X-ray Imaging for Nanoscience: Opportunities and Challenges

NNI Workshop on X-rays and Neutrons:
Essential Tools for Nanoscience

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June 16-18, 2005, Washington DC

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Opportunities

- Background
  - X-rays are important (R&D spending > $600M/year and X-ray related business > $5B/year)
  - Seeing is believing (>$3B/year market for imaging tools)
  - X-Ray Based Techniques are Powerful structure, chemistry, crystallography, texture, magnetism ----
- Opportunities
  - Nanoscience and nanotechnology requires new tools with a resolution approaching 1 nm
  - X-ray imaging offers powerful and unique capabilities by adding spatial resolving power to well established x-ray techniques

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Important parameters of Imaging

- Resolution
- Exposure time
- Desired Information
- Sample requirement

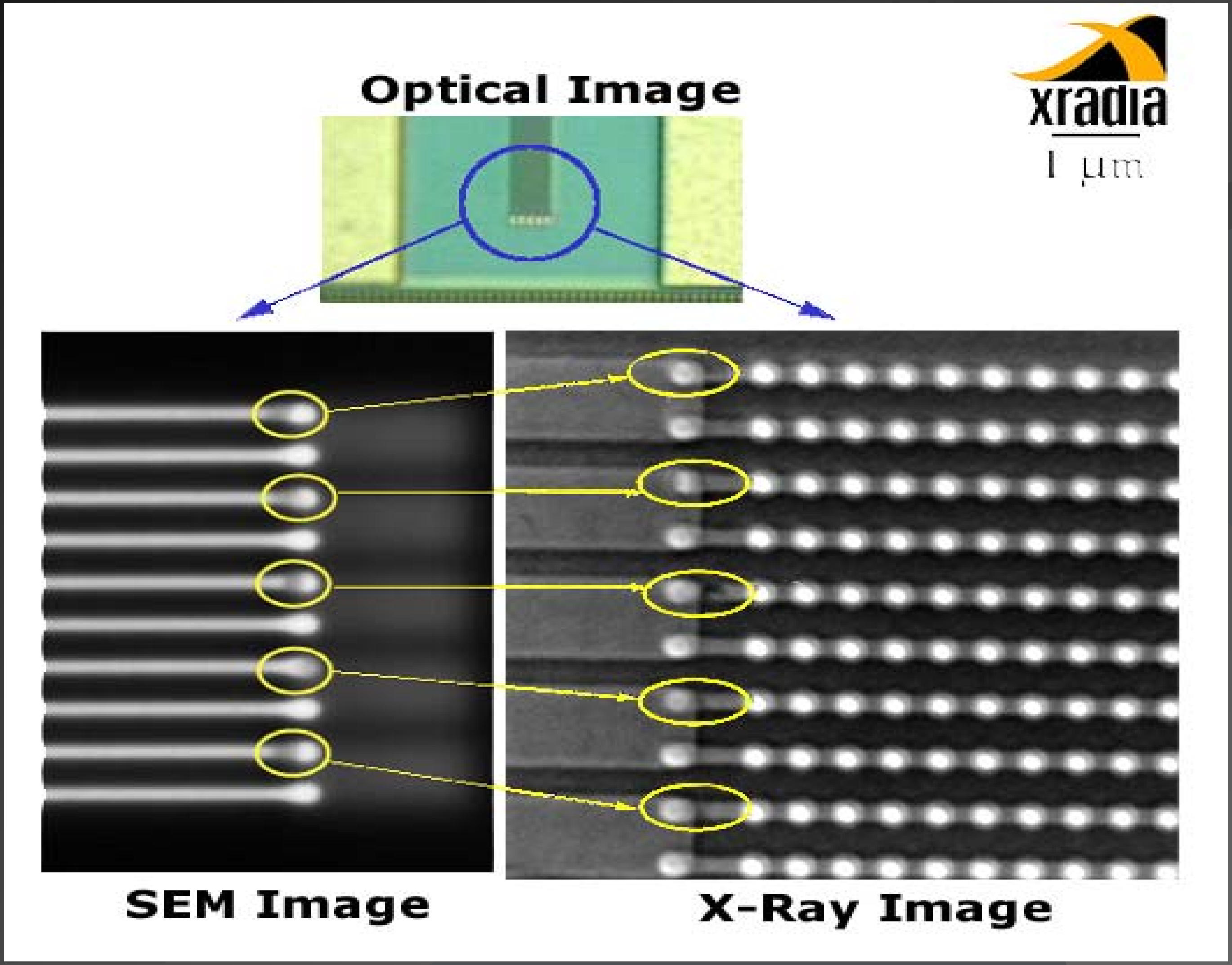
Generation Definition of Imaging?

