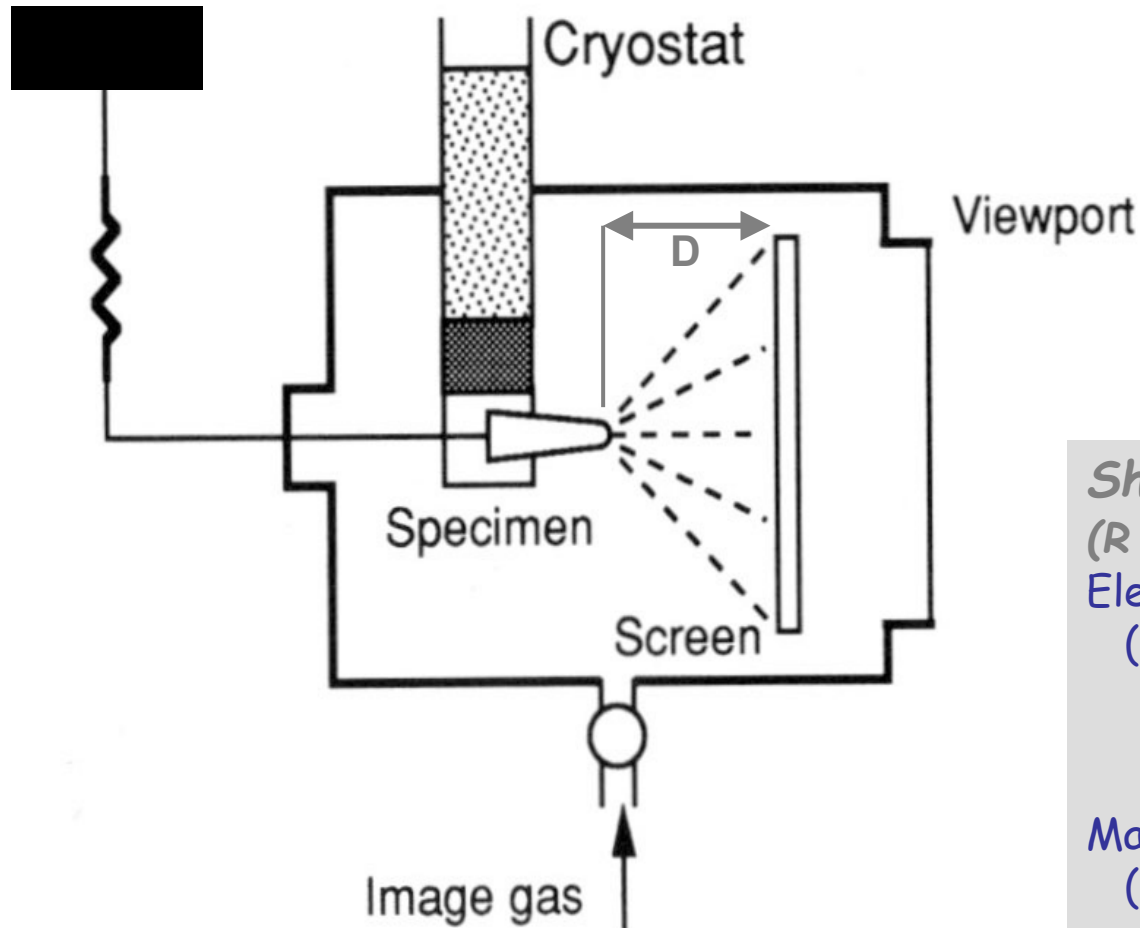


14) 3D Microscopy/Tomography

3D Microscopy

- Atom probe Tomography (origin/applications)
- X-ray Tomography (Medicine, Materials science)
 - Radon transform
 - Back projection
- Tilt-series tomography in TEM

Field emission (electron, ion) microscopy



*FEM: vacuum,
electrons
voltage < 0*

*FIM: low pressure rare gas,
ionized gas molecules
voltage > 0*

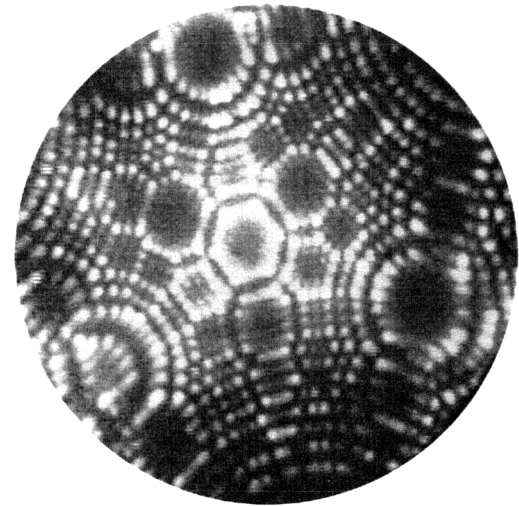
*Sharp tip
(R radius of curvature):*

Electric field $E = V/R$
($E \approx 10^9$ V/m) $V \approx 1$ kV
 $R \approx 0.1$ μm

Magnification $M = D/R^\beta$
($M \approx \dots 10^6$) $D \approx 0.3$ m
 $\beta \approx 1.5$ (tip shape)

Field Ion Microscopy

Gas Field Ionisation Source (GFIS)



- atoms (molecules) are trapped by polarizations forces
- Trapped atoms hop on the surface until they are ionised

Ionisation: tunnelling process with probability D :

$$D \propto e^{\frac{-c(I-\Phi)}{V}}$$

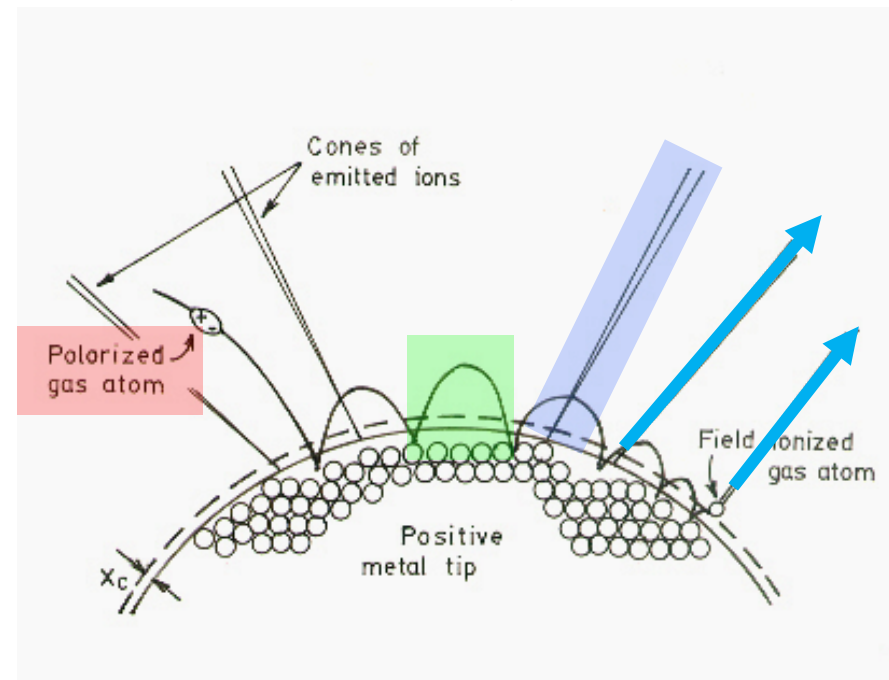
I : Ionisation potential

Φ : Work function of emitter

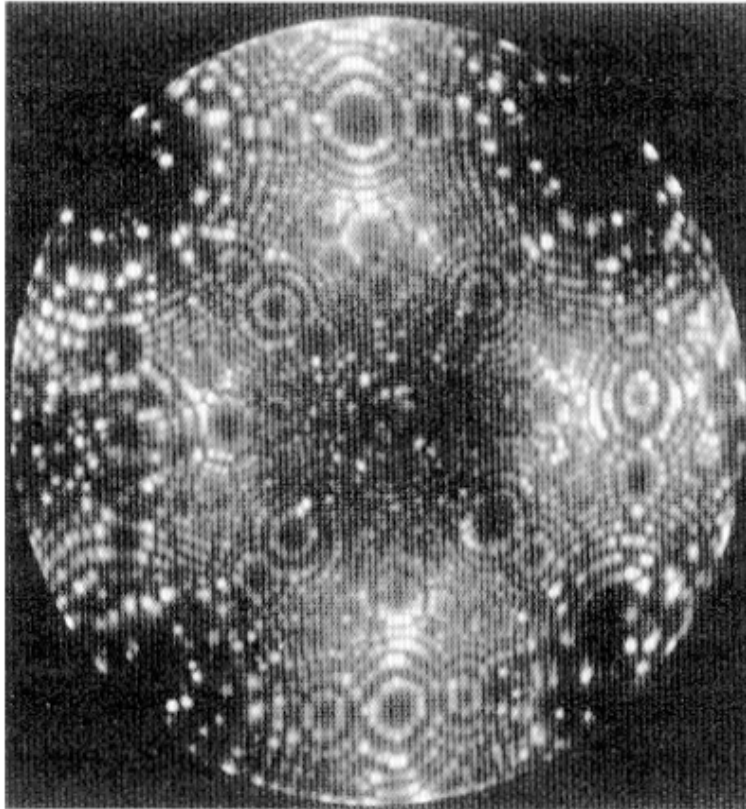
V : El. Potential

c : constant

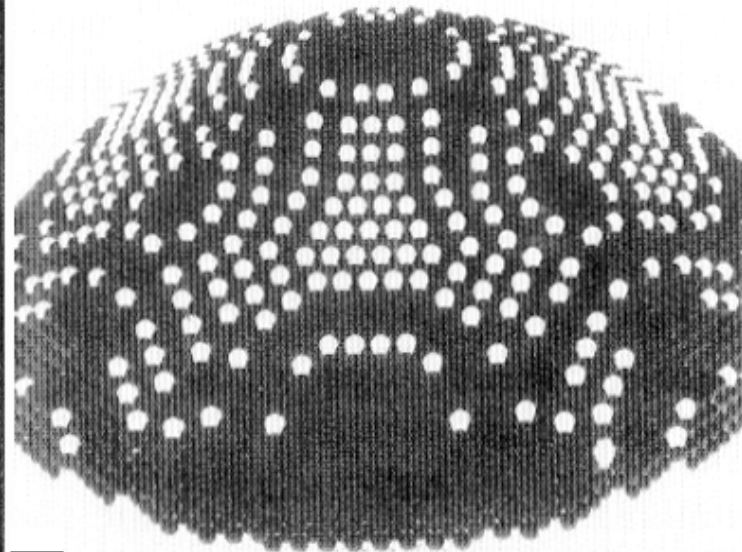
- Ions are ejected from the surface



Field emission (electron, ion) microscopy FEM, FIM



Field ion micrograph from a [100] oriented specimen of Pt 17 at.% Rh catalyst material imaged in Ne gas with an applied voltage of approx. 10 kV at 80K. Each of the bright spots in the left picture is the image of an individual atom on the specimen surface.

