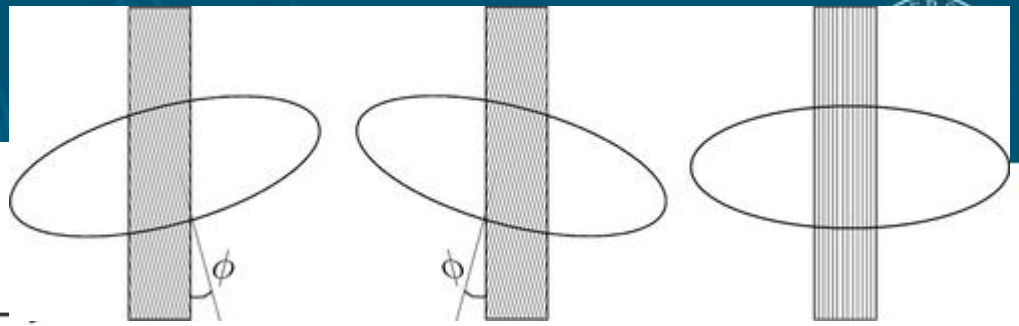


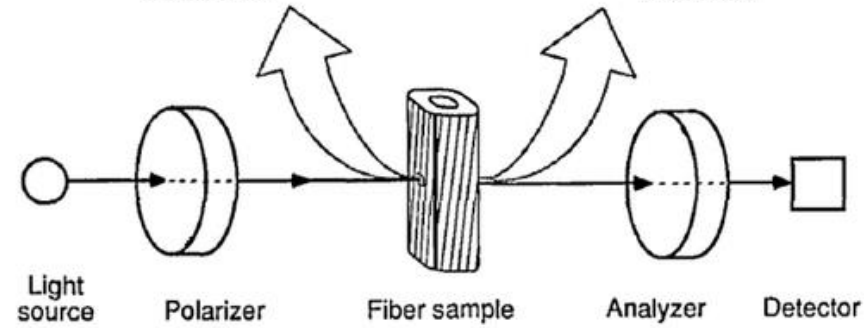
b



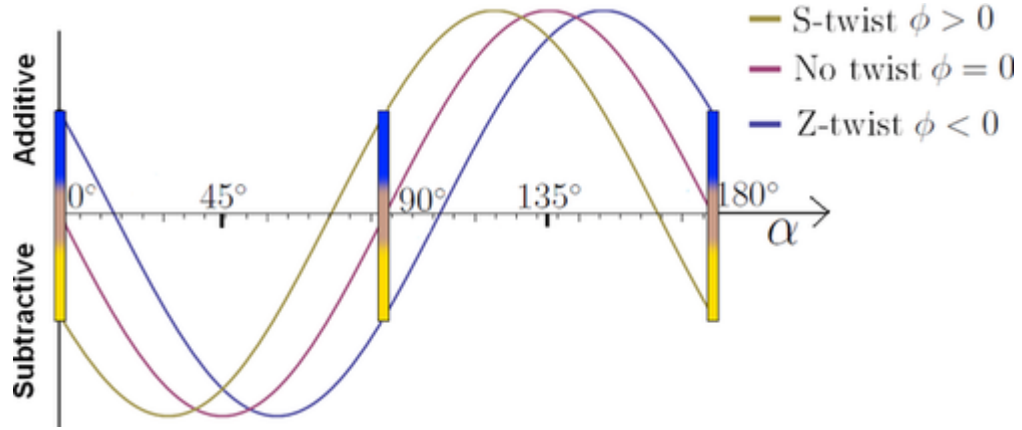
Calcium Oxalate Crystals are
Present in nettle and not in flax







E. Haugan, B. Holst: Determining the fibrillar
Orientation of bast fibres with polarized light microscopy: the modified Herzog test (red plate test) explained
Journal of Microscopy, 252, 159-168 (2013)



By inserting a so called full wave compensator, the phase shift is turned into a colour change (designed to introduce a fixed amount of retardation between the orthogonal wavefronts) - giving an interference pattern.