- Spatial distribution (2D&3D) of specific information about an object, e.g.,
  - Structure
  - Electronic states
  - Chemistry (composition and chemical states)
  - Crystalinity
  - Texture
  - Magnetism
  - Strain
  - -------

Comparison of Imaging Tools

Feature	X-ray	Optical	SEM	TEM
Spatial resolution	Current: 15 -25 nm Ultimate: ~1 nm	200-300 nm	1-10 nm typically >500 nm for elemental analysis	0.1 nm
Information Achievable	Outstanding	Good	OK	Good
Probing depth	1-50 μm for E<10 keV, 20 mm for E=100 keV	<100 nm for metal, Opaque or transparent materials	< 10 nm typically < 3000 nm for element analysis	<100 nm
Material class	Insulator, semiconductor, and conductor	Insulator, semiconductor, and conductor	Conductive path required	Conductive path required
Vacuum requirement	No	No	Yes	Yes

The X-ray Advantages: High resolution+3D information

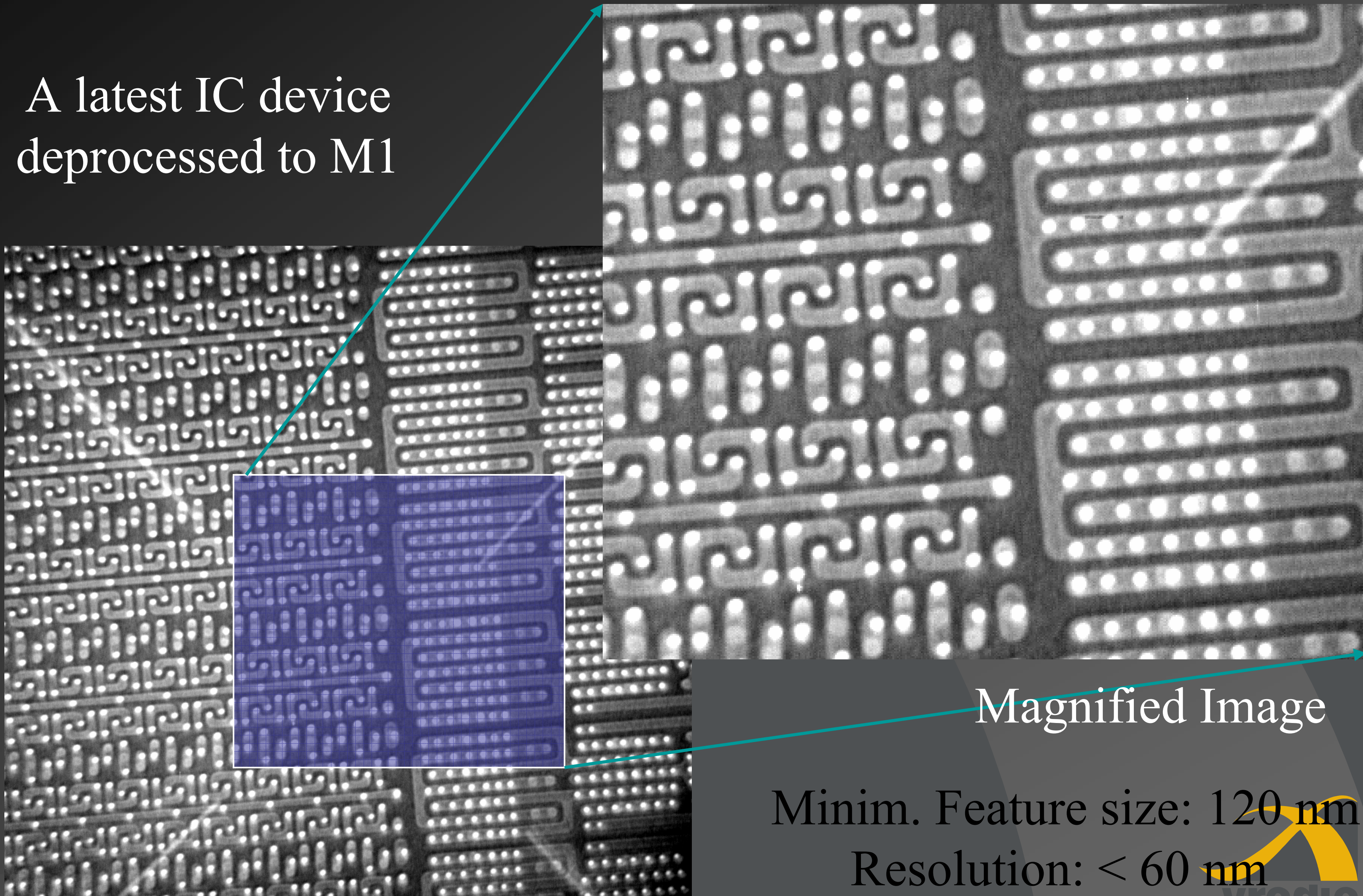


Advantages of X-ray Imaging

- High penetration for nondestructive and in-situ imaging
- Short wavelength for high resolution
- Rich image contrast mechanism: absorption, chemical state, phase, diffraction, polarization
- Element specific: absorption edges and characteristic fluorescence
- No charging effect on image resolution

