# A Study of EUV/SXR/XR Grazing Incidence Collectors for Metrology Sources

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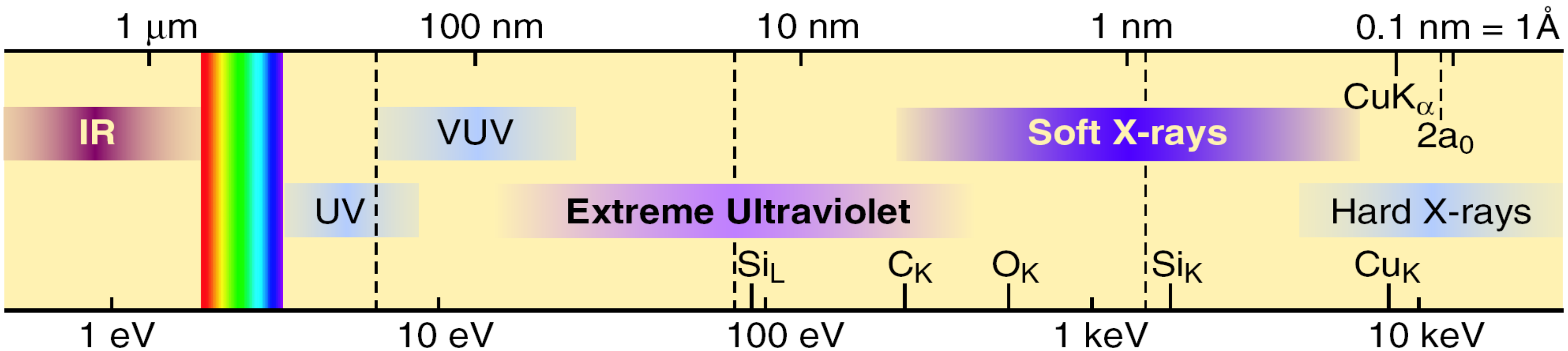
# Electromagnetic radiation spectrum



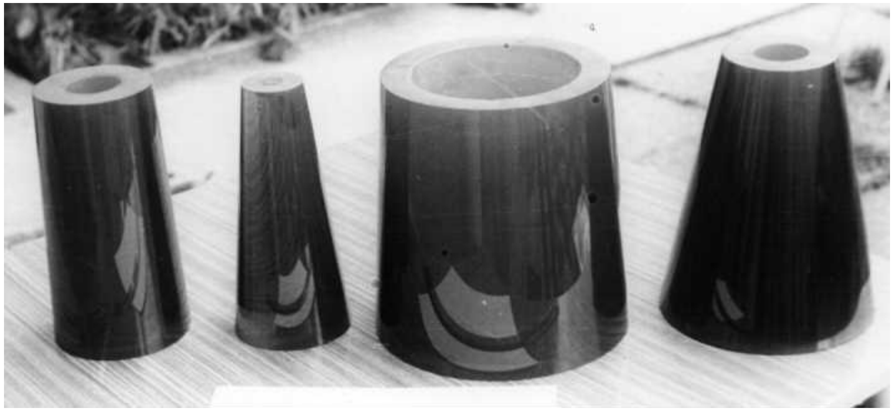

**D. T. Attwood *Soft X-rays and Extreme Ultraviolet Radiation: Principles and Applications* (Cambridge University Press, Cambridge, 1999)**

| | |
|---|---|
| **13.5 nm / 92 eV** | **EUV Lithography** |
| **6.2 nm / 200 eV** | **BEUV Lithography** |
| **2.34 – 4.39 nm / 283 – 531 eV** | **Water Window Microscopy** |
| **5 – 17 keV** | **X-ray Microscopy** |

**Microscopy → Tomography**

# Grazing Incidence (GI) replicated X-Ray Optics
# 47 years of research and development in Prague

## Replication Technology for X-ray Optics Manufacturing



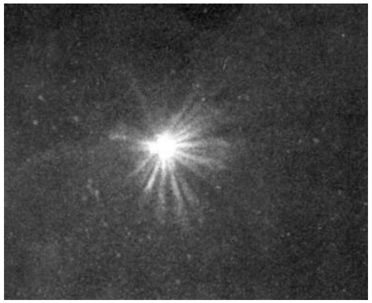
Mandrels used for the manufacture of X-ray mirrors (Glass ceramics Sital, Acad of Sci, Prague, 1969)

Replicated X-ray mirrors (hyperbolas, Ni surfaces, Acad of Sci, Prague)

# History – milestones and examples of projects
## (Academy of Science, Czech Technical University, Reflex, Rigaku)

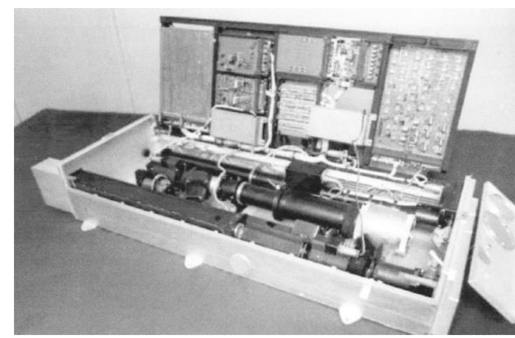
- 1969 First considerations started
- 1970 First X-ray mirror produced (Wolter 1, 50 mm)
- 1971 Wolter 1, 80 mm
- 1976 Wolter 1, 115 mm
- 1979 First mirrors flown in space (two Wolter 50 mm, Vertikal 9 rocket)
- 1980 Vertikal 11 rocket (two Wolter 50 mm)
- 1981 First large Wolter mirror (240 mm)
- 1981 Salyut 7 orbital station (Wolter 240 mm nested)
- 1985 Applications for plasma physics, EH 17 mm, PP 20 mm
- 1987 First high quality X-ray foils for foil mirror X-ray telescope (SODART)
- 1988 Fobos 1 Mars probe, TEREK X-Ray Telescope
- 1989 KORONAS I X-ray mirror, Wolter 80 mm
- 1990 First Micromirror (aperture less than 1 mm, Bede Ltd.)
- 1993 Collaboration with SAO, USA, WF X-ray optics started
- 1996 First Lobster Eye test module produced, Schmidt geometry
- 1997 Double-sided X-ray reflecting flats (SAO MA USA, CTU Prague)
- 1997 Lobster Eye Angel geometry project started
- 1999 First Lobster Eye test module produced, Angel geometry
- 2001 Thin segmented X-ray mirrors
- 2005 Replicated Image Slicers for LEO, EU FP6 projects, Cambridge
- 2006 MFO Kirkpatrick-Baez optic, University of Boulder, CO, NASA, USA
- 2007 Innovative technologies for X-ray telescopes, PECS, ESA XEUS projects
- 2008 – 2016 EUV/BEUV/WW/SXR/XR Grazing Incidence mirrors …

# Examples of Imaging GI X-ray optics



X-ray image of the laser plasma by the 17 mm EH microscope (IPPLM Warsaw)

1985 — Applications for plasma physics
(EH 17 mm, PP 20 mm)

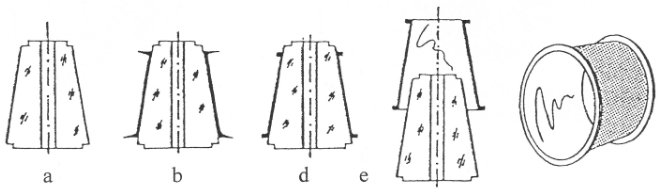


1988 — FOBOS 1 Mars probe,
TEREK X-ray telescope

1989 — KORONAS I
(Wolter 80 mm)

# Replication technology

- Replication technology developments in the Czechoslovak Acad. of Sci., National Research Institute for Materials (1969)
  - 2-3 mirrors from one master




a - master,
b - master with electroformed nickel layer
d - cutting/finishing of the edges
e - removing the Ni mirror shell

- Improvement of replication technology:
  - less damage of mandrel
  - reduced weight

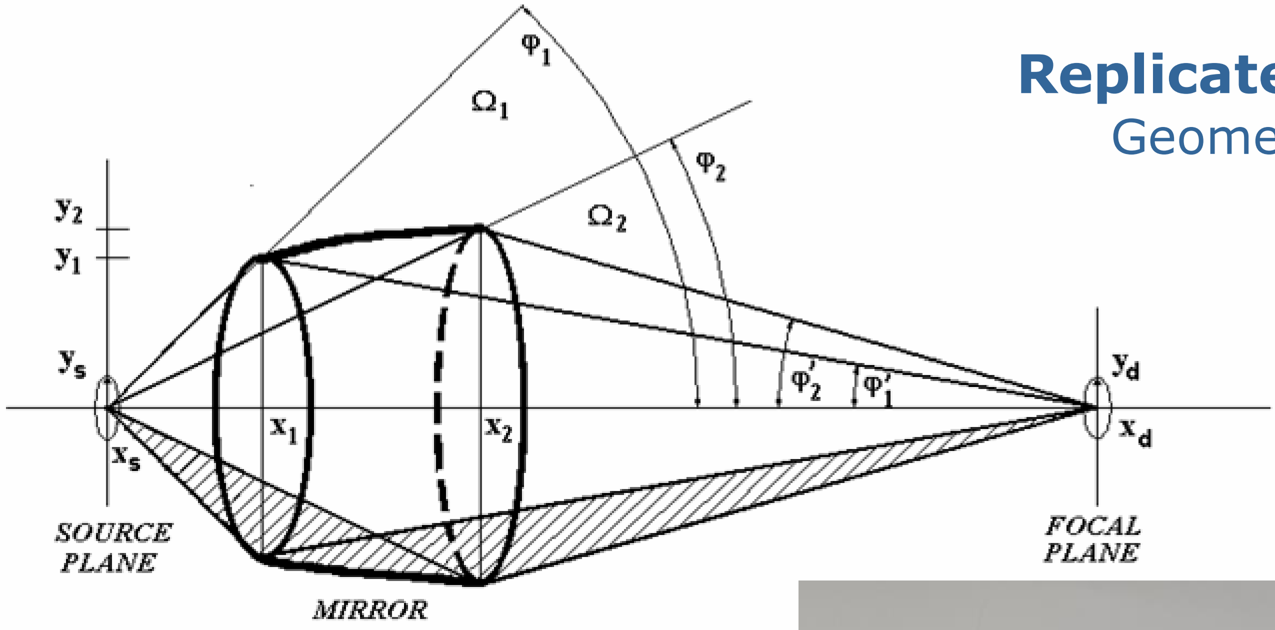
- Laboratory and space applications
  - Wolter objectives 17 mm and 20 mm dia
  - EH Wolter used (1985) for taking photographs of laser plasma in Institute of Plasma Physics and Laser Microfusion in Warsaw

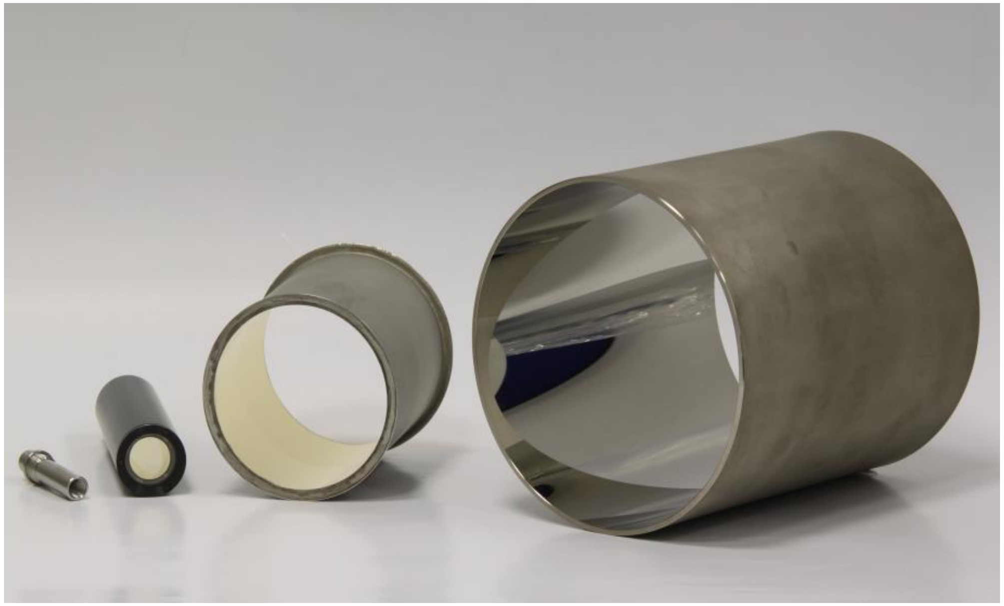
# Replication technology

MANDREL
with Au surface layer

Ellipsoidal mirror for spectral region
10 – 15 nm

# Replicated GI Mirrors
## Geometry and size



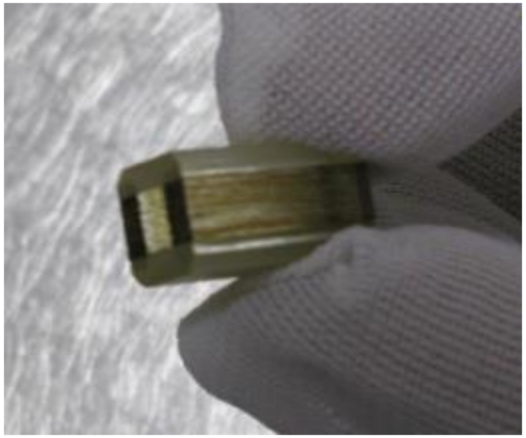



**Example: Elliptical mirror**

- **Mirror surface has shape of rotational ellipsoid**
- **Source is placed in left focus**
- **Detector or sample is placed in right focus**
- **Radiation strikes mirror surface at grazing angles 0,5° ÷ 20°**
- **Mirror is focusing radiation from left focus on right focus**

# Now manufactured by
# Rigaku Innovative Technologies Europe

- A part of Rigaku Corporation group (Tokyo, Japan)
- Established in 2008 as European center for the design, development and manufacturing of **X-ray optics**, **X-ray detectors** and **X-ray sources**
- Colaboration with Czech academic institutions and high-tech companies
- Ellipsoidal and parabolic optics for EUV/BEUV/WW/SXR/XR
  (laser plasma research, EUVL, WW and X-ray microscopy, space, …)
- Slope error < 10 arcsec (5“), microroughness < 2 nm (0.5 nm)

# Replicated GI Mirrors

**Ellipsoidal GI mirror for WW application (2.3 – 4.4 nm)**



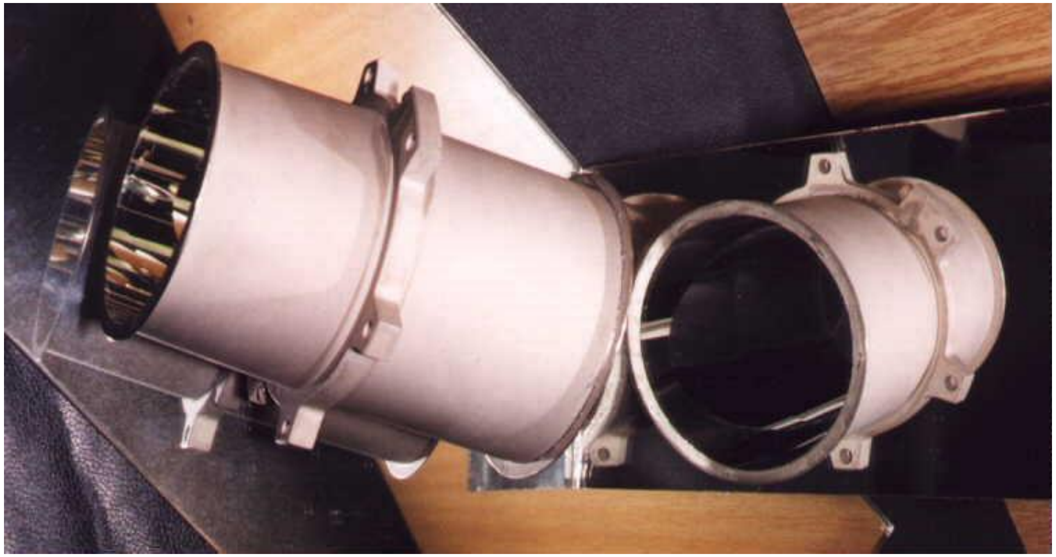
**Ellipsoidal GI mirror For EUVL applications (10-15 nm)**