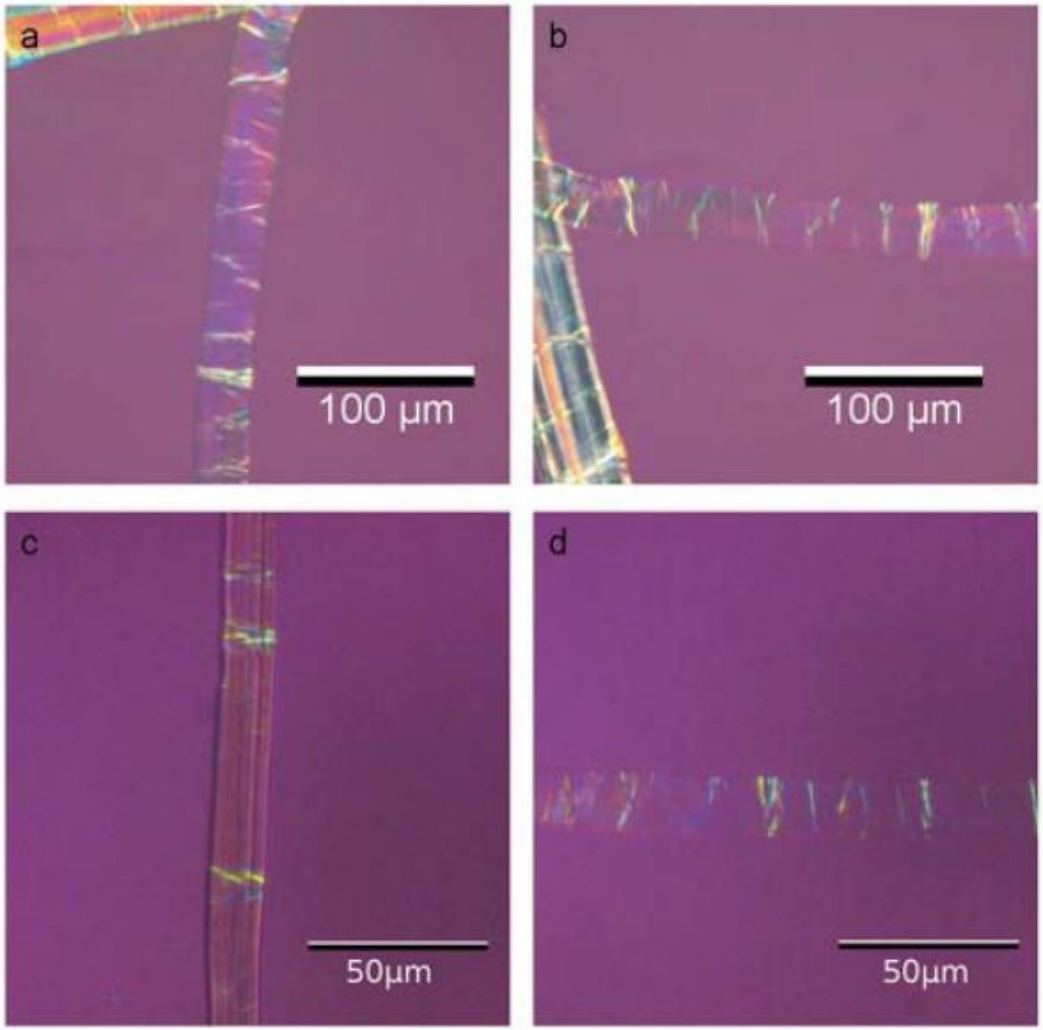


Figure 6 | The modified Herzog Test performed on the Överhogdal Viking wall-hanging piece Ia. The two top pictures (a and b) show a Z-twist fibre (Hemp) and the two bottom pictures (c and d) show a S-twist fibre (Flax). The analyzer is oriented vertically.

