CROWN

Confocal micRoscopy at sOft x-ray WaveleNghts

Proposal for 2024-2025 [LNF ……. Trieste]

We propose a new concept for soft-x-ray/UV microscopy based on the development of a confocal microscope using bent meta-lenses (Micro-Channel Plates – MCPs) suitable for SR, FELs, betatrons and conventional sources.

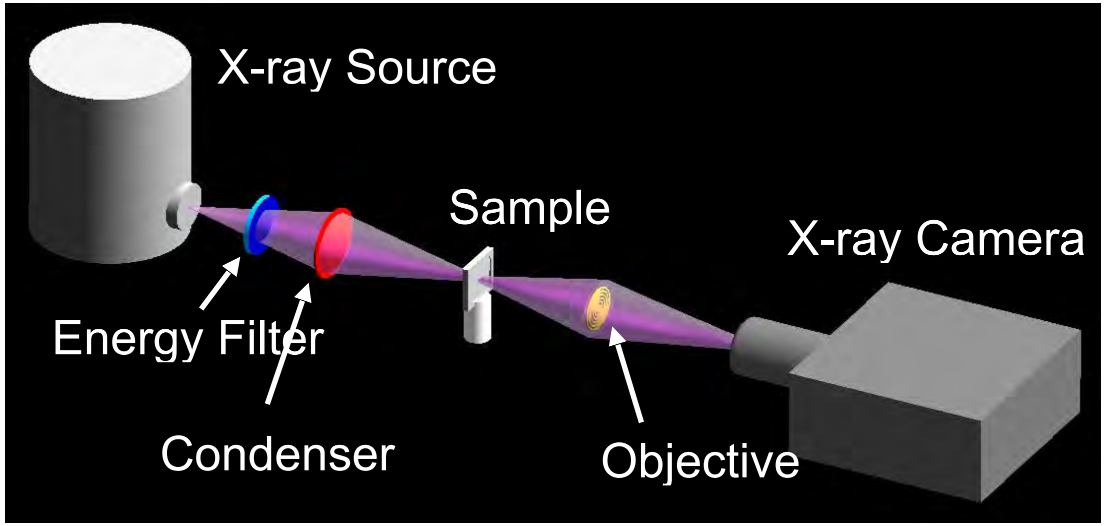
Following the tests performed with synchrotron radiation and the instrumentation already available the microscope will demonstrate the feasibility to image objects/samples placed inside the focal spot of a bent MCP. The microscope considers a full field transmission layout and two low cost-low weight meta-lenses.

Facilities for the R&D are available at the INFN in the Laboratori Nazionali di Frascati and in Trieste. In particular the set up will be assembled and tested at the Circular Polarization beamline (CiPo) of ISM-CNR at Elettra. The existing layout is based on two available six axes manipulators to align the meta-lens in the confocal geometry