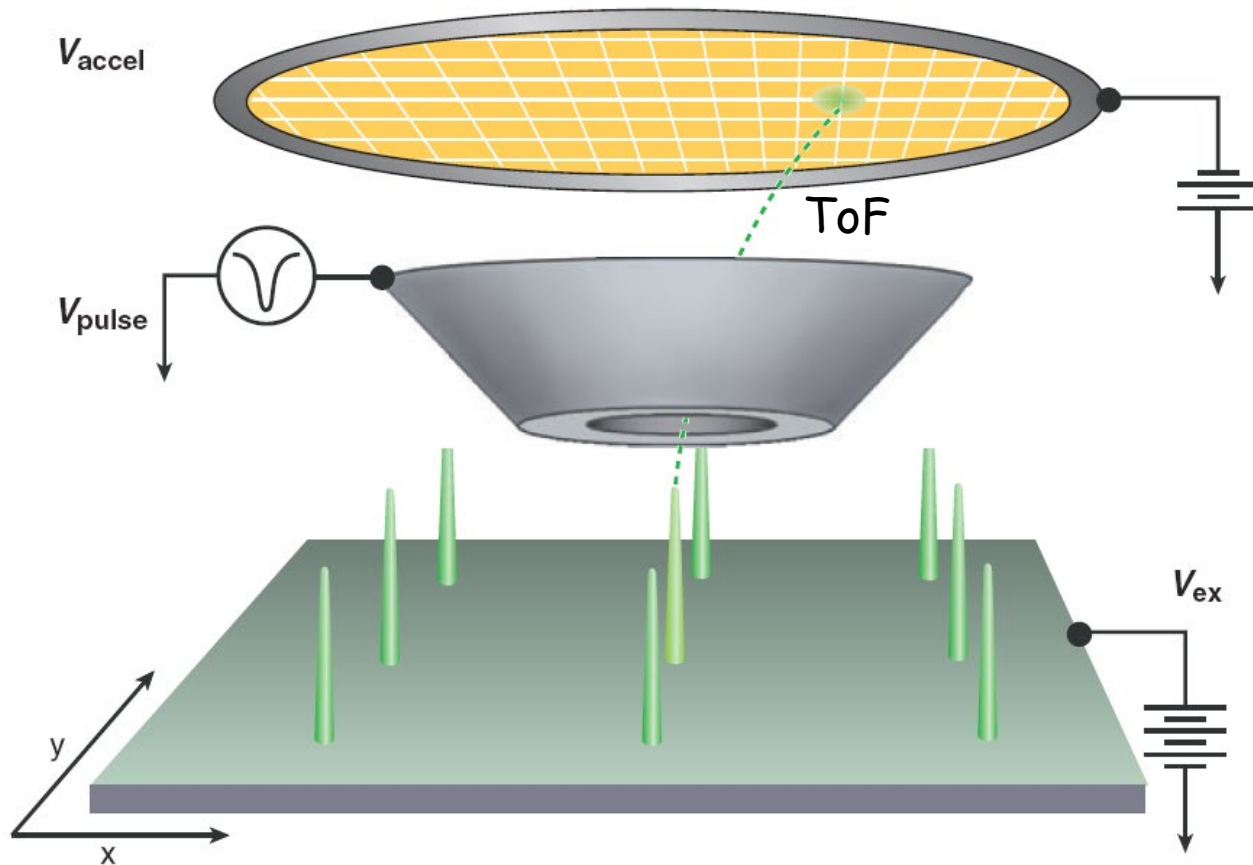


Ball model . The prominent sites, representing kink site atoms on the surface are shown in white. These form a serie of concentric rings similar to those seen in the field-ion picture

Atom probe Tomography

- Use the sample atoms as imaging ions....!
 - APFIM: Atom Probe Field Ion Microscopy
- Measure the Time Of Flight (ToF), to determine the mass of the ion...!
 - elemental analysis on atomic level
- Use Laser to assist ablation of ions
 - LAWATAP: Laser Assisted Wide Angle Tomographic Atom Probe
 - Insulating samples become possible

Wide Angle position sensitive detector



Sample needs to have tip shape: metals: etching, insulators: FIB

Three-Dimensional Atom-Probe Tomography: Advances and Applications

Annu. Rev. Mater. Res. 2007.37:127-158

David N. Seidman

Department of Materials Science and Engineering, Northwestern University Center
for Atom-Probe Tomography, Northwestern University, Evanston, Illinois
60208-3108; email: d-seidman@northwestern.edu

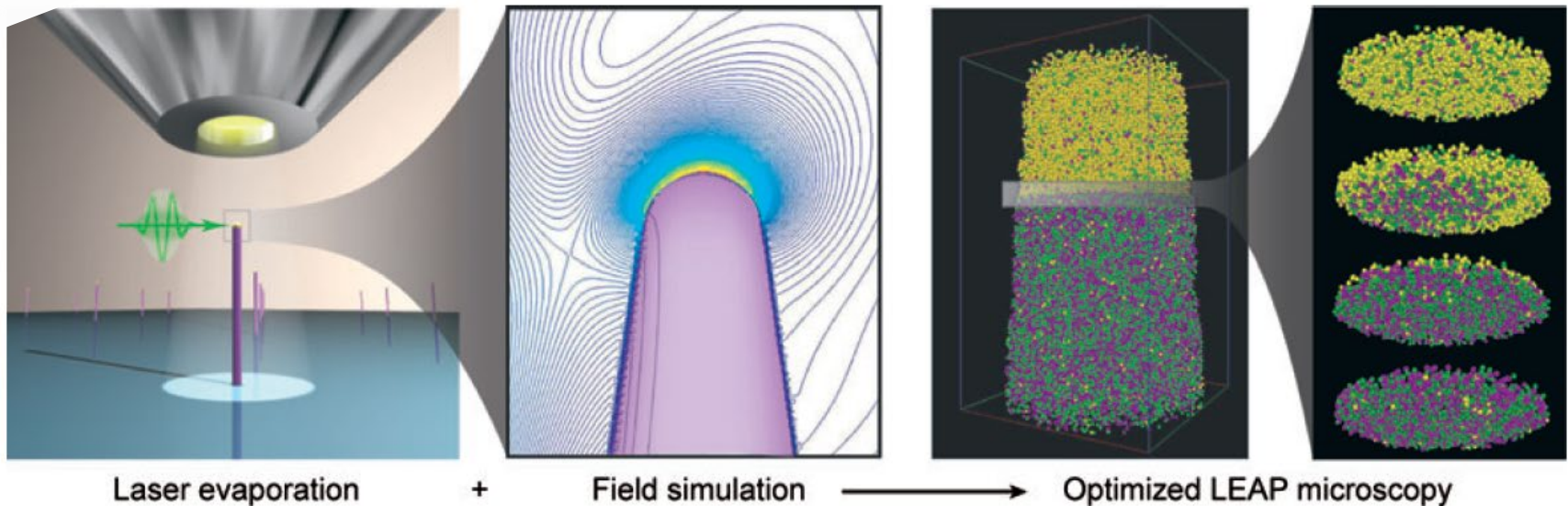


Figure 2

(Left) An array of microtips sitting on a conducting substrate with green laser light impinging on a single microtip. (Middle) The calculated electric field at a microtip that results from the E-component of the laser beam. Courtesy of Prof. Tamar Seidman (Chemistry Department, Northwestern University). (Right) The results of an analysis of an InAs nanowire grown by the vapor-liquid-solid (VLS) technique.

Detection speed

- **Electrical pulsing**

The pulse repetition rate is variable in discrete steps from 1 kHz to 250 kHz, and a detection rate of up to $2 \times 10^6 \text{ ion min}^{-1}$ ($120 \times 10^6 \text{ ion h}^{-1}$) can be achieved. This implies that a data set containing 10^9 atoms can be obtained in 8 h from a single cooperative specimen. For electrical pulsing, the full-width half-maximum (FWHM) value of $m/\Delta m$ is 500.

- **Laser (picosecond) Pulsing**

For the LEAP 3000X Si XTM, the laser pulse repetition rate is variable in steps from 1 kHz to 500 kHz, and a detection rate of up to $5 \times 10^6 \text{ ion min}^{-1}$ ($300 \times 10^6 \text{ ion h}^{-1}$) can be obtained. Therefore, a data set containing 10^9 atoms is attainable from a single very cooperative specimen in 3 h, which is a factor of 2.5 faster than with electrical pulsing.

Precipitates in Ni Alloy

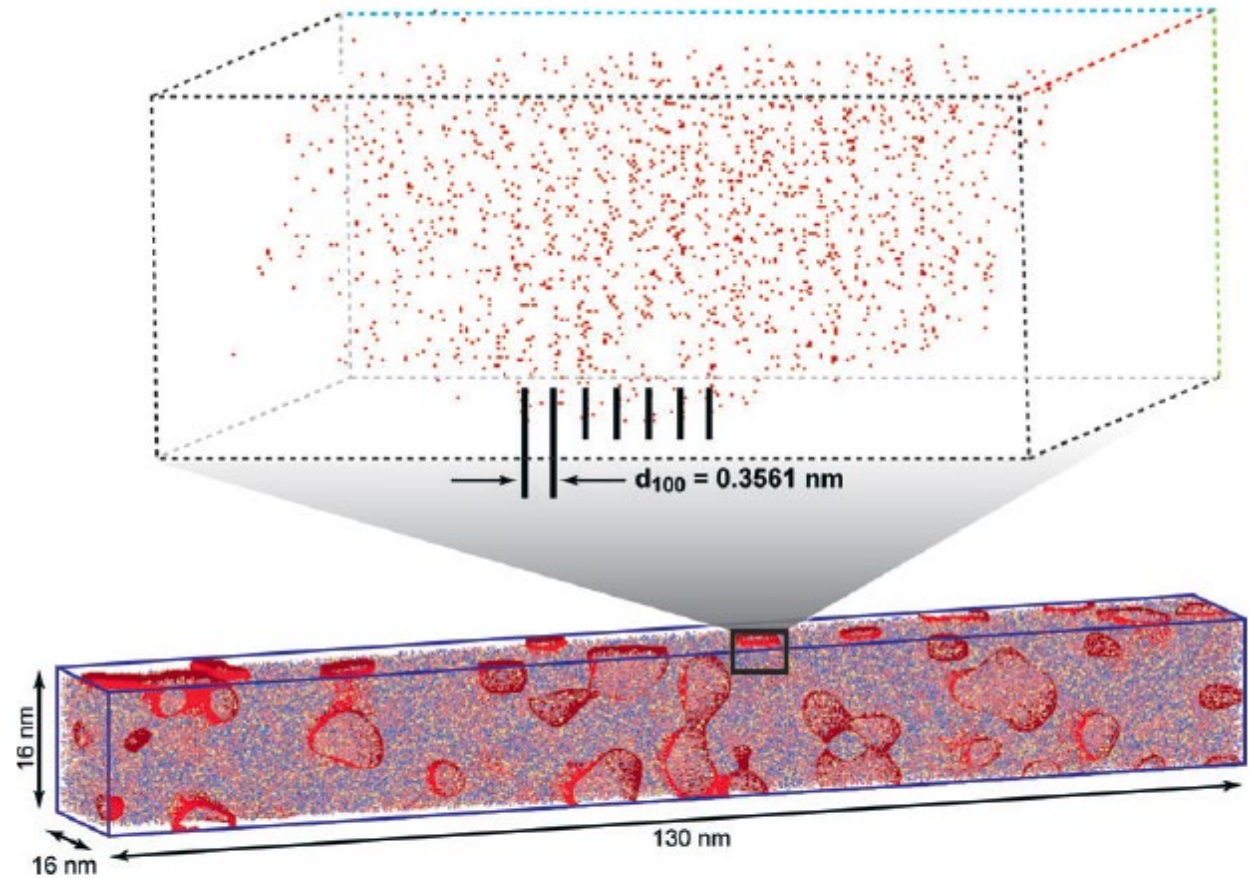


Figure 9

APT reconstruction of a Ni-10 at.% Al-8.5 at.% Cr-2 at.% Re alloy in the as-quenched state, which indicates the presence of γ' -precipitates. A 12 at.% Al isoconcentration surface is used to indicate the γ/γ' interfaces. A portion of the γ' -precipitate is magnified, with Al (*red*), Cr (*blue*), and Re (*orange*) atoms shown to display the alternating Al planes in the $[100]$ direction of the ordered $L1_2$ structure. Ni atoms are not shown for the sake of clarity. Adapted from Reference 129.