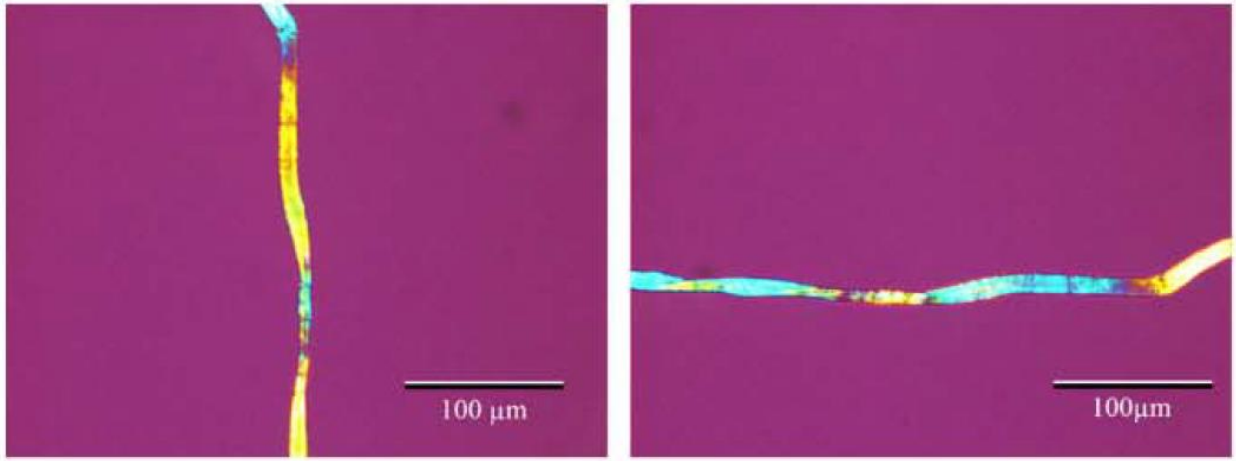


Fig. 13. The modified Herzog test performed on cotton. Cotton is not a bast fibre with well-defined fibrillar orientation hence it will normally not be possible to bring it to extinction and it will show a rapid colour change along the fibre, regardless of the orientation angle. In some cases it may appear as if there is no colour change at all with orientation angle.

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Journal of Microscopy

www.journalofmicroscopy.org

Volume 252, Issue 2, November 2013

- Often the tendency has been simply to assume, based on a rather superficial examination that a sample was made of flax.
- <u>This still happens even in one of the worlds best scientific journals!!!!!!</u>
- The paper claims (among others) that they have identified flax fibers. They have done no such thing!

30,000-Year-Old Wild Flax Fibers

Eliso Kvavadze,[1] Ofer Bar-Yosef,[2*] Anna Belfer-Cohen,[3] Elisabetta Boaretto,[4] Nino Jakeli,[5] Zinovi Matskevich,[2] Tengiz Meshveliani[5]

Here, we report an identification of wild flax fibers from a series of Upper Paleolithic layers at Dzudzuana Cave, Georgia (*1*, *2*), indicating that prehistoric hunter-gatherers were making cords for hafting stone tools, weaving

We found the flax fibers during analyses of 86 clay samples of 50 g each collected from five locations within the excavated area in 2007 and 2008 (table S2). The clay deposits are rich in carbonate and produced large amounts of nonpollen paly-