A X-Ray Image from NanoXCT-60

A latest IC device deprocessed to M1

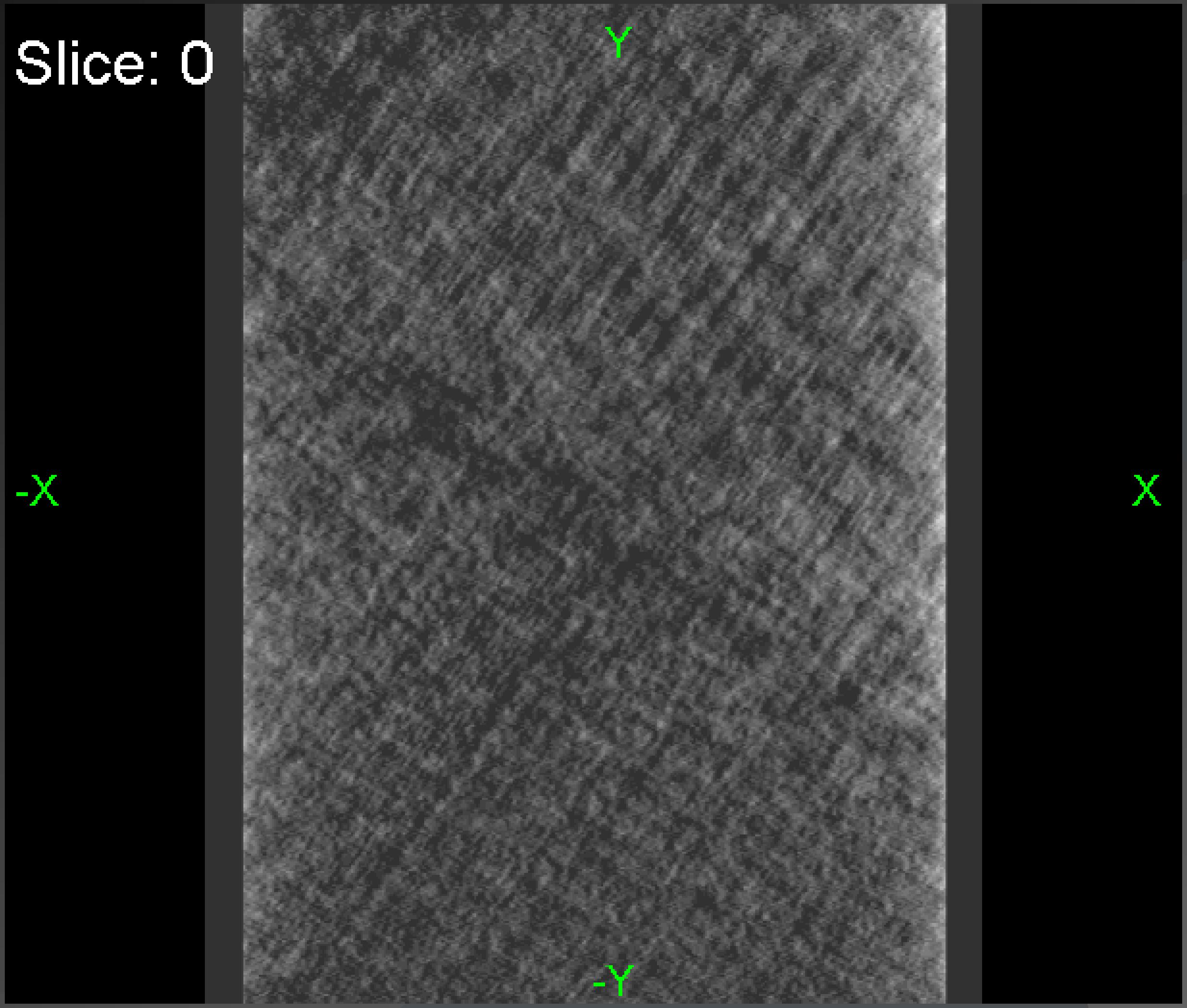


Magnified Image

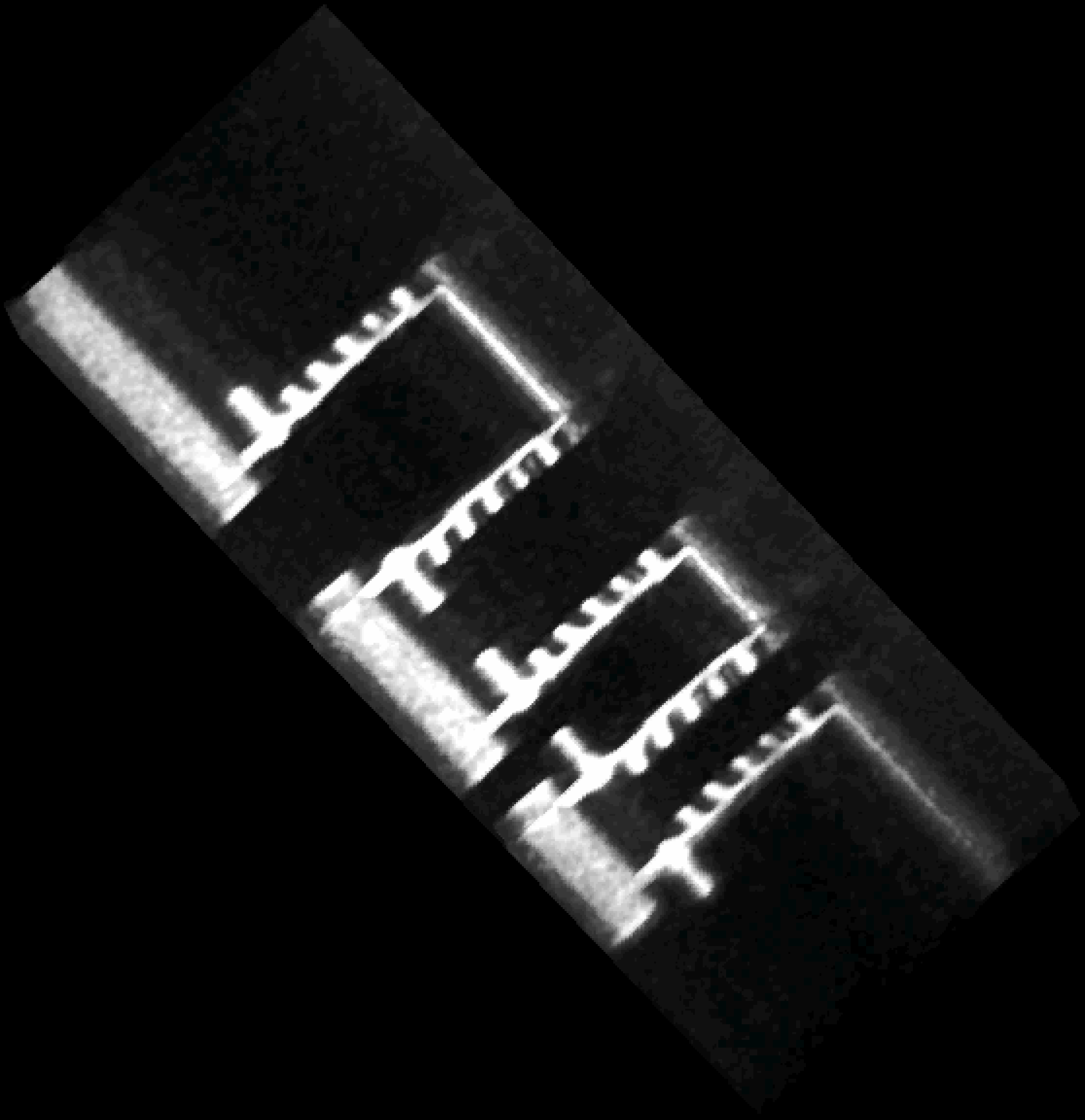
Minim. Feature size: 120 nm

Resolution: < 60 nm

Virtual Slices through the XY Planes of a Reconstructed Tomography Image of an IC Device

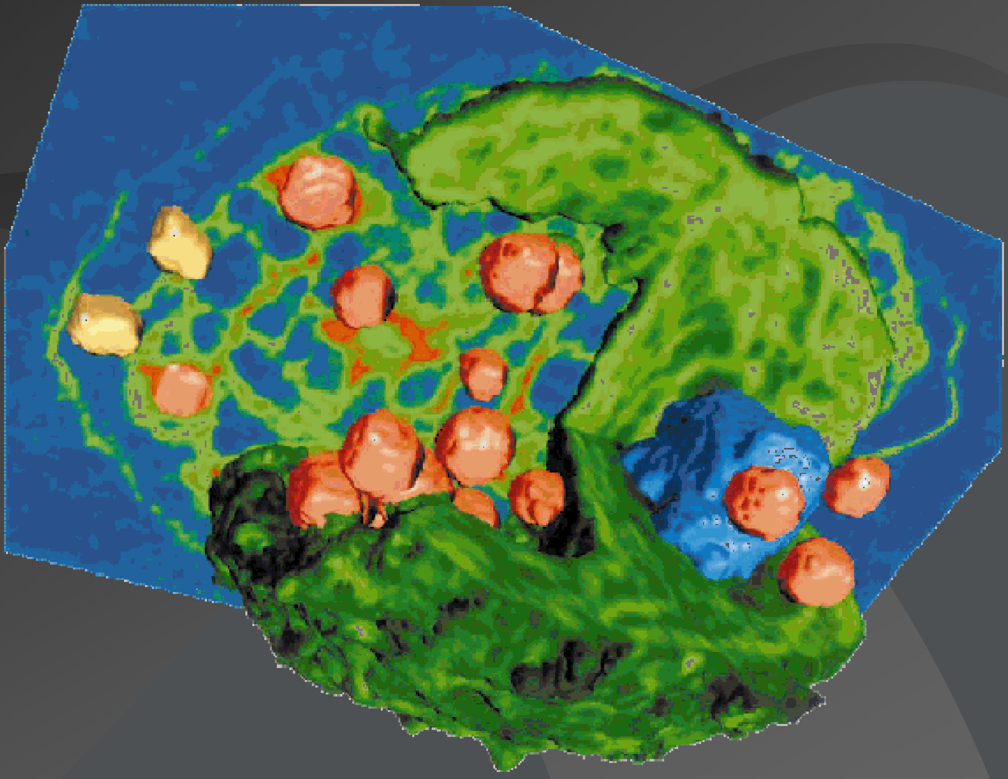


A Rendered 3D Image of a 8-level Cu Device Showing Electromigration Defects



Tomography in a TXM

- TXM is *much* faster for tomography!
- D. Weiß *et al.*, *Ultramicroscopy* **84**, 185 (2000). See also G. Schneider *et al.*, *Surf. Rev. Lett.* **9**, 177 (2002)
- *Chlamydomonas reinhardtii*, frozen in liquid nitrogen.
- Rendering: organelles highlighted by optical density



Requirements and Challenges for X-ray Imaging Microscope with 1 nm Resolution

Requirements for high resolution x-ray imaging

- Adequate image contrast
- Sound methodology: many well established concepts
- Bright source: available, adequate, and improving
- High resolution objective lens: conceptually possible for sub-10 nm but far from reality

Major challenges:

- High resolution objective lens: Tremendous effort is required to produce 1 nm resolution objective lens with an acceptable efficiency
- High NA condenser: generally more challenging to make
- High speed x-ray detector for synchrotron sources
- Tomography: image alignment, imaging system stability, depth of focus
- Cryo?