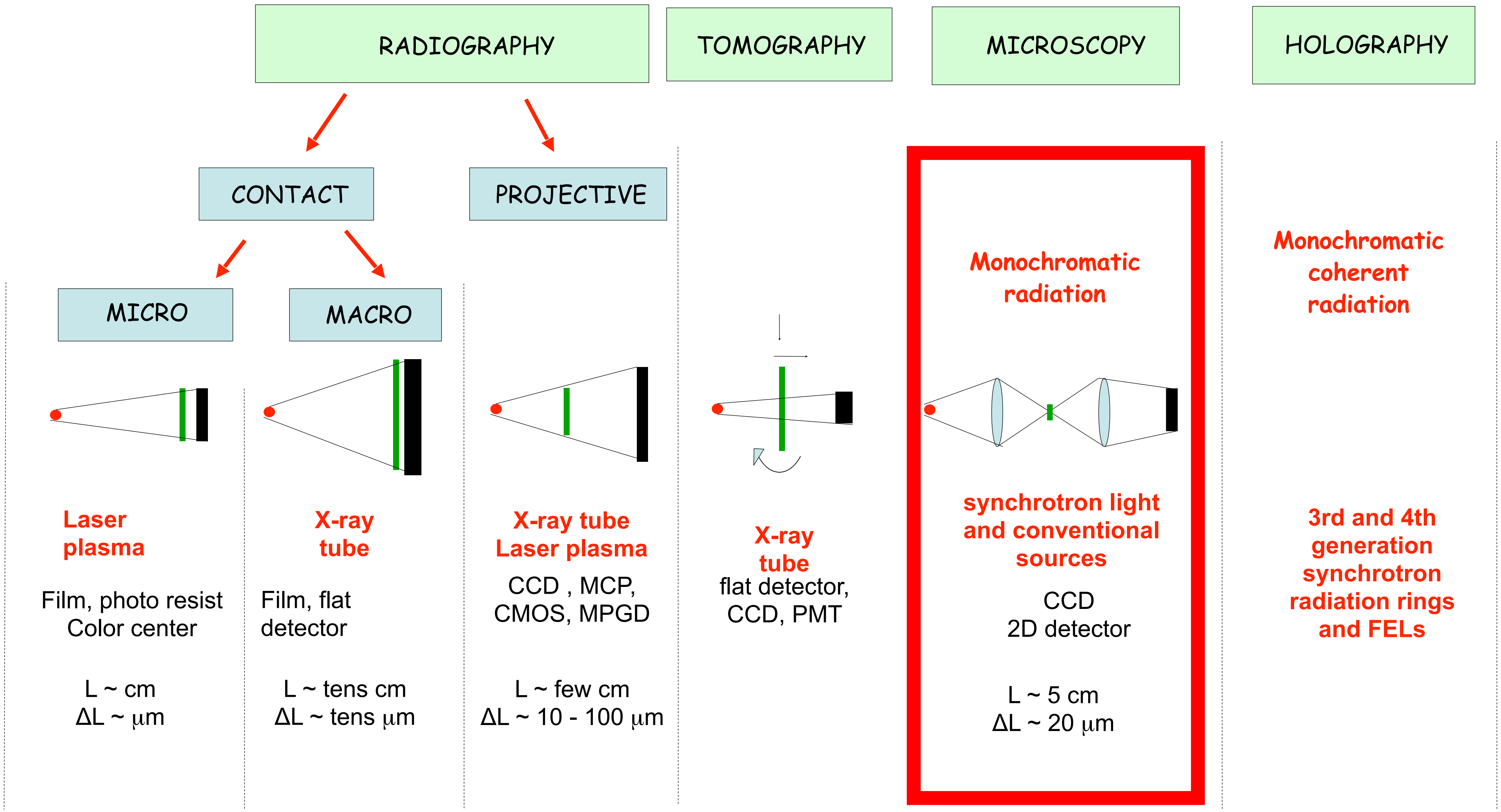
Technical Plan

Year 1: Assembly of the optical layout inside the HV chamber and characterization and tests of the bent meta-lenses.

Year 2: Acquisition of microscopy images to reach an almost TRL4 phase for components/system and validation in the laboratory environment.