Rigaku

Ellipsoidal GI EUV mirror for 13.5 nm

## GI EUV Mirrors

Ellipsoidal GI EUV mirror for 13.5 nm

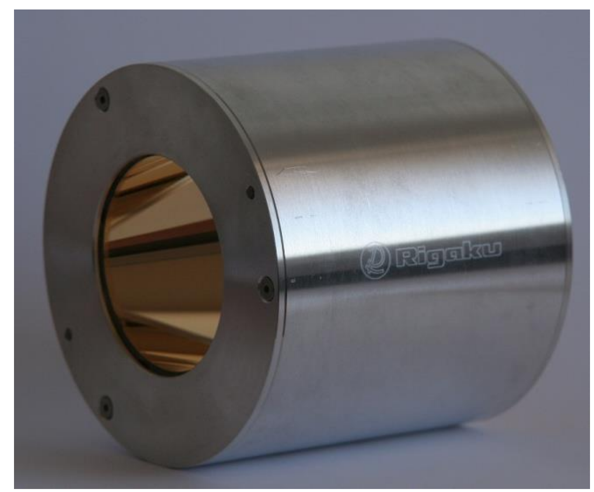


# Range of Applications

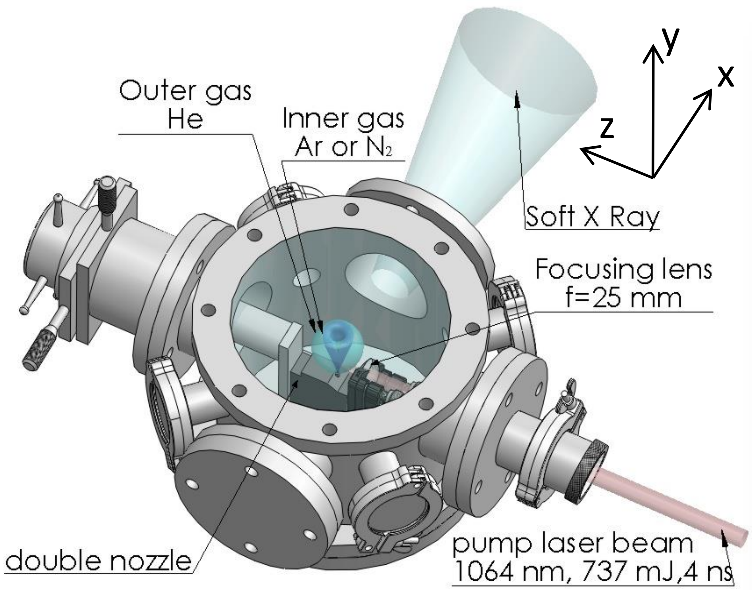
Replicated Wolter X-ray mirrors for space research (aperture 80 mm)

Replicated X-ray mirror for XRD (input aperture 0.4 mm, 8 keV, grazing angle 0,5°)

EUV Condensor for Laser Plasma Research and EUV Litography



# LPP - Gas puff target EUV laser-plasma short wavelength source




Scheme of the gas-puff target source

Photograph of the setup

| | |
|---|---|
| **Pumping laser** | Nd:YAG laser (EKSPLA), 4 ns/500mJ pulses, repetition rate **10Hz** |
| **Nozzle** | Inner: circular 0.4mm in diameter<br>Outer: ring 0.7mm/1.5mm diameters |
| **Gasses** | Working gasses: **Ar, Kr, Xe, $O_2$, $N_2$**, outer gas : **He** |

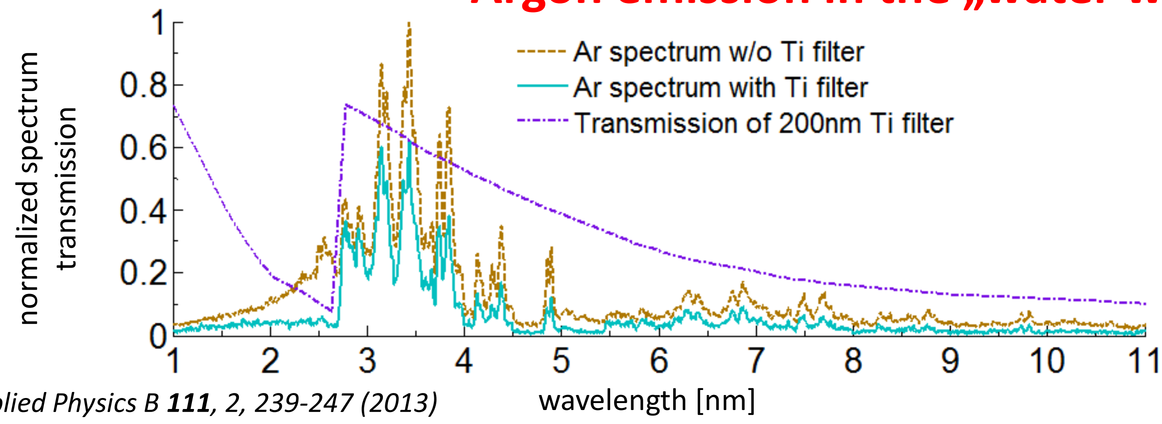
54
Xe
131.29

## Advantages:

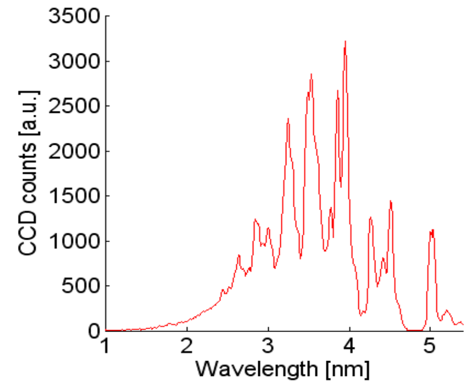
- ✓ no debris from gaseous targets
- ✓ compact construction, high repeatability
- ✓ high conversion efficiency, very robust – thousands of shots/day

# LPP - Gas puff target EUV laser-plasma short wavelength source spectra

## Argon emission in the „water window"

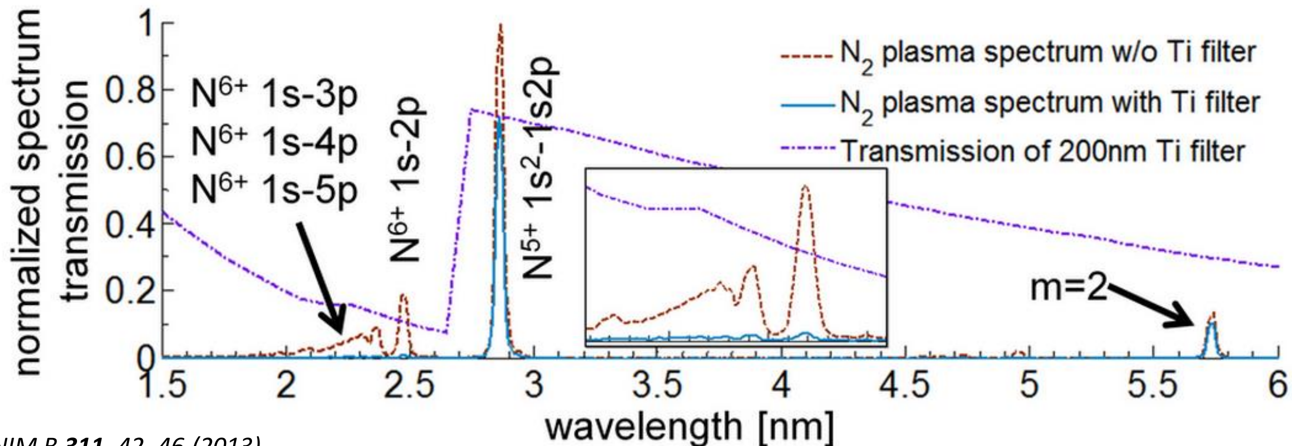



*Applied Physics B* ***111****, 2, 239-247 (2013)*

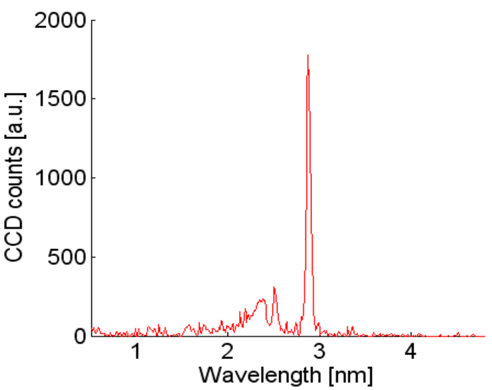



*NIM B* ***268****, 10, 1692-1700 (2010)*

## Nitrogen emission in the „water window"

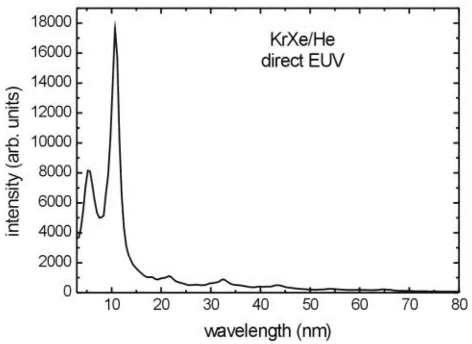


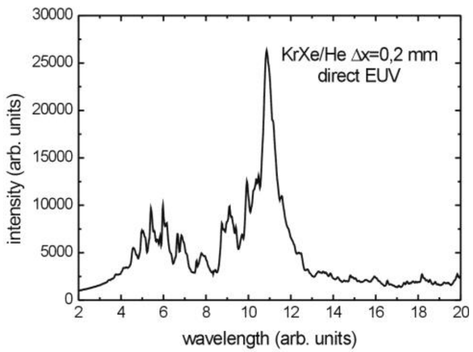

*NIM B* ***311****, 42–46 (2013)*



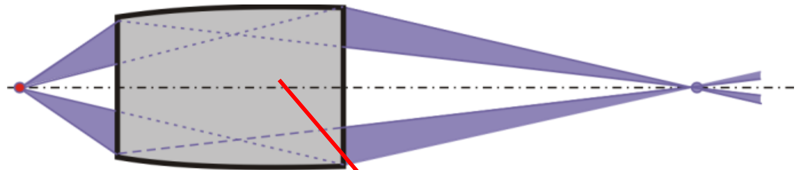

*NIM B* ***268****, 10, 1692-1700 (2010)*

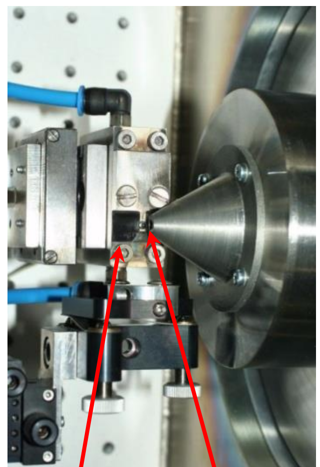
# Laser-plasma produced EUV source: laser 0.8 J / 4 ns

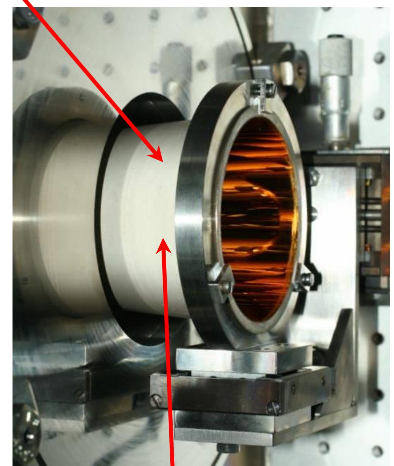



Low and high resolution EUV spectra of plasma radiation



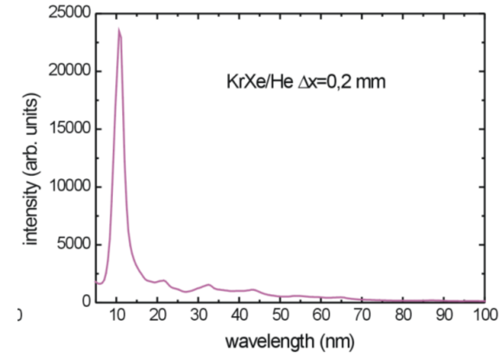


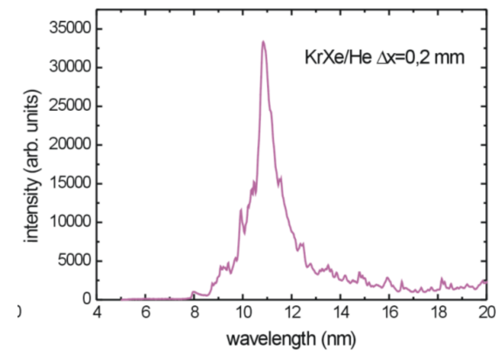


Gas puff valve

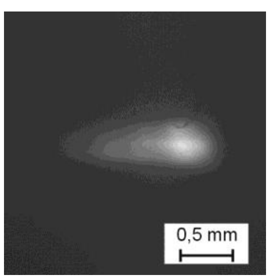
Orifice for differential pumping

Ellipsoidal EUV collector



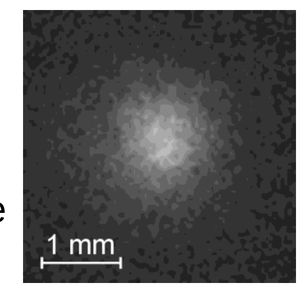




Low and high resolution EUV spectra of reflected radiation



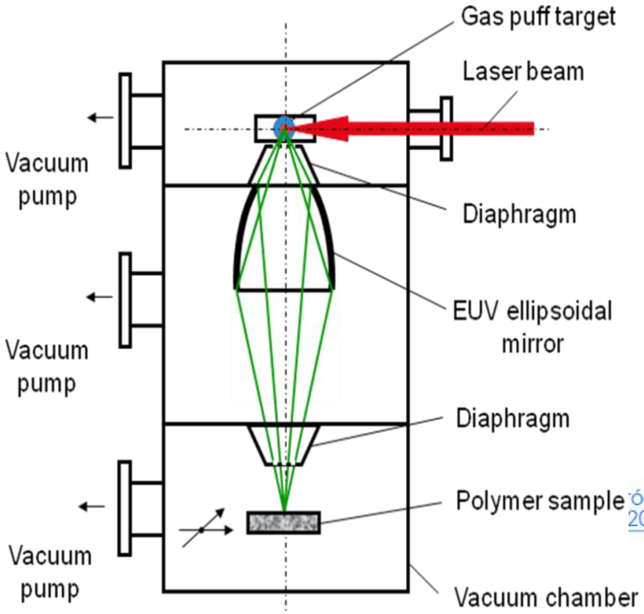

Plasma image

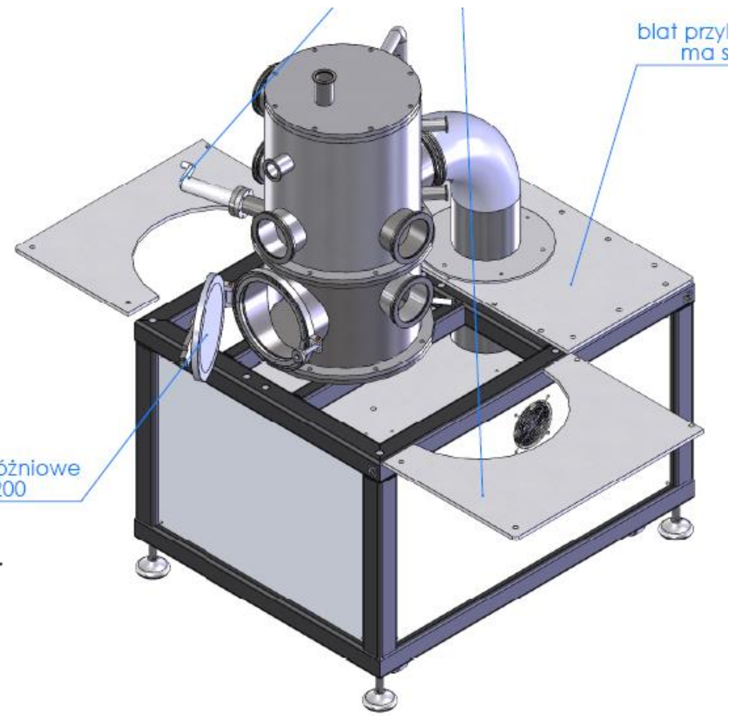
Focal spot image



# Laser plasma EUV source for processing polymers

**Laser plasma EUV source dedicated for processing polymers has been designed at IOE and was built in co-operation with EKSPLA, RIGAKU and PREVAC high-tech companies**