InAs nano-wire

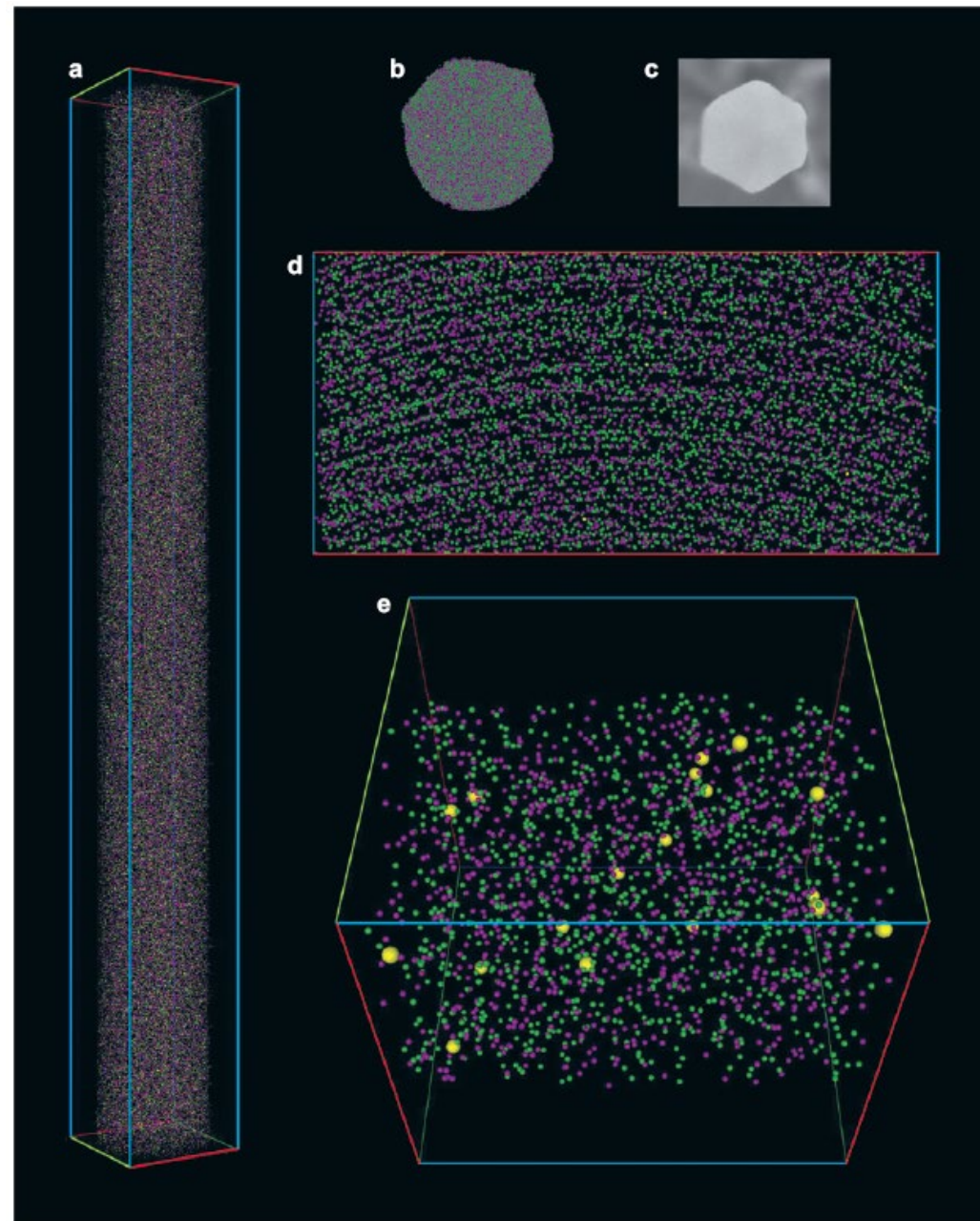


Figure 12

Three-dimensional reconstruction of an InAs nanowire. The sample temperature was at a fixed value between 50 K to 100 K, and the ambient pressure in the LEAPTM tomograph was 10^{-10} Torr during its atom-by-atom dissection. (a) Side view (perpendicular to growth axis) of a $25 \times 25 \times 300$ nm³ reconstruction of the nanowire. In, As, and Au atoms are rendered as green, purple, and yellow dots, respectively. Only 5% of the atoms are displayed to provide a sense of depth. (b) A 21×21 nm² end-on view of nanowire reconstruction showing hexagonal faceting. (c) SEM micrograph of an InAs nanowire displaying a hexagonal cross section ($1.7 \mu\text{m}^2$). (d) Magnified side view of the nanowire displaying (0001) atomic planes. The dimensions are 23×14 nm². The slight curvature of the atomic planes is an artifact; the software used for the reconstruction assumes a hemispherical end-form for the field-evaporating nanowires. (e) For clarity, a $27 \times 27 \times 29$ nm³ reconstruction of the nanowire with Au atoms is enlarged, and 2% of In and As atoms are shown; the growth axis runs left to right. The 18 Au atoms within the volume correspond to a concentration of 100 atomic parts per million. From Reference 121.

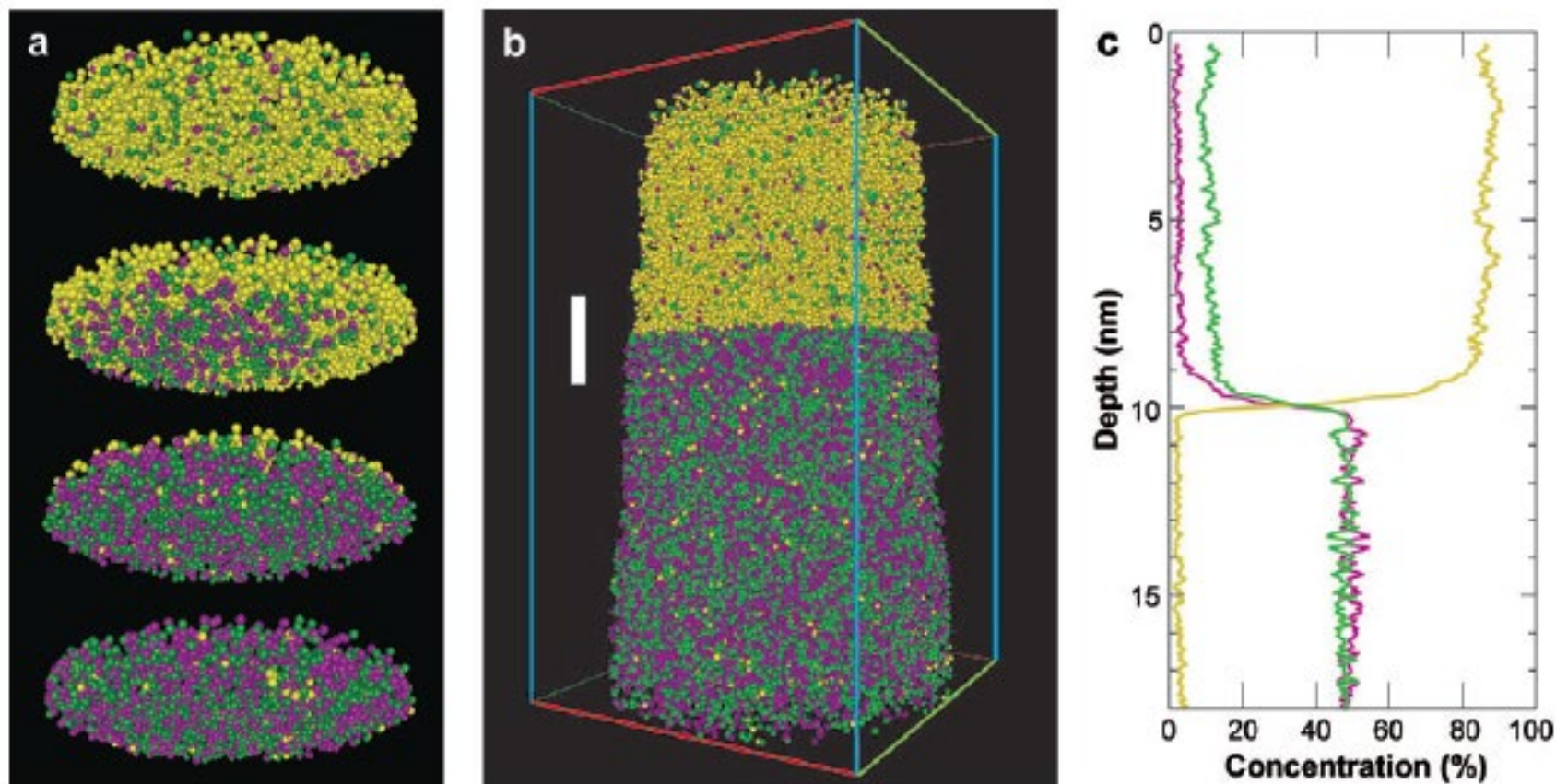


Figure 13

Catalyst nanowires interface in 3-D. (a) One-nm-thick slices through the nanowires over the region defined by the white bar in *b*. The diameter of the slices is 10 nm. In, As, and Au atoms are rendered as green, purple, and yellow dots, respectively. (b) A $14 \times 14 \times 23 \text{ nm}^3$ reconstruction of an InAs nanowire tip showing a Au catalyst particle at the top. (c) Composition profile in 1-D plotted along the growth axis and through the catalyst/nanowire interface. The plotted composition is a radially average value within a 4-nm-diameter cylinder centered in the middle of the nanowire. From Reference 121.

Introduction to Tomography

- **Tomography** is imaging by sections or sectioning. A device used in tomography is called a **tomograph**, while the image produced is a **tomogram**.
- The method is used in [medicine](#), [archaeology](#), [biology](#), [geophysics](#), [oceanography](#), [materials science](#), [astrophysics](#) and other sciences.
- In most cases it is based on the mathematical procedure called [tomographic reconstruction](#).
- The word "tomography" is derived from the [Greek](#) tomos (slice) and *graphein* (to write).

Wikipedia

Introduction to Tomography

- **Tomography** is a method in which a 3-D structure is reconstructed from a series of 2-D projections (images) acquired at successive tilts (Radon 1917).
- First developed for use in medical imaging (1963, Nobel Prize for Medicine in 1979) using X-rays, ultrasound and magnetic resonance (e.g. 'cat-scans')..
- Found further application in geology, astronomy, materials science, etc...

Introduction to Tomography

Recording

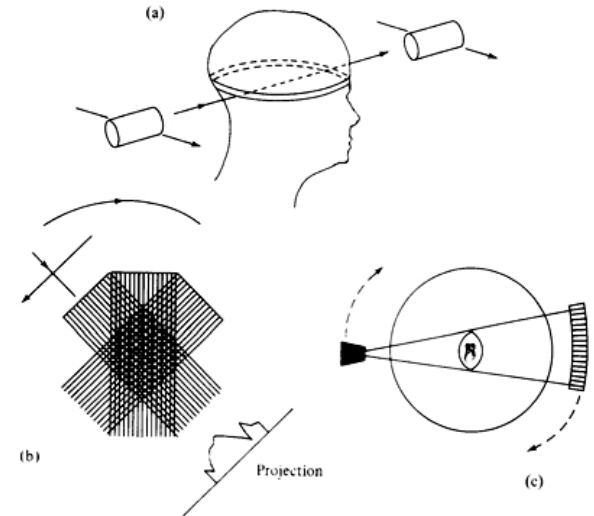
- Series of 2D images
- Destructive: serial sectioning, FIB, LEAP
- Non-destructive: X-rays, TEM

Reconstruction and « viewing »