How Small Can be Imaged with X-rays

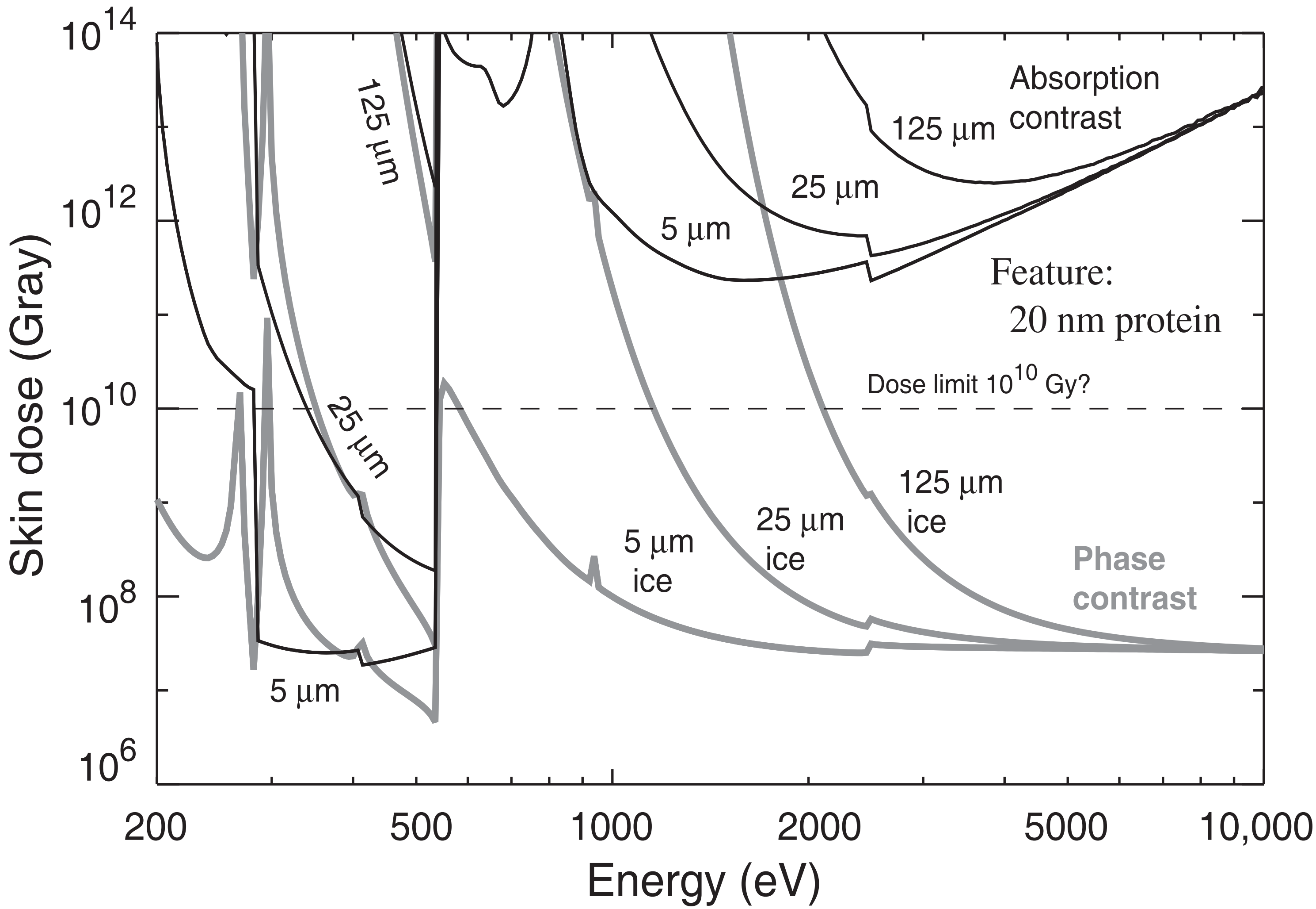
- Exposure time T ~ $10/K^2$

  K = image contrast ~ 1/t , t = thickness of a feature

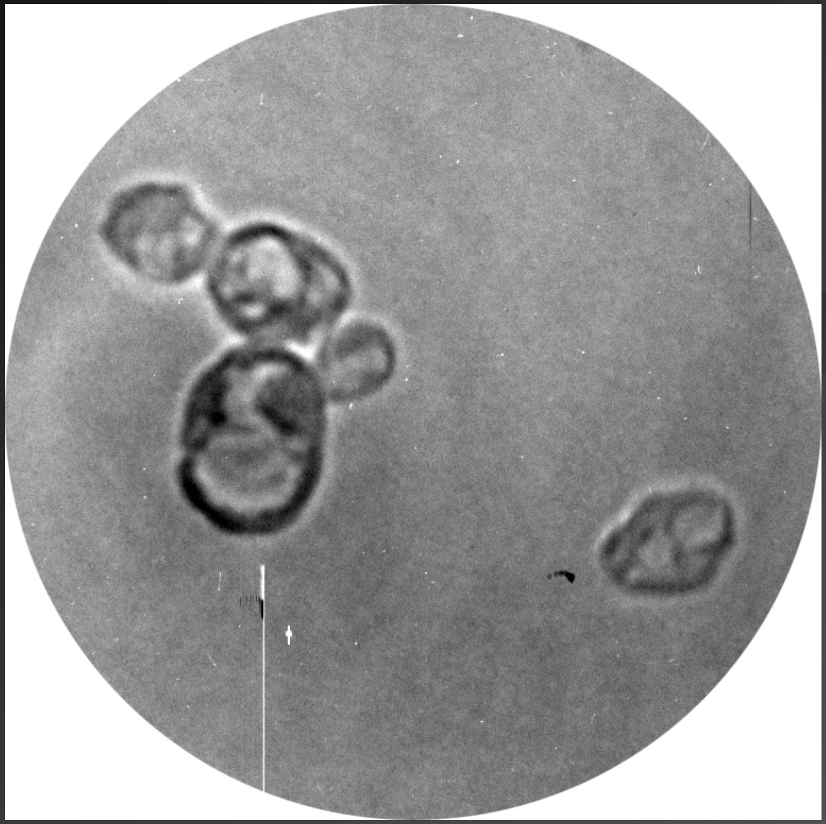
- Phase image contrast of 1 nm features @1.8keV

