Liquid-jet/droplet x-ray sources for laboratory nano-scale bio imaging

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AND

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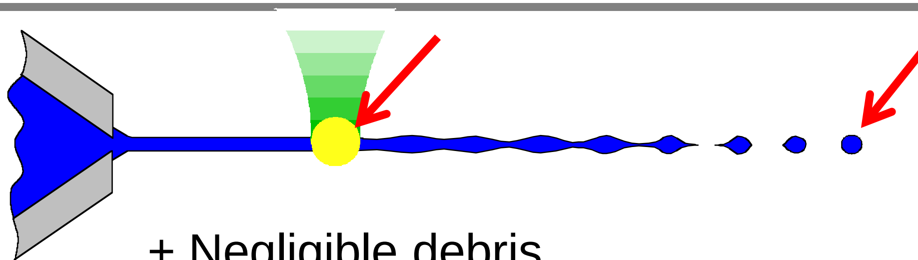
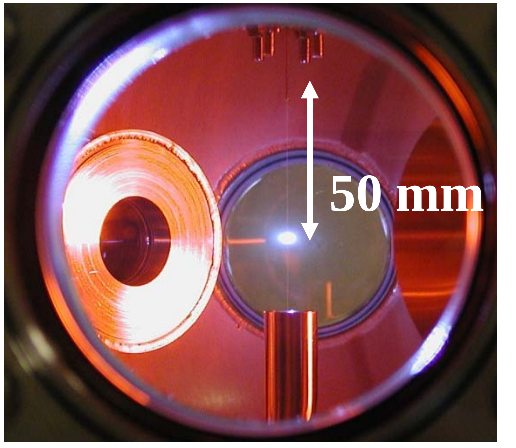
Excillum AB, Stockholm: O. Hemberg, M. Otendal, T. Tuohimaa et al
Karolinska Inst., Stockholm: M. Vita and M. Henriksson
Uppsala Uni: S. Svärd, J. Jerlström-Hultquist
FhG, Jena: S. Yulin et al
ILT, Aachen: D. Esser, M. Hoefer et al
MBI, Berlin: H. Stiel et al
X-Ray Physics, Göttingen: J. Thieme et al
UoB, Barcelona: J. Fernandéz-Varea

Laboratory soft x-ray sources:

Liquid-jet/droplet laser plasmas

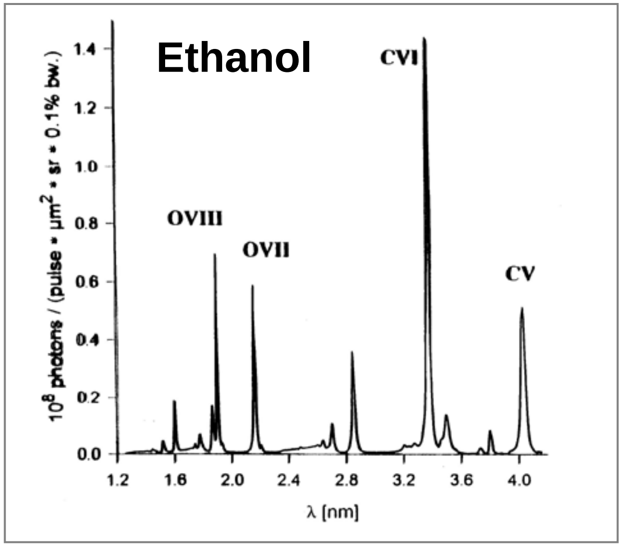




+ Negligible debris
+ Regenerative target
+ High rep.-rate operation
+ High-power operation possible
+ Tailored spectral emission

Rymell et al, Opt. Commun. (1993); Malmqvist et al, RSI (1996)

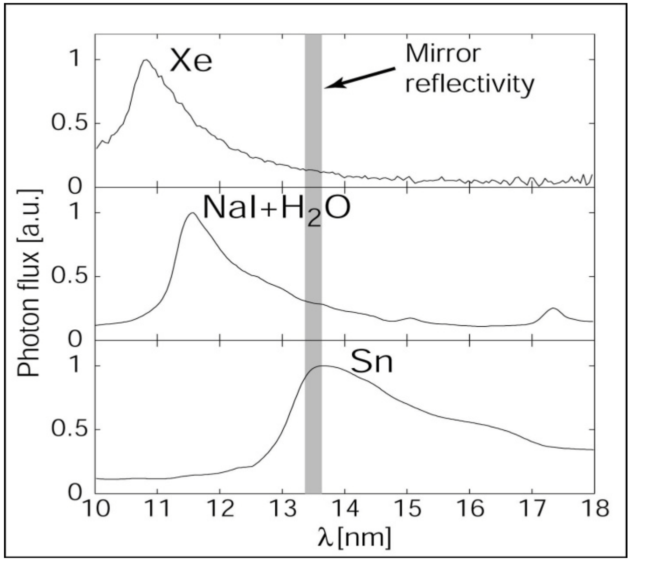
$\lambda \approx$2-4 nm: Water-window



+ Line emission $\Rightarrow$ fixed wavelength
+ $\lambda/\Delta\lambda$>500
+ High brightness

Rymell et al, APL (1995), Berglund et al APL (1997)

λ=13 nm: EUV Lithography



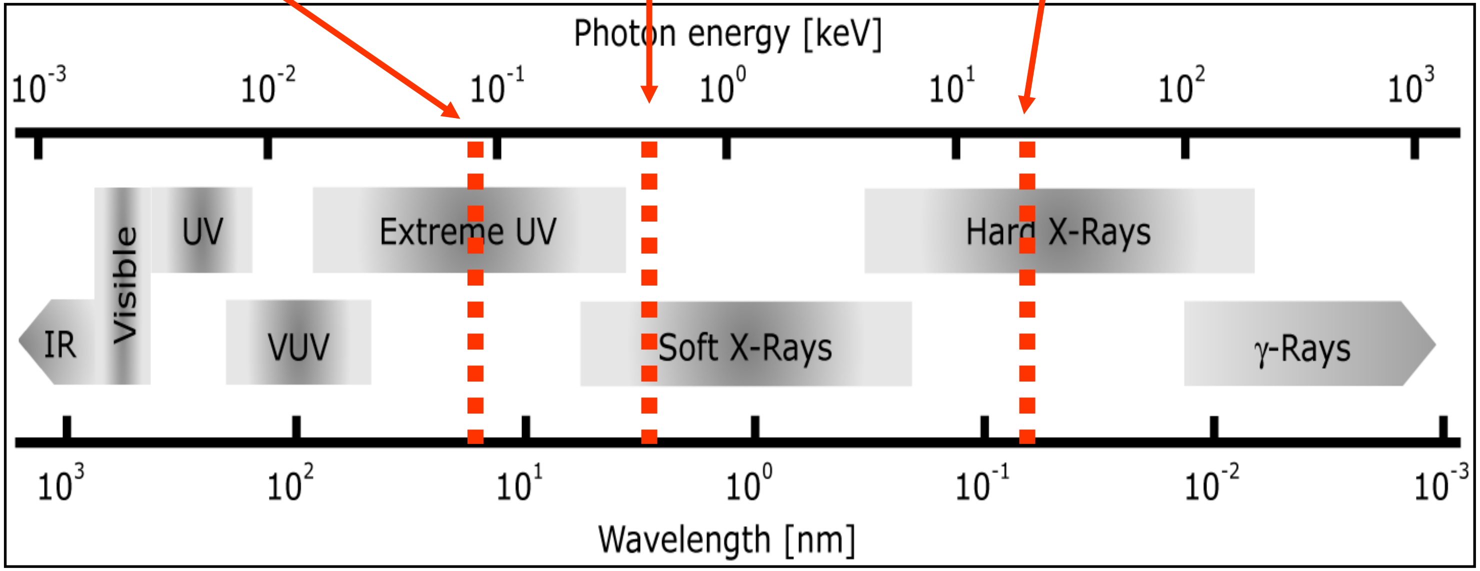
Hansson et al, MNE (2001) ; Jansson et al APL (2004)

Today:
Liquid-jet/droplet sources

2. Water window soft x-rays :
Laser-plasma liquid-jet sources
X-ray microscopy

1. EUV :
Laser-plasma liquid-jet sources
Lithography & Metrology

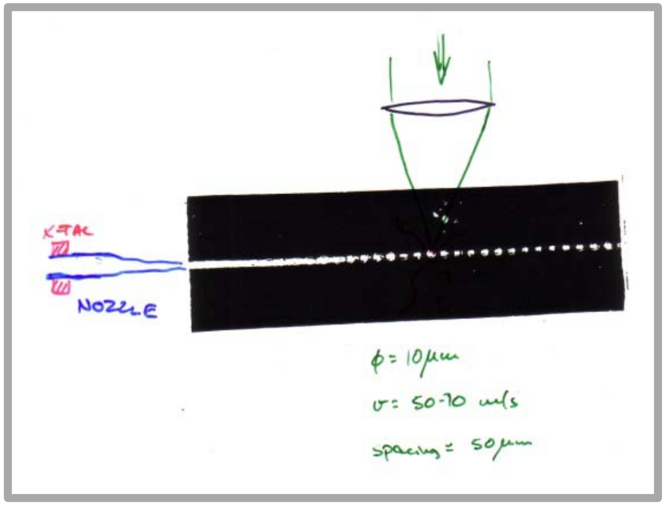
3. Hard x-rays :
Electron-impact liquid-jet-anode sources
Phase-contrast imaging



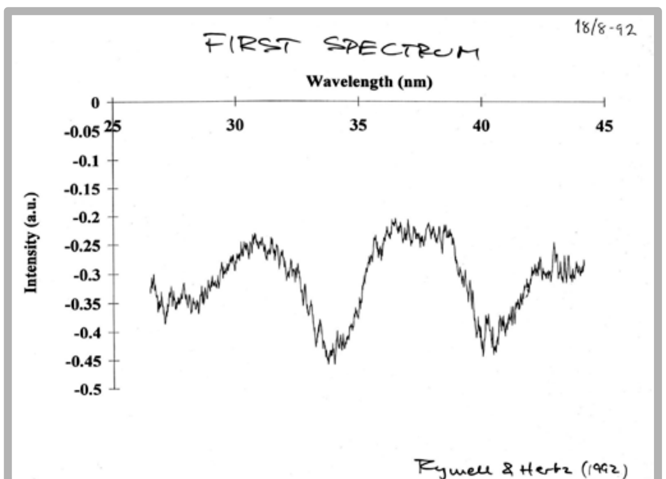
Liquid-jet/droplet laser plasma sources:

1993: Water-window & ethanol droplets

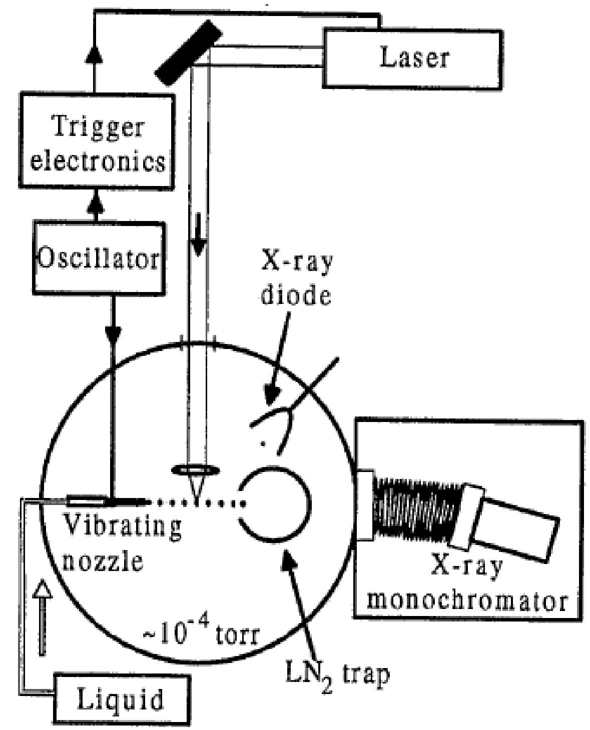
1990 : First exp'l result

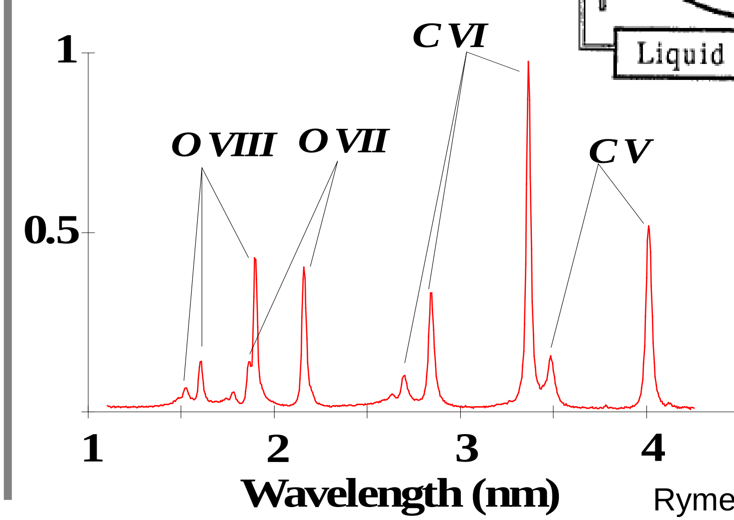


1992 : First droplet spectrum



100 ps, 70 mJ,
10 Hz Nd:YAG



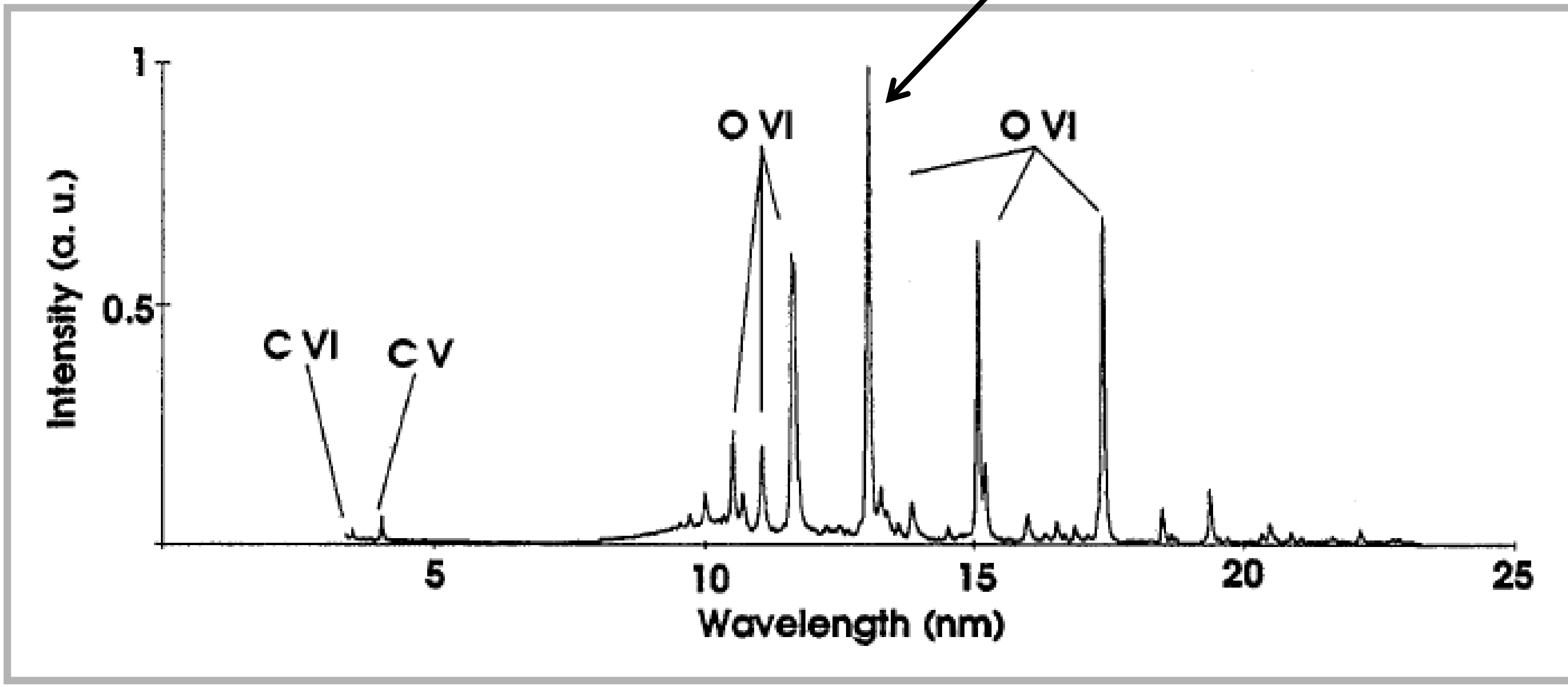


Rymell et al, Opt. Commun. (1993)

Liquid-jet/droplet laser plasma sources:

1995: EUV & water droplets

λ≈13.0 nm



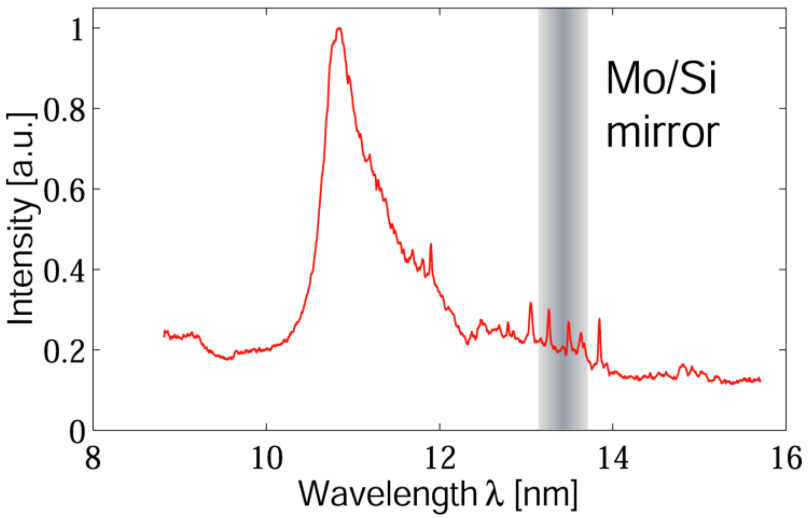
8 ns Nd:YAG

Hertz et al, SPIE **2523** (1995)

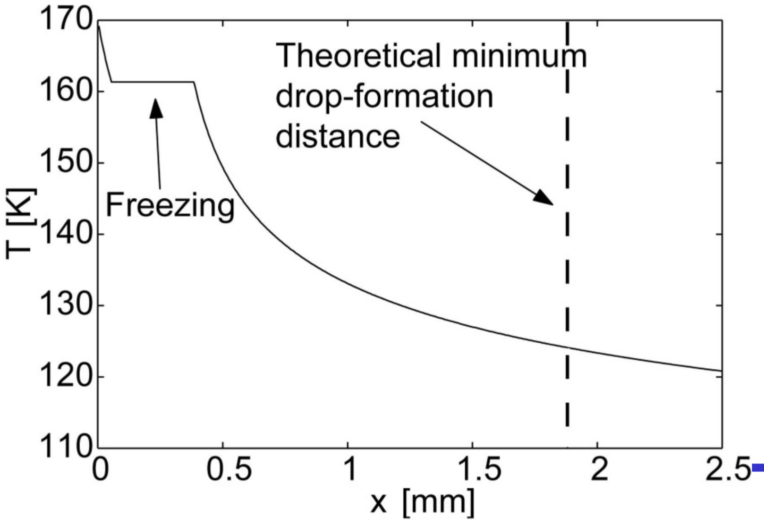
Liquid-jet/droplet laser plasma sources:

2000: EUV & xenon jet

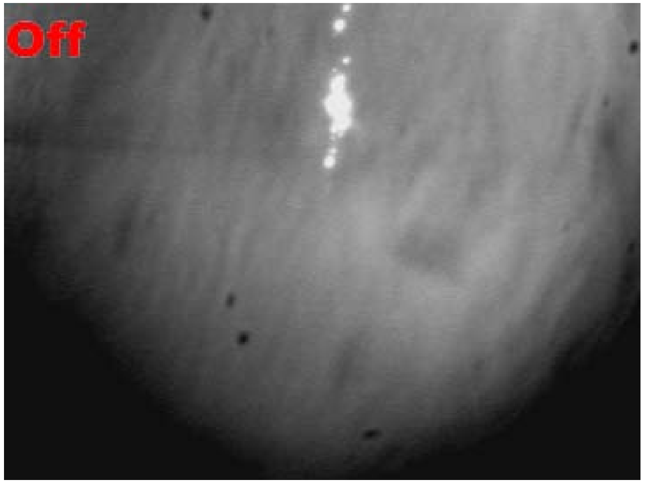
Spectrum

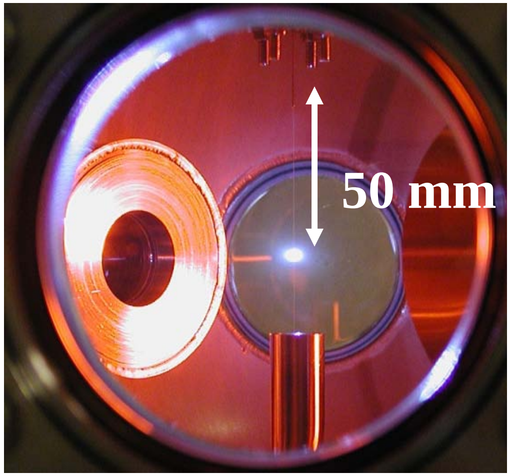


Jet cooling



Stability





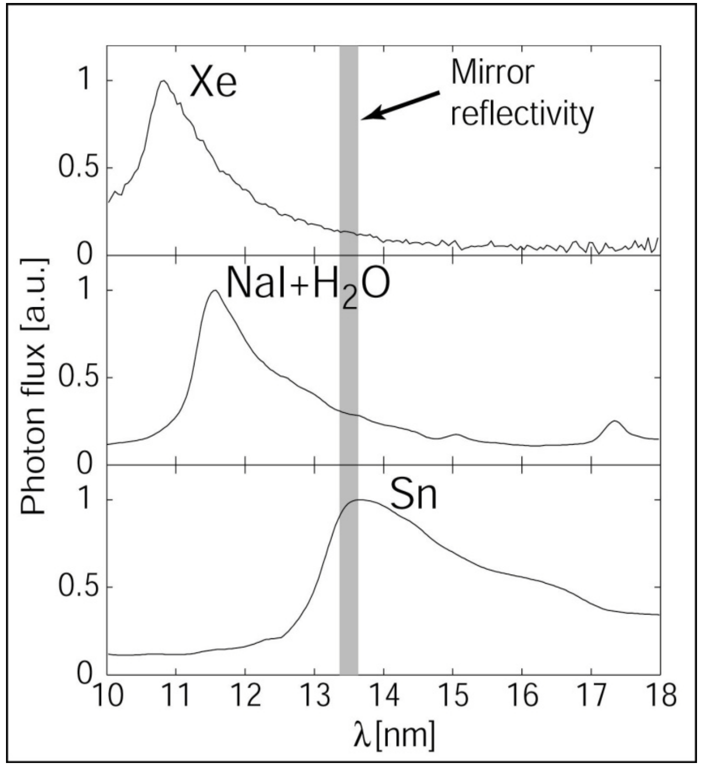
Hansson et al, Microel. Engin. (2000);

Liquid-jet/droplet laser plasma sources:

2004: EUV & tin jet

Stable jet @ >250 C

Spectral match



CE: 2.5% into ($2\%BW \times 2\pi \times sr$)

Debris:

1 h gave coating
Mitigation need: ~10^8

Jansson et al . Appl. Phys. Lett. (2004)