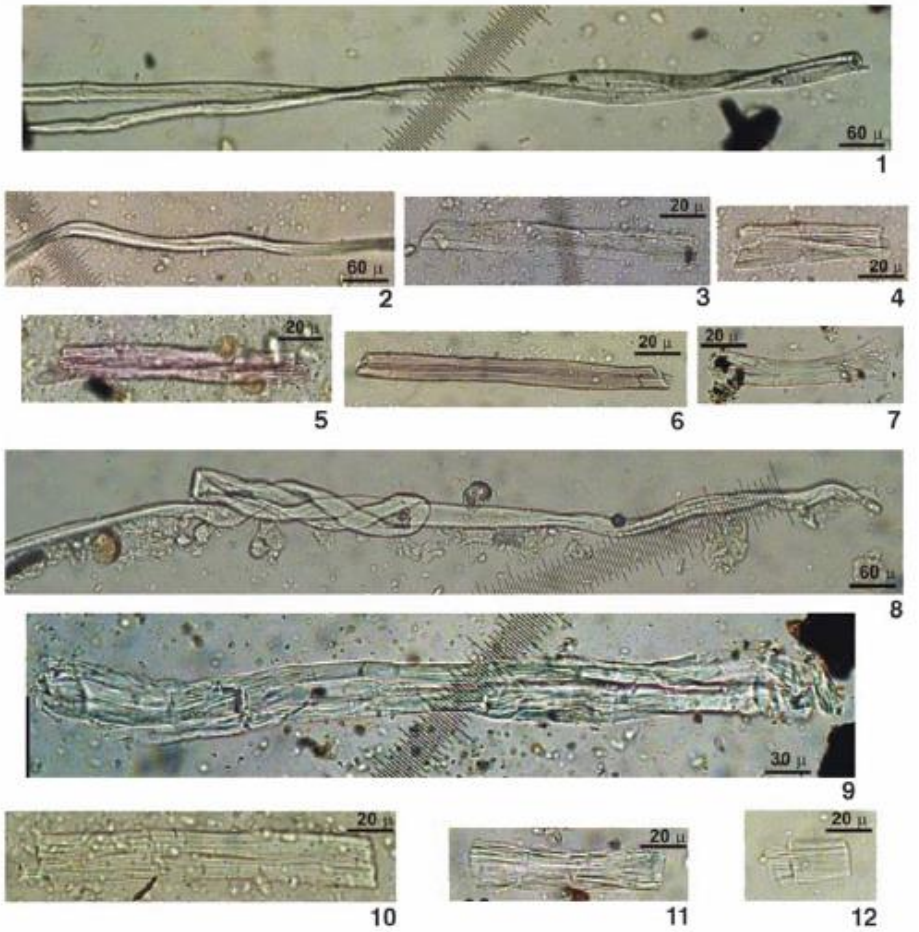


Fig. 1. (**1** to **7**) Fibers from Dzudzuana, Georgia, unit D. 1, twisted flax fibers; 2 to 4, flax fibers; and 5 to 7, unraveled flax fibers. (**8** to **12**) Fibers from Dzudzuana, unit C. 8 and 9, twisted flax fibers; 10 and 12, flax fibers; and 11, dyed flax fibers.

SCIENCE VOL 325 11 SEPTEMBER 2009

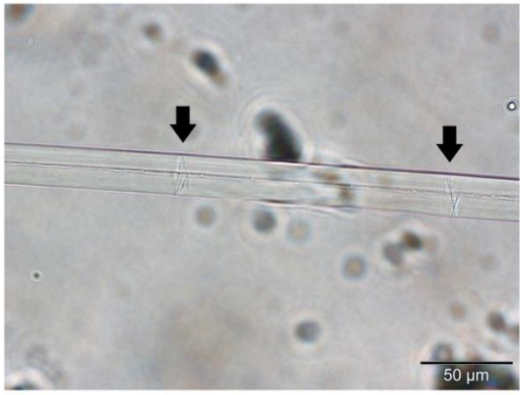
This figure is taken from:

Is It Really Flax? On The Identification of Bast Fibers

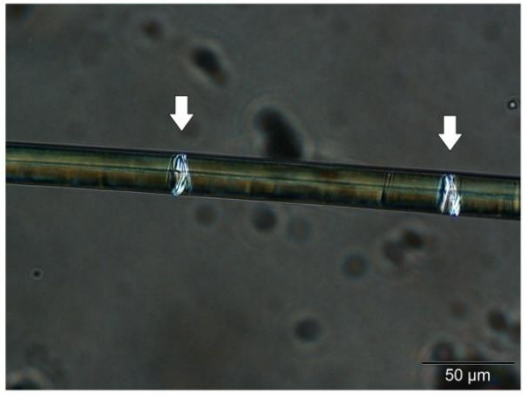
”

C. Bergfjord, S. Karg, A. Rast-Eicher, M-L. Nosch, U. Mannering, R. G. Allaby, B. M. Murphy and B. Holst·

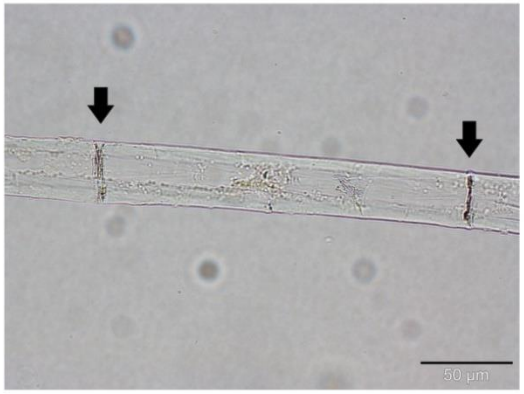
Technical Comment,
Science, **328**, 1634-b (2010)