Material	Au	Cu	Si
Contrast %	1	0.5	0.1

Imaging Large Biological Specimens with Hard X-rays



Phase Contrast Image of Biological Specimens



Phase contrast image of yeast cells (a) and 3T3 fibroblast cell (b) using TXM 54-60

Performance Comparison of X-ray Focusing Optics

	KB Mirror	Refractive Lens	Zone Plate
Resolution (nm)	40-60	110	15 for 0.5 keV 25 for 4-20 keV
Flux Density Gain	>500,000	10000	>500,000
Coherence preservation	Acceptable ?	Acceptable?	Good
Imaging Optic	No	Yes	Yes
Chromatic Aberration	No	$1/\lambda^2$	$1/\lambda$
Theoretical resolution limit (nm)	~10	10-20	~1
Mini. focal length for 100 $m\mu$ aperture (mm)	~30	100 (?) for 10 keV	0.5 for 0.5 keV 20 for 10 keV

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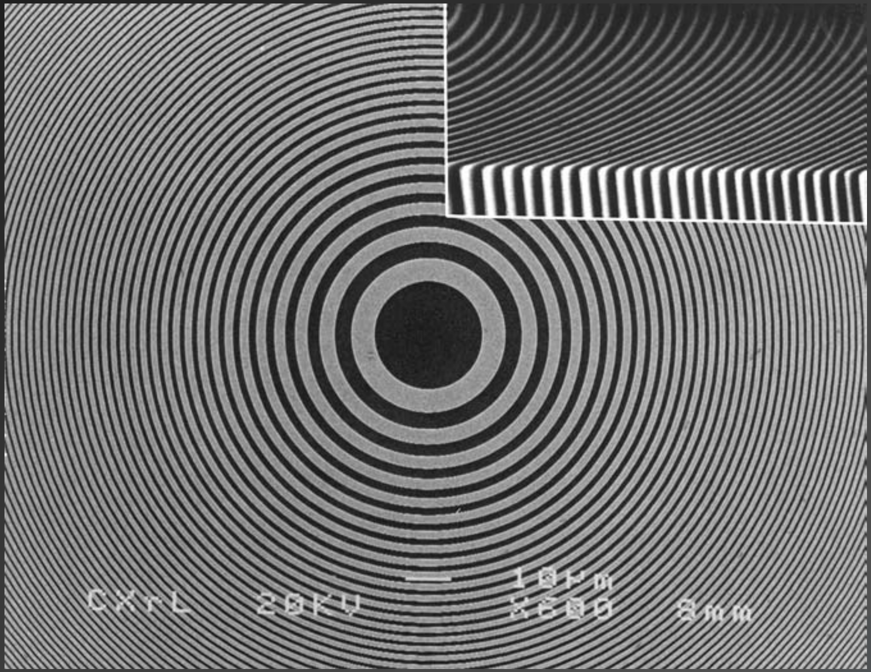
Zone Plate's Key Parameters

Zone plate consists of concentric rings (zones) with zone width decreasing with radius



SEM Image of a ZP and its zone profile

Key parameters:

Number of zones
- > 100 required for good focusing

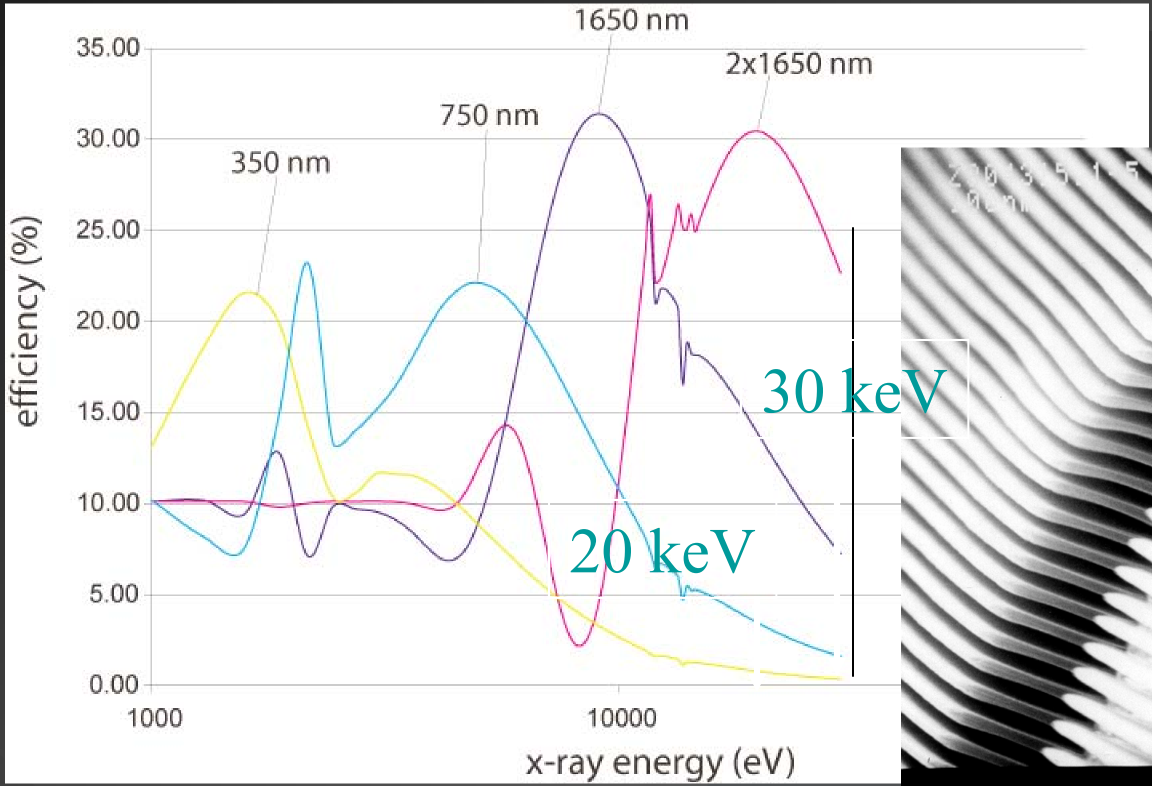
Outermost (smallest) zone width
- Determines resolution and NA