Liquid jet/droplet sources

2. Water window soft x-rays :

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Lithography & Metrology

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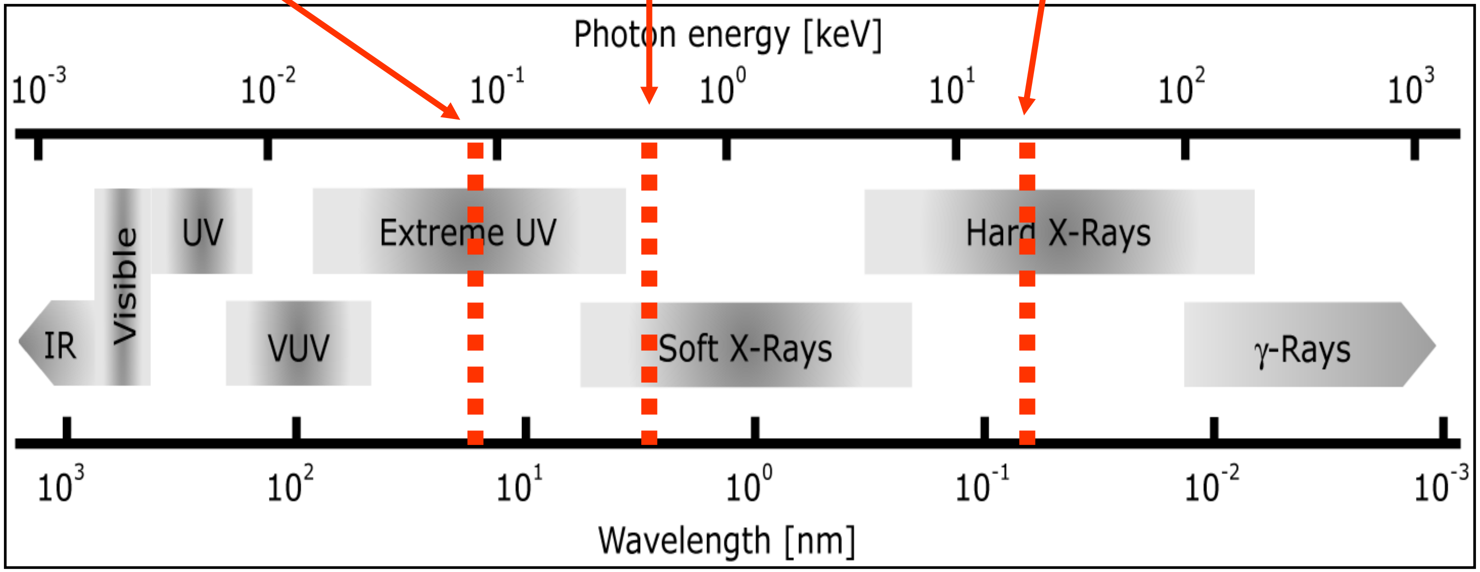
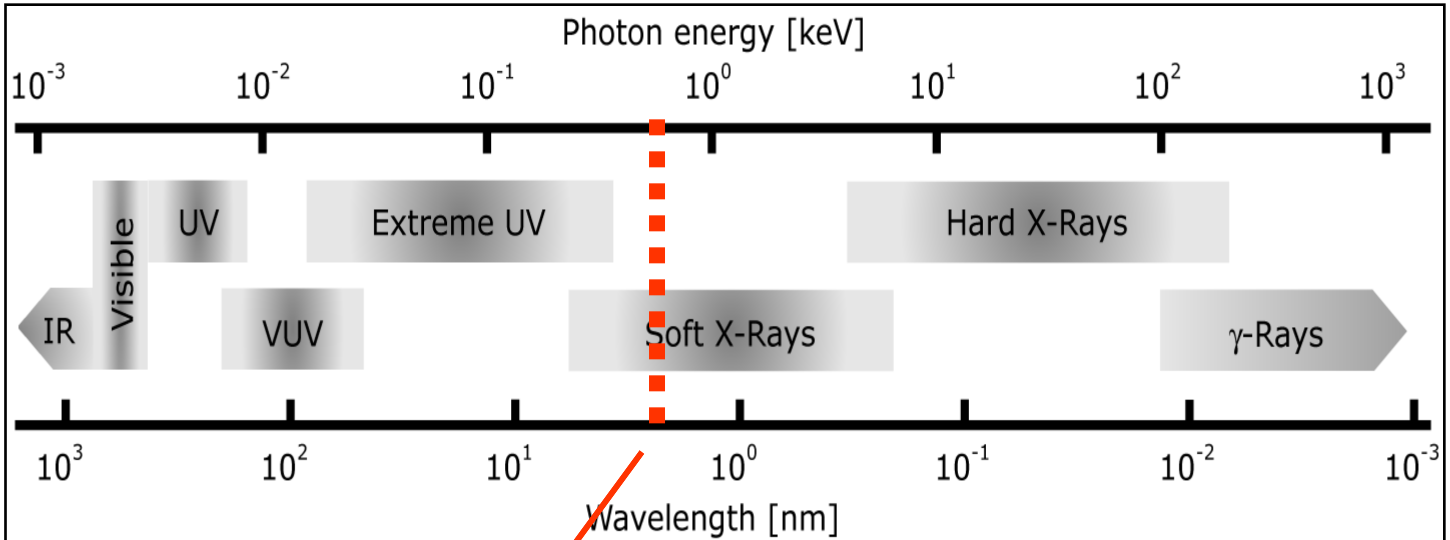
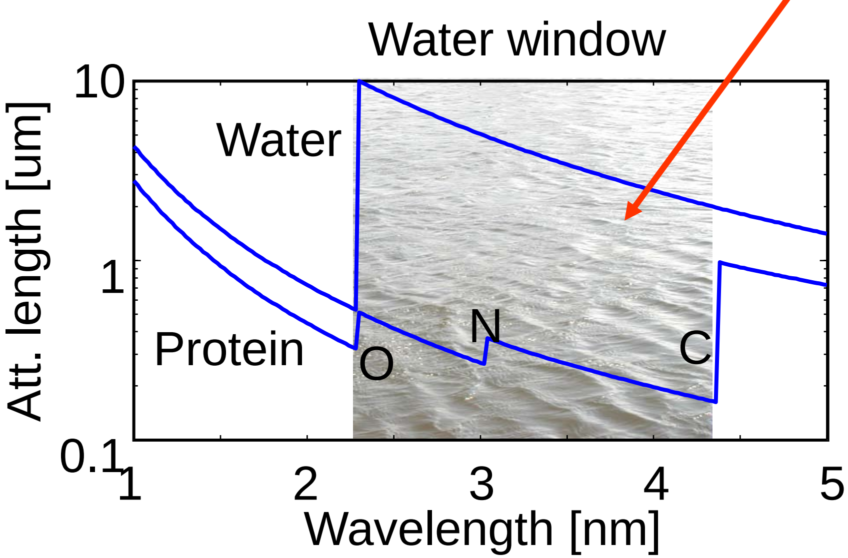


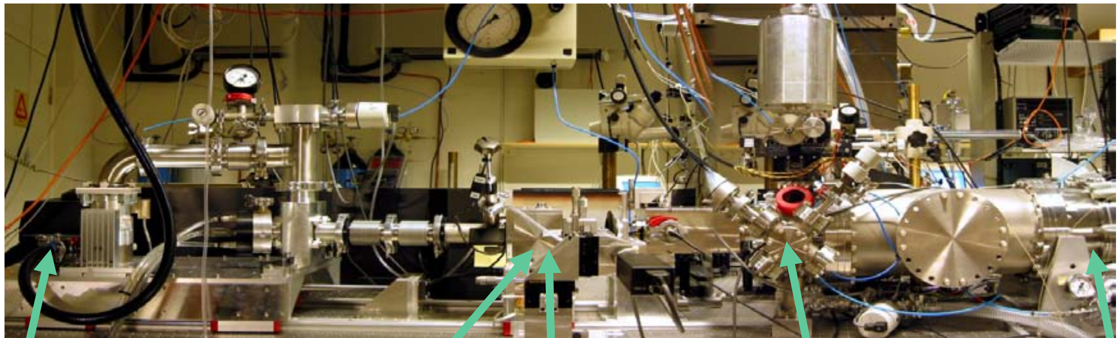
Water-window x-ray microscopy

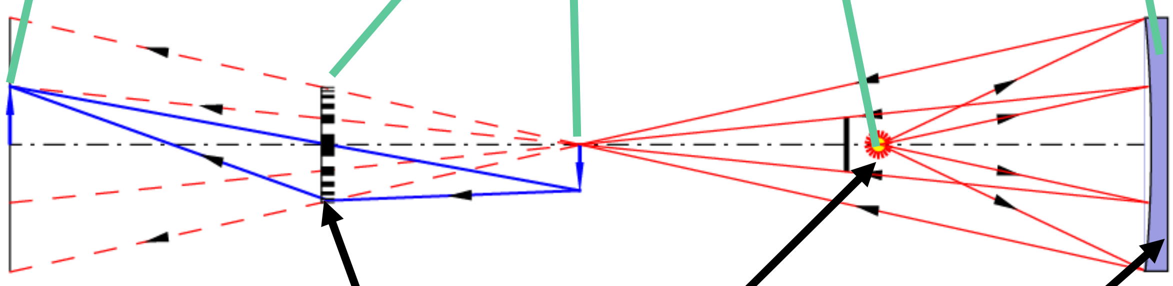




+ Resolution: $0.61\lambda/NA$
+ Natural contrast for wet/frozen specimen
+ Possibility to study thick (~10 μm) objects
- Lack of laboratory high-brightness sources
- Inefficient optics

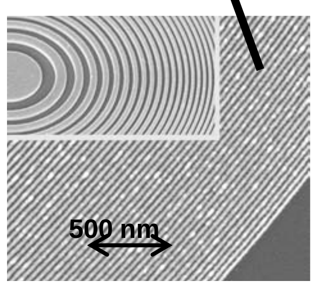
Laboratory water-window x-ray microscopy

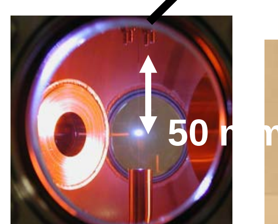


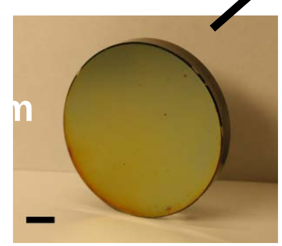


λ=3.37 nm &
λ=2.48 nm

Micro zone plates for high-resolution imaging





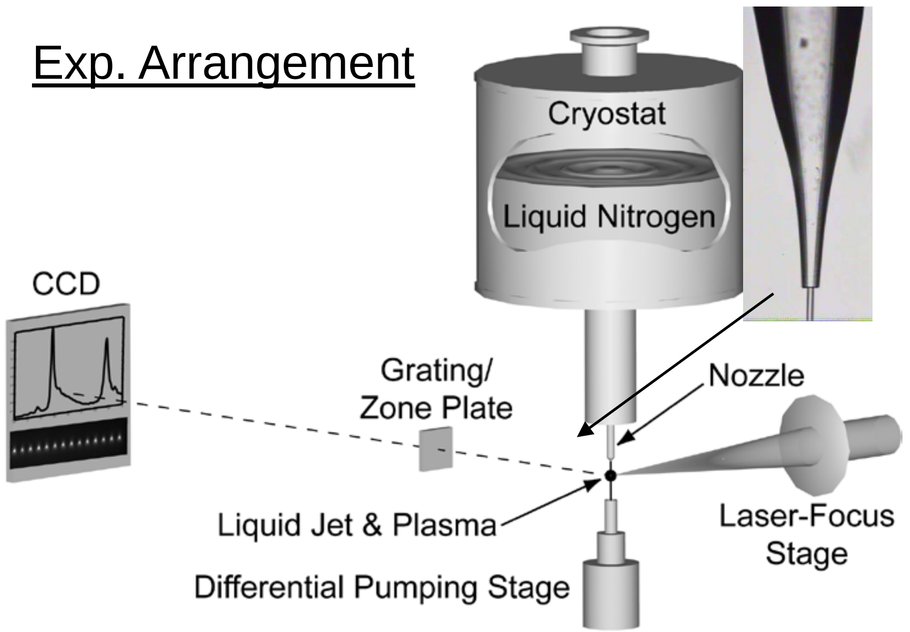


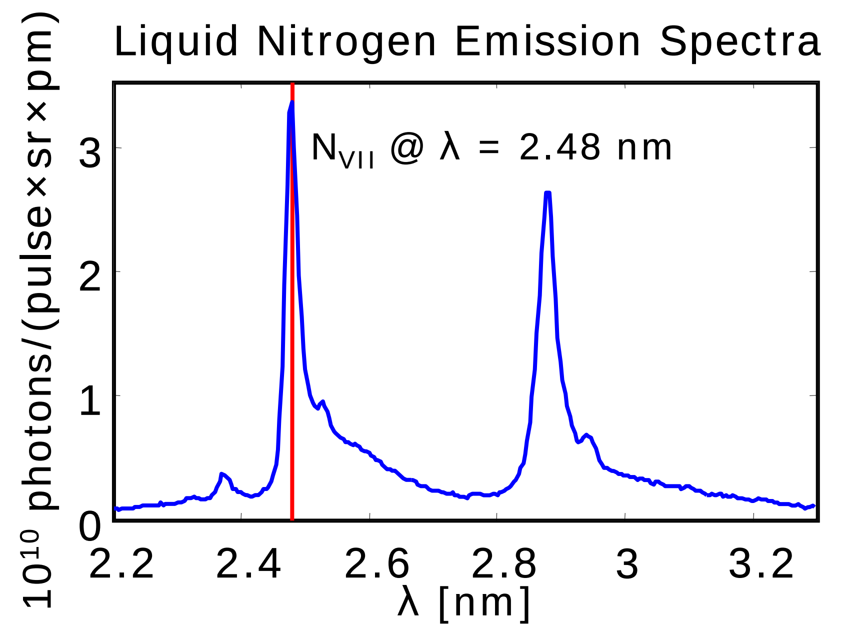
Normal-incidence multilayer mirrors as condensors

Berglund et al, J. Microsc. (2000), Johansson et al, RSI (2002) Takman et al, J. Microsc. (2007)