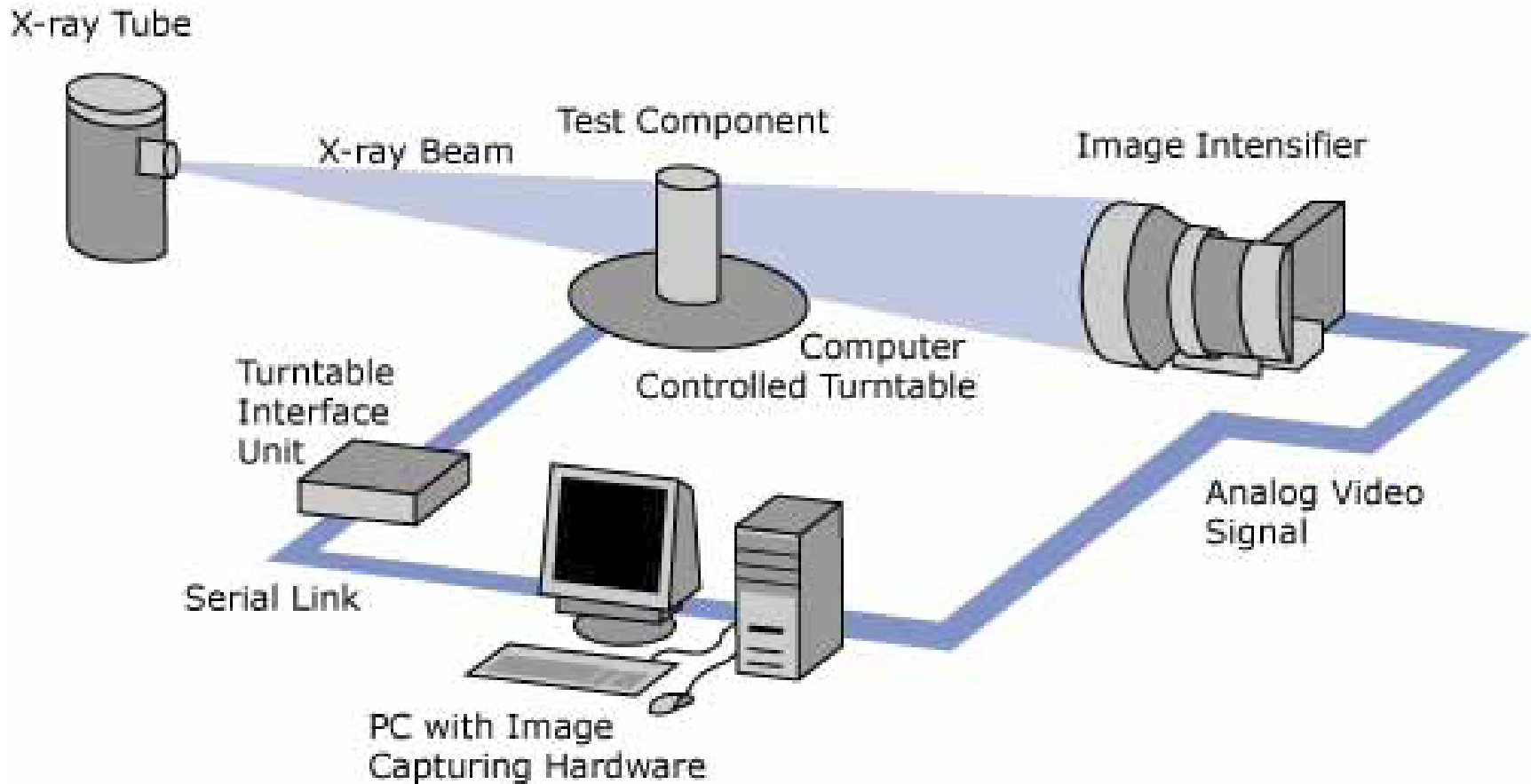
- Registration (alignment of images)
- Back-projection, reconstruct (tilt-series)
- **Tomogram**
- Segmentation (image processing), extraction of the desired information

3D imaging in medicine

- Non-invasive methods are preferred!
- The disadvantage of conventional X-radiographs is its inability to discriminate between organs of close absorptivity or overlapping organs in the viewing direction.
- X-ray computed tomography overcomes that limitation:
- X-radiographs are made in many different directions and combined mathematically to reconstruct cross-sectional maps.
- reconstruction tomography or computer assisted tomography.



Tomograph



Radon 1917



Über die Bestimmung von Funktionen durch ihre Integralwerte längs gewisser Mannigfaltigkeiten.

Von

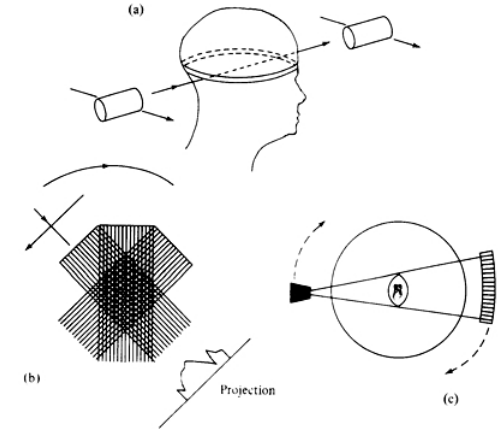
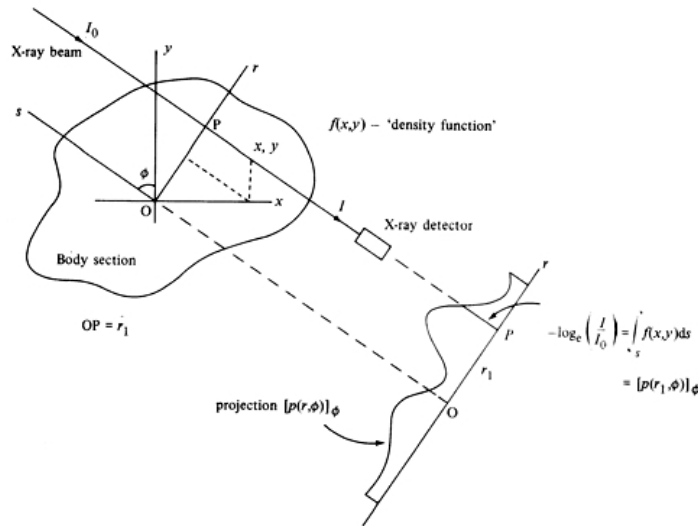
JOHANN RADON.

Integriert man eine geeigneten Regularitätsbedingungen unterworfenen Funktion zweier Veränderlichen x, y — eine *Punktfunktion* $f(P)$ in der Ebene — längs einer beliebigen Geraden g , so erhält man in den Integralwerten $F(g)$ eine *Geradenfunktion*. Das in Abschnitt A vorliegender Abhandlung gelöste Problem ist die Umkehrung dieser linearen Funktionaltransformation, d. h. es werden folgende Fragen beantwortet: kann jede, geeigneten Regularitätsbedingungen genügende Geradenfunktion auf diese Weise entstanden gedacht werden? Wenn ja, ist dann f durch F eindeutig bestimmt und wie kann es ermittelt werden?

Ber. Sächs. Akad. Wiss. Leipzig, Math. Phys. Kl. 69, 262 (1917)

English translation in: Deans, S.R. (1983) The Radon transform and its applications. John Wiley & Sons, NY)

Radon Transform



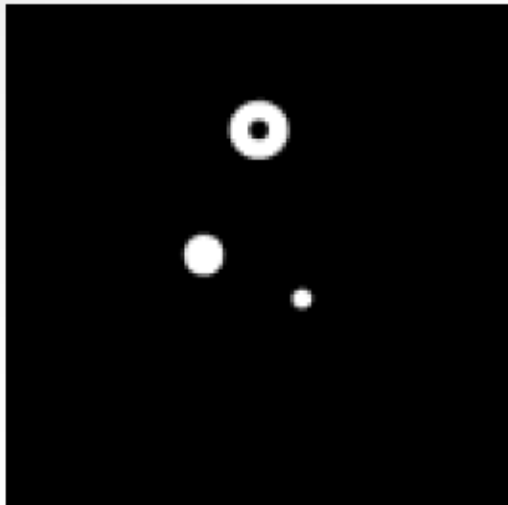
The paper defines the Radon transform R as the mapping of a function $f(x, y)$, describing a real space object D , by the projection, or line integral, through f along all possible lines L :

$$Rf = \int_L f(x, y) ds,$$

A discrete sampling of the Radon transform is geometrically equivalent to the sampling of an experimental object by some form of transmitted signal: a projection. **The consequence of such equivalency is that the reconstruction of an object $f(x, y)$ from projections Rf can be achieved by implementation of the inverse Radon transform**