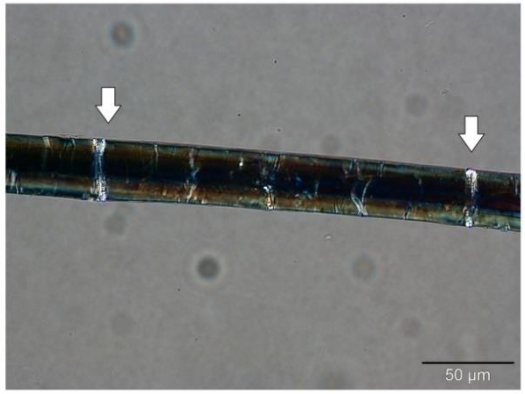
Flax, White light microscopy



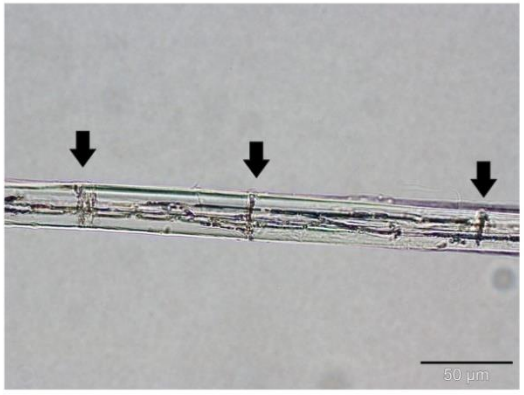
Flax, Polarised light microscopy



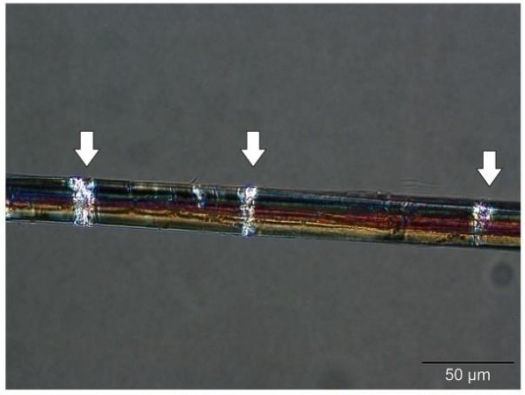
Nettle, White light microscopy



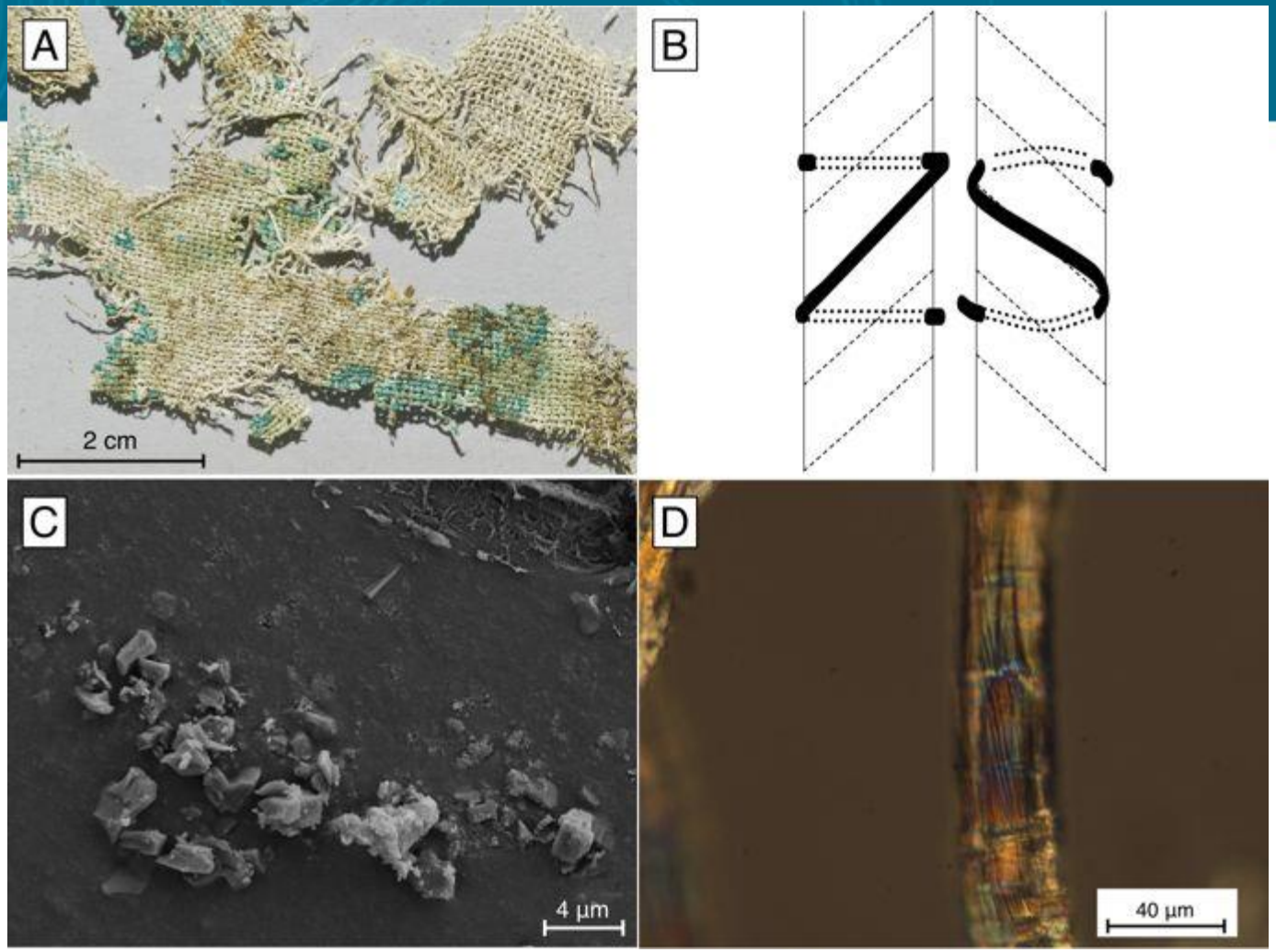
Nettle, Polarised light microscopy



Hemp, White light microscopy



Hemp, Polarised light microscopy



C. Bergfjord et al Scientific Reports 2, 664 (2012)

(A) The 2800-year-old Lusehøj Textile. Photo by Roberto Fortuna, The National Museum of Denmark. (B) A diagram of Z- (left) and S-twist (right). (C) Scanning Electron Microscopy image of calcium oxalate crystals found in association with the fibres. The sample has been plasma ashed to reveal the crystals. The remains of a fibre can be seen in the top right corner. (D) The fibrillar orientation in the ancient fibre is visible in a polarising microscope. As can be seen, the fibrillar orientation corresponds to an S-twist.