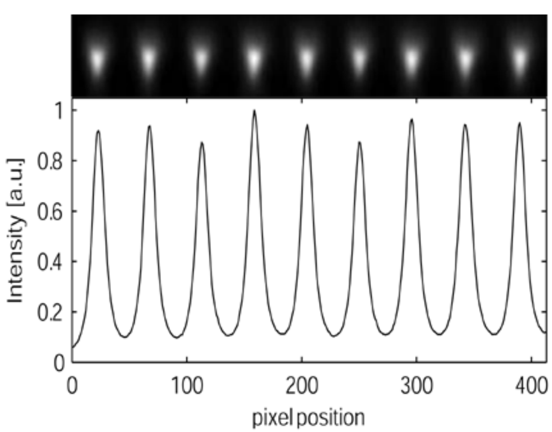
The liquid-nitrogen-jet source

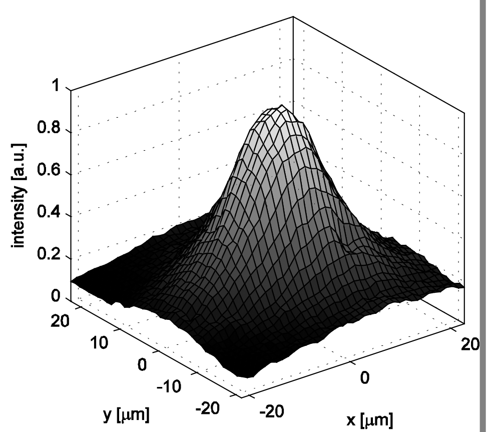
Exp. Arrangement





Stabiliy & Size



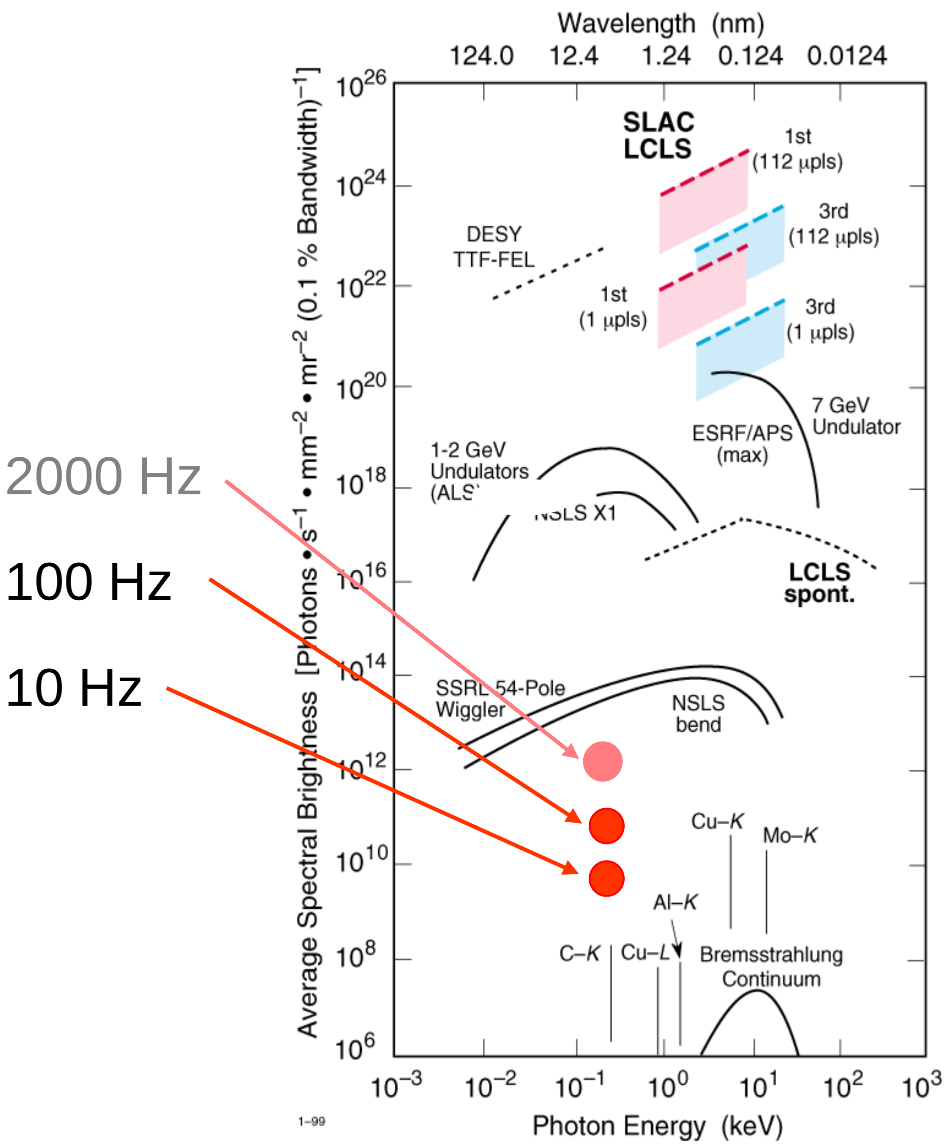


Laser: 20W, 100 Hz, 3ns

Flux: 1×10^{12} ph/pulse×sr×line
Stability: ±2 μm
Brightness: 4×10^{8} photons/(pulse×sr×μm²×line)
Illumination Uniformity:10%

Berglund et al, RSI (1998), Jansson et al RSI (2005)

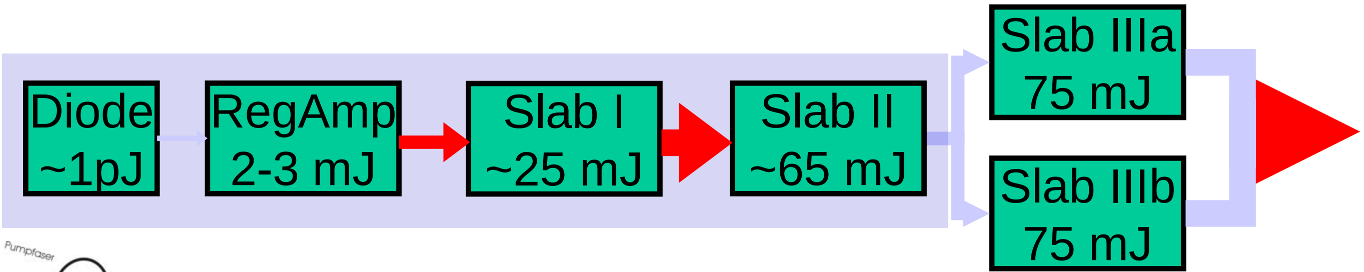
Liquid-jet laser-plasma sources:
Brightness

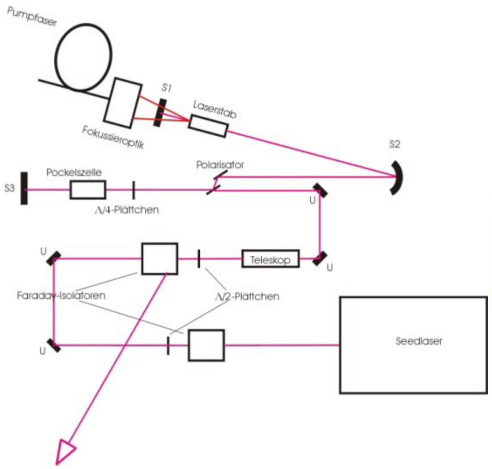


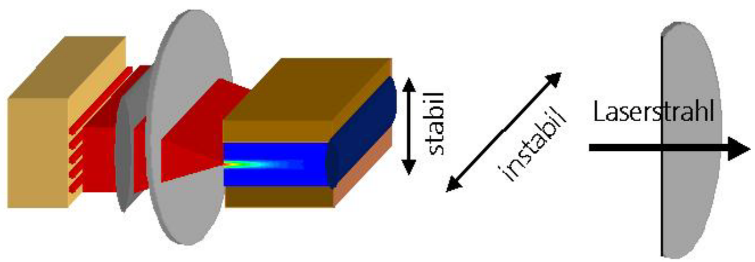
Bright,
but not like a synchrotron

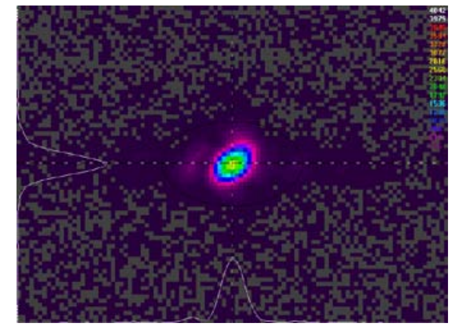
Next-generation liquid-jet laser plasmas:

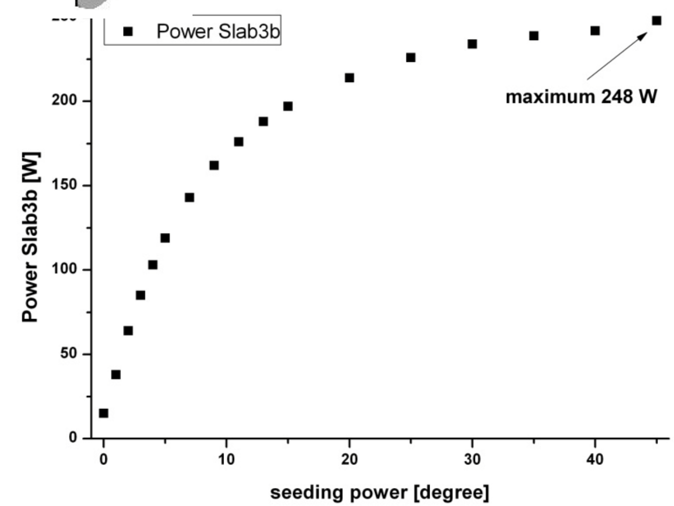
260 W, 800 ps, 2 KHz DPSS

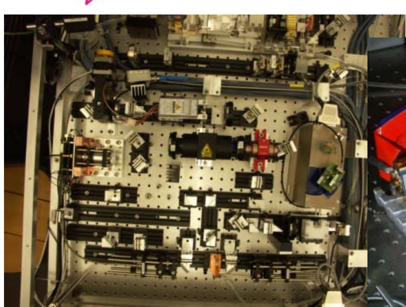


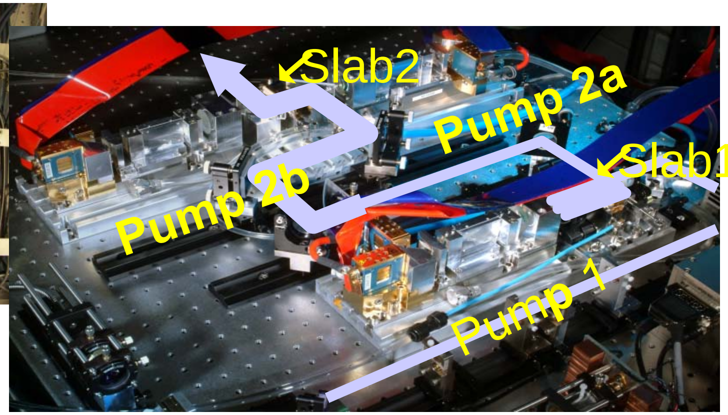






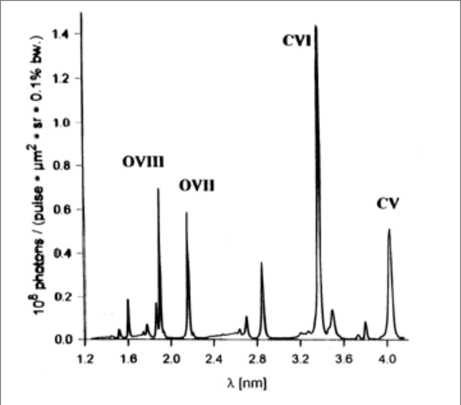


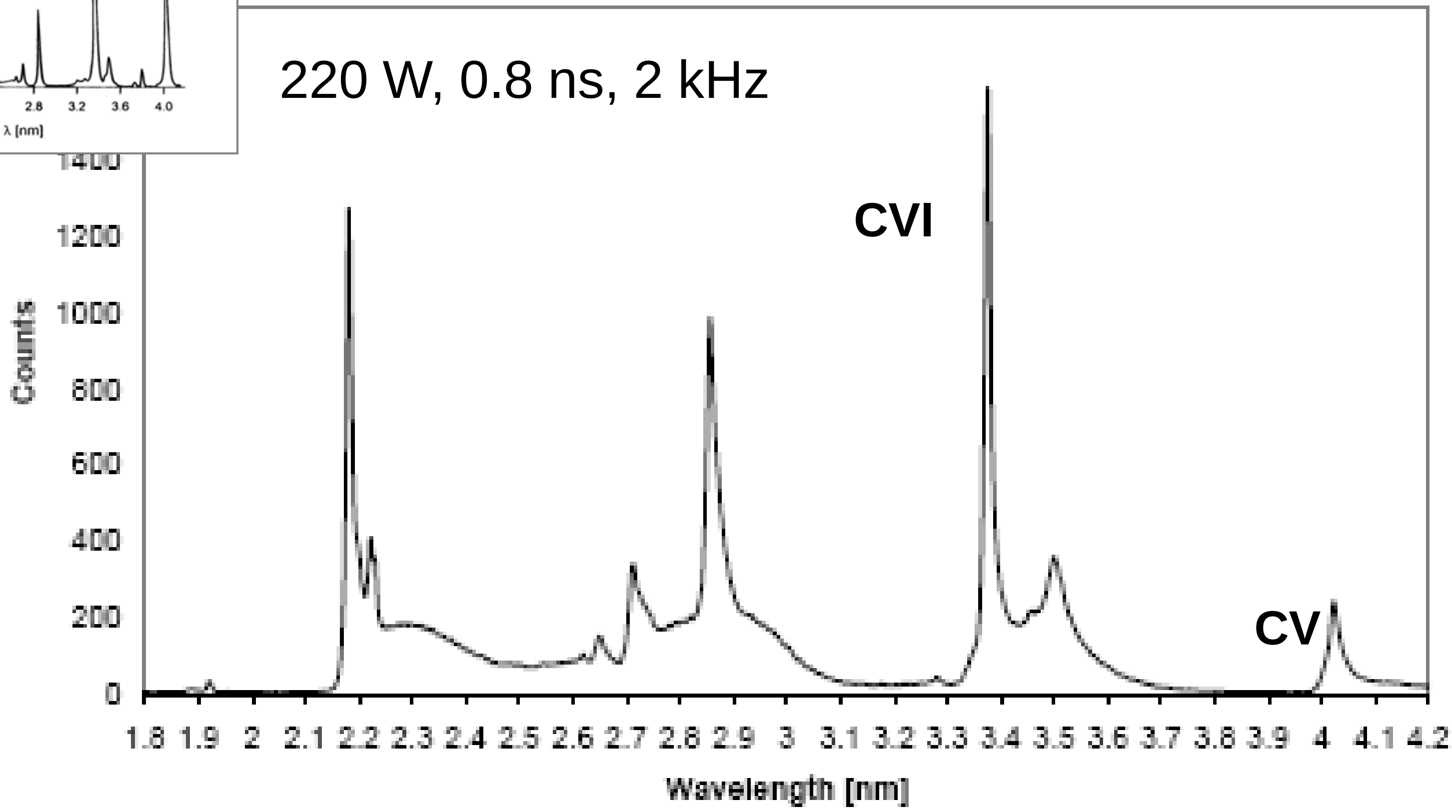




Thanks to D. Esser et al, FhG ILT, Aachen

First methanol-jet spectrum





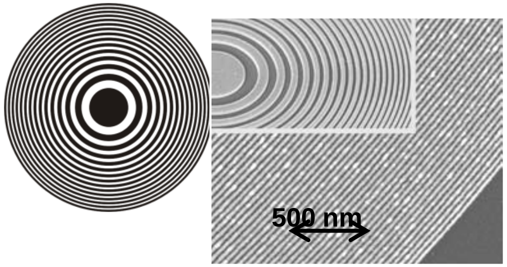
Jet is stable
Hot plasma

Selin et al, in progress,

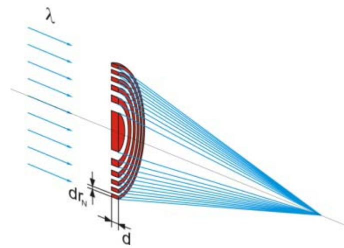
Soft x-ray optics:

Zone plates & Multilayers

Zone plates

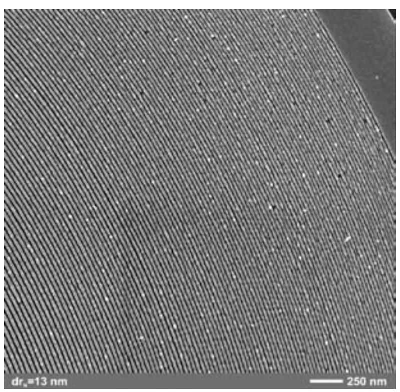


Circular diffraction gratings



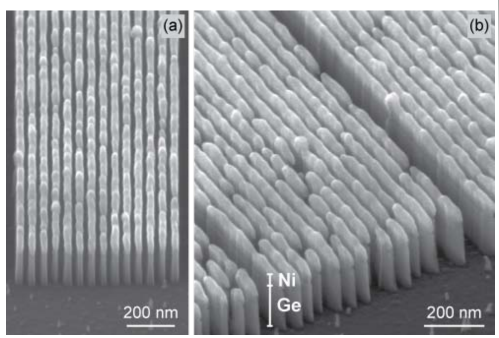
Resolution:

$$\Delta r_{Rayl.} = 1.22 dr_N$$



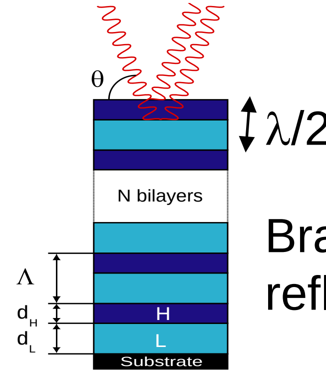
Single-write 13 nm Ni ZP

Ni-Ge zone plate, 15 nm

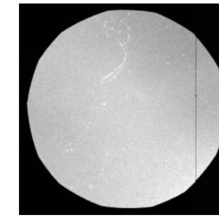


Reinspach et al, JVST (2009), MNE (2010); Lindblom et al JVST (2009) etc

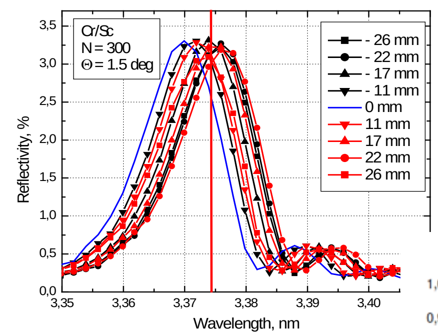
Normal-incidence multilayers



Bragg reflection



Uniformity

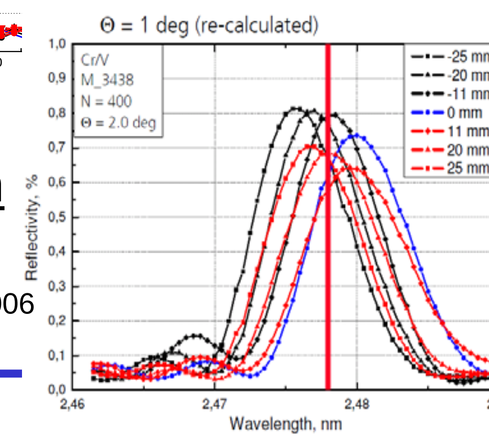


Cr/Sc @ 3.374 nm

R=3%

Cr/V@ 2.48 nm

R=0.7%



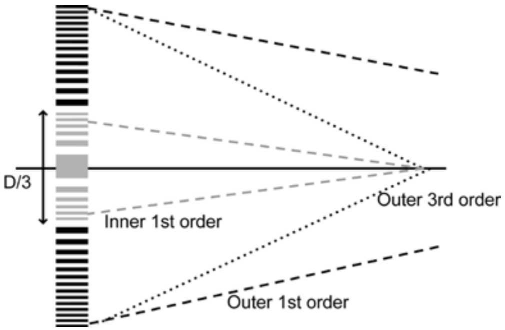
Stollberg et al, Appl Opt (2006

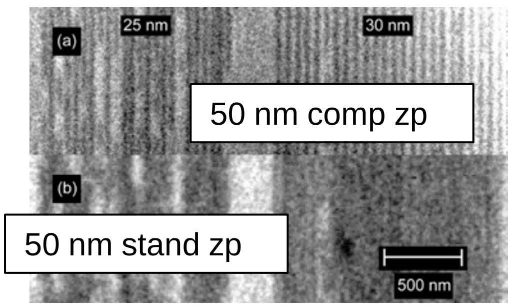
Yulin et al, FhG, Jena

Laboratory water-window x-ray microscopy

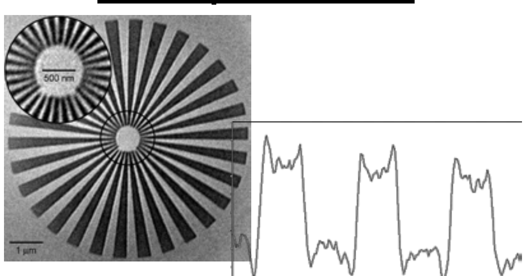
Compound ZP⇒

<25 nm lab. XRM!

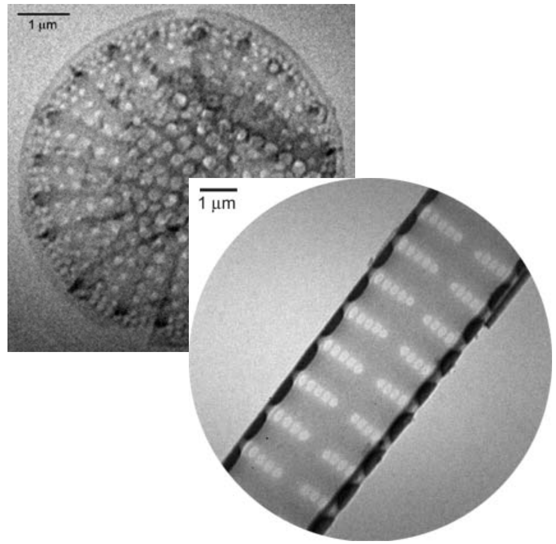




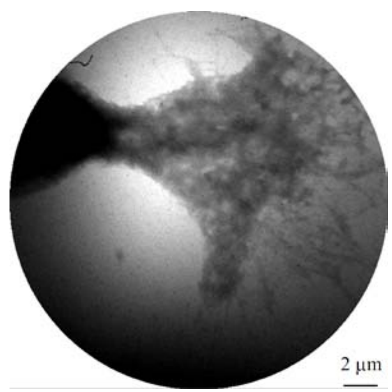
Test patterns:



Diatoms:



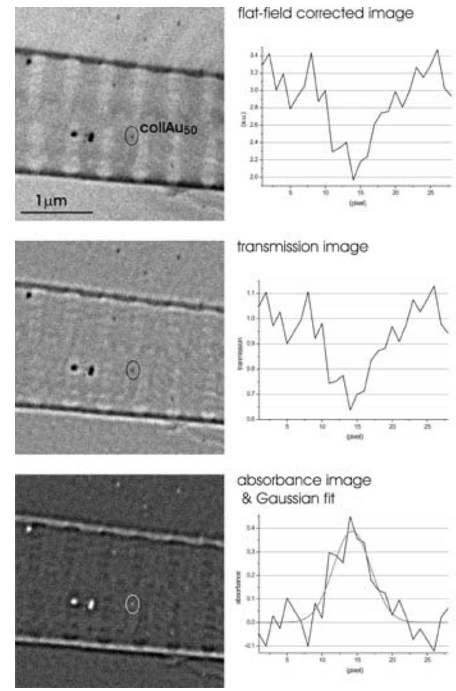
COS-7 cells

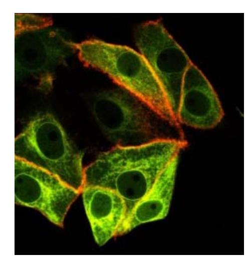


Function:

Size-selective coll.