Radon Transform

Object



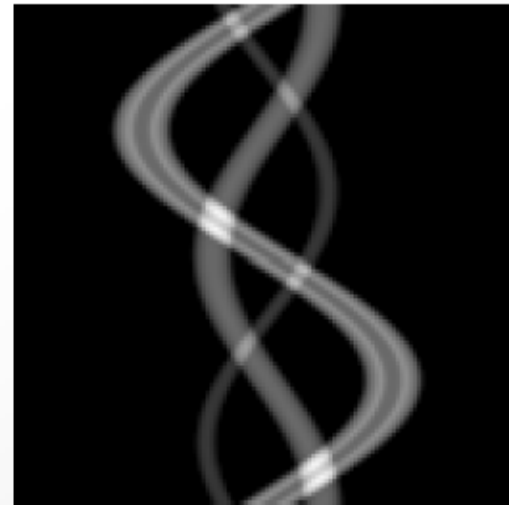
Radon transform
= Sinogram

-180°



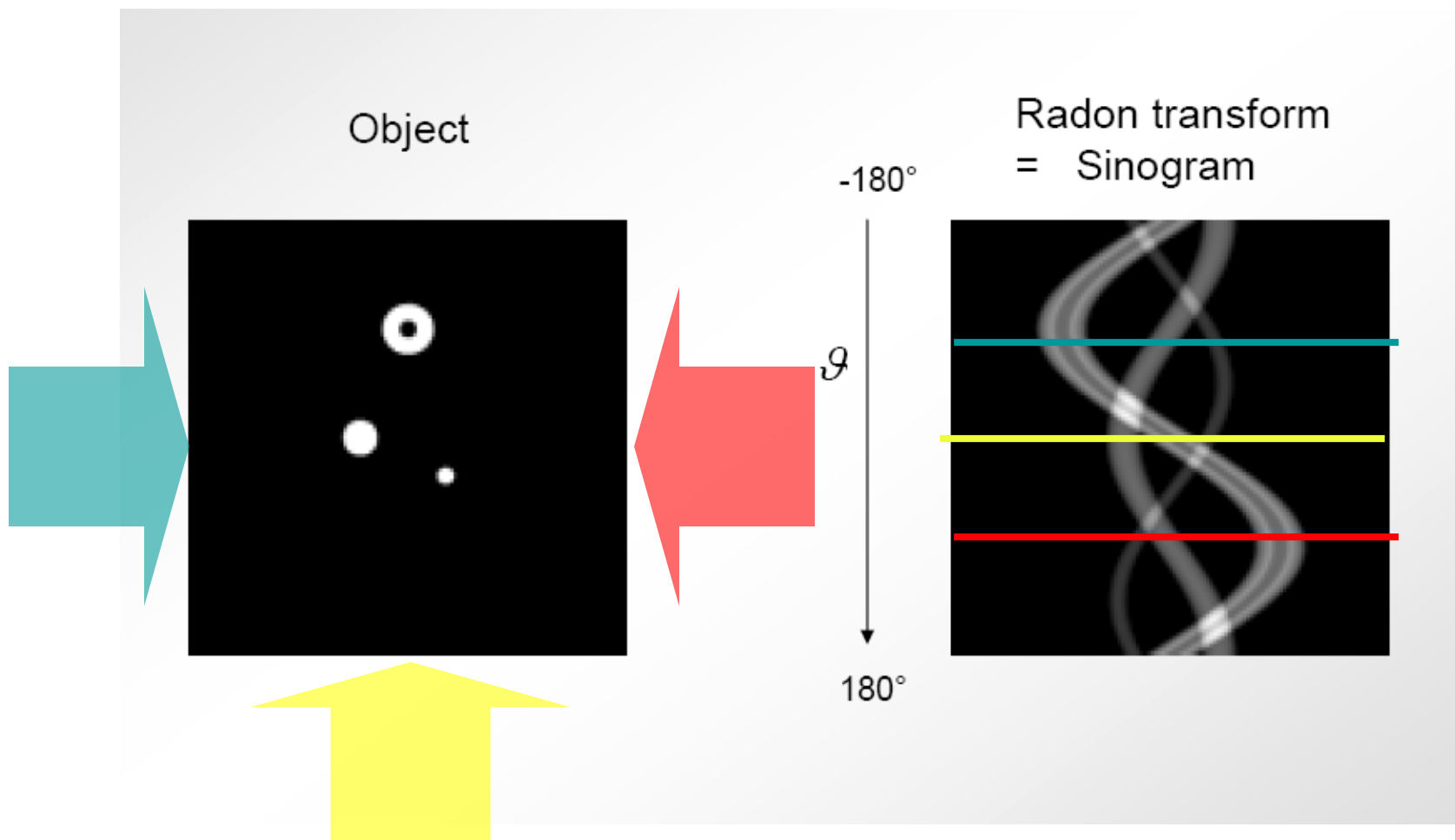
ϑ

180°



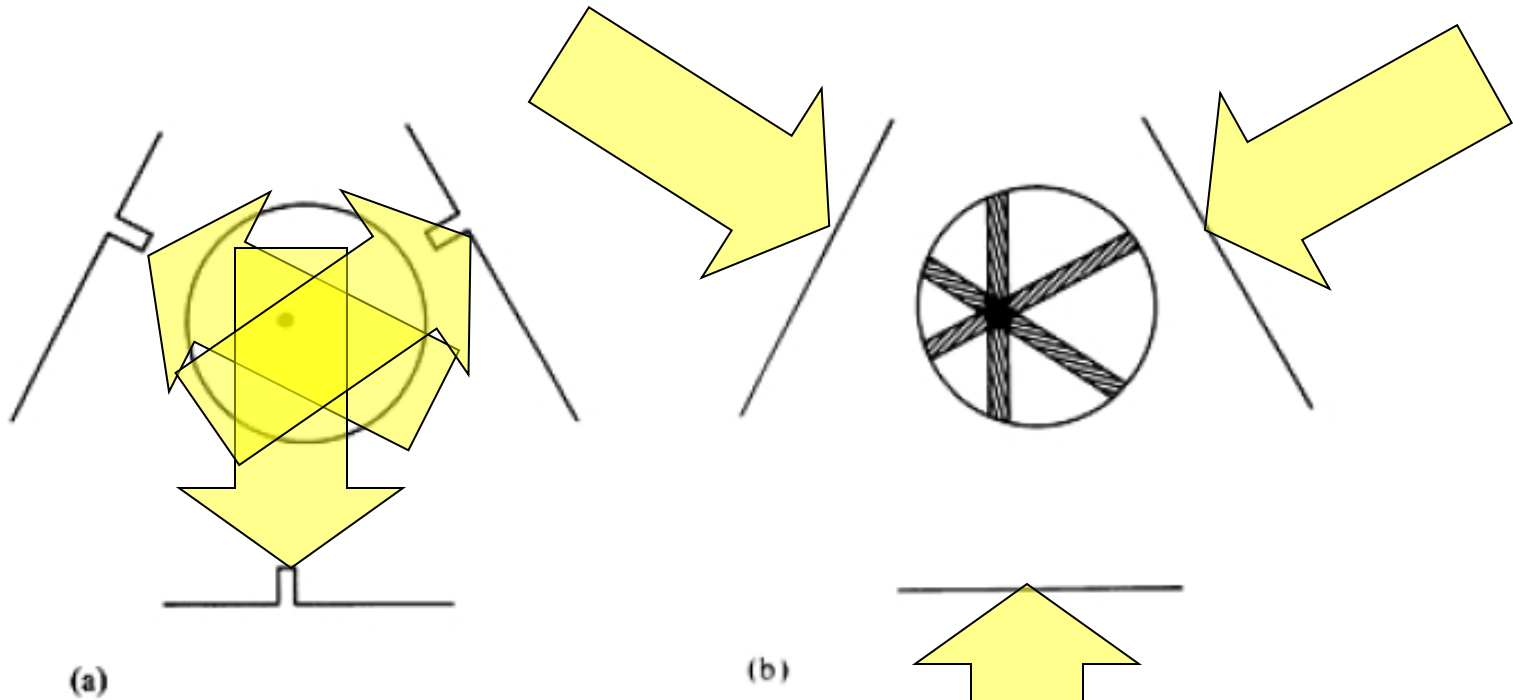
Radon Transform

(projection in different directions)



Back projection

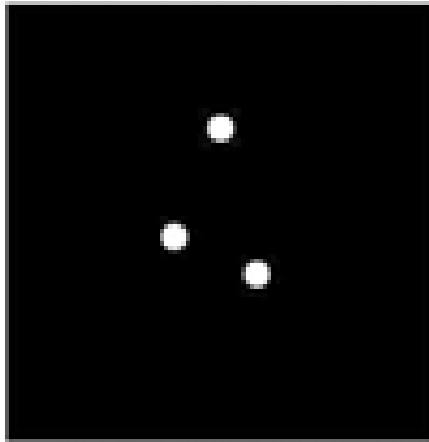
(« reconstruction »)



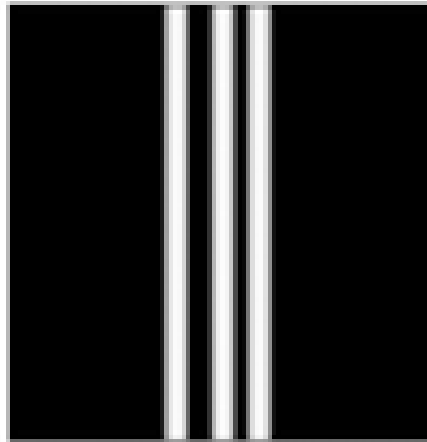
Projection
recording

Back projection
reconstructing

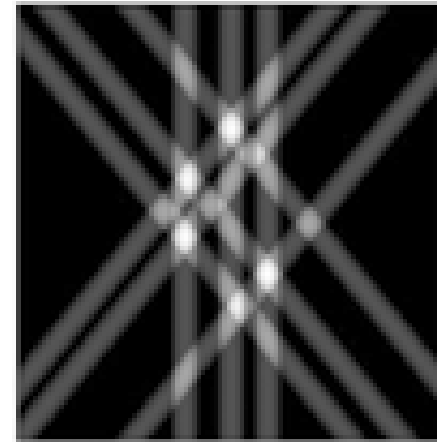
Back projection



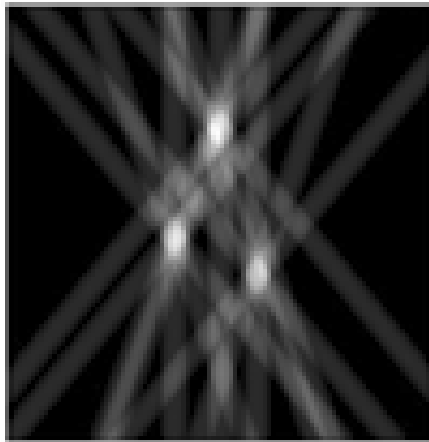
Object



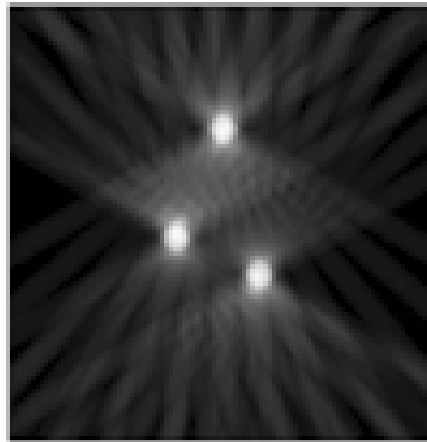
1 Proj.



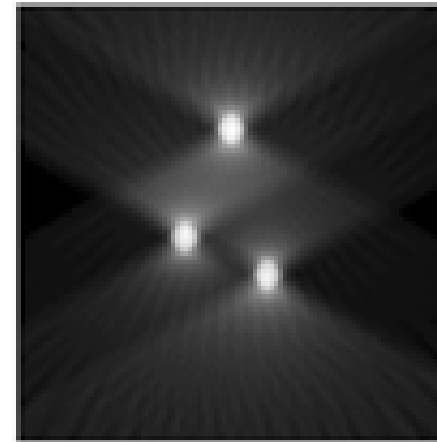
3 Proj./40 deg.



5 Proj./20 deg.

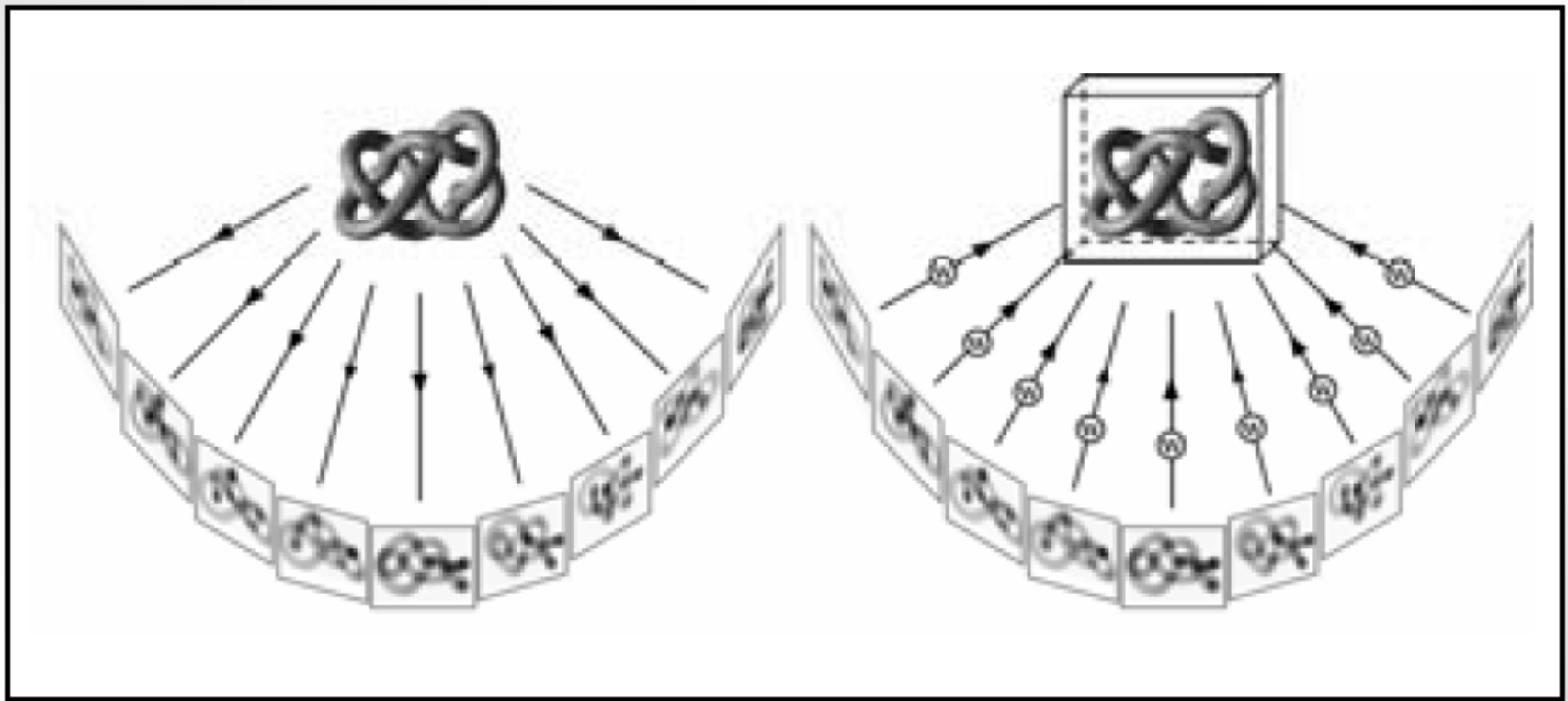


13 Proj./10 deg.

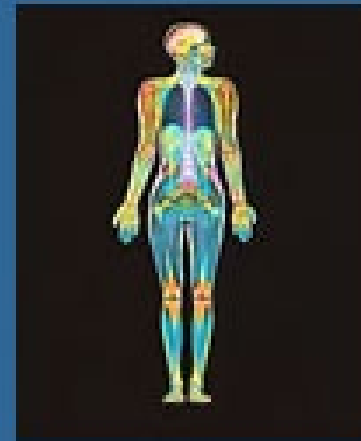
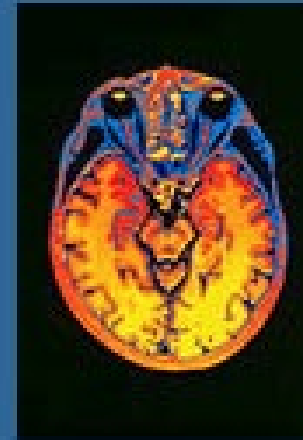
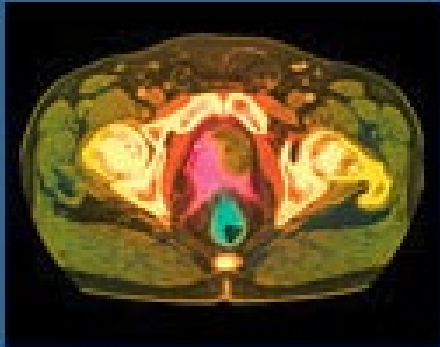