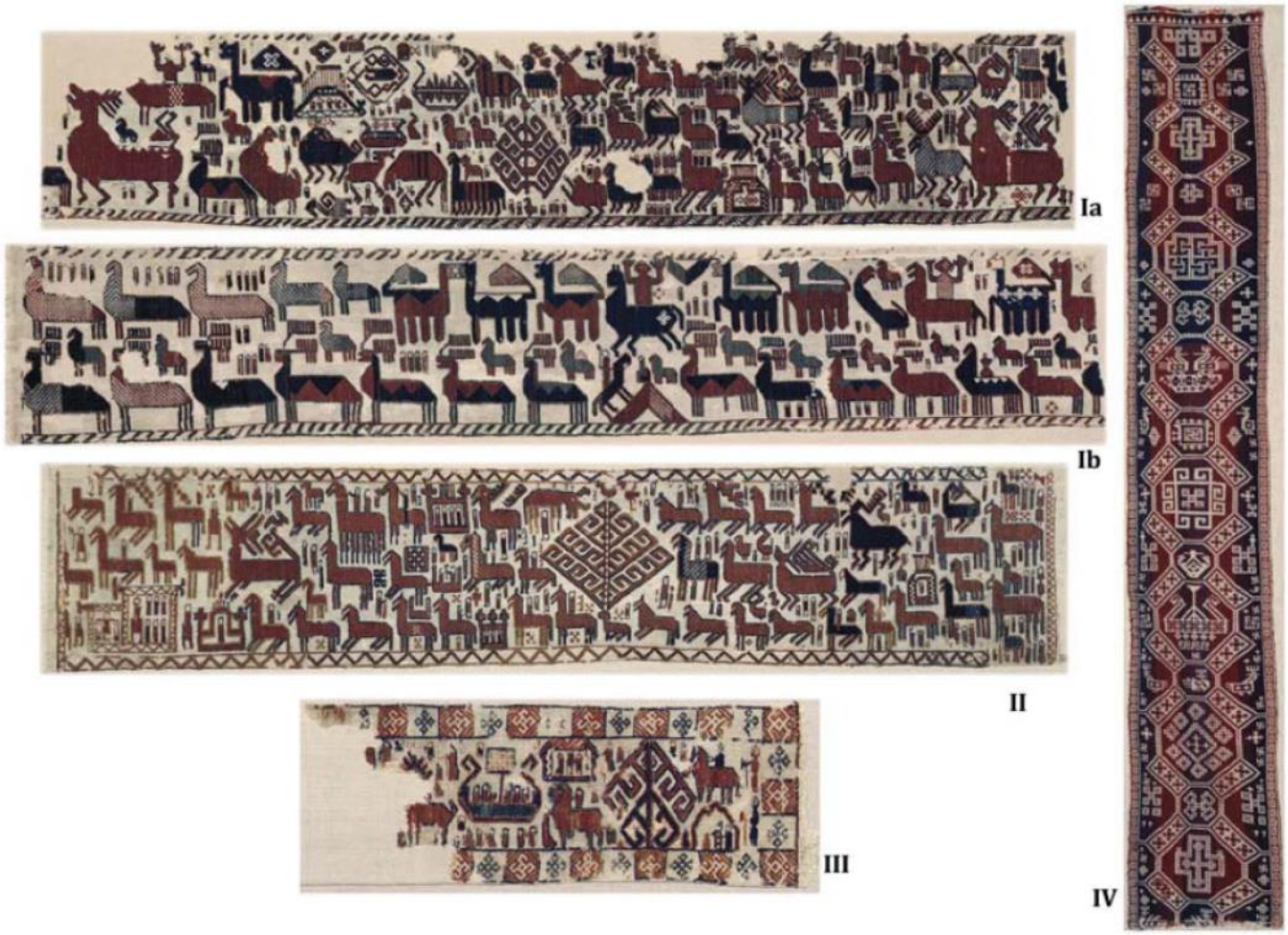


Figure 2 | The Överhogdal Viking wall-hangings. The two top pieces Ia (Length: 164 cm (top edge, Width: 33, 4–33, 5 cm) and Ib (Length: 195 cm, Width: 33–35 cm) are shown to be made of a mixture of hemp and flax. For the other pieces only flax were found (see Table 1). Published with permission of Jamtli Museum, Sweden.

– Viking and early middle ages northern Scandinavian textiles proven to be made with Hemp Mittleage. G. Skoglund, M. Nockert, B. Holst, Scientific Reports 3, 2686 (2013)

H. Lukesova, A. S. Palau, B. Holst, Identifying plant fibre textiles from orwegian Merovingian period and Viking age agraves: The late iron age collection of the University Museum of Bergen, Journal of Archaeological Science Reports: 13, 281-285 (2017)

SEEKING COMFORT IN THE IRON AGE

PHILIP BALL

The sophistication of Viking material culture, such as the fine quality of their decorative metalwork, has long given the lie to the old idea that they were barbaric brutes — a reputation that attests to the trauma left in Europe by the Viking raids of the ninth to the eleventh centuries. There have even been suggestions that Vikings used advanced navigation techniques based on the ability of certain local minerals — such as calcite or Iceland spar — to influence polarized light[1,2], although there is no archaeological evidence to support the idea.

But a new discovery must surely sink the image of unreconstructed brutishness once and for all: Vikings preferred soft underwear. They made their underclothing not from coarse hemp, which the Vikings used for sailcloth and wall hangings[3], but from finer flax.

Along with wool and nettle, hemp and flax were the only choices available for textile manufacture in Scandinavia in the late Iron Age of the sixth to the eleventh centuries. Cotton could theoretically have been acquired from late Roman culture, but there is no evidence of it. Wool, needless to say, would be rather itchy for clothing worn against the skin, but the other options all seem viable in principle.