EKSPLA Rigaku

EUREKA

PREVAC PRECISION & VACUUM

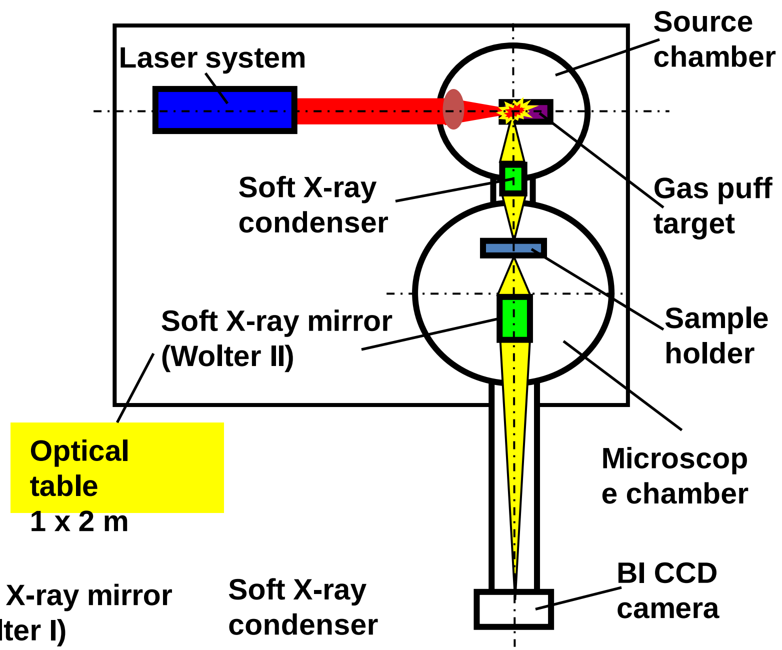




Bartnik et al. *Nucl. Instr. Methods A* 647 (2011) 125

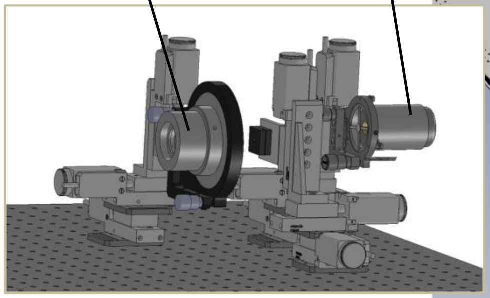
# NANOIMAGING USING SOFT X-RAYS

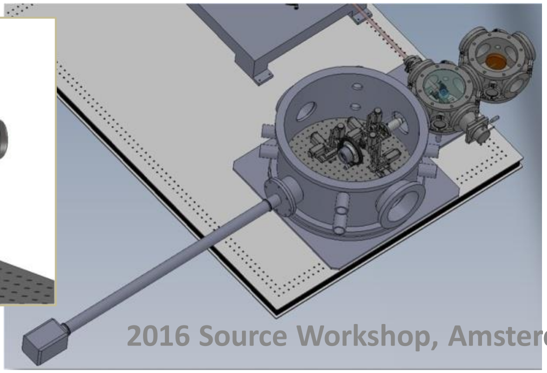
**Desk-top soft X-ray microscope with a laser plasma source**




Soft X-ray mirror
(Wolter I)

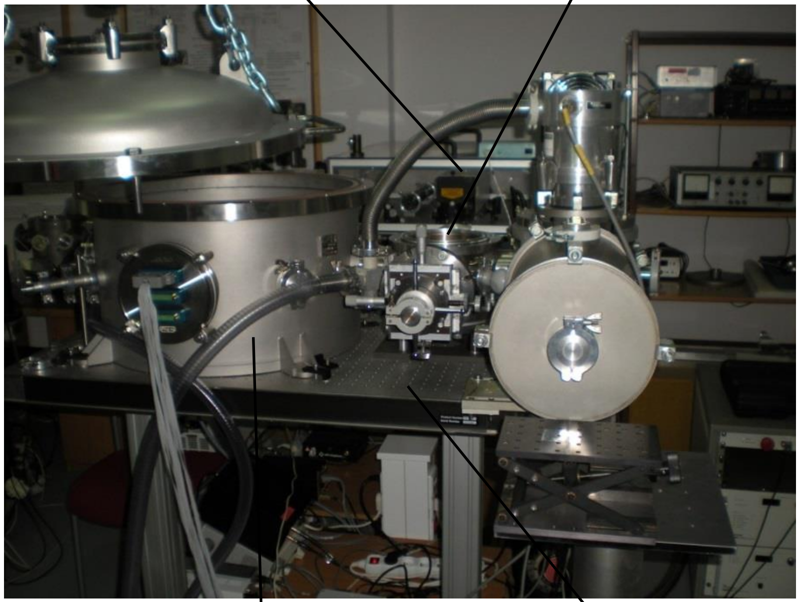
Soft X-ray
condenser





Source chamber

Nd:YAG laser

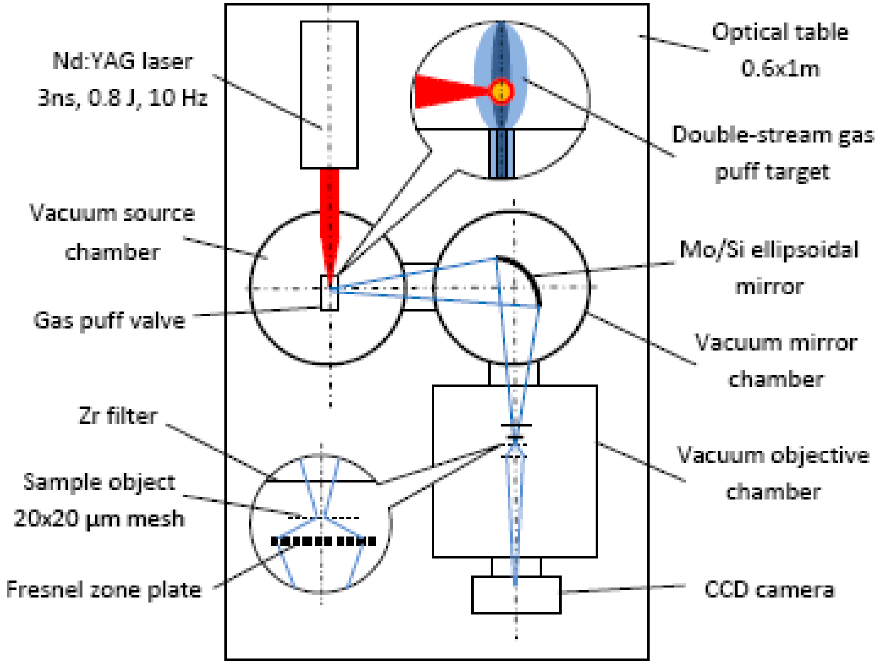


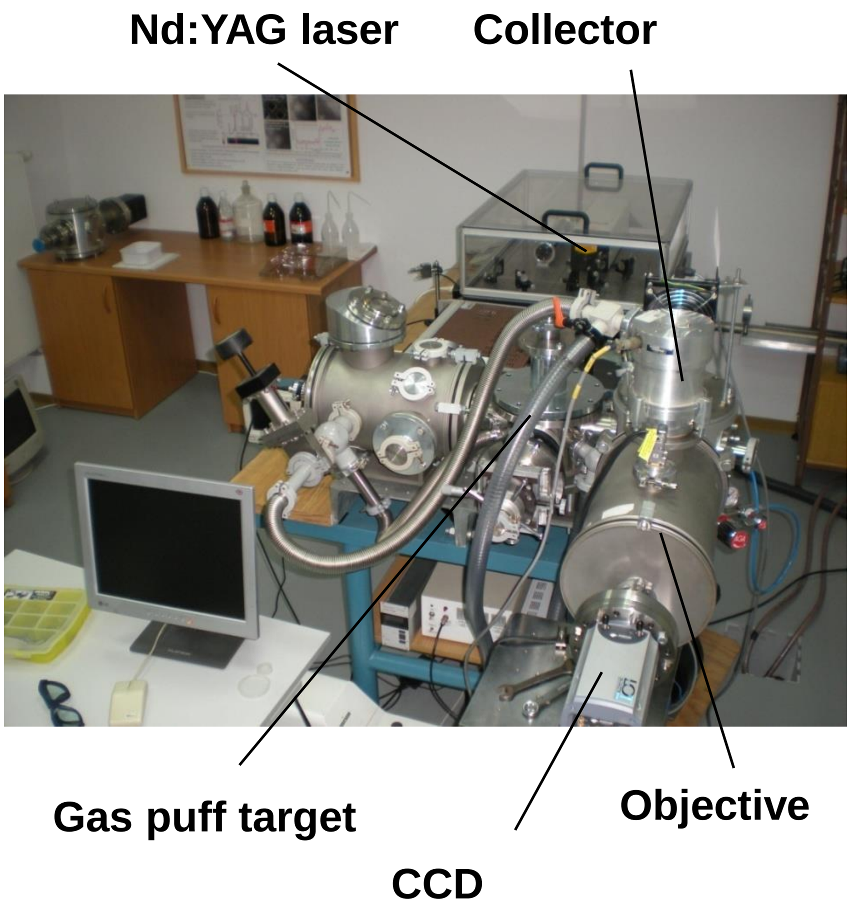
Microscope
chamber

Optical
table

# EUV IMAGING IN A NANOSCALE

**Desk-top soft X-ray microscope with a laser plasma source**

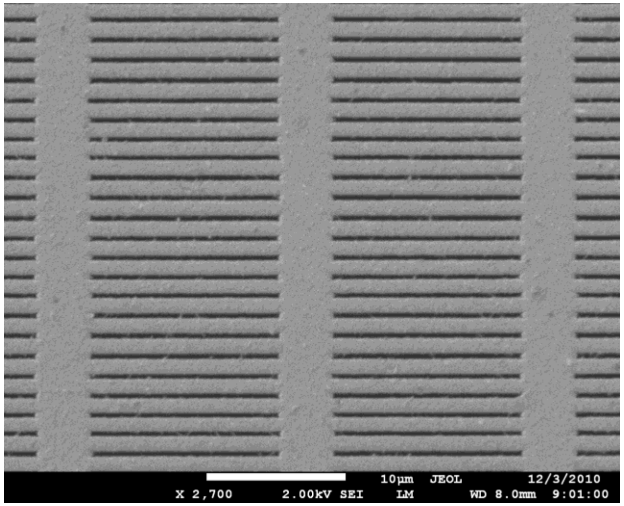


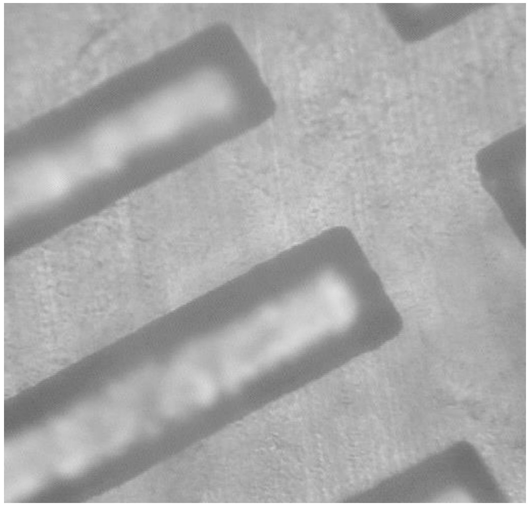



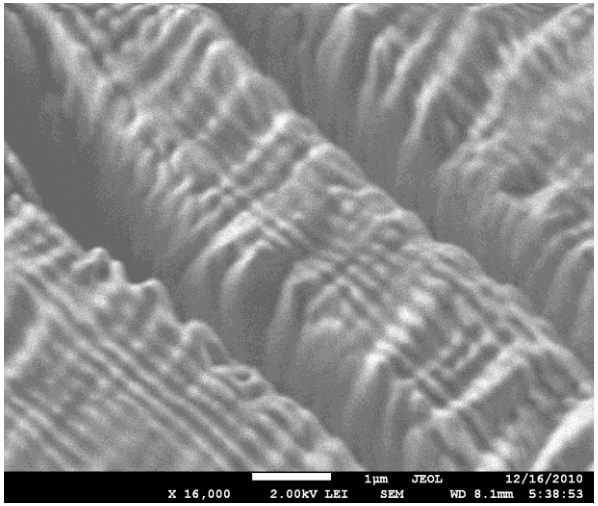

**Optical table 0.6x1m**

# EUV beam intensity amplification - joint experiments of CTU Prague and WAT Warszaw

## (EUV ablative lithography)



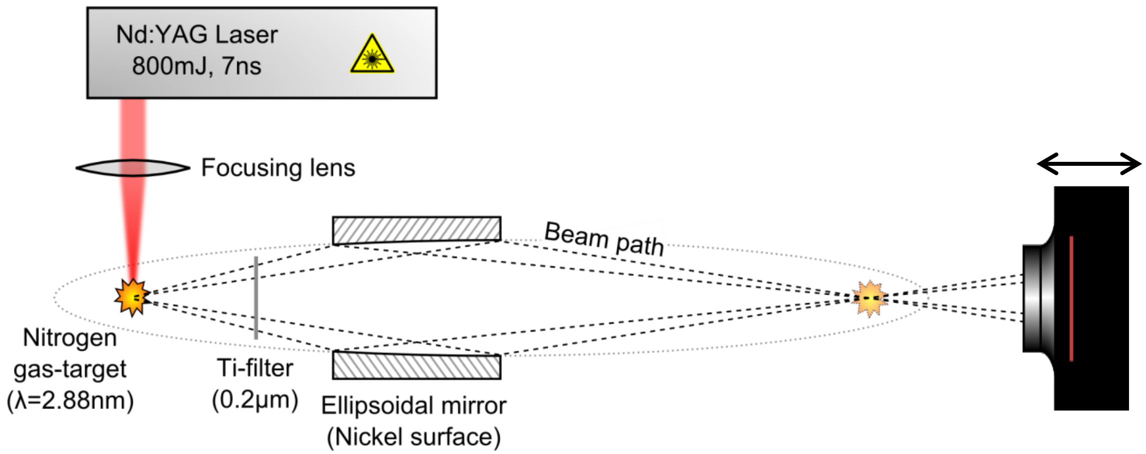



**Microstructure made in PTFE by EUV lithography. EUV radiation from gas-puff laser plasma transmitted through a metal mask**

**Multi–foil (MFO) XUV bifacial Kirkpatrick-Baez condenser**

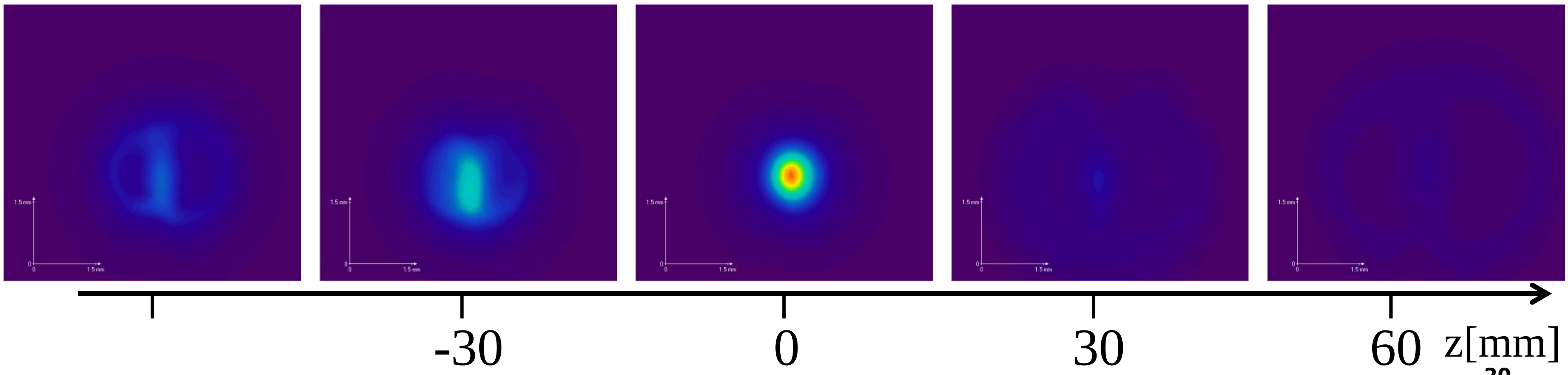
# Focusing of soft X-ray radiation

Setup at LLG with Rigaku ellipsoid

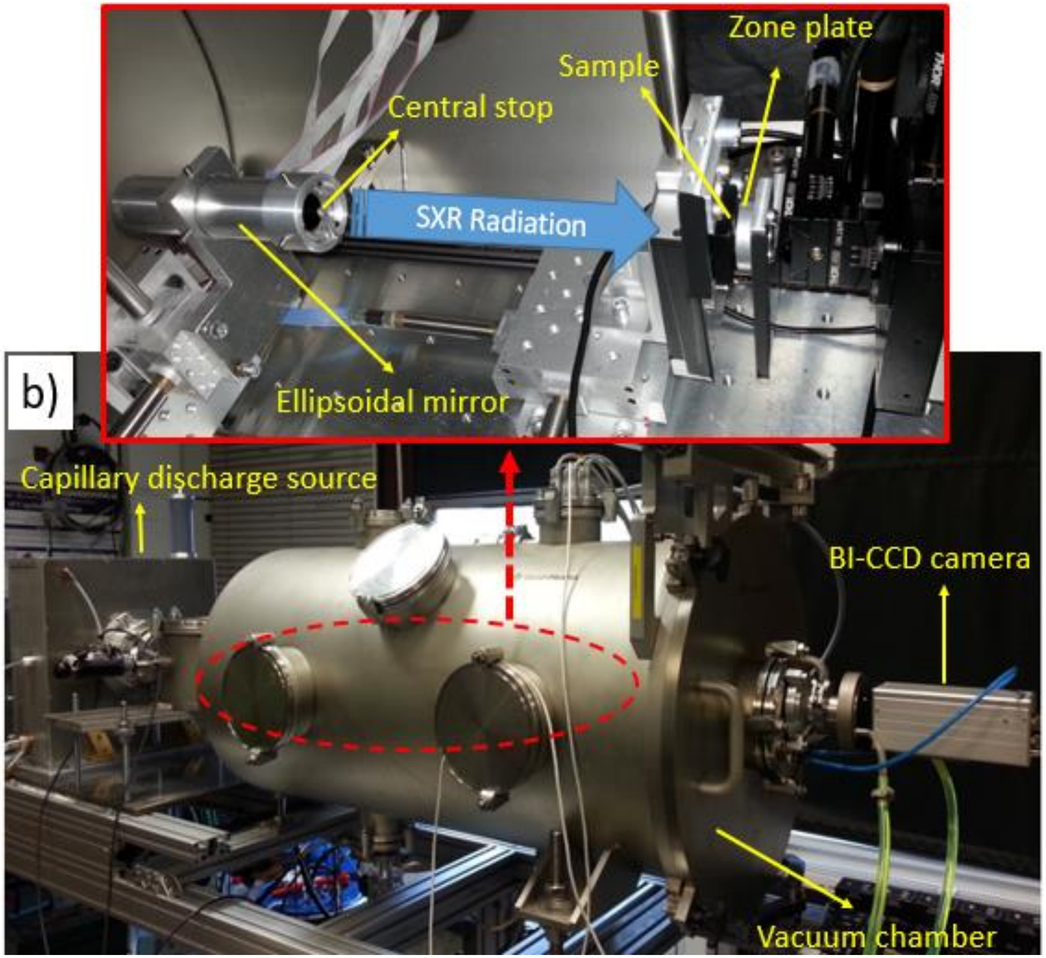



- 200 nm Ti filter
  → T = 72%
- (before: 1200 nm, T=14%
  = 1/5 · 72%)
- phosphor coated CCD
  → exposure level ~ 80%
  in focal plane