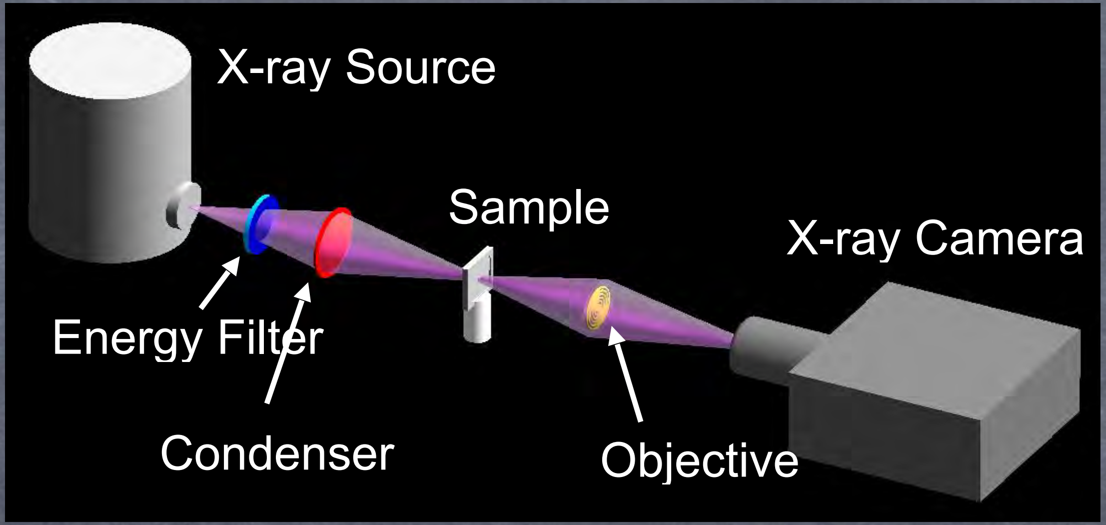


(X-ray) imaging techniques



The microscope

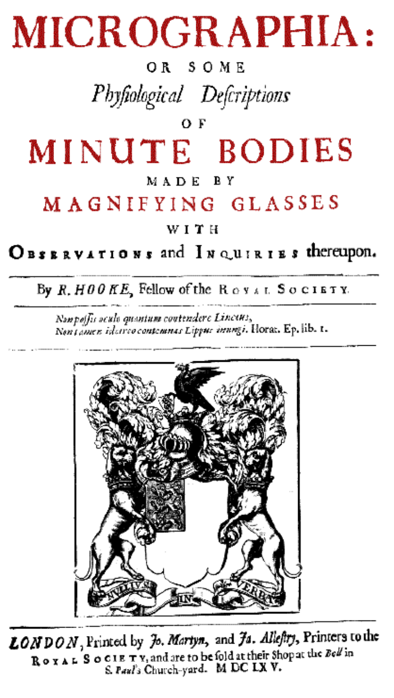
- in a microscope, the radiation emitted by the source is focused on the sample using a "condenser"



- the light transmitted from the sample is collected in the primary focal plane using an "objective" that forms a magnified image of the sample
- the image is then "projected" on the detector

Microscope time-line

- 14th century - born and develop in Italy the lens manufacture technology
- 1595 - Hans e Zacharias Jannsen built the first two lenses microscopy
- 1675 - Anton van Leeuwenhoek observes blood, insects and many other small objects (e.g., cells and bacteria) with a simple microscope
- 18th century - many technological advancements improve microscopy and microscopes (chromatic correction, spherical aberration correction, etc.). Abbe introduces in 1878 the equation that determines the maximum resolution of a microscope
- 19th century - diffusion of the microscopy technique. 5 Nobel prizes are awarded to advancements of microscopy and new microscopy techniques



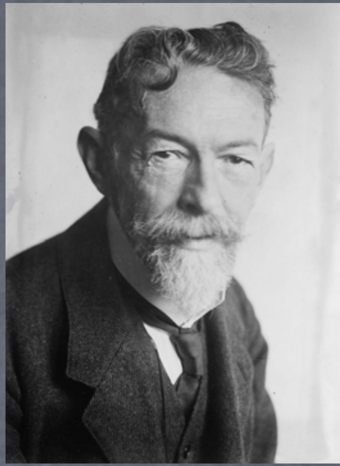
MICROGRAPHIA:
OR SOME
Physiological Descriptions
OF
MINUTE BODIES
MADE BY
MAGNIFYING GLASSES
WITH
Observations and Inquiries thereupon.

By R. HOOKE, Fellow of the Royal Society.

Non possis oculo quantum contendere Linceus,
Non tamen idcirco contemnas Lippus inungi. Horat. Ep. lib. 1.