Lukešová and colleagues[4] have investigated the fibres in ten textile items — nearly all of them from the tenth century — collected from excavations of Viking graves in various Norwegian provinces and kept at the University Museum of Bergen. All are made from plant fibres and, with the exception of a purse, all are items of clothing. One appears to be a man's shirt, while the others are women's clothing, mostly shifts worn as underwear. The sex of the wearer could be identified from other items in the respective graves, which indicate also that the bodies were high-ranking members of Viking society.

Plant fibres can be distinguished by polarized-light microscopy. The fibres contain bundles of cellulose chains called fibrils, which wind helically around the cell walls in either a left-handed (S) or right-handed (Z) twist (the same terminology is used for the twisting of macroscopic strands in textile threads). Flax and nettle have an S twist, hemp a Z twist, and these different fibre orientations show up as differences in the colour of the light transmitted through crossed-polar filters under the microscope: a so-called modified Herzog test. Nettle and flax can usually be differentiated, meanwhile, by the telltale presence of calcium oxalate crystals in the former.

With the exception of one ambiguous sample, all of those studied were made of flax. It is easier to make softer fabrics from this plant, since on average its fibres are finer — to achieve the same qualities with hemp, one would need to sort the fibres to remove the coarser strands. The same is true of nettle, which has the added drawback that it can't be cultivated as a textile crop but must be collected from the wild.