Dr. Klaus Mann, LLG Gottingen



## EUV microscope with capillary discharge plasma source (Nitrogen, $\lambda$ = 2.88 nm), ellipsoidal grazing incidence condenser and Fresnel Zone objective (Czech Technical University in Prague)



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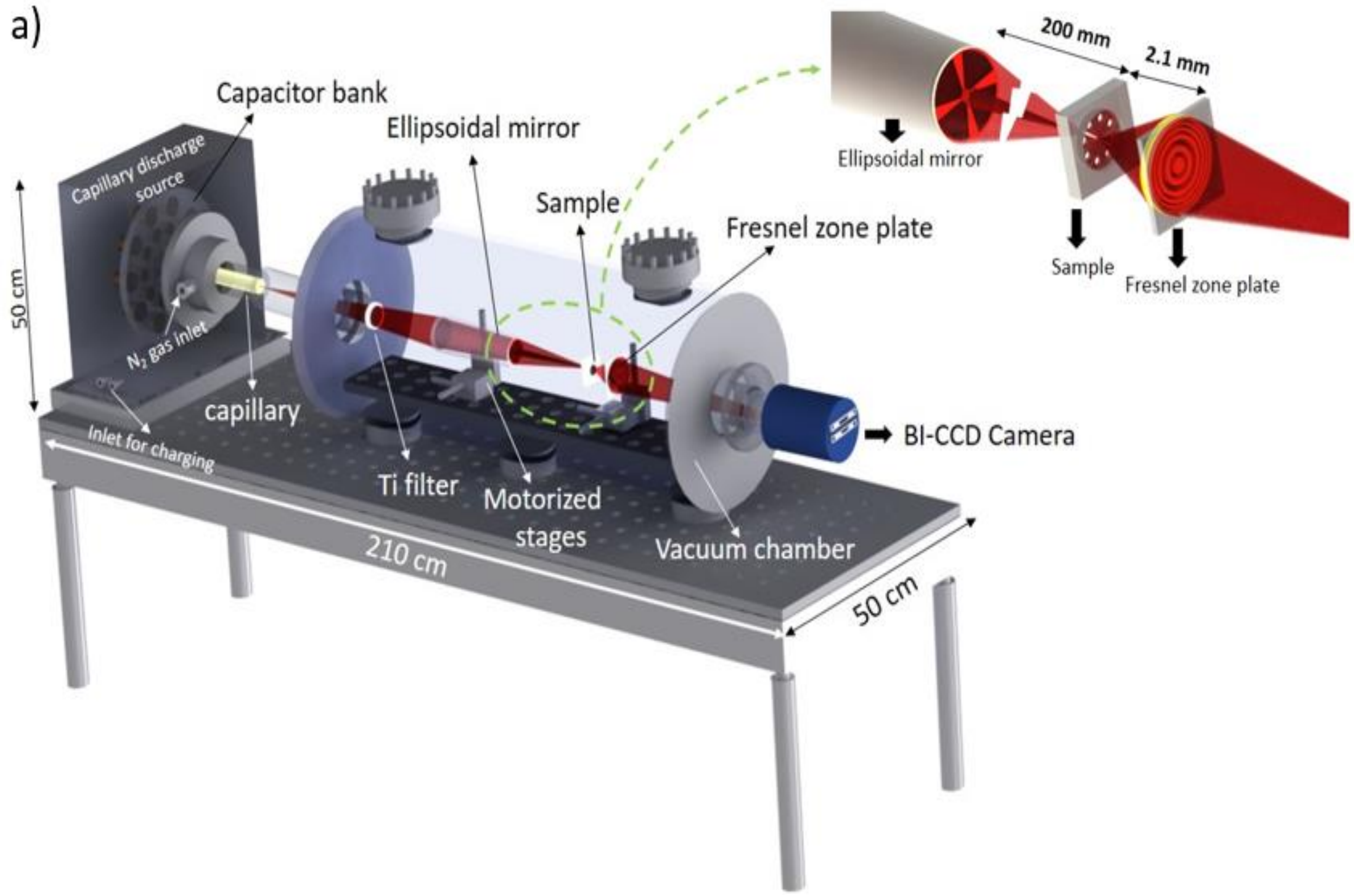

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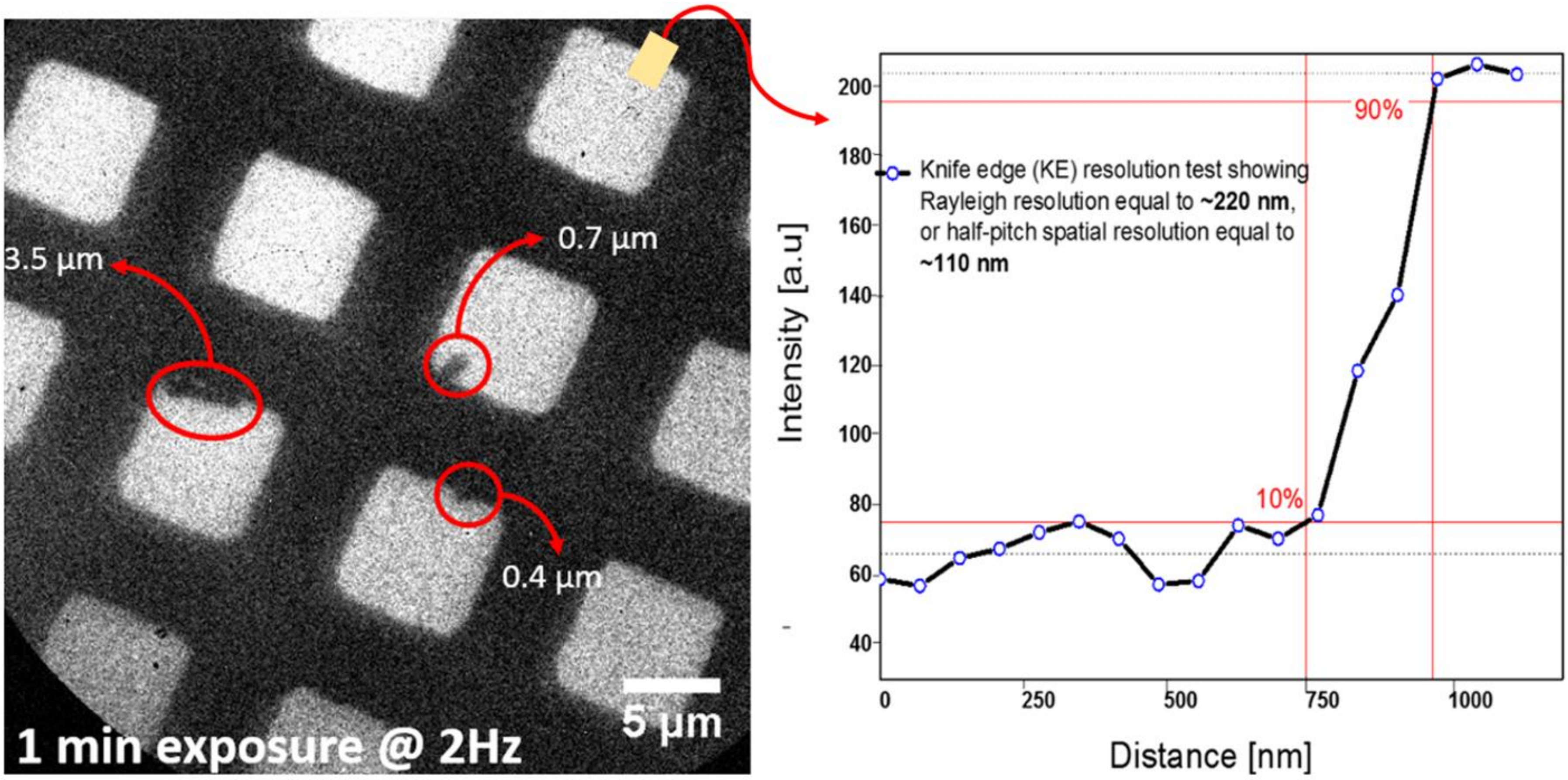
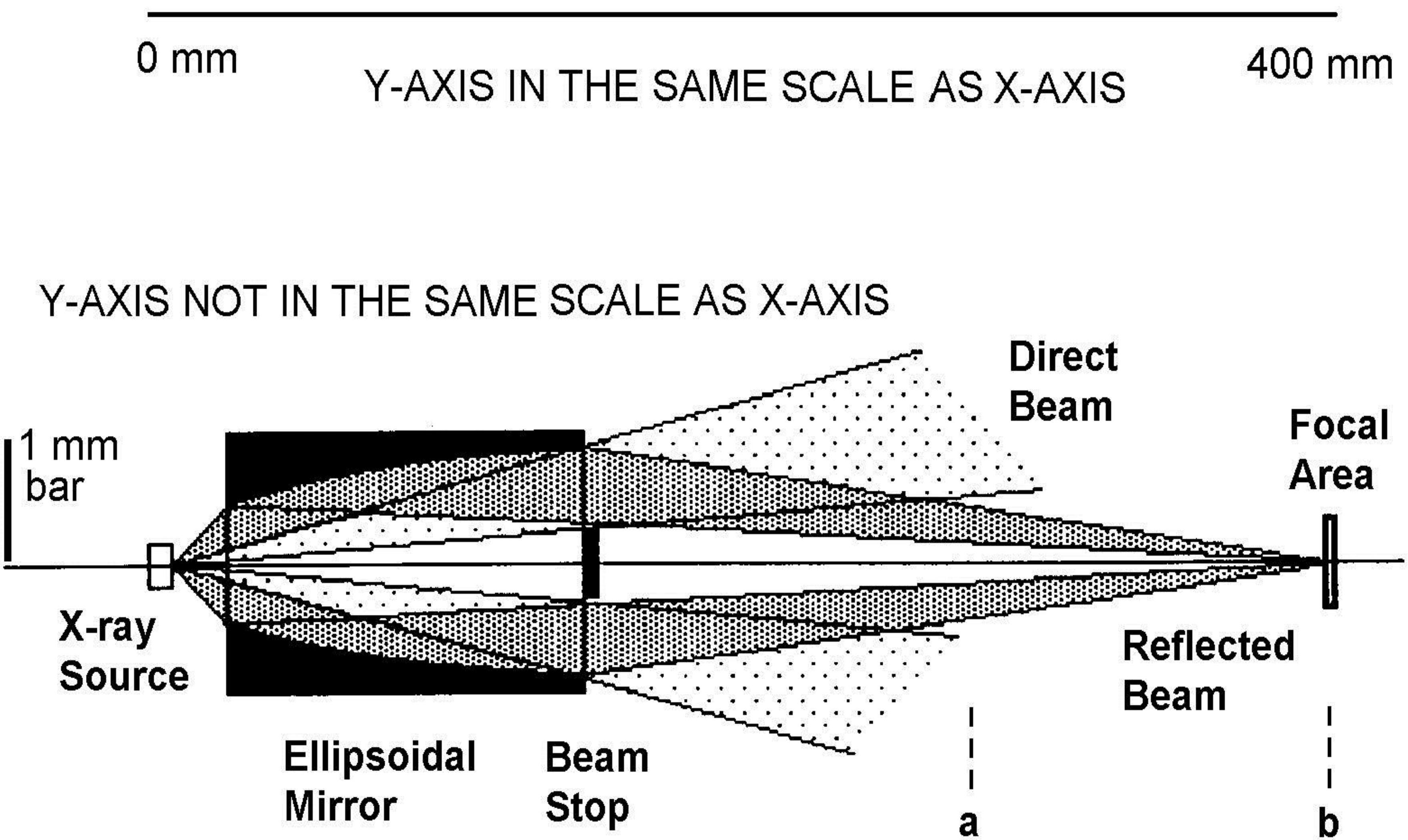


# Replicated GI Mirrors Spectral and Focusing Analysis

# Ellipsoidal optic for 8 keV microfocus source



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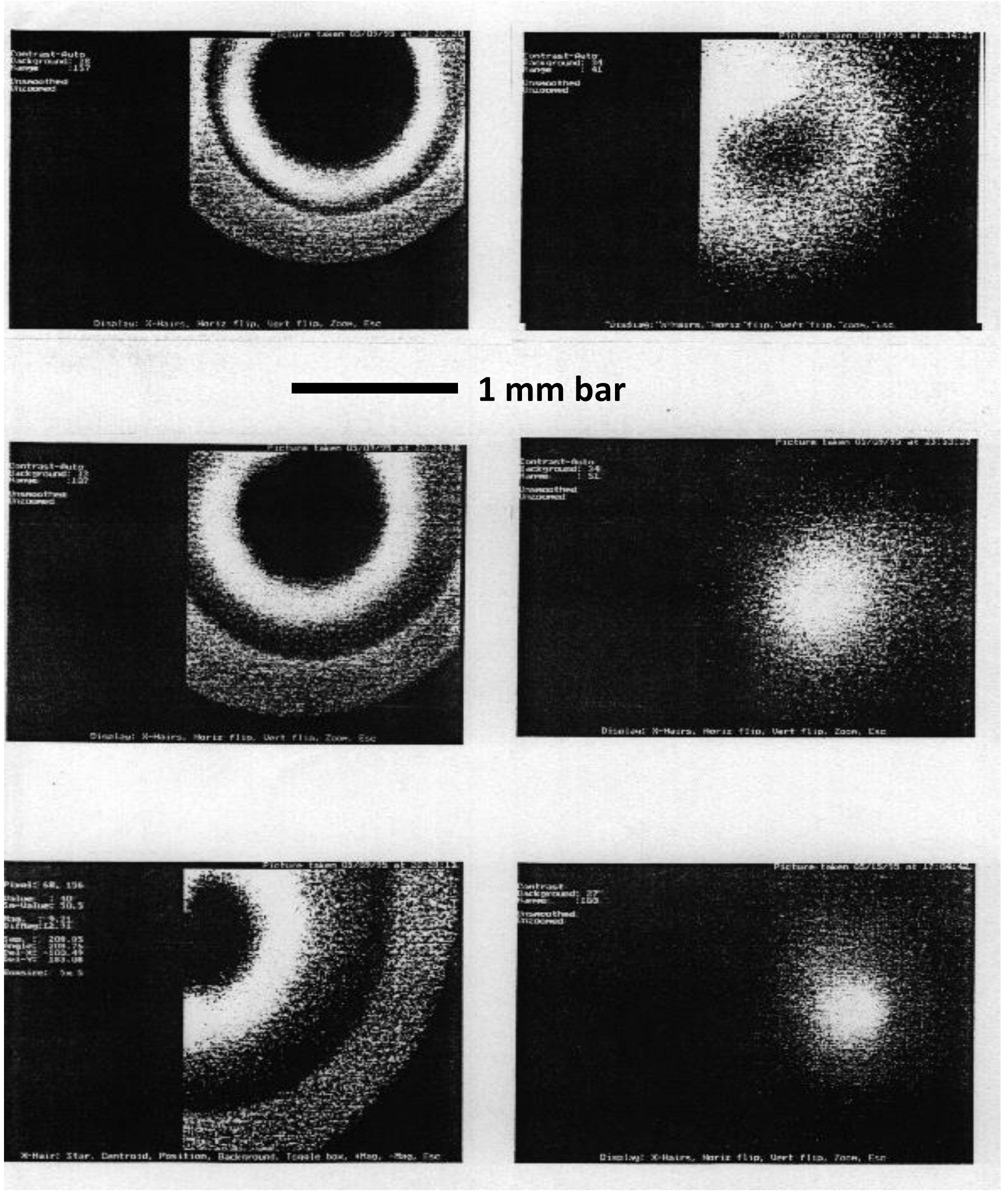
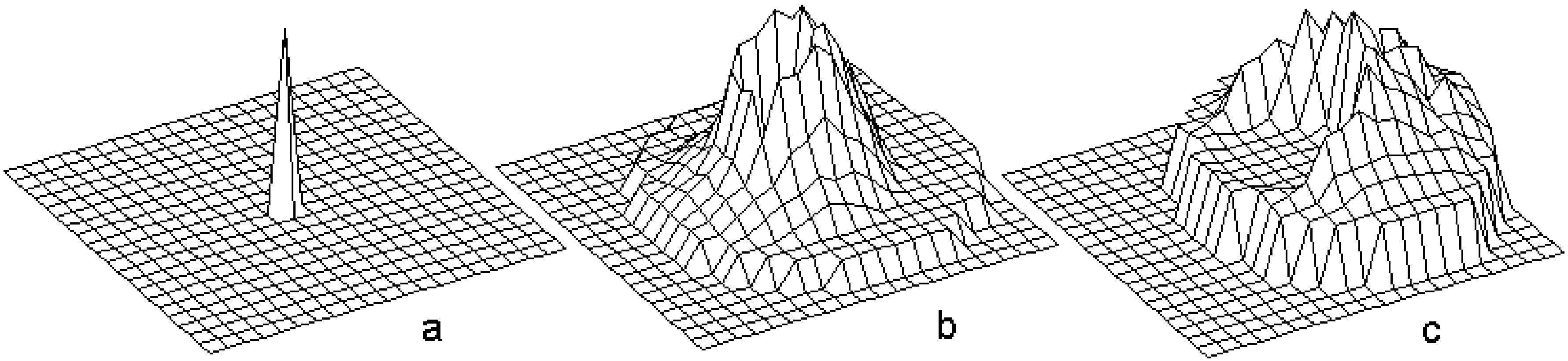