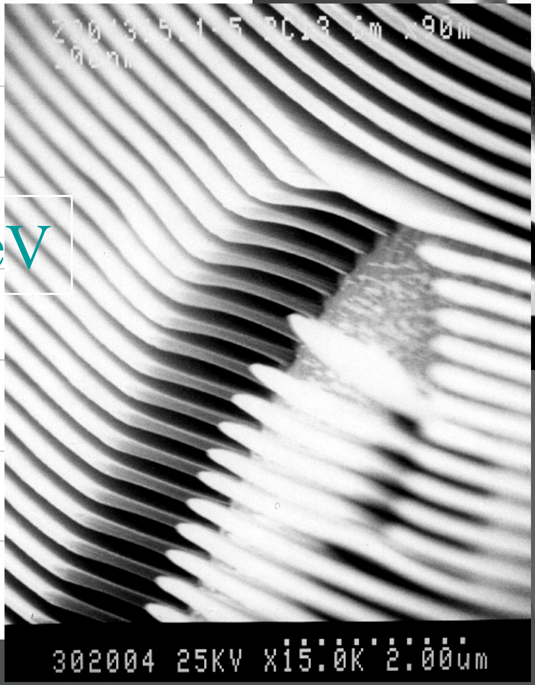
Zone materials, thickness, profile
- Focusing efficiency

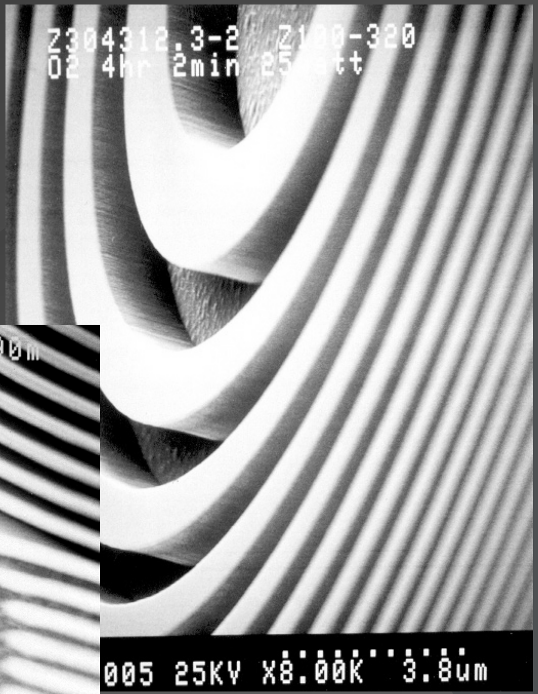
Focal length
- Working distance

Zone plates fabricated at Xradia

Theoretical efficiency of zone plates made by Xradia. Zone plates made by Xradia are guarantees to perform with at least 75% of the theoretical efficiency.







Center and outer zones of a zone plate with 50 nm outer zone width and 700 um thickness.

Xradia's zone plate production capabilities

Energy (eV)	Outermost Zone width (nm)	AR	Efficiency (%)	Diameter (μm)	Material
500	25	6	10	80	Au
1500	35	12	21	80/320	Au
5400*	35	20	17	80/320	Au
9000	50	18	16	100	Au
9000	100	16	31	160/320	Au
9000	100	16	31	4000	Au

Note:
1, Xradia warrants >75% of calculated focusing efficiency for its zone plates
2, Xradia is currently developing zone plates with finer zones and large diameters,
3, Xradia is developing zone plates for 50-100 keV applications

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Path to sub 10-nm x-ray imaging