As ever with such archaeological studies, the findings shouldn't be generalized too readily. And it remains unclear whether the fibre choice was aesthetic or simply dictated by local availability. All the same, when it comes to underwear, given the choice we would probably have the same preference as the Viking noblewomen. ❐

References

1. Ropars, G., Gorre, G., Le Floch, A., Enoch, J. & Lakshminarayanan, V. *Proc. R. Soc. A* **468,** 671–684 (2011).
2. Ropars, G., Lakshminarayanan, V. & Le Floch, A. *Contemp. Phys.* **55,** 302–317 (2014).
3. Skoglund, G., Nockert, M. & Holst, B. *Sci. Rep.* **3,** 2686 (2013).
4. Lukešová, H., Palau, A. S. & Holst, B. *J. Archaeol. Sci. Rep.* **13,** 281–285 (2017).

Confocal Microscopy

A

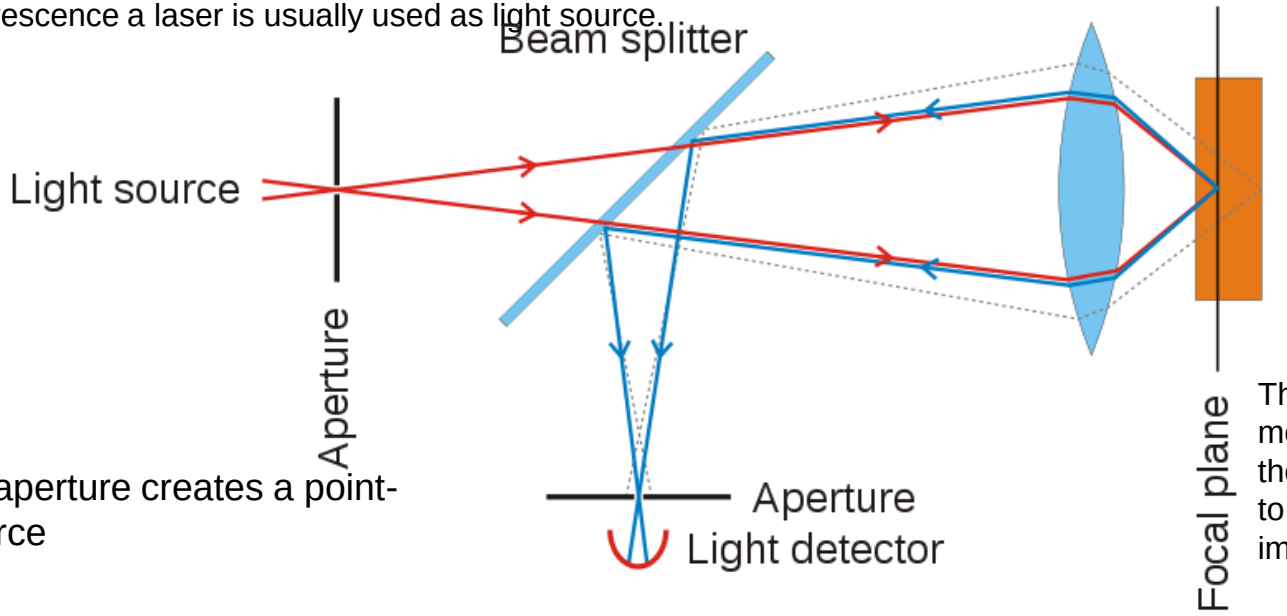
A Confocal Microscope is constructed to achieve 3D imaging, funnily enough this is done by essentially going back to the oldest type of microscopy illumination: The light source is focused onto the sample. The difference to old microscopes is that the light source used is a point source.

To obtain a 3D image the sample is scanned across the focussed point source.

Confocal microscopy normally relies on flourescence to get 3D imaging. Otherwise it would not be possible to image areas lying within the object. There would not be light enough.

How well the 3D imaging works depends on how well the light exiting the fluoropores and the light emitted from the fluoropores is transmitted through the sample

Resolution is still diffraction limited (determined by the numerical aperture). To achieve the best resolution and selective flourescence a laser is usually used as light source.



Pihole aperture creates a point-like source

The sample is moved through the focal plane to create a 3D image.

Pinhole aperture in front of light detector
Ensures that only light from the image point on the sample is detected