**A series of X-ray beam images behind the output of ellipsoidal mirror with beam stop on the axis.**

**Converging reflected beam and diverging direct beam are clearly distinguishable.**

# Ellipsoidal optic for 8 keV microfocus source

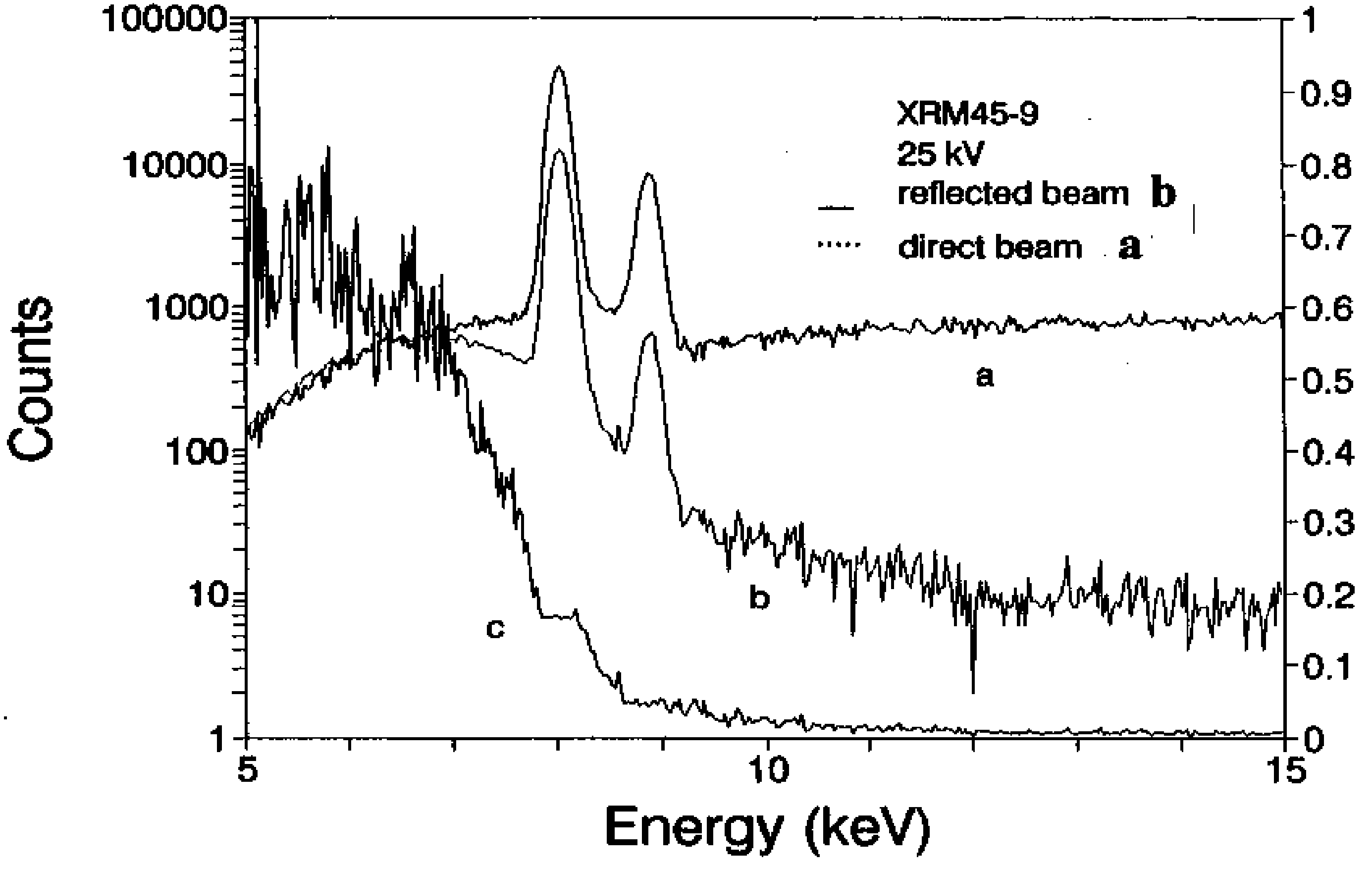
## Focal spots for off-axis source position (ray-tracing model)



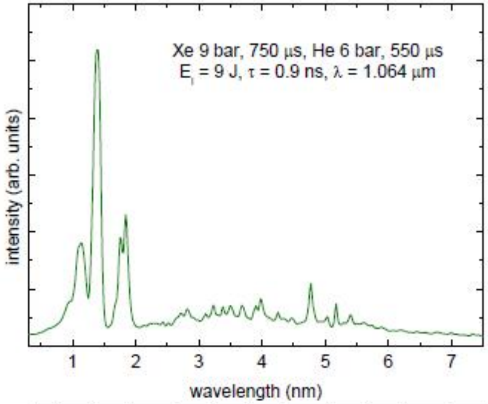

Graphs a to c showing the effect of point-like X-ray source off-axis displacement on the detector intensity distribution for ellipsoidal mirror.

a – 0 μm source displacement,
b – 200 μm displacement,
c – 400 μm displacement.

# Ellipsoidal X-ray Mirror as a Spectral Filter



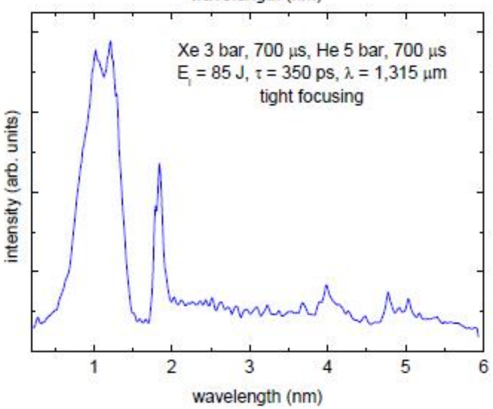
## Focusing system prepared for a soft X-ray plasma source based on Xe gas target, driven by a 10 J/ 1ns/ 10 Hz Nd:YAG laser system




10J laser system

IOE, Warsaw

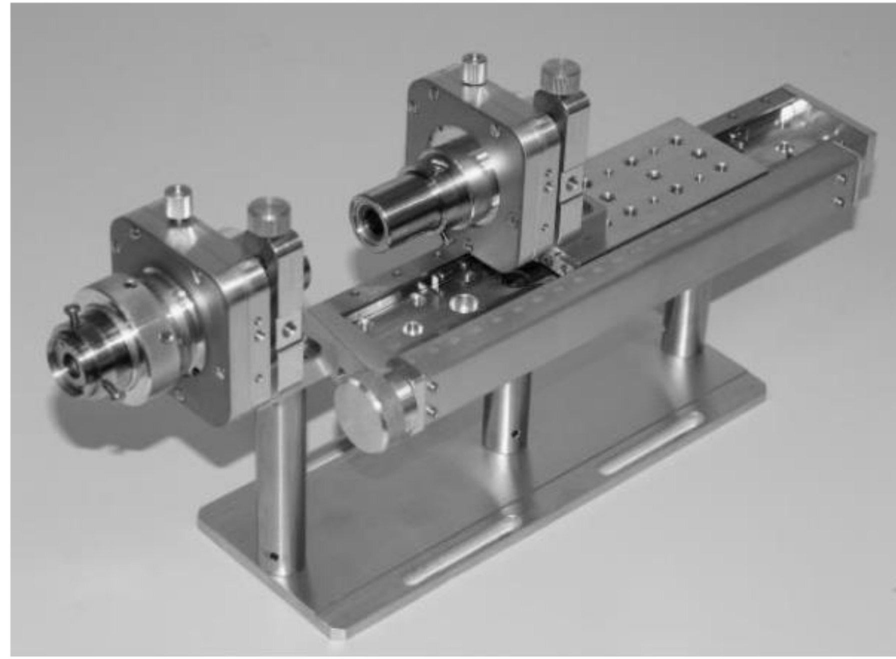
Spectrum for 9J



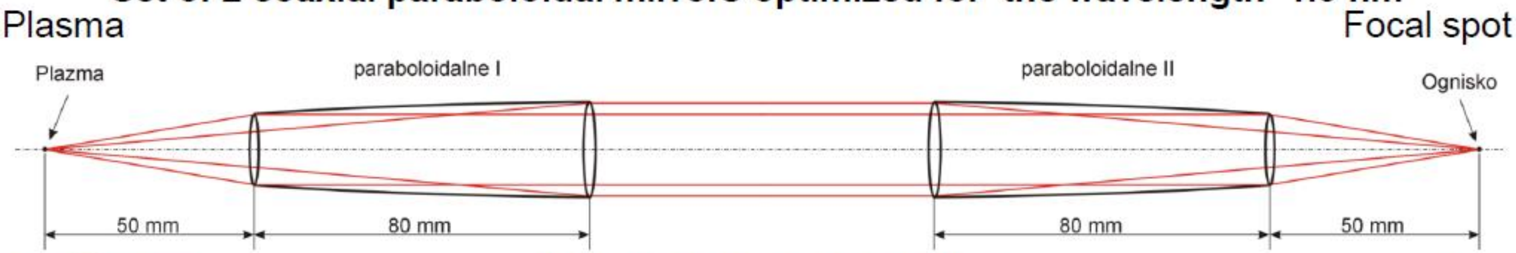

600J laser system

PALS , Prague

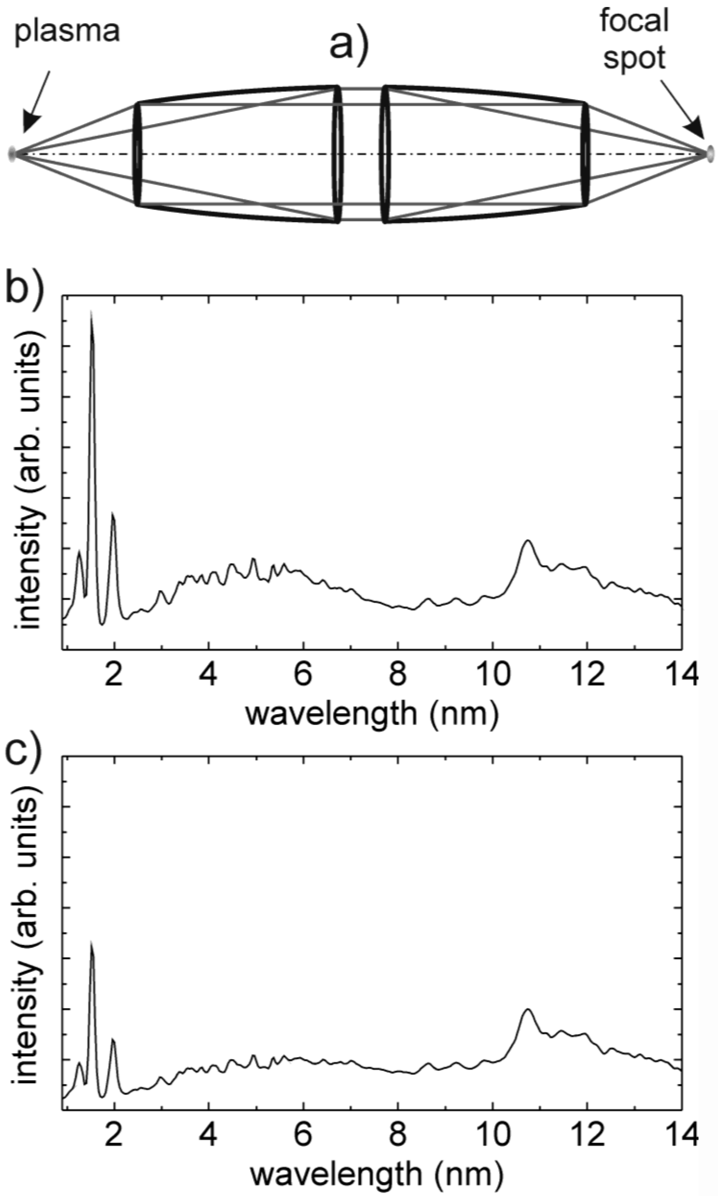
Spectrum for 85J



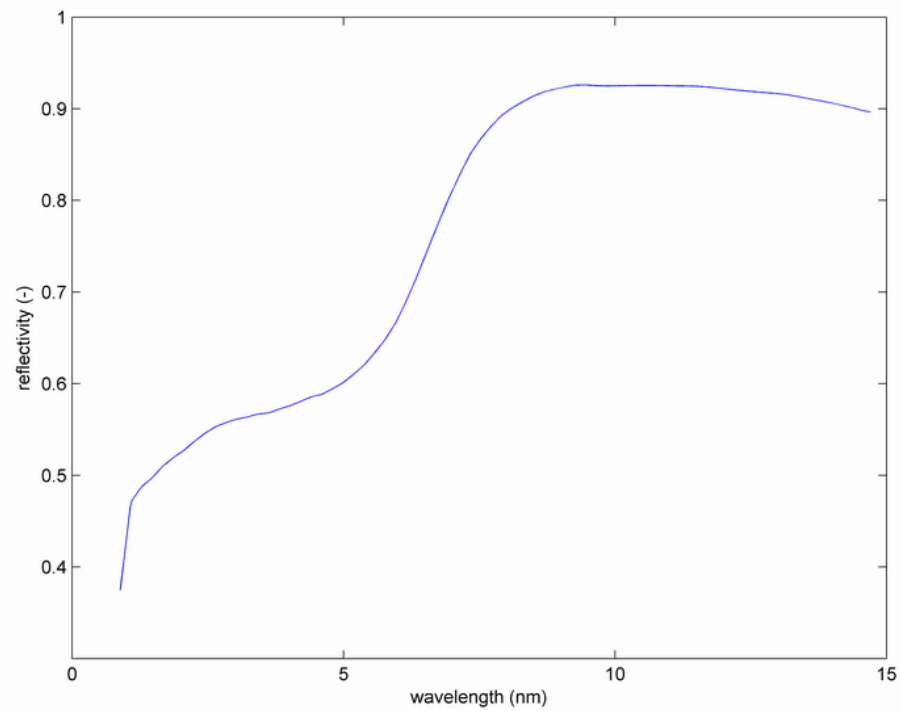
### Set of 2 coaxial paraboloidal mirrors optimized for the wavelength 1.5 nm



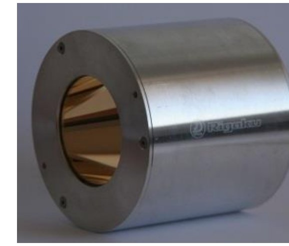
# Measurement of SXR GI mirror spectral reflectivity