- Improving zone plate fabrication technology
- Using higher order diffraction m

  $\delta = 1.2\ \Delta R/m$ ΔR = the outermost zone width

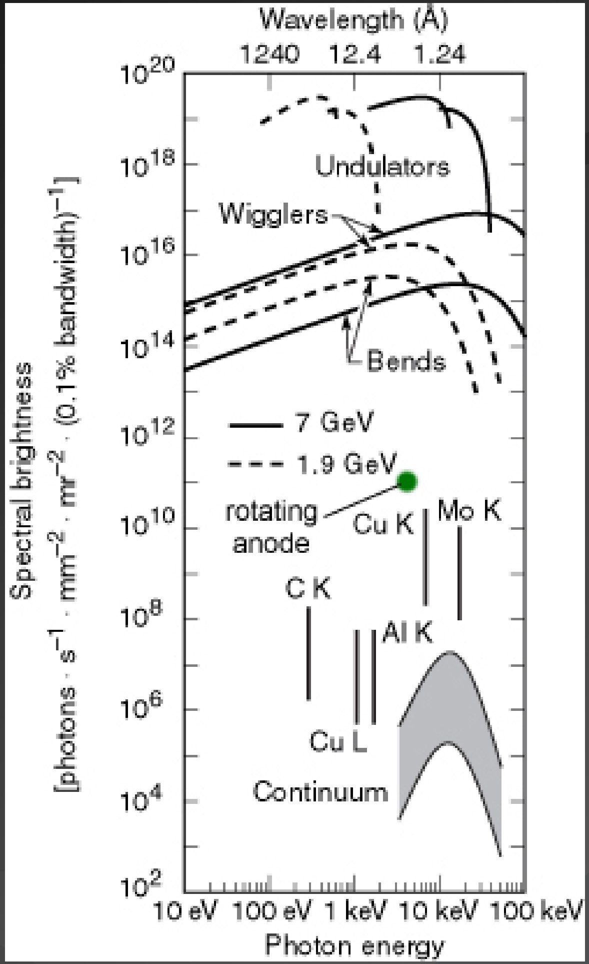
  Example: δ = 20 nm for ΔR=50 and m = 3
  = 12 nm for ΔR=30 and m = 3

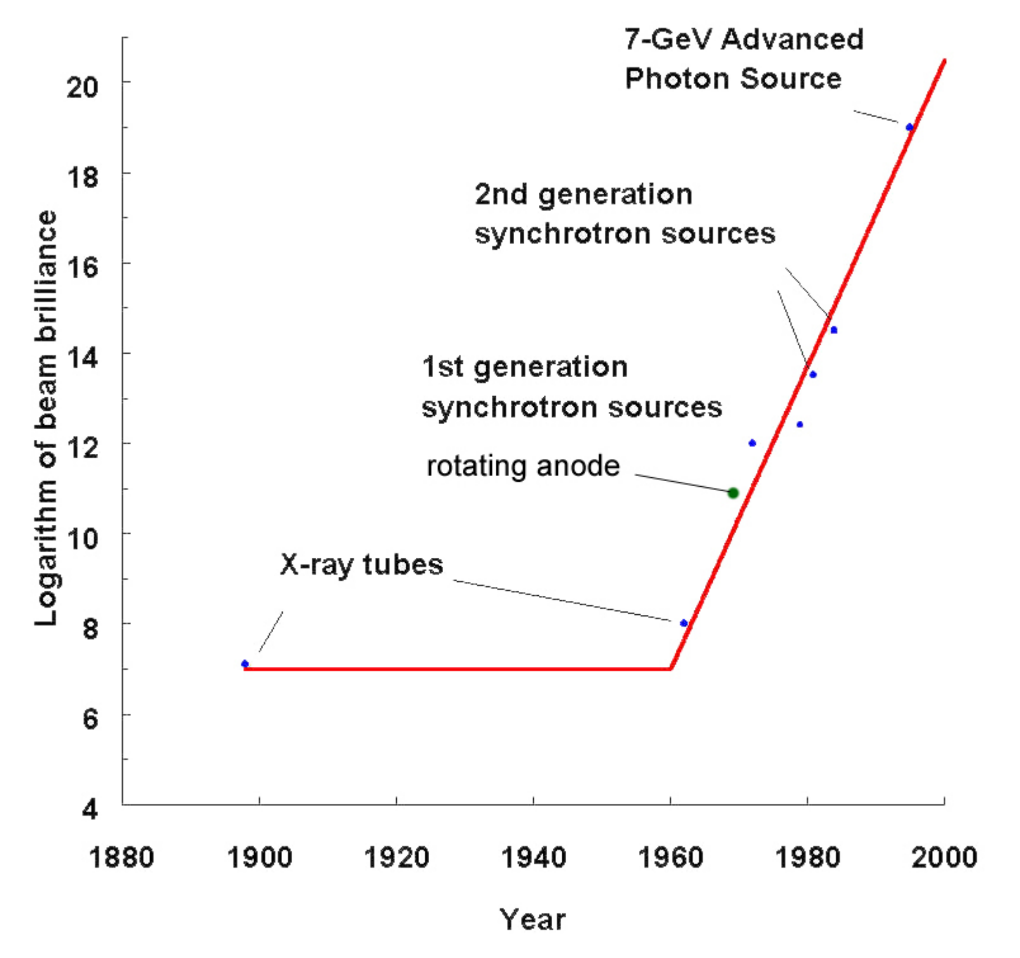
  Note:

  1. m > 3 can be used to get better resolution
  2. Focusing efficiency ~ $1/m^2$, acceptable for synchrotron sources

System Components – X-ray Source

X-ray source of TXM 54-80:	A commercial rotating anode source from Rigaku with Cr anode - 5.4 keV x rays from Cr *K*α-line.





Summary Remarks

- X-ray imaging offers important and unique characterization capabilities to nanoscience, e.g. in-situ nondestructive 3D imaging
- X-ray microscopy is evolving rapidly in recent years
- ~1nm resolution is achievable in principle but requires substantial development and a lot of investment, recommending collaboration between RD labs. and industry.