Au identification





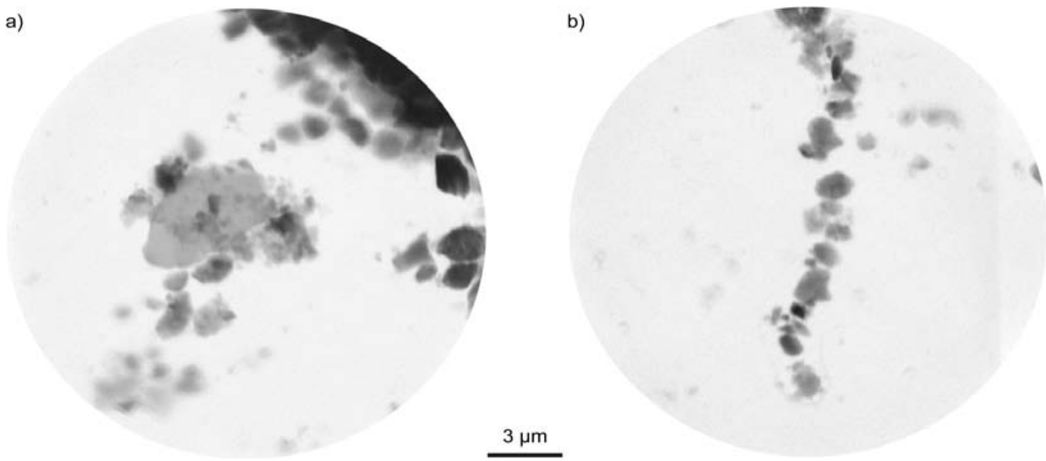
Goal: protein co-localization

Takman et al, J. Microsc. (2007), v. Hofsten et al, Ultramicr (2007) Stollberg et al, J Micr. (2006); Stollberg et al, J Micr. (2007); v. Hofsten et al Ultramicr. (2008), v. Hofsten et al., Opt. Lett. (2009) etc etc

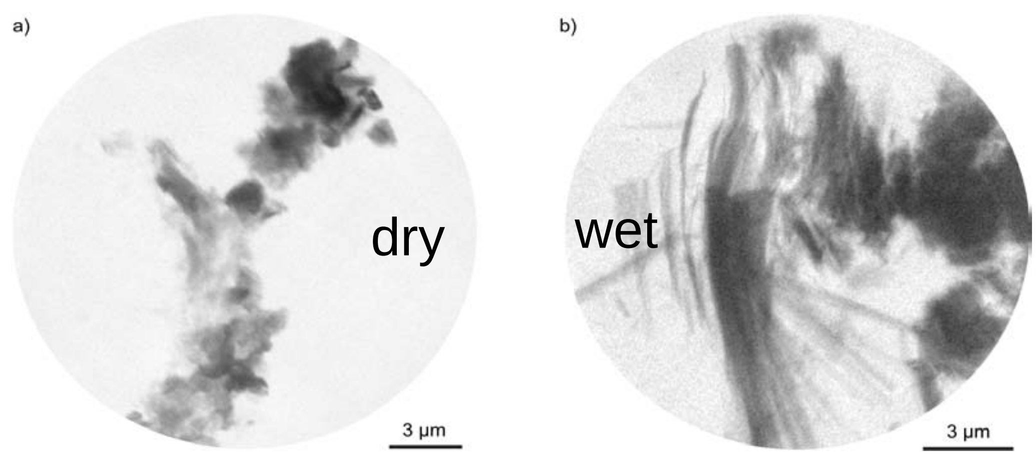
Recent results:

Environmental colloids

Chernozem, wet



Nontronite

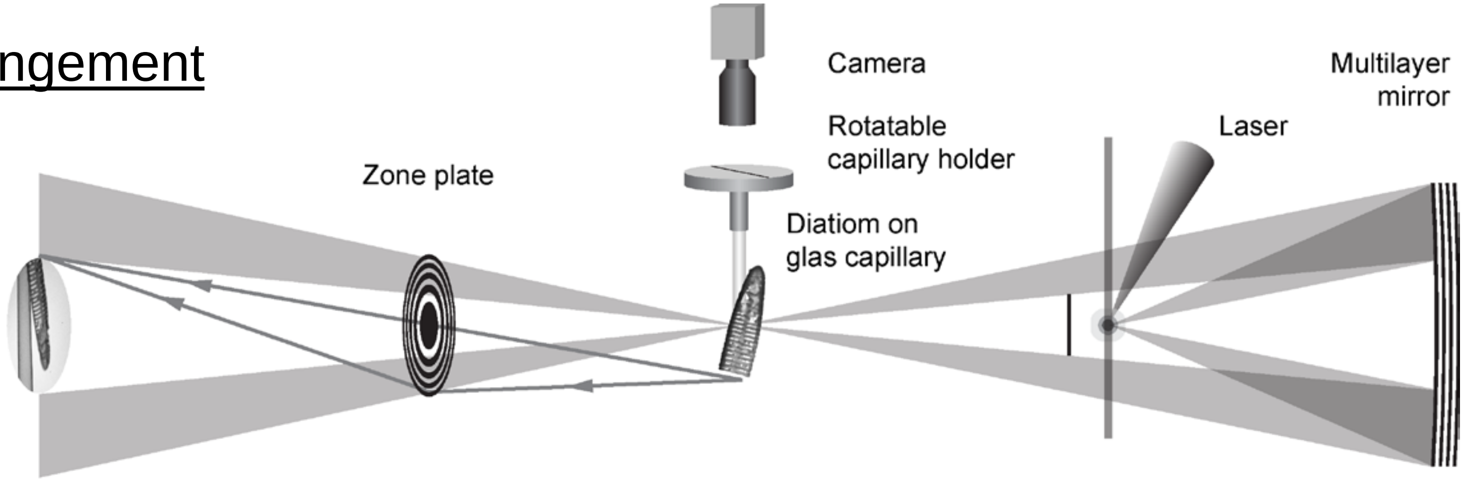


Thanks to J Thieme et al.!

Hertz et al, submitted Chem Geol (2010)

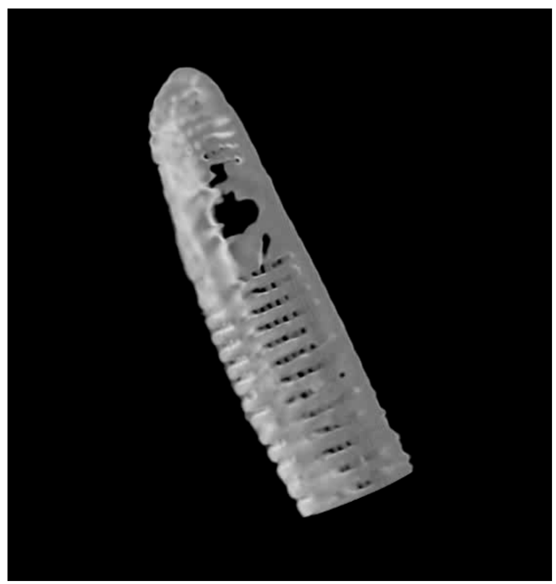
Micro-tomography w/ lab. water-window XRM

Arrangement



λ= 3.37 or
λ= 2.48 nm

Diatom reconstruction



λ=3.37 nm
Filtered. back. proj.
53 projections
140 nm resol. (DPR)

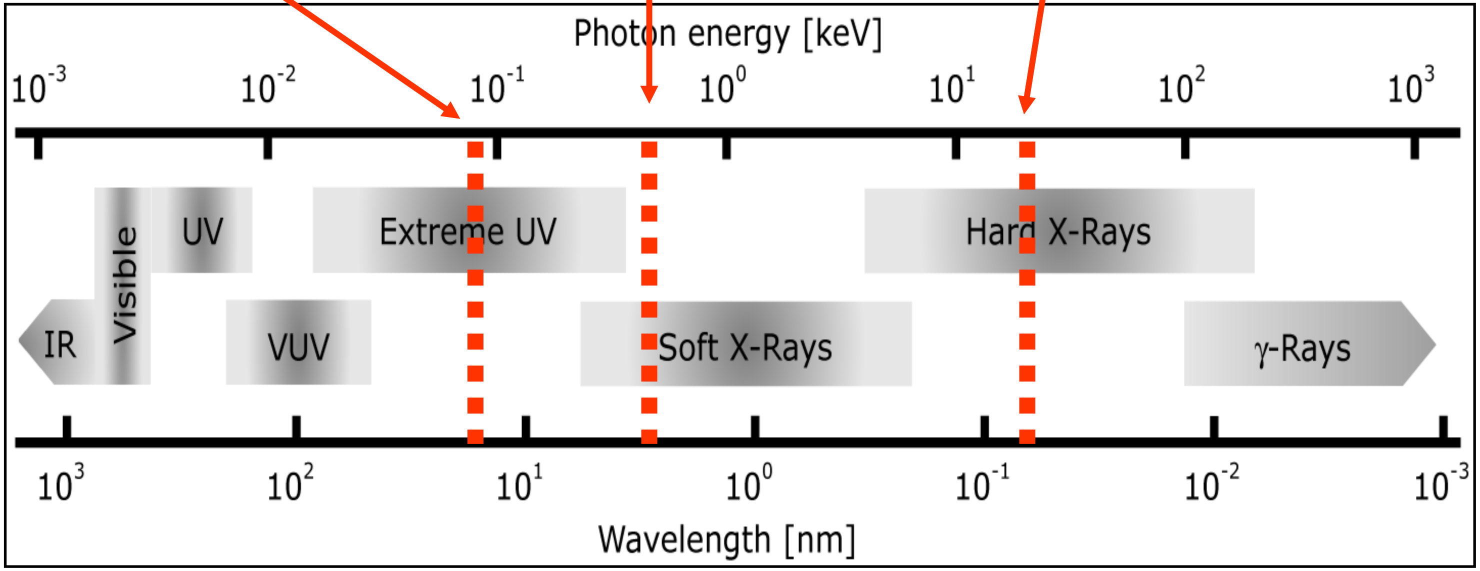
Bertilsson et al, Opt Expr (2009)

Liquid jet/droplet sources

<u>Water window soft x-rays :</u>
Laser-plasma liquid-jet sources
X-ray microscopy

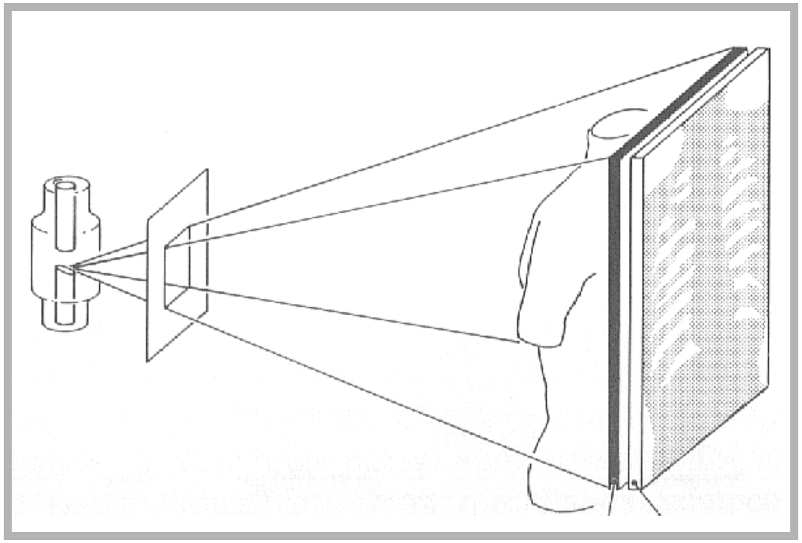
<u>EUV :</u>
Laser-plasma liquid-jet sources
Lithography

<u>Hard x-rays :</u>
Electron-impact liquid-jet-anode sources
Phase-contrast imaging



Hard X-Rays:

Laboratory hard x-ray imaging



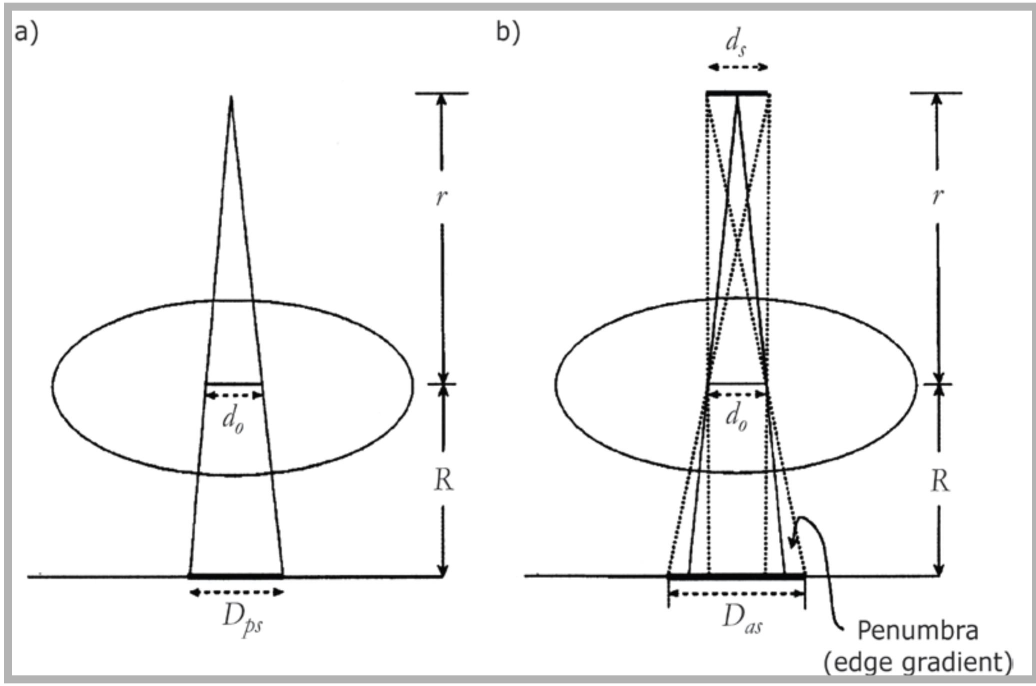
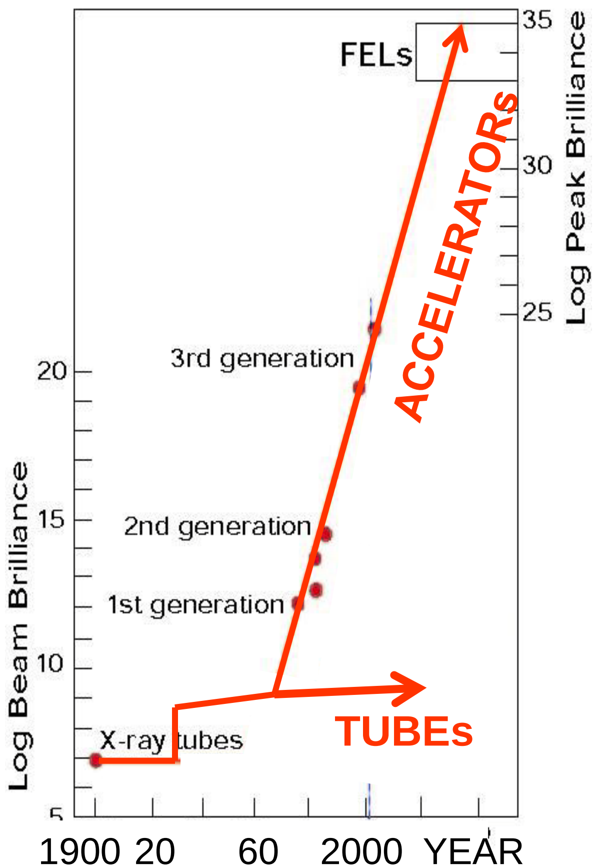


Image quality is source limited

Hard X-Rays:

Electron-Impact X-Ray Sources

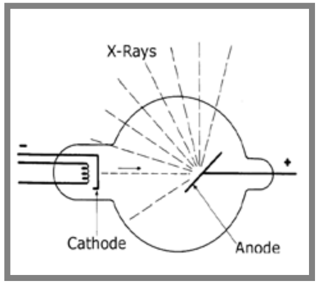
X-Ray Brightness



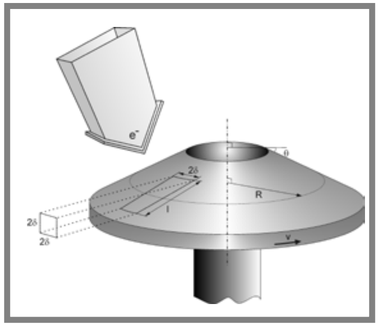
History: Electron-impact sources

E-beam power density ⇔ brightness

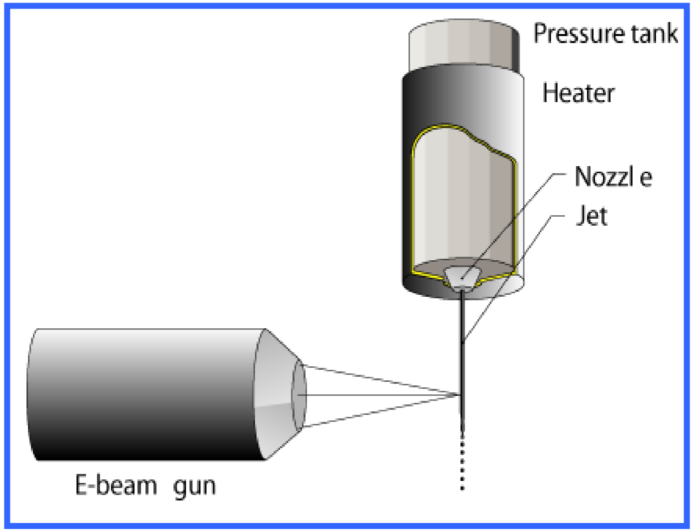
Thermally limited



Classic x-ray tube
(1895)
~1 kW/mm^2



Rotating-anode source
(1929)
~10 kW/mm^2

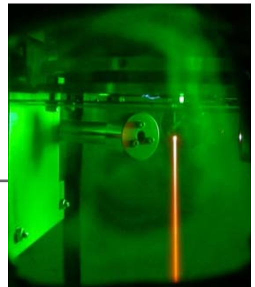


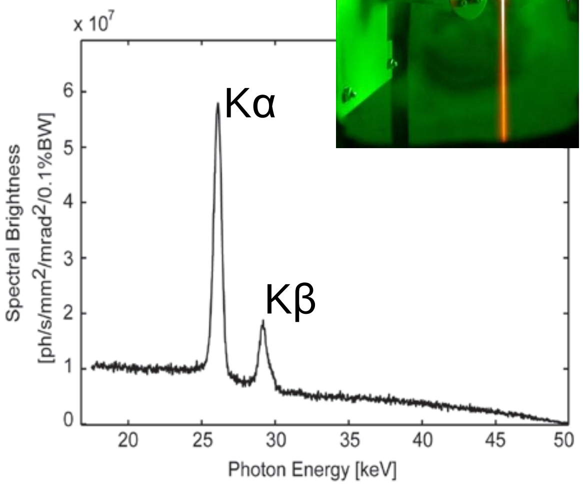
Liquid-metal-jet-anode source
(2003)
>10 MW/mm^2

Hemberg et al, APL (2003); Hemberg et al, Opt. Eng. (2004)

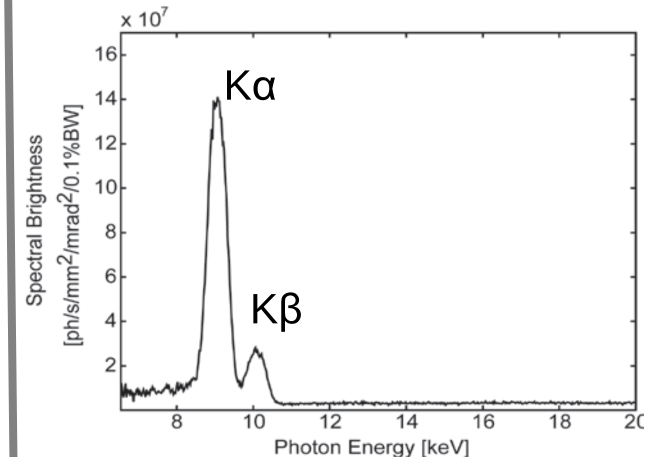
The liquid-(metal)-jet-anode x-ray source:
Present status

Tin

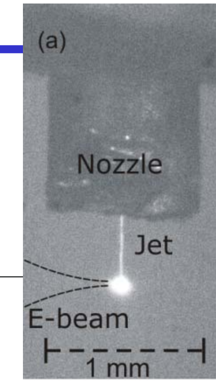


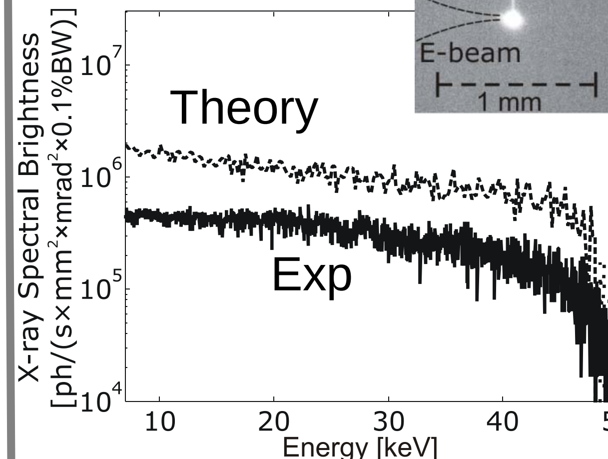


Gallium



Methanol





Present data:

Jet diameter: 15-75 µm
Jet speed: 10-100 m/s
Source size: >5 µm

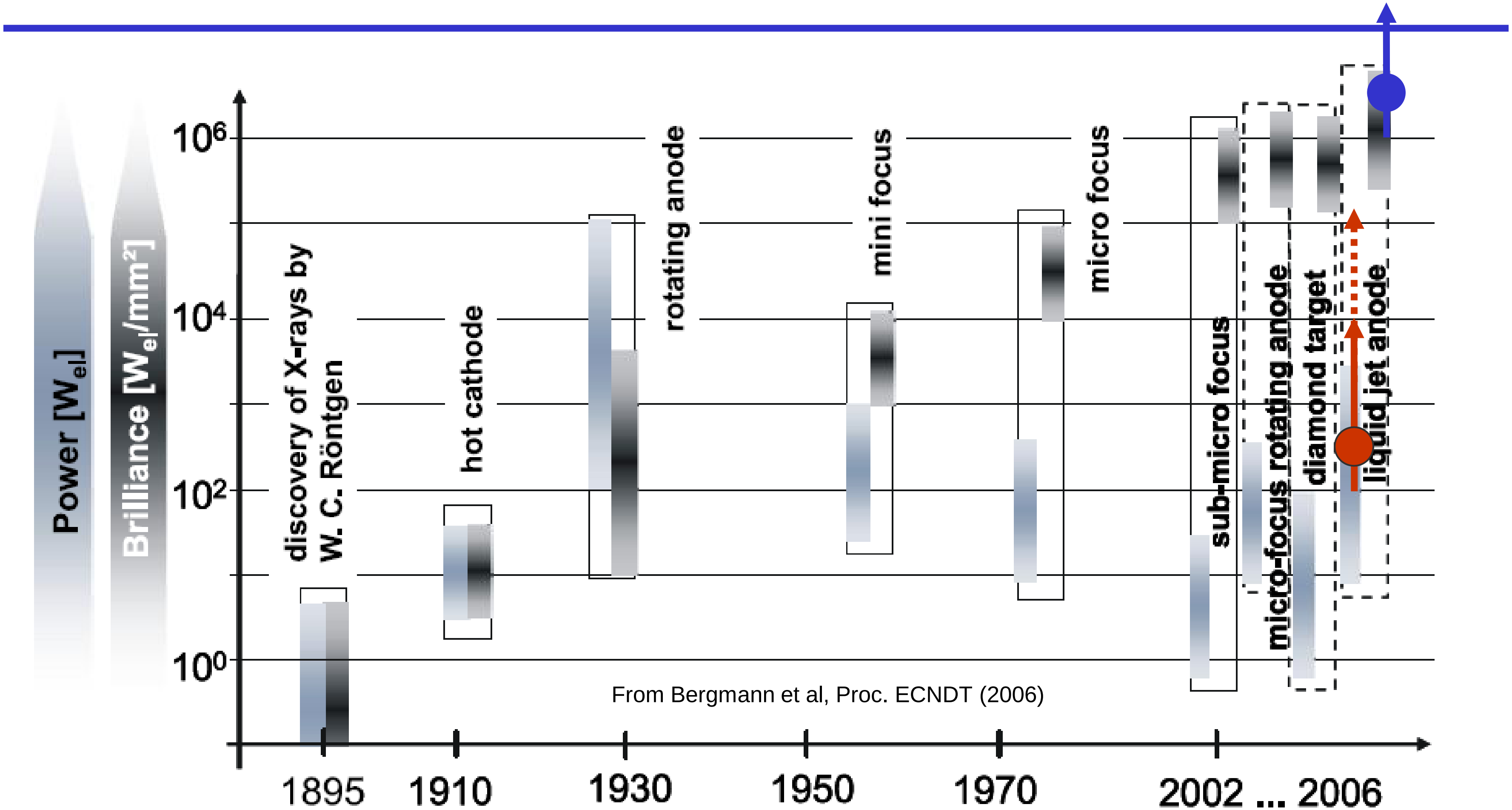
Power: 50-300 W
Power density: >2 MW/mm²
(cf. ~10-100 kW/mm² existing sources)

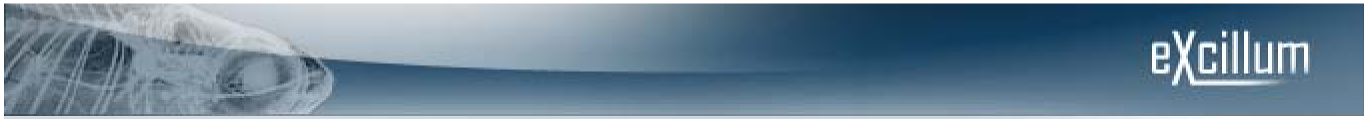
Future:

Power scalability: >100×
Power dens. scal.: >10×

Otendal et al, Exp. Fluids (2005); Otendal et al JAP (2007); Otendal et al RSI (2008); Touhimaa et al. APL (2008)

Laboratory hard x-ray sources



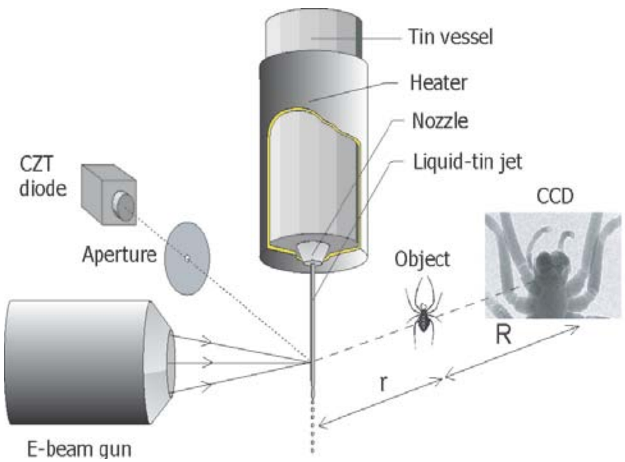


Excillum presently: 50 W/5 μm⇒few MW/mm^2 or
100 W/10 μm or 200 W/20 μm

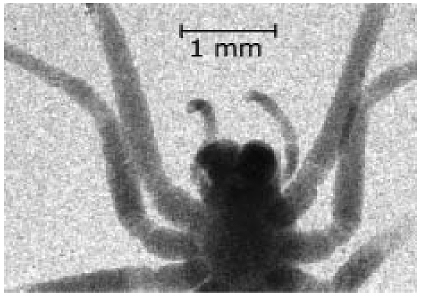
Hard x-rays:

Laboratory high-resolution phase-contrast imaging

Imaging arrangement.