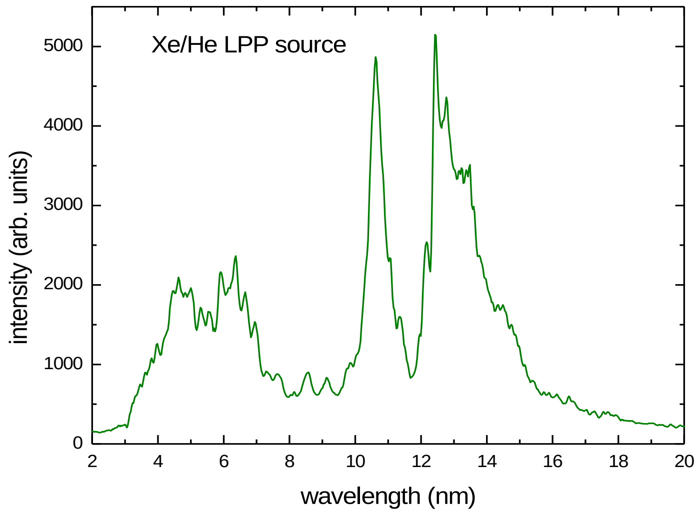



(a) Schematic view of the paraboloidal collector
(b) Spectrum of the unaltered Xe plasma radiation
(c) Spectrum of Xe plasma radiation focused using the paraboloidal collector
(d) Calculated spectral reflectivity in 1-15 nm range



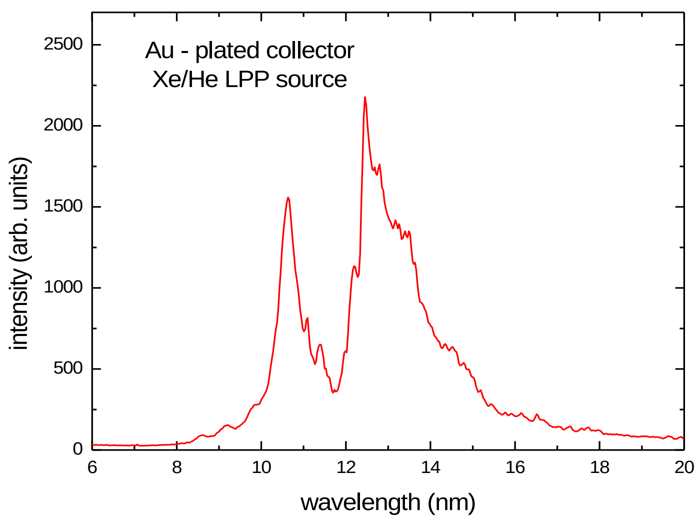
# Comparison of Au and Ru coated EUV collector reflectivity

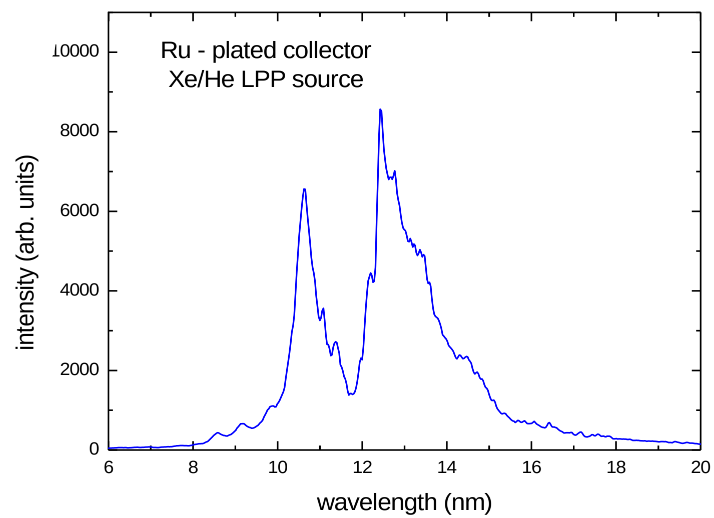





**Spectra of Xe/He plasma radiation:**

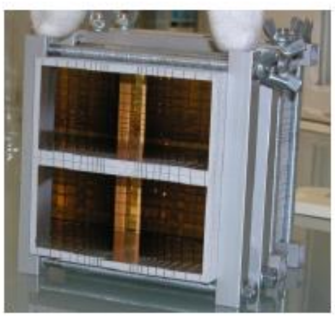
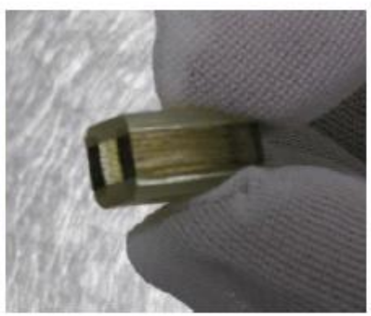
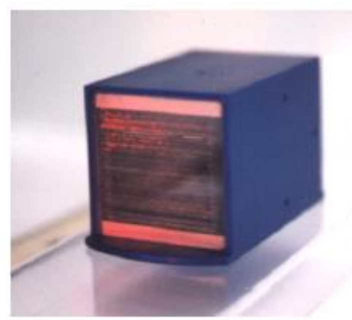
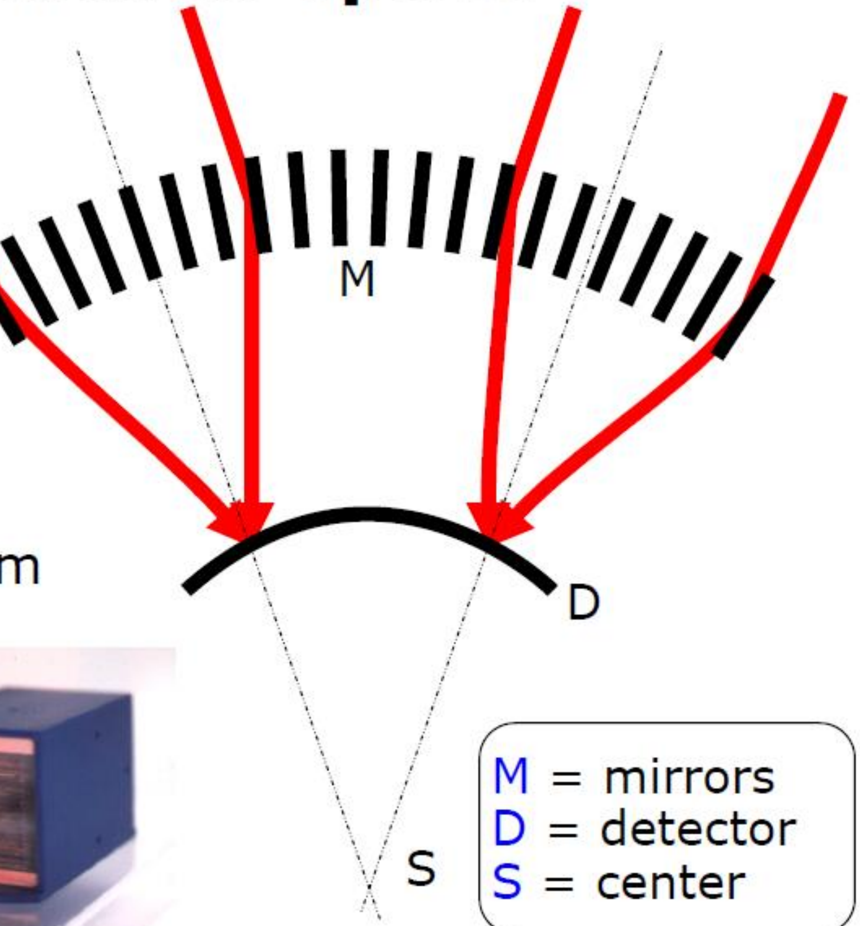
a) **Xe plasma emission**
b) **Radiation reflected from the Au coated collector surface**
c) **Radiation reflected from the Ru coated collector surface**



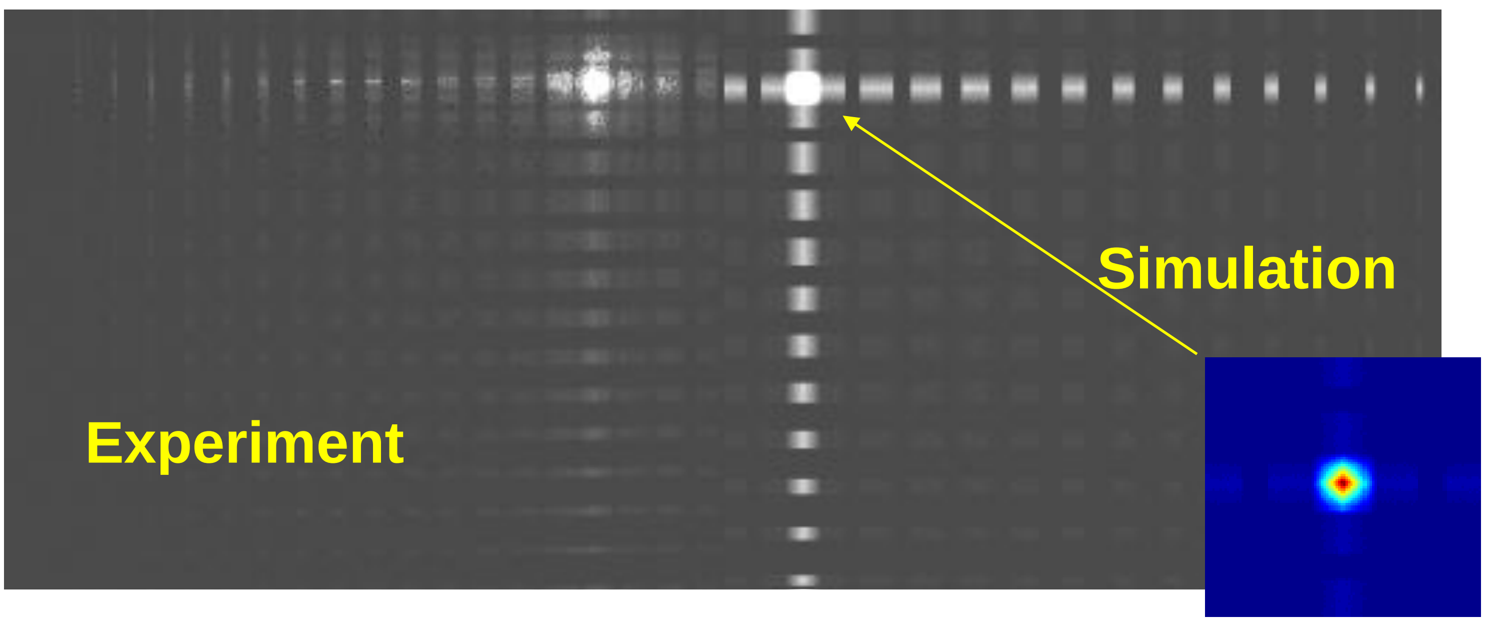
# Lobster eye & multifoil optics

- Wide FOV
- Glass and/or silicon substrate for soft X-rays
- Planar & ellipsoidal mirrors
- Foils 3x3 mm to 300x300 mm
- Foil thickness from 30 μm to 1 mm

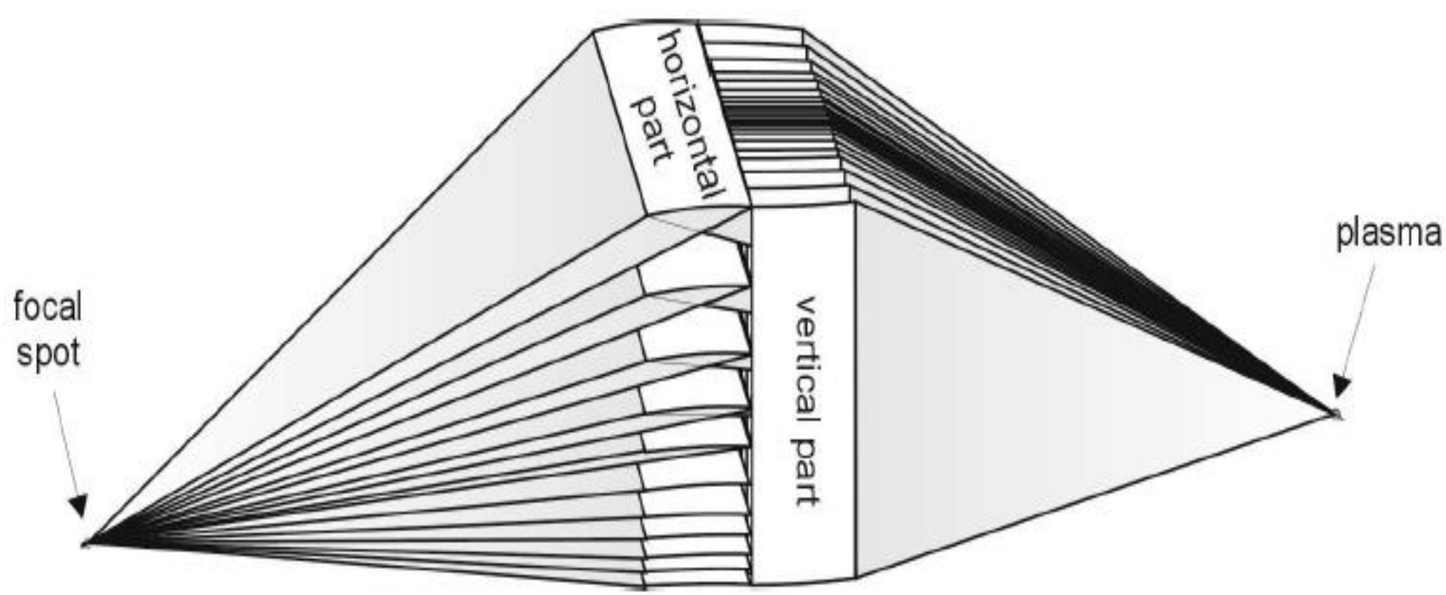





# X-ray LE - experiment vs theory

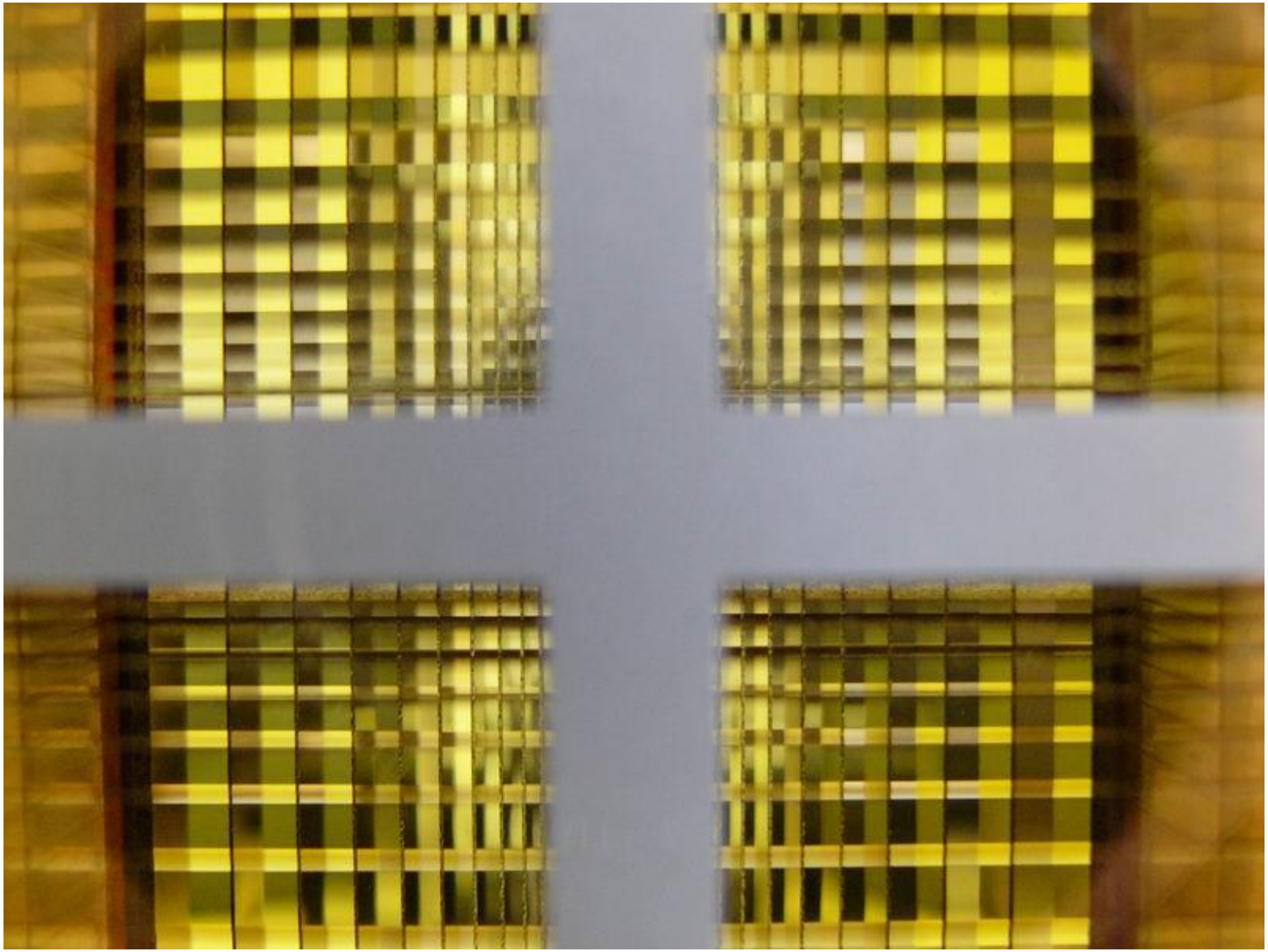
- Point-to-point focusing system
- Source: 20 μm size, 8 keV photons
- Source-detector distance: 1.2 m, 8 keV photons
- Detector: 512x512 pixels, 24x24 μm pixel size
- Intensity Gain: G=570 (experiment) vs. G=584 (comp. simulation)