25 Proj./5 deg.

Projection and Back Projection



Tomography in medicine

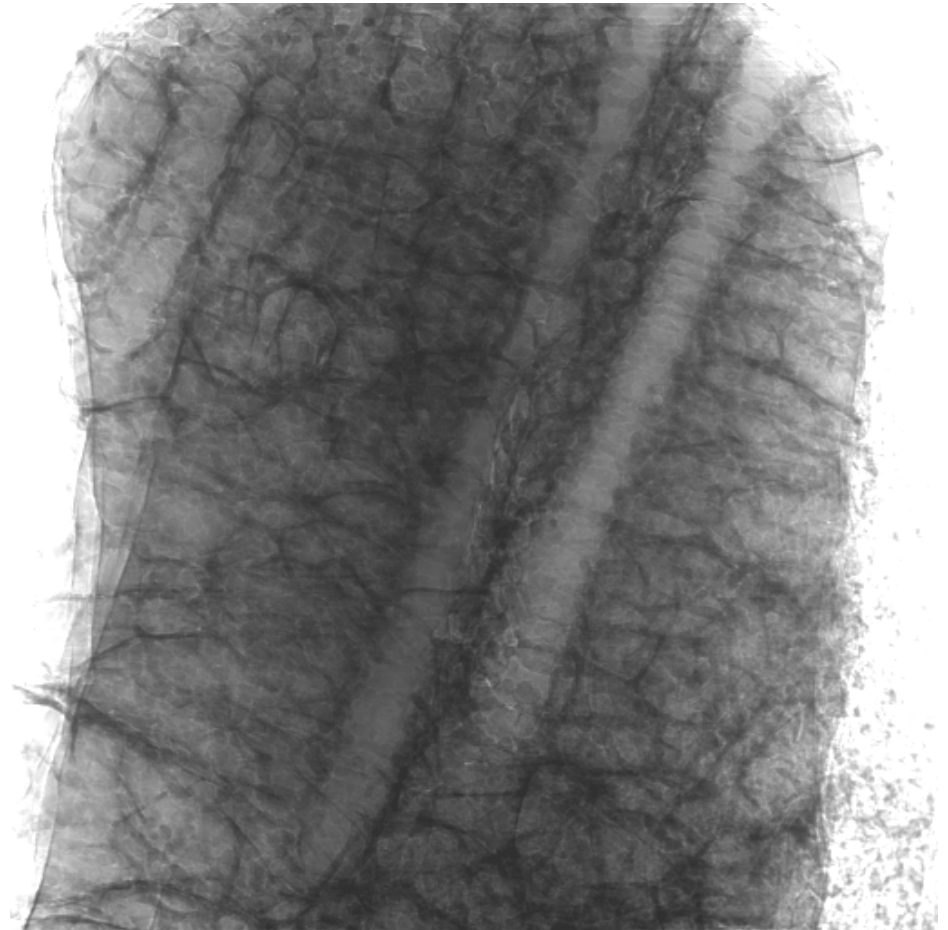


3D imaging in materials science

360degree X-ray tomography
on synchrotron

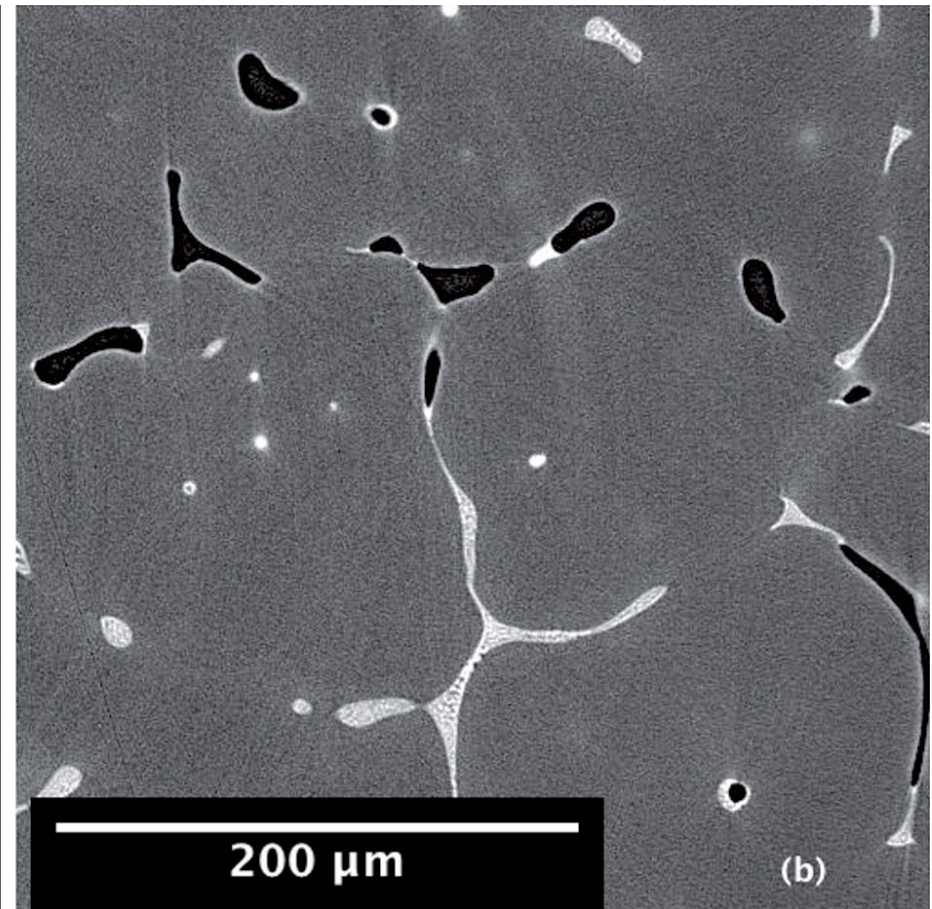
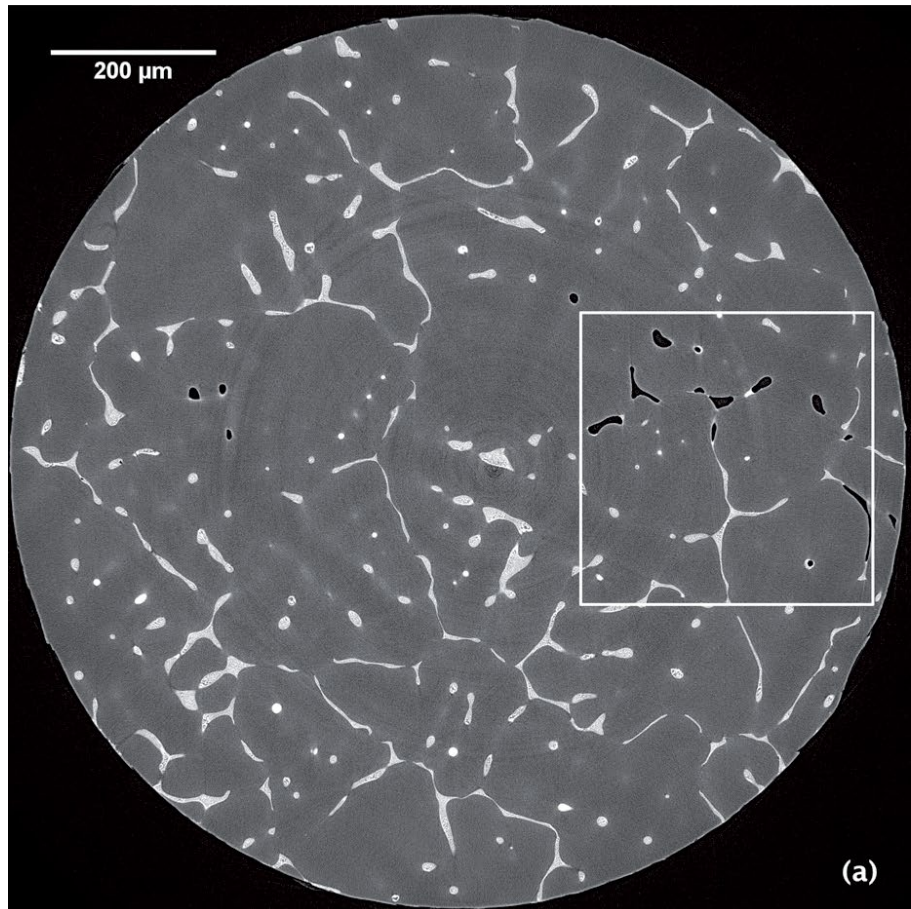
Milan Felberbaum
STI-IMX-LSMX

Cylinder of an Al-Cu Alloy



3D imaging in materials science

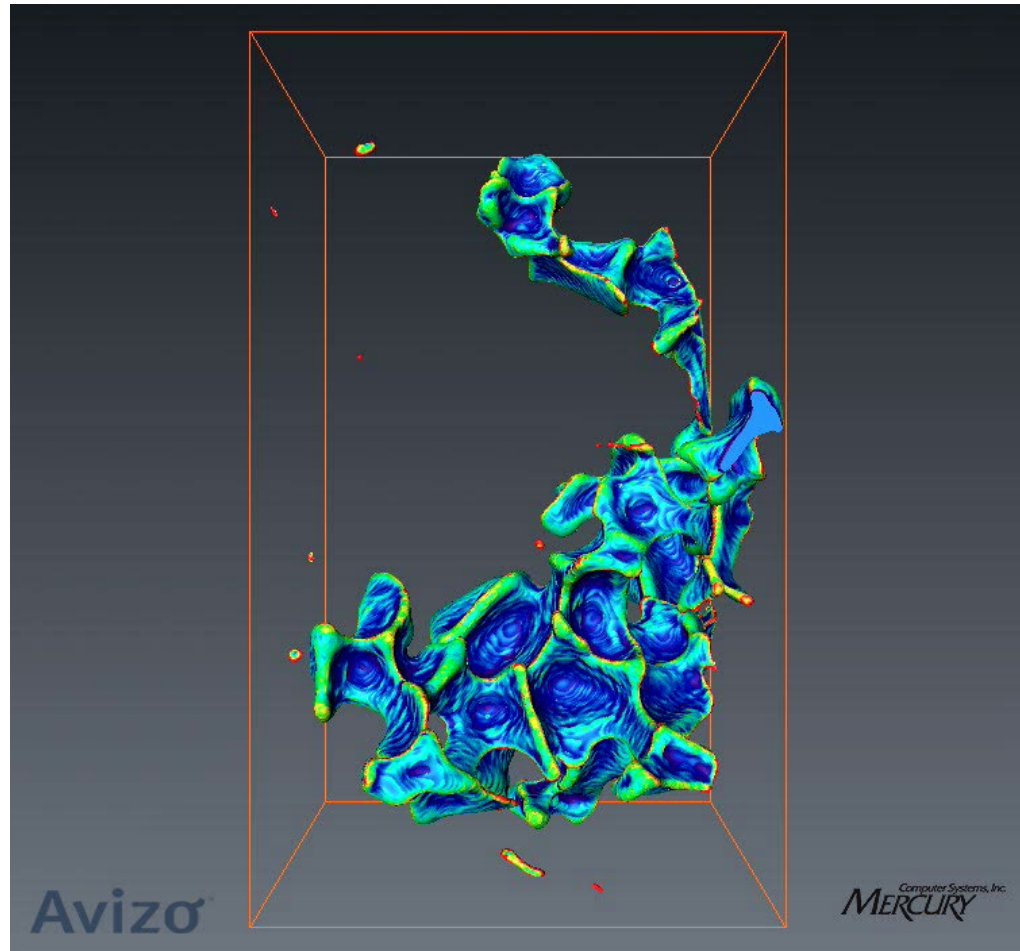
Tomogram (reconstructed volume)



3D imaging in materials science

(rendering, visualisation)