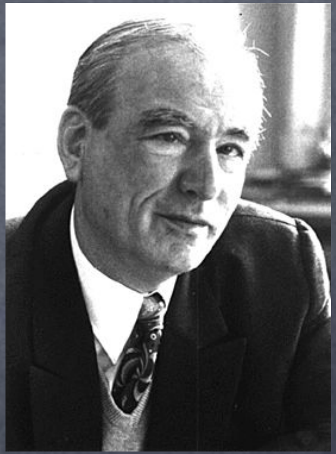
LONDON, Printed by Jo. Martyn, and Ja. Allestry, Printers to the Royal Society, and are to be sold at their Shop at the Bell in S. Paul's Church-yard. M DC LX V.

Nobel prizes



1903 - R. Zsigmondy introduces the ultramicroscopy an instrument that allows to investigate objects smaller than the wavelength of the visible light (Nobel in Chemistry - 1925)

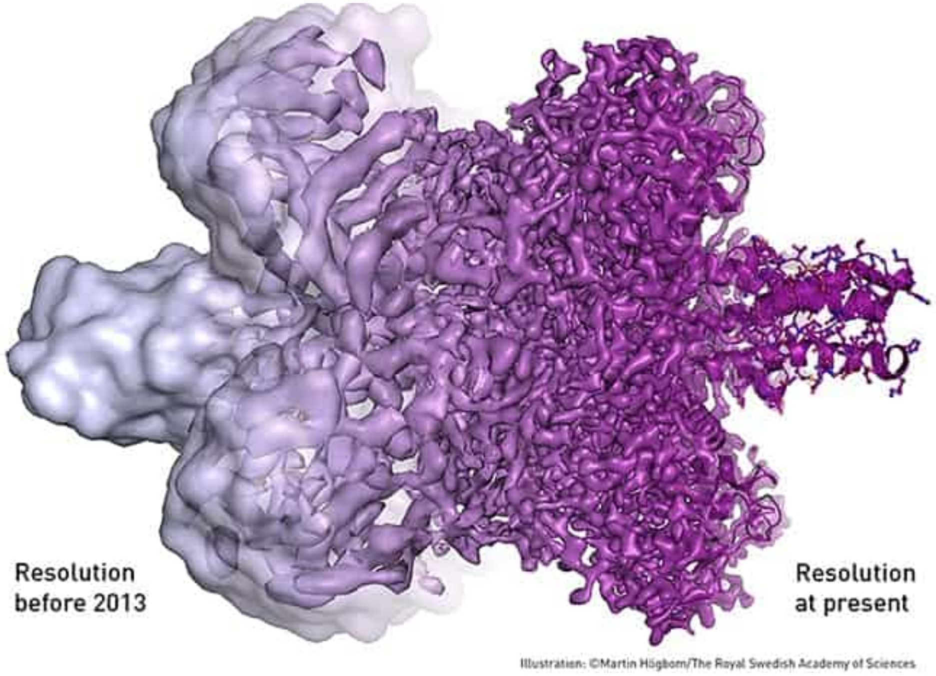
1932 - F. Zernike built the first phase contrast microscope that allows to investigate biological materials (Nobel in Physics - 1953)



1938 - E. Ruska develops the electron microscope. This instrument allows to push forward by several orders of magnitude the achievable spatial resolution of a microscope. (Nobel in Physics - 1986)

1981 - G. Binnig and H. Rohrer set up the first scanning electron microscope. The observation is based on the tunnel effect and allows to collect 3D images at atomic resolution (Nobel in Physics - 1986)

Nobel in Chemistry 2018



Jacques Dubochet, Joachim Frank and Richard Henderson received the prize for their part in developing cryo-electron microscopy (cryo-EM), a technique that fires beams of electrons at proteins that have been frozen in solution, to deduce the biomolecules' structure even in systems not suitable for X-ray crystallography.