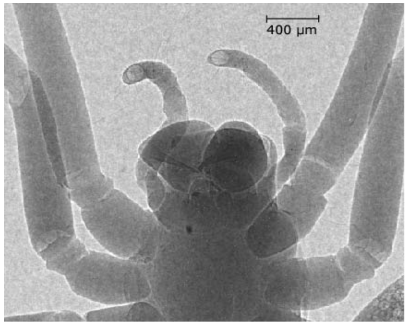


Absorption contrast

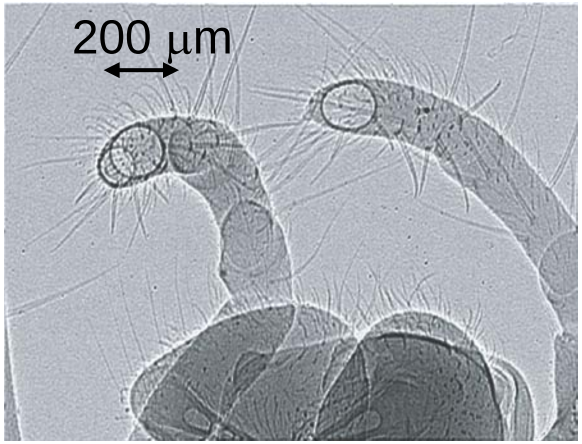


Phase-contrast imaging

M=1.3×

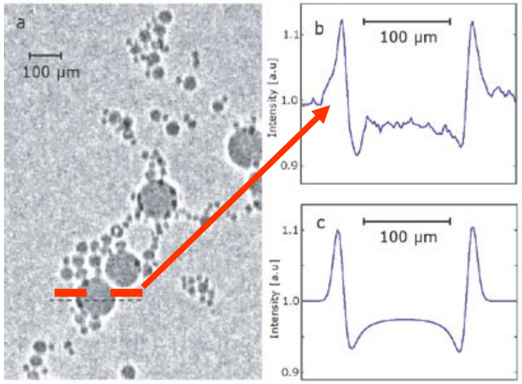


M=4.5×



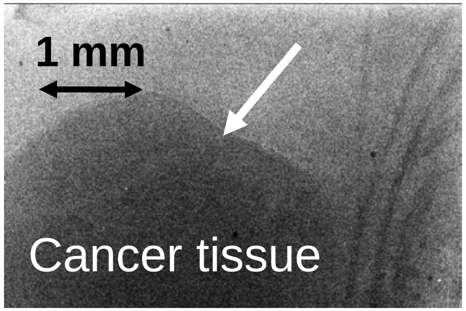
Tuohimaa et al, APL (2007)

Theoretical modeling



Small-animal studies

Tumour detection

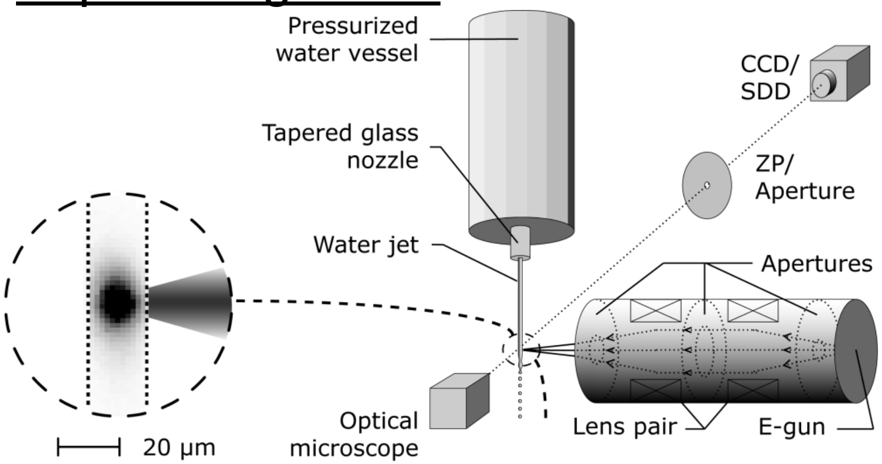


Lundströml, in progr

Recent results:

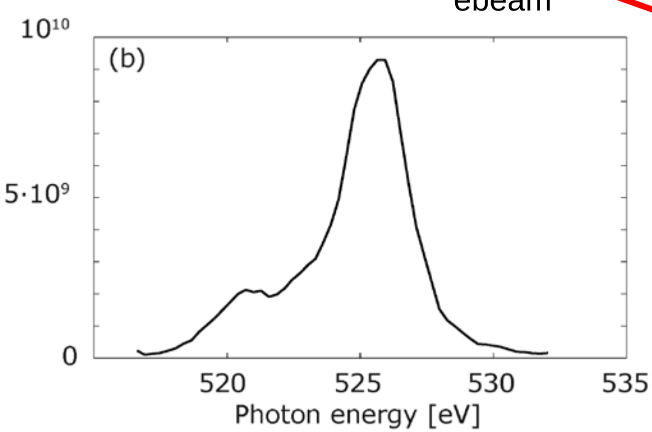
Electron-impact water-jet soft x-ray generation

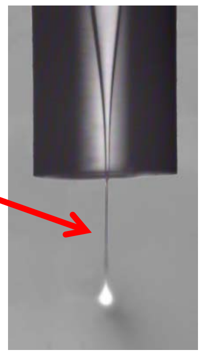
Exp. arrangement



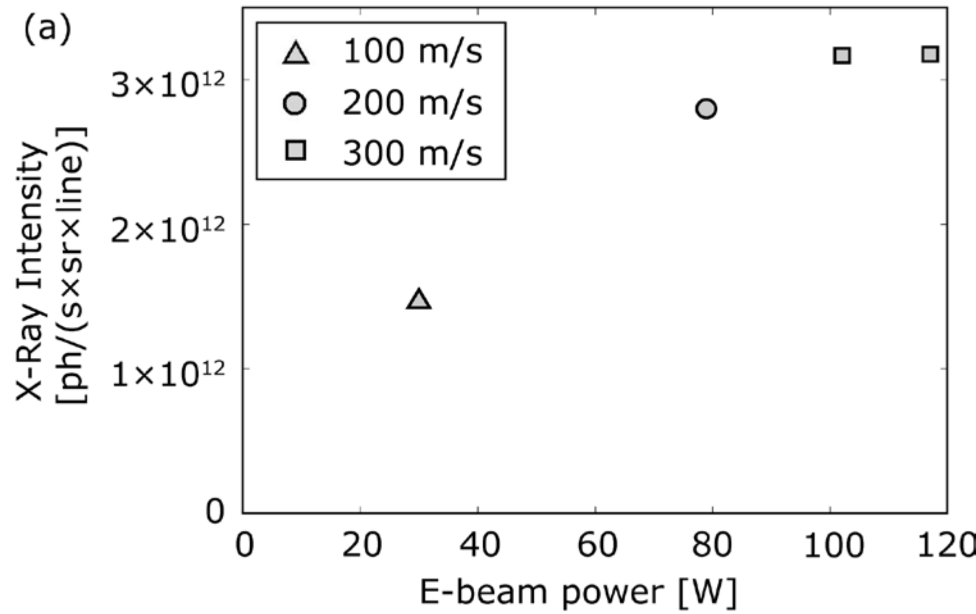
525 eV emission

100 m/s
50 W_{ebeam}





Power scaling



Scaling at low power:

~0.5×10^{11} ph/(s×sr×line×W_{ebeam})
~4×10^{8} ph/(s×sr×μm^2×line×W_{ebeam})

Cf. 100 Hz LN2 LPP:
2-4×10^{10} ph/(s×sr×μm2×line)

Summary & Future

- EUV:
  - Liquid jets and droplets are still a preferred target modality
- Soft x-rays:
  - Laboratory x-ray microscopy approaches synchrotron quality
    - Resolution: <25 nm features
    - Contrast: Phase optics
    - Cryo 3D imaging
  - Next:
    - Improved laser-plasma source w/new laser for shorter exposure time
    - Reliable e-beam liquid-jet source
    - New diffractive optics for higher resolution and improved contrast
    - Applications: Soils, Colloids, Cells, Carbon content
- Hard x-rays:
  - Liquid-metal-jet lab source promises 100$\times$ higher brightness
  - Small spot and high power $\Rightarrow$
    - High-resolution imaging
    - Improved contrast with phase
    - Adequate exposure times
  - Next:
    - High-resolution imaging in thick tissue
    - Small-animal imaging w/ and w/o tumors
    - Source development for diffraction, harder x-rays....

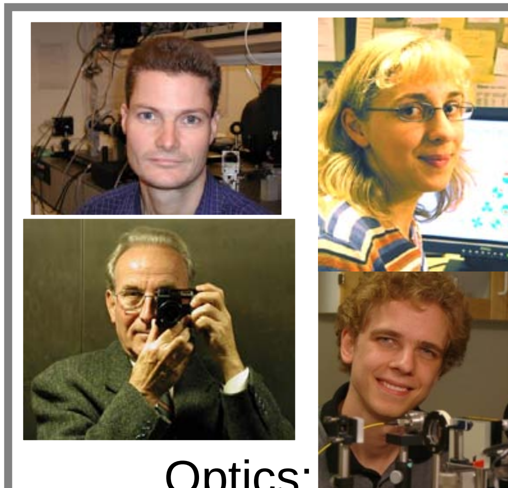
Biomedical & X-Ray Physics group

Thanks!

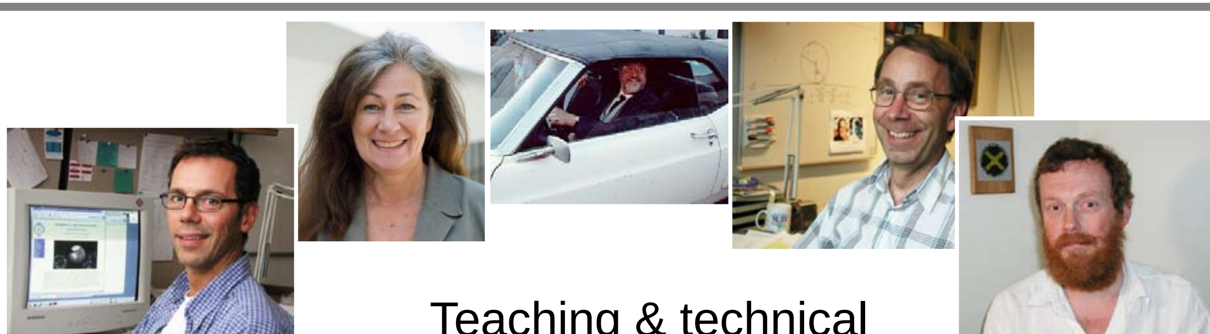
Soft x-rays:
Sources, optics, and microscopy

Hard x-rays:
Sources and (medical) imaging

Ultrasonics & μ-fluidics:
Bio-analytics and cell biol.



Optics:
Peripheral vision



Teaching & technical