# Focusing of XUV radiation and XUV modification of materials (experiments at CTU, PALS and WAT)




**Schematic view of one half of the multi–foil (MFO) XUV bifacial Kirkpatrick-Baez condenser – experiments at WAT, Warsaw.**



MF K-B system for EUV lithography

# Summary

- **EUV/BEUV/SXR/XR grazing incidence mirrors have been studied and analyzed**
- **Selected applications of EUV/BEUV/SXR/XR mirrors and detectors were presented**

# THANK YOU FOR ATTENTION

**Prague**

# Appendix

# X-RAY MICROSCOPY

# and

# X-RAY μTOMOGRAPHY

# SUBMICRON RESOLUTION IMAGING REQUIREMENTS

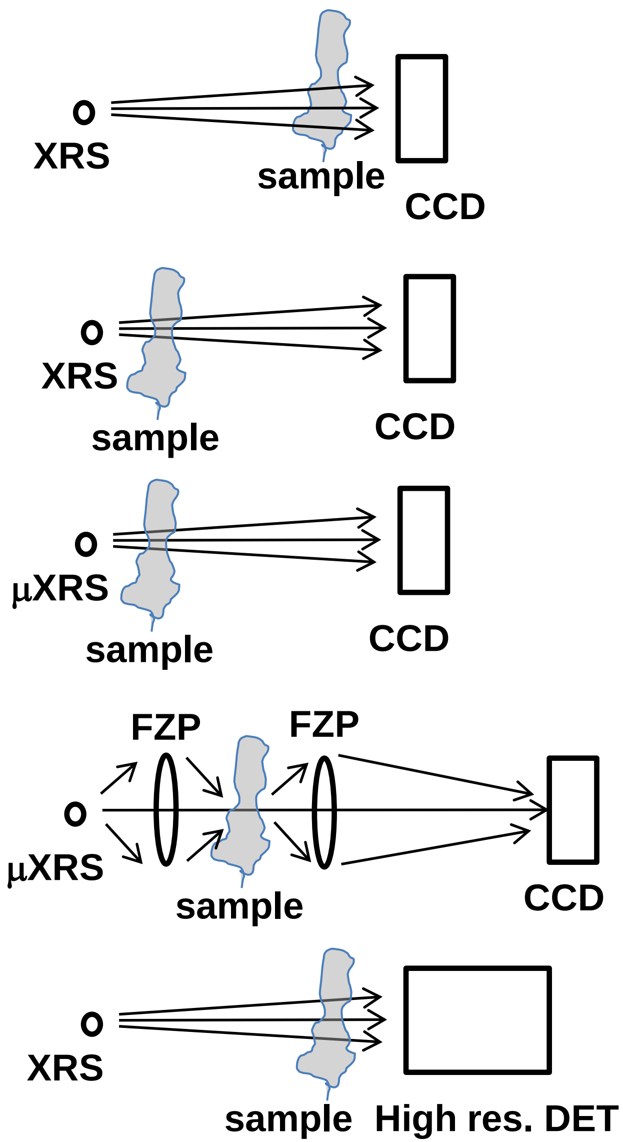
**SOURCE – small size or divergence**

**OPTICAL SYSTEM – magnification >> 1**

**DETECTOR – small pixels**

**high resolution**

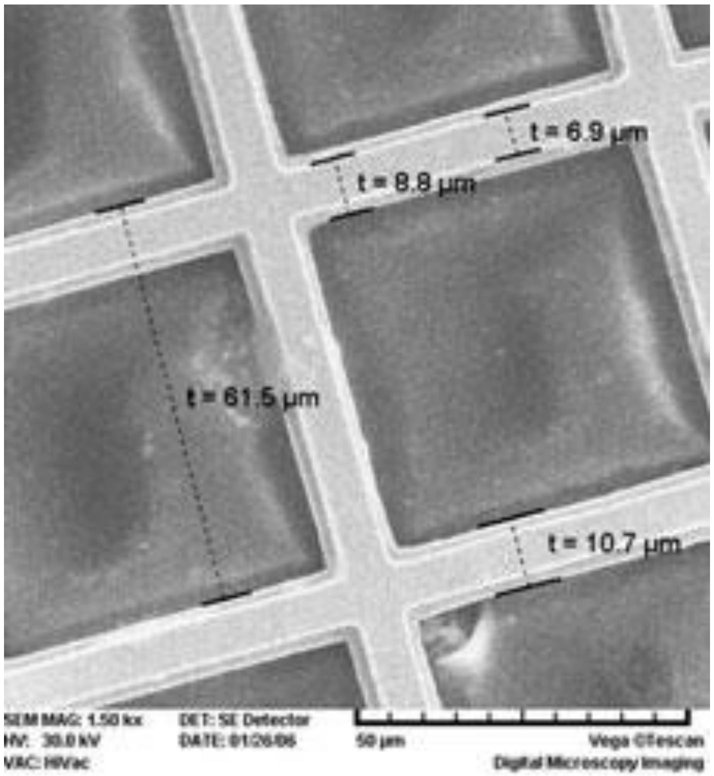
**High resolution imaging in EUV and X-ray region requires novel advanced imaging system components and methods**



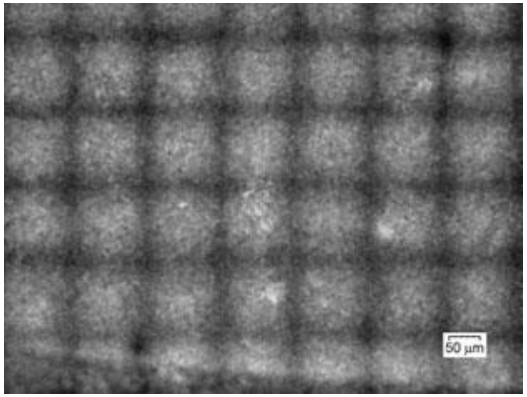

## EUV/XR IMAGING - METHODS

| Source size/power | Optics | Detector pixel size | Resolution | Signal | Cost |
|---|---|---|---|---|---|
| Large/ High | No | Large | Low | High | Low |
| Large/ High | No | Large | Very Low | High | Low |
| Very Small/ Very Low | No | Large | High | Low | High |
| Small/ Low | Yes | Large | Very High | Very Low | Very High |
| Medium/High | No | Small | High | High | High |

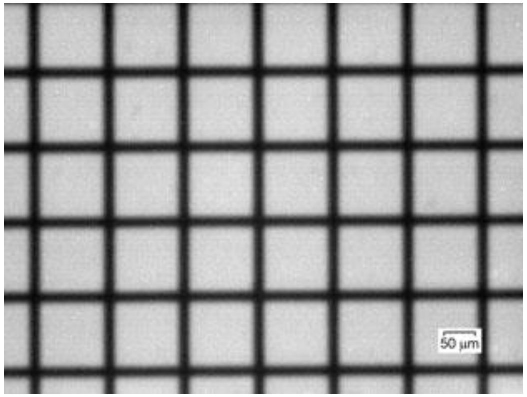
# Powder scintillator and monocrystal scintillator image convertor comparison




The gold mesh sample - SEM image




X-ray image of the gold mesh (P43 phosphor screen)

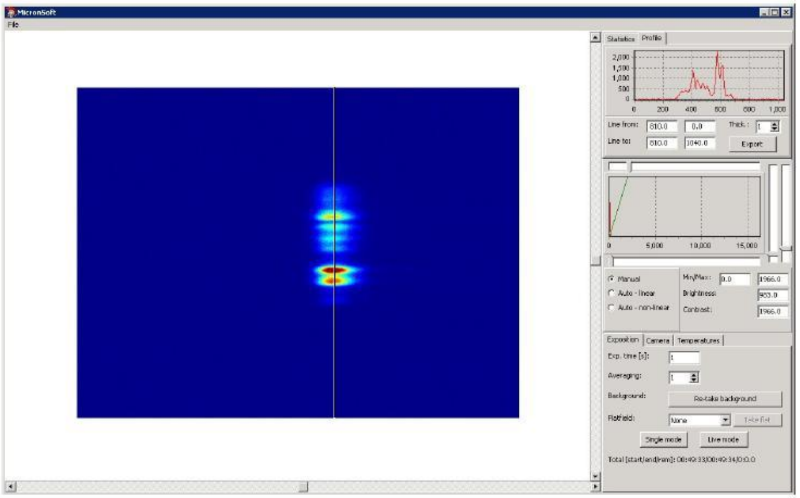



X-ray image of the gold mesh (YAG:Ce scintillator screen)

# Xsight™ Micron X-ray CCD Camera

**Applications:**,

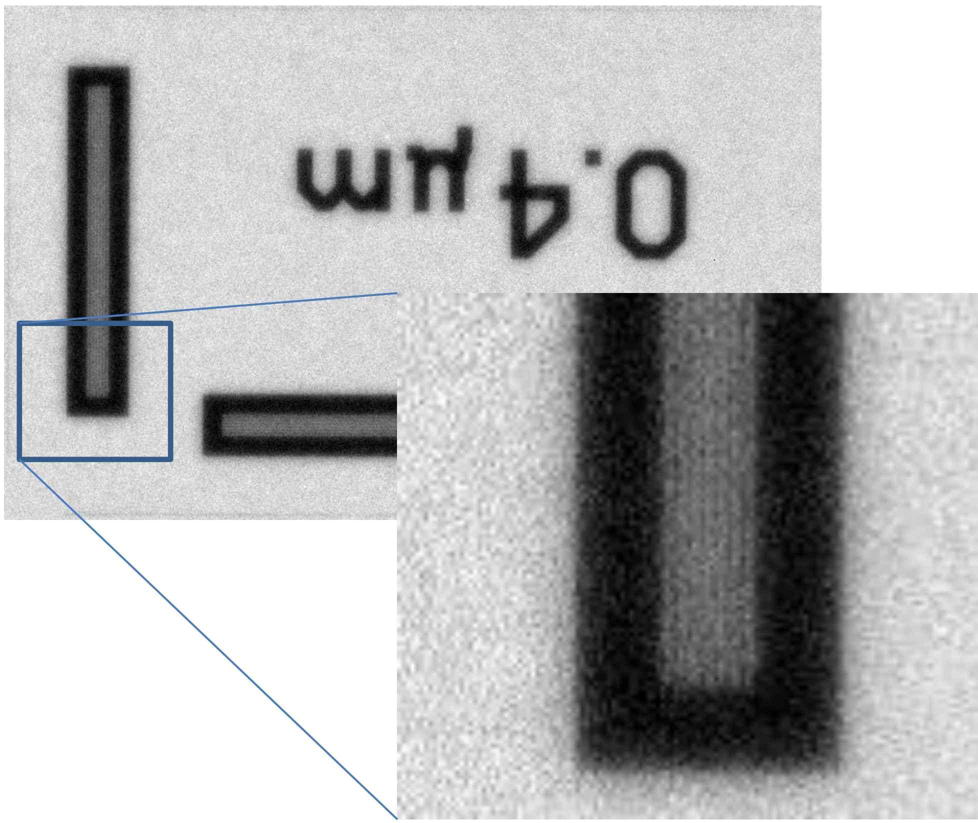
- **X-ray microscopy**
- **X-ray microtomography**
- **X-ray optics adjustment & metrology**
- **Phase contrast X-ray imaging**

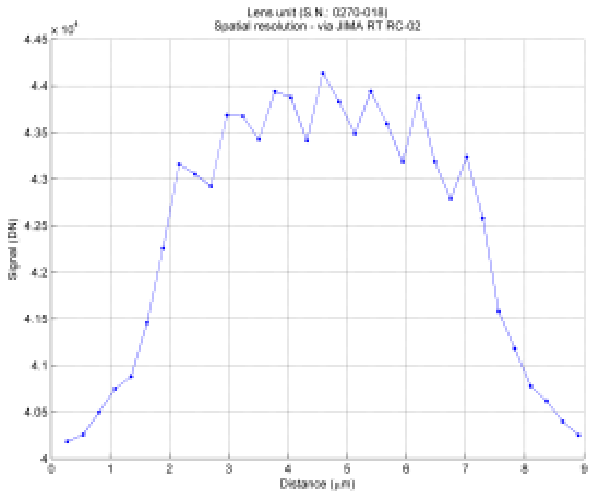



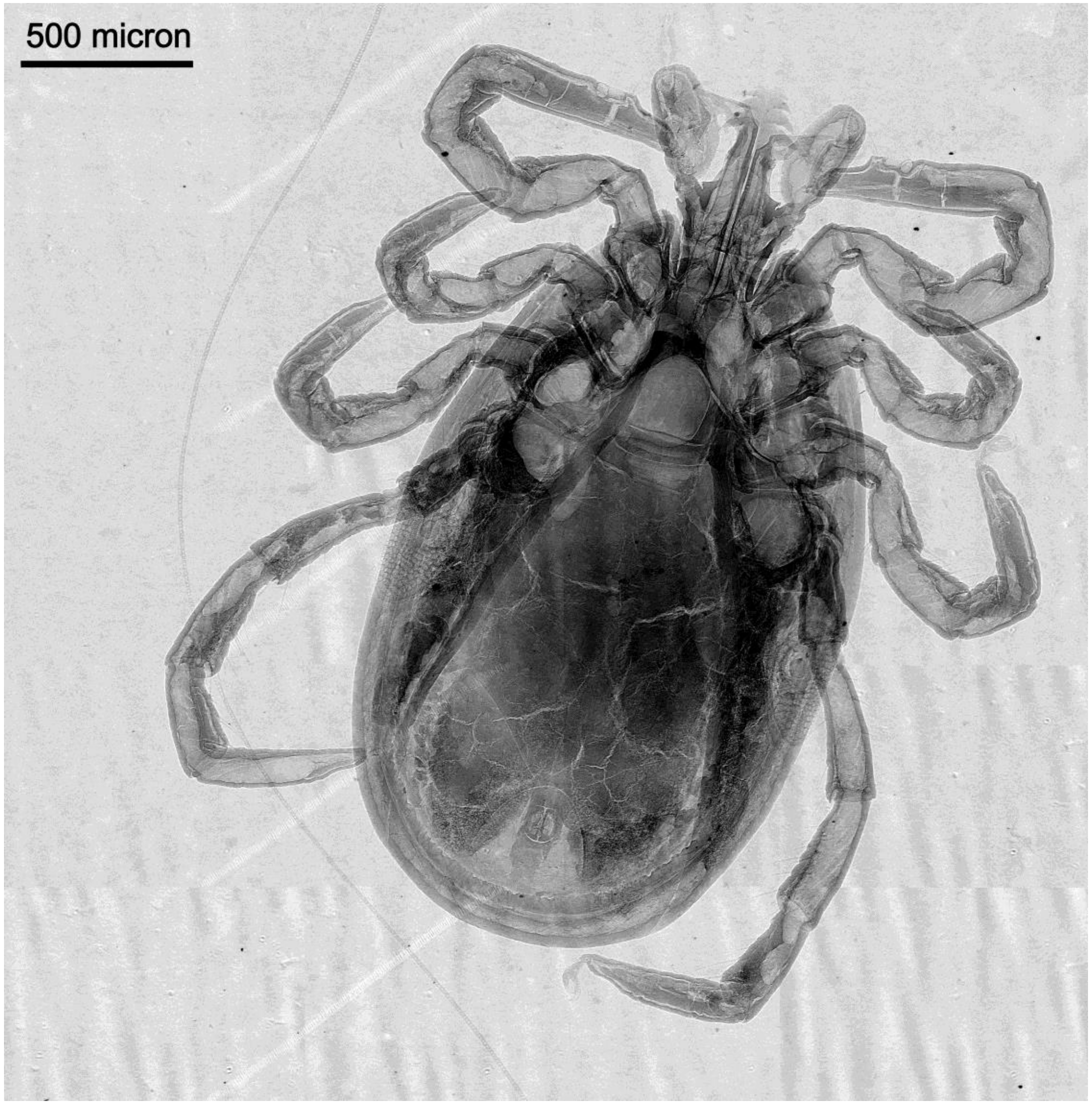
**Field of view: 0,90 mm x 0,67 mm**
**Resolution: ≤ 1 µm (@ 8 keV)**
**Spectral range: 50 eV to 35 keV**
**Exposure time range: 20 µs to 500 s**
**Dynamic range: 70 dB**
**Dimensions: 60 x 70 x 250**
**Weight: 2.5 kg**

# Xsight™ Micron X-ray CCD Camera Vacuum front end version

Spatial resolution of the X-ray camera evaluated
with JIMA RT RC-02 high resolution X-ray chart at 8 keV.