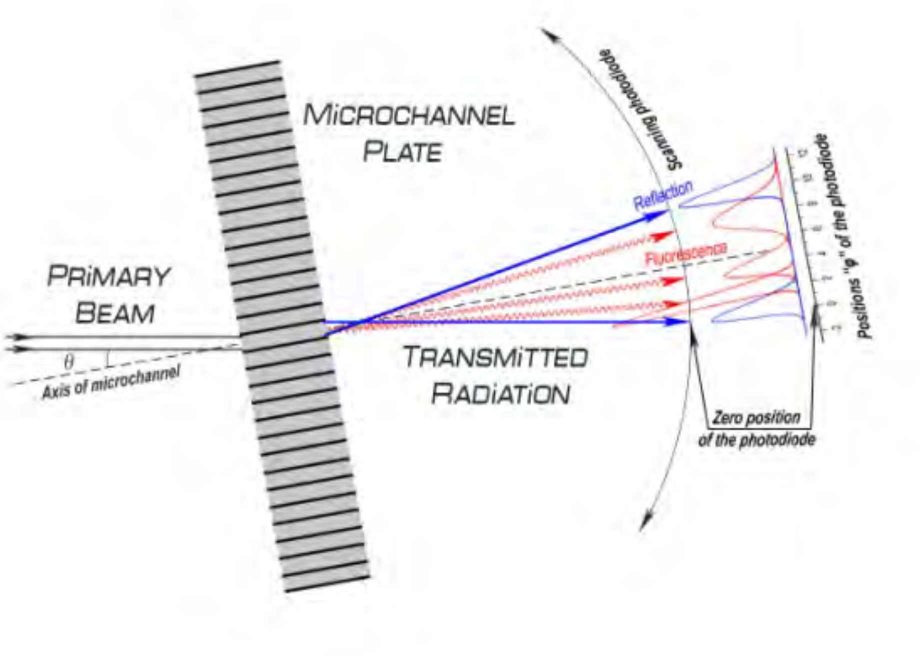
Meta-lenses

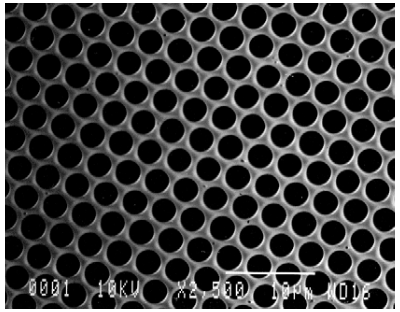
- *Capillary optics is a powerful optical technologies because of its superior capacity of generating high flux density x-ray beams in the μm - and sub μm range, their gain, high spatial resolution and high temporal resolution. Actually, channeling based devices may guide and shape a radiation beam and control intensity, spot size, divergence and spatial distribution.*

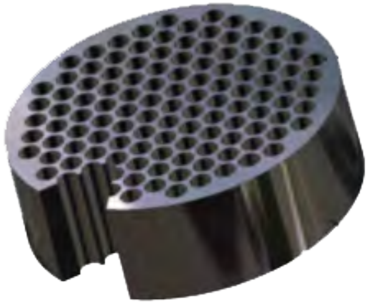
- *A polycapillary device consists of compact and low weight optics made by an array of a large number of small hollow glass tubes of circular or squared shape. Hollow cylindrical microcapillaries work also as waveguides for X-ray radiation. This optical device collects radiation emerging from a radiation source within a large solid angle and guides radiation in order to have a focused or a parallel beam.*

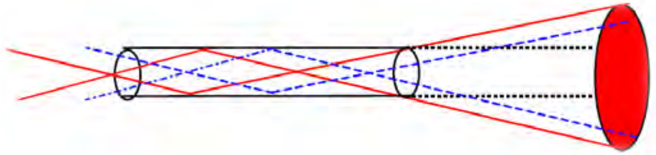
- *Based on experimental and theoretical data, the study of the channeling phenomenon may gives unique information on the nature of X-ray wave interaction and propagation of radiation. Indeed, both experimental and theoretical data point out the presence of propagation radiation modes in such glassy waveguides and the interference between incident and reflected (fluorescence) waves inside microcapillaries.*