Reconstructed pore

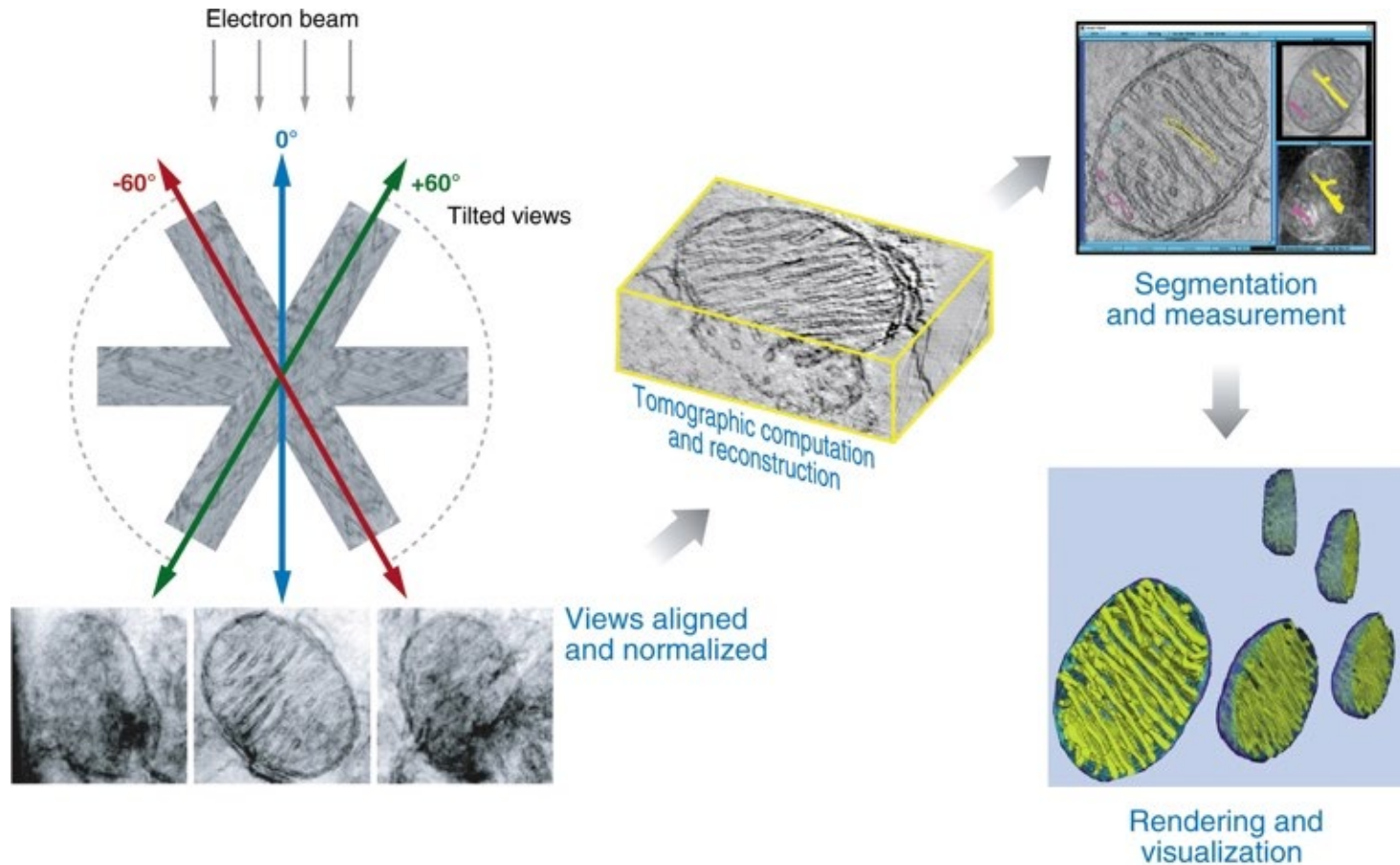


Tomography with electrons

Stopping range for electrons (99% absorbed)

Element (specific weight)	4-Be 1.84 g/cm ³	13-Al 2.7 g/cm ³	29-Cu 8.93 g/cm ³	82-Pb 11.3 g/cm ³
X-rays Cu-K α $\lambda=1.54 \text{ \AA}$ Mo-K α $\lambda=0.71 \text{ \AA}$	16 mm 83 mm	0.35 mm 3.3 mm	0.10 mm 0.10 mm	0.017 mm 0.034 mm
Neutrons $\lambda \approx 1.08 \text{ \AA}$	89 m	6 m	0.26 m	14 m
Electrons $\lambda=0.037 \text{ \AA}$ à 100 kV $\lambda=0.020 \text{ \AA}$ à 300 kV	39 μm	42 μm ~330 μm	11 μm	0.6 μm

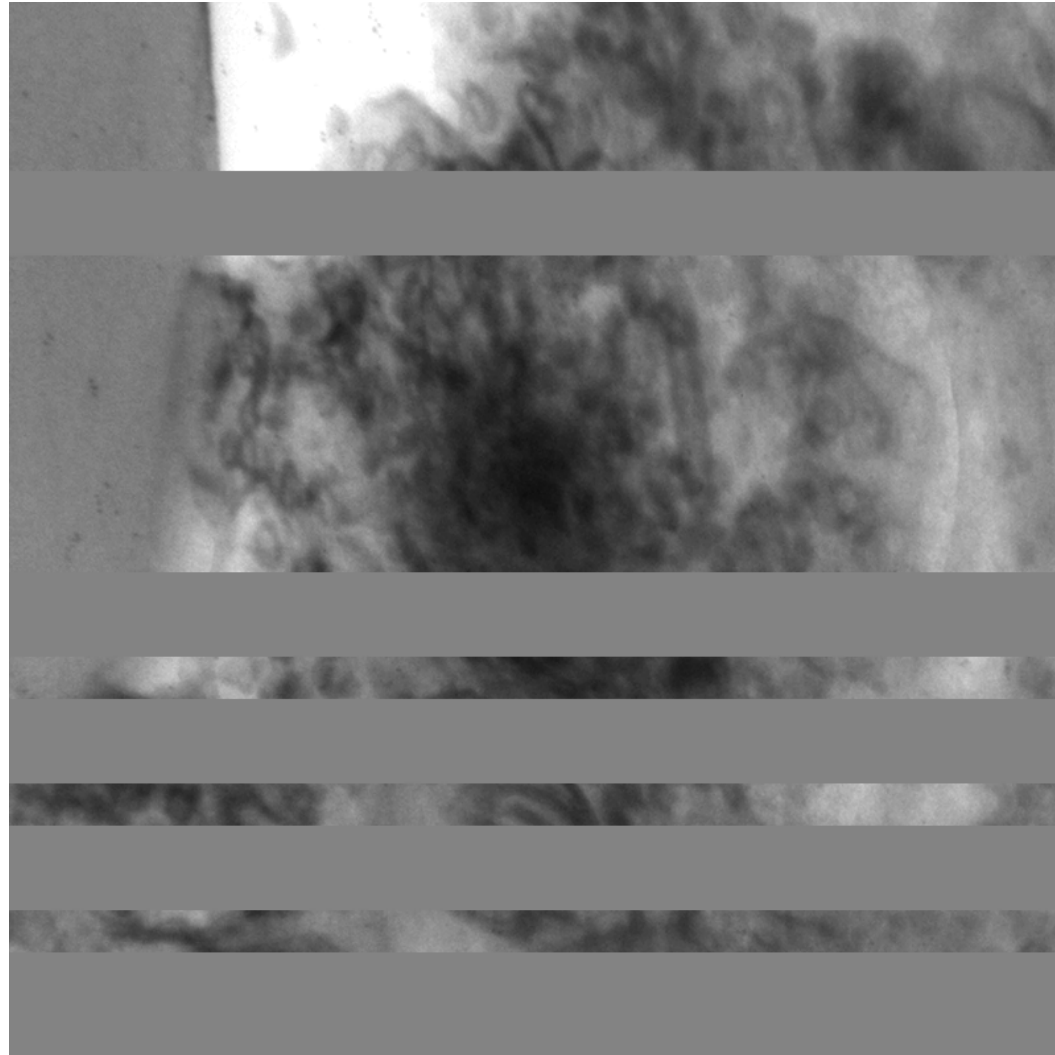
Tomography in TEM, Bio-EM



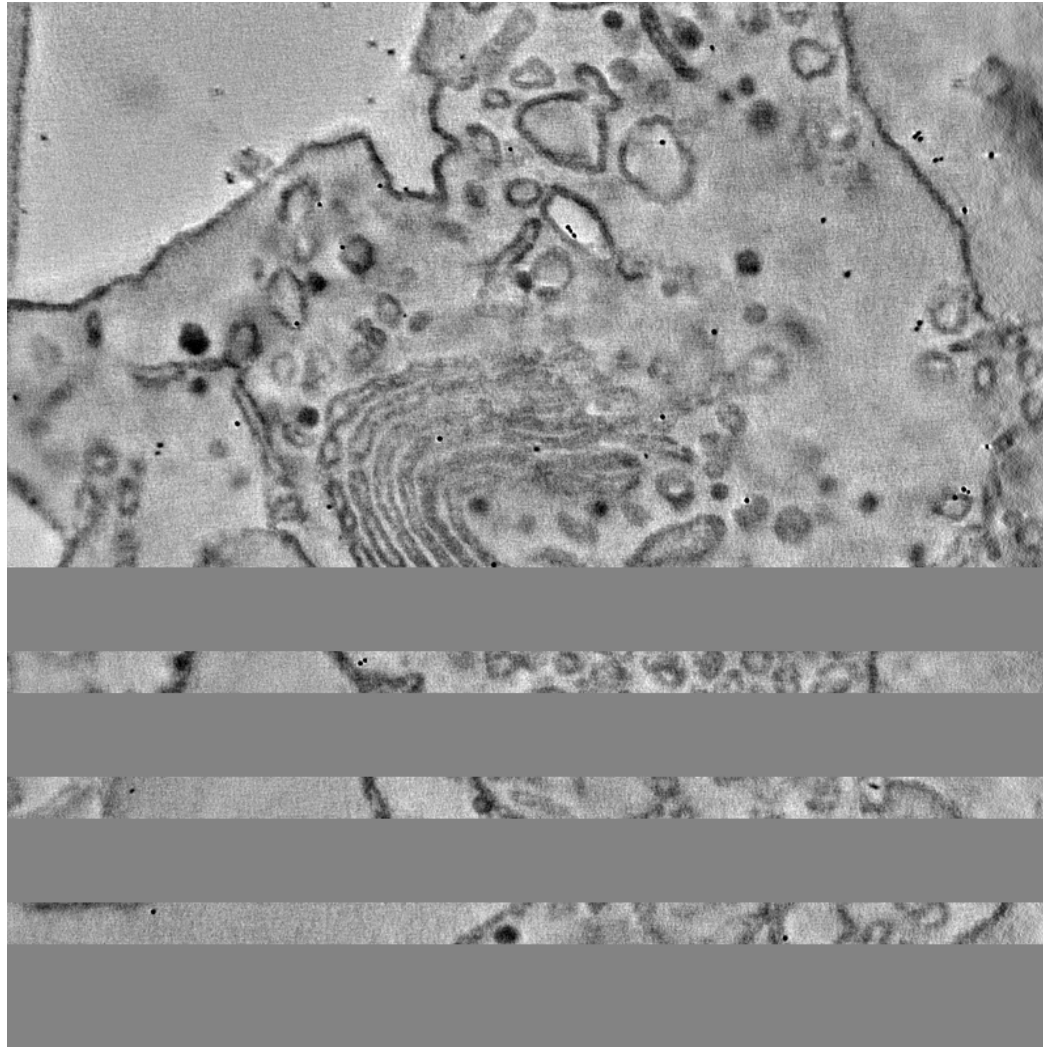
Frey TG, et al. 2006.