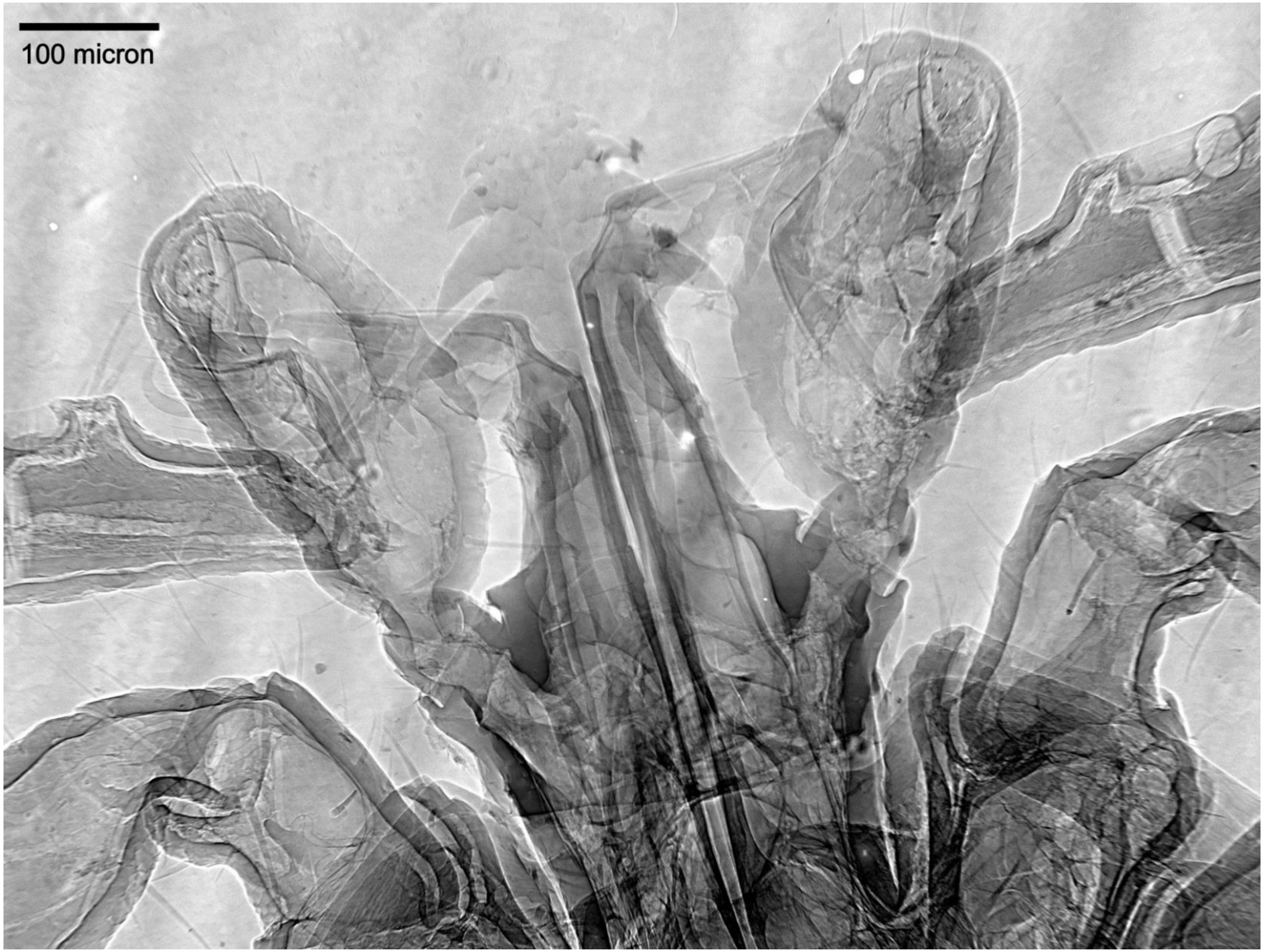





**X-ray image of Ixodes Ricinus**

**(Taken by XSight Micron at RITE laboratory using 80 W microfocus X-ray tube with Cu target**




**X-ray image of Ixodes Ricinus**

**(Taken by XSight Micron at RITE laboratory using 80 W microfocus X-ray tube with Cu target)**

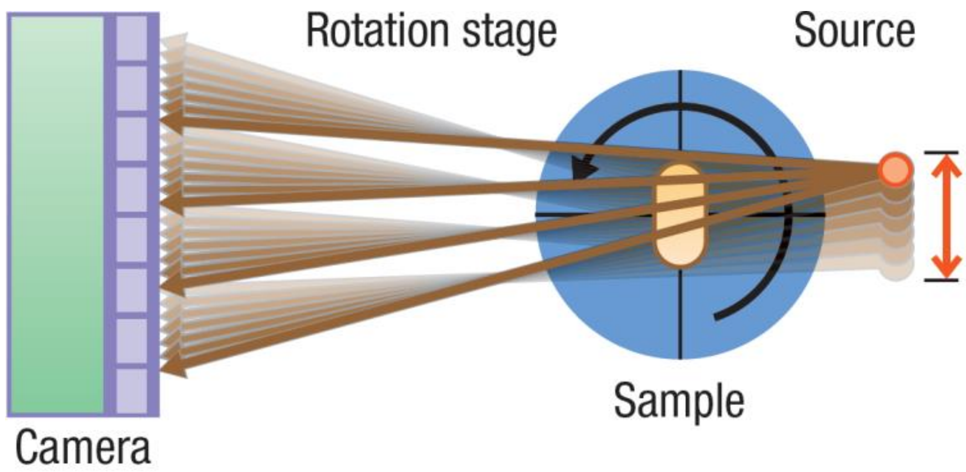
# Rigaku nano3DX

# High Resolution 3D X-ray Microscopy

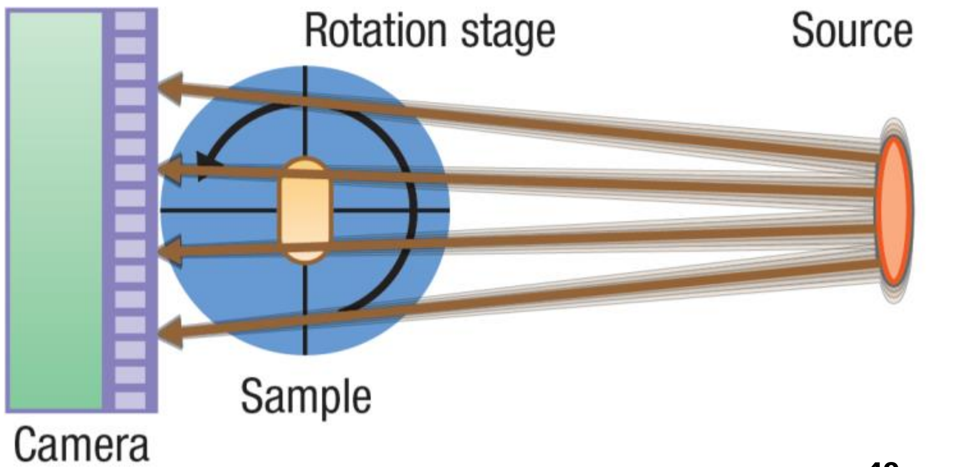
| | |
|---|---|
| **POWER :** | **Ultra High flux, up to 1200 W** |
| **ENERGY:** | **Cr, Cu , Mo** |
| **DETECTOR:** | **3300 x 3300 x 2500 Matrix** |
| **OPTICS:** | **No projection magnification** |
| **EASY:** | **Minimal Alignment or optimisation** |

# Increased resolution by novel design (Rigaku)

Conventional design requires a small source, operating at low power, and extreme stability to prevent smearing.




The Rigaku design places the sample close to a high resolution detector allowing for a near parallel beam experiment making the system robust to environmental changes.



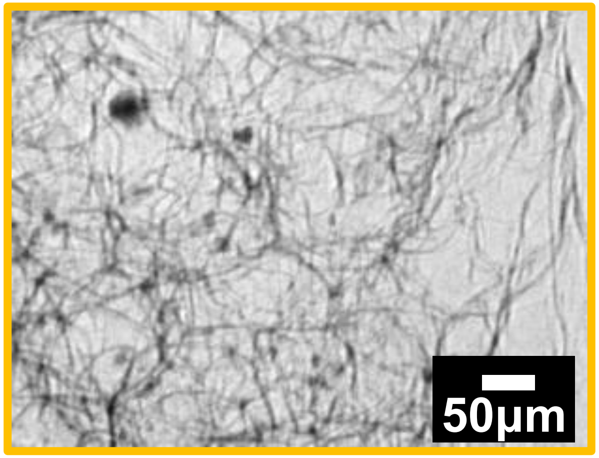
# High Contrast for Light Elements



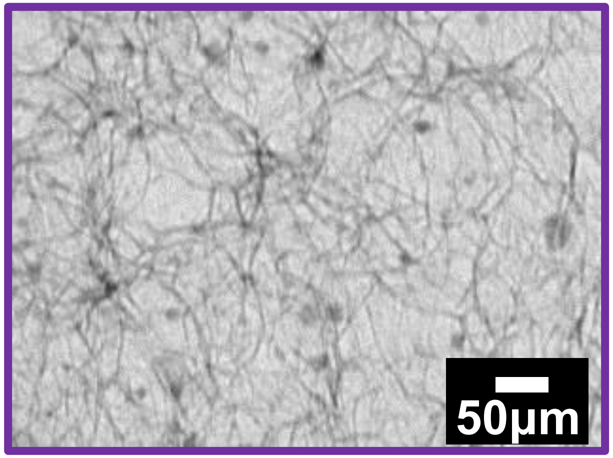
## Selectable target materials:

**Images taken with different X-ray energies**

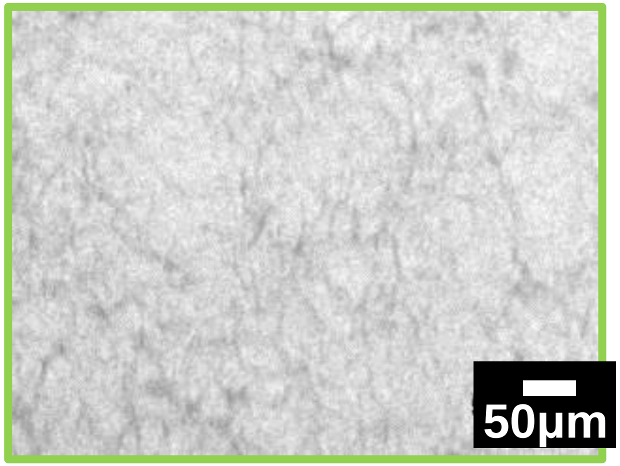
Projection images of resin foam
(1.08 μm/pixel; Exposure 1 sec)




**5.4 keV Cr radiation**




**8 keV Cu radiation**




**17 keV Mo radiation**

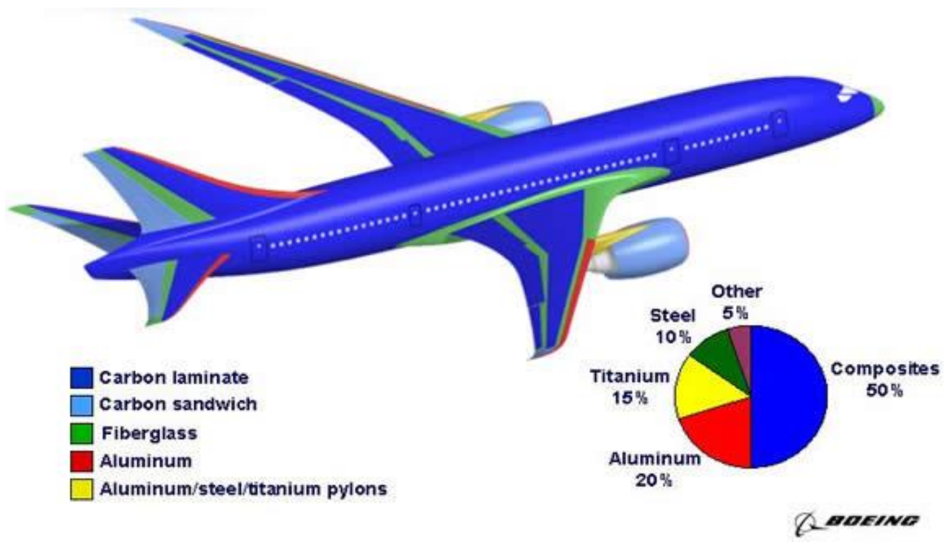
← Suited for organic resins, etc.

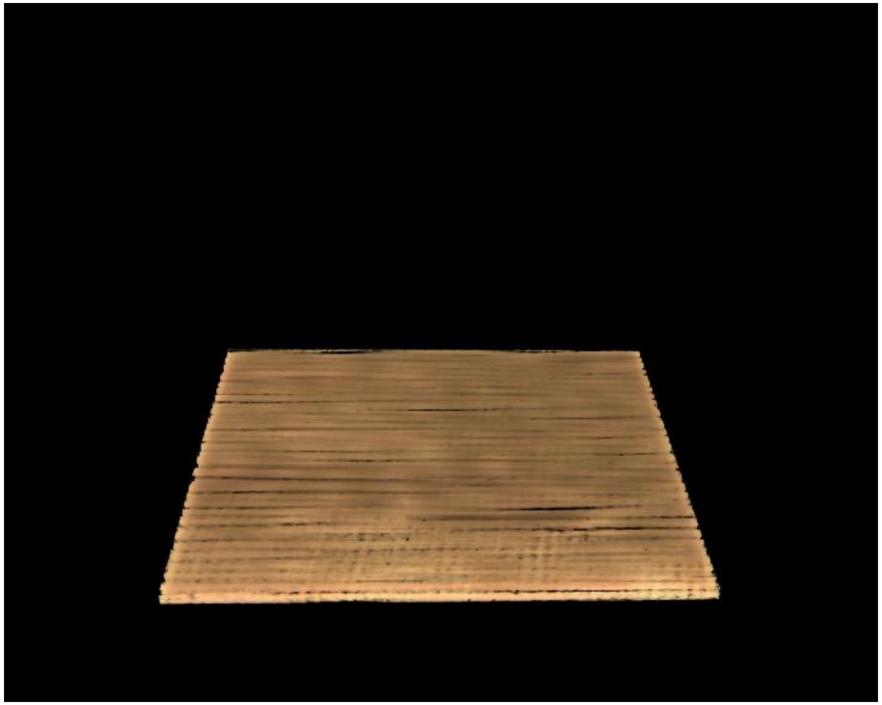
Suited for light metals, etc. →

**By using low energy characteristic x-rays, nano3DX gives a clear detail of a resin sample, which is difficult to measure by using continuous white radiation**

# Carbon Fiber Reinforced Plastic (CFRP) is Now Coming

Body for future car



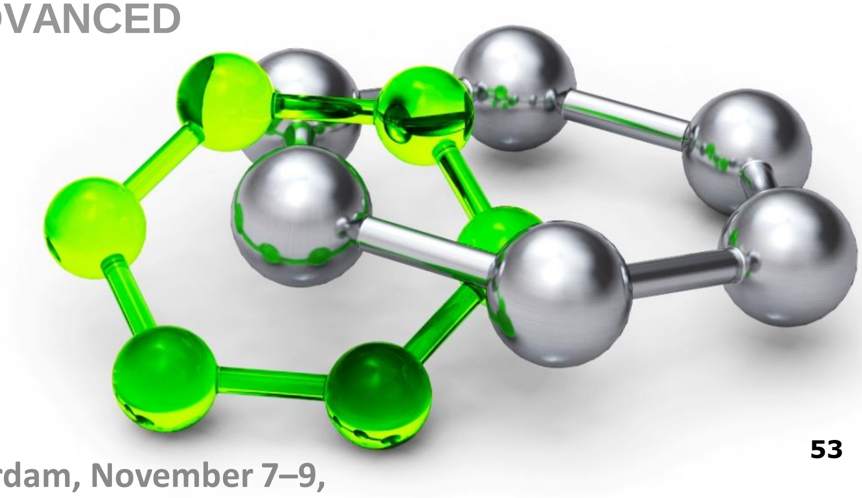
CFRP is introduced for aircraft body

CEITEC

Central European Institute of Technology
BRNO | CZECH REPUBLIC

# CEITEC X-ray micro CT and nano CT

MATERIAL CHARACTERIZATION AND ADVANCED COATINGS RESEARCH GROUP



EUROPEAN UNION
EUROPEAN REGIONAL DEVELOPMENT FUND
INVESTING IN YOUR FUTURE

2007–13
OP Research and Development for Innovation

# Summary

- **Submicron resolution EUV/BEUV/SXR/XR imaging detectors for microscopy and tomography have been developed and characterized**

# THANK YOU FOR ATTENTION

Prague