Micro-Channel Plates







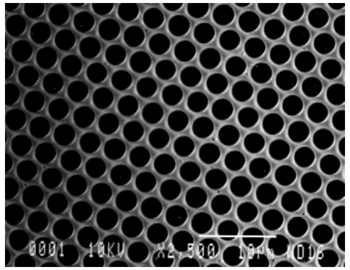


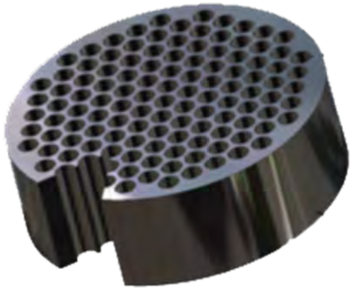
Radiation can be transmitted in a wide energy range at the exit of micro-channel plates (MCP). We have studied both energy and angular distribution spectra of X-rays for large MCPs (20 mm and 33 mm of diameter) with a thickness of ~0.3 mm, characterised by long micro-channels with a length-to-diameter ratio ~80:1 (~12-13 mrad).

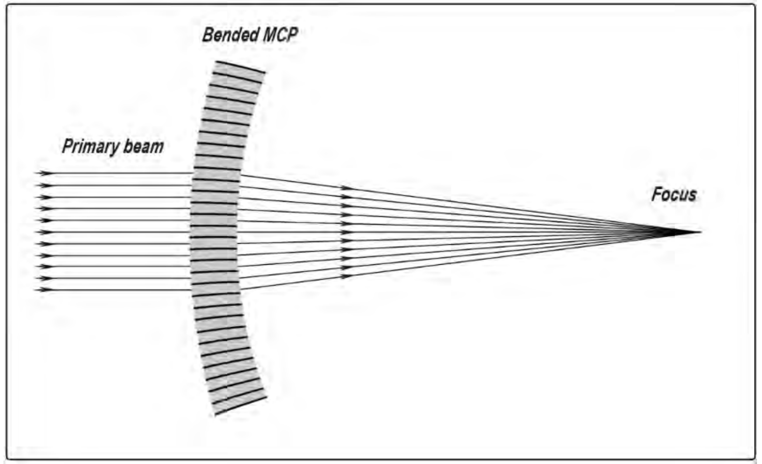
MCPs have spatial regular empty channels (pitch size of 4.2 μm) with a hexagonal symmetry in the transverse cross-section. Micro-channels with a diameter of 3.4 μm have their walls oriented parallel to the normal of the MCP surface.

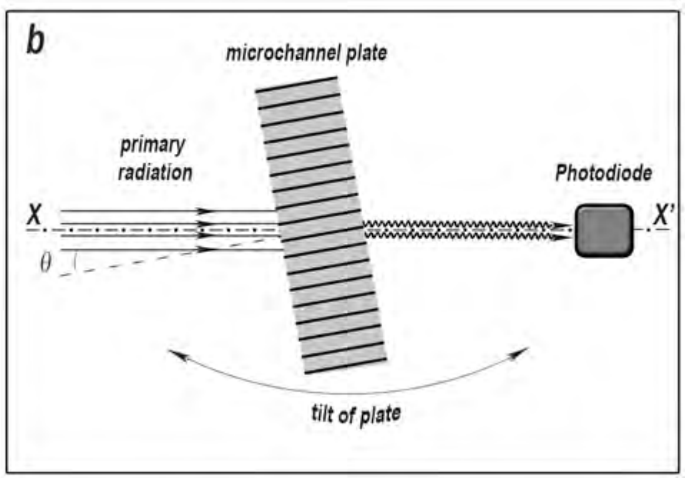
What we can do MicroChannel Plates