Annu. Rev. Biophys. Biomol. Struct. 35:199–224

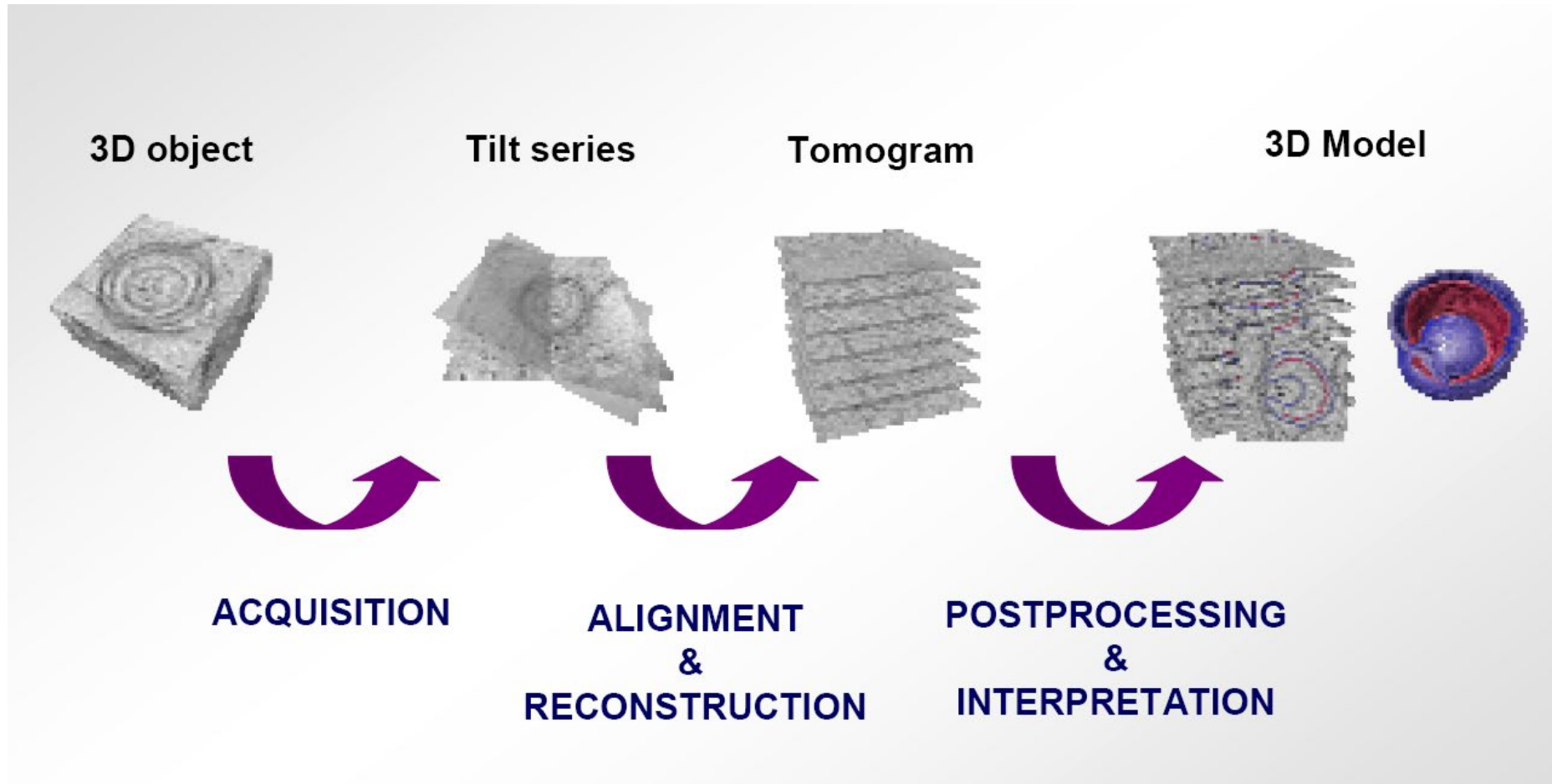
Tilt series, -60 ... +60 degree tilt



Tomogram



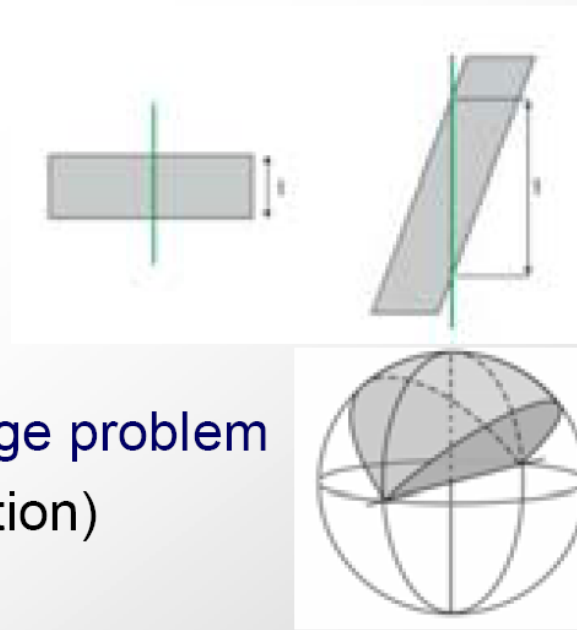
Tomo workflow



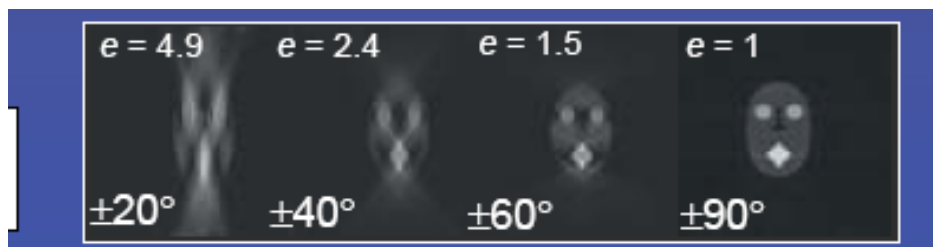
geometrical limit, the missing wedge

- There is a limit in the tilt angle we can reach ($\sim \pm 70^\circ$) due to:

- Design of the holder
- Grid bars
- Increasing thickness of the specimen with high tilt angles



- This is known as the **missing wedge problem** (missing information, loss of resolution)



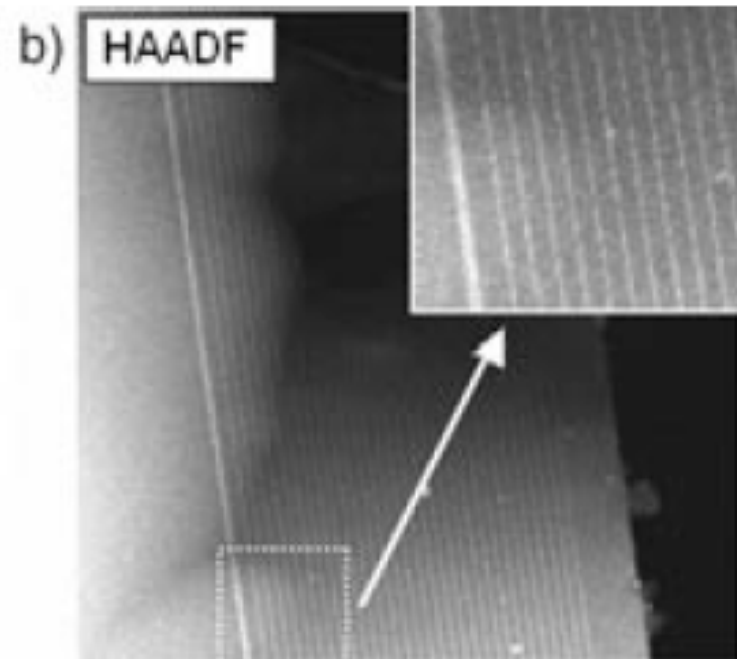
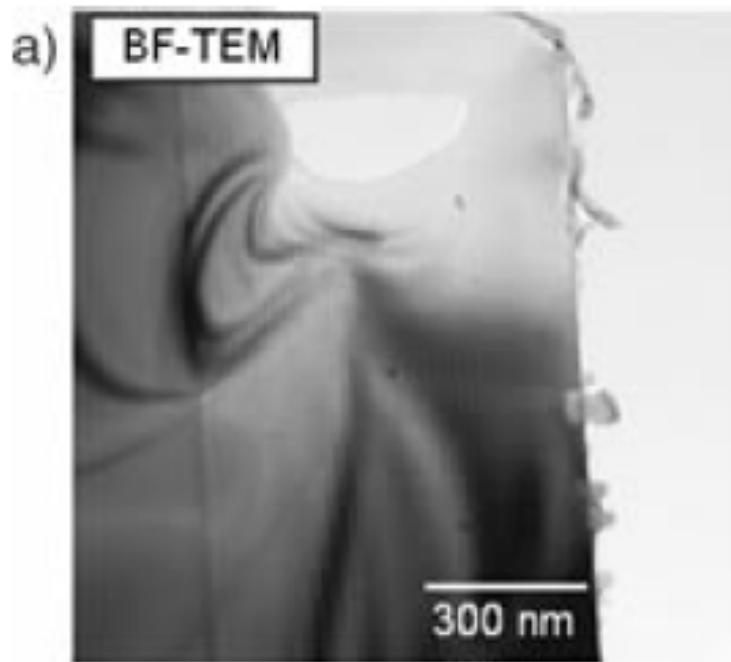
projection requirement

Radon: projection requirement: **monotonically varying** function of a physical property: mass-thickness dominant in biological samples !

Diffraction contrast in BF TEM not suitable

Diffraction contrast ☹

Z- contrast ☺

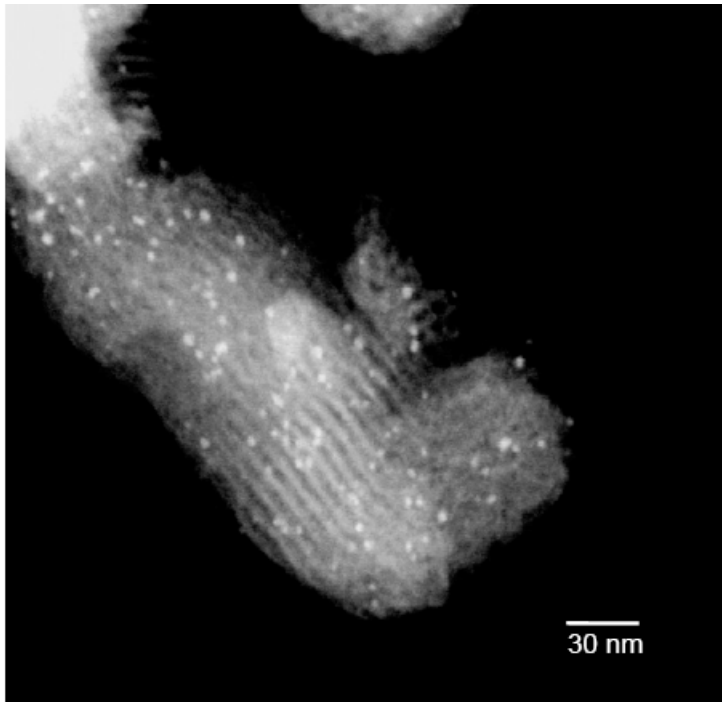


Si-Ge multiple quantum well structure

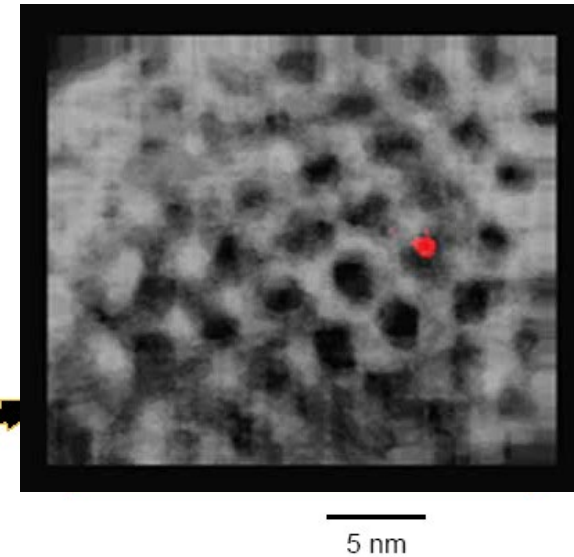
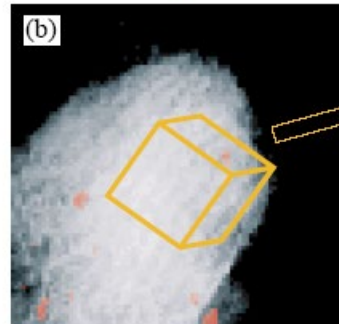
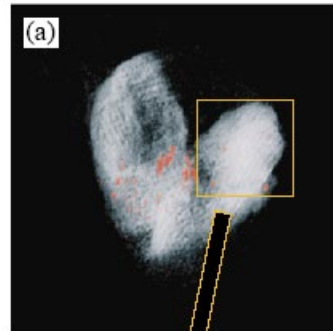
Tomography with HAADF (z-contrast)

nanoparticle bimetallic catalysts
supported on mesoporous silica

*Dogan Ozkaya, Paul Midgley;
Catalysis Letters 60 (1999) 113–120*



STEM HAADF: heterogeneous catalyst
composed of Pd_6Ru_6 nanoparticles (~ 1 nm)
on mesoporous silica support with
mesopores of ~ 3 nm diameter.



Pd_6Ru_6 nanoparticles
anchored to the wall
of mesopore

Electron Tomography in Materials Science

- Acquisition and alignment of typically 2x71 tilted images
- Back-projection: missing wedge
- Projection requirement (image contrast)
- Thickness limitation (samples rather thin)
- ...
- FIB/SEM Nano-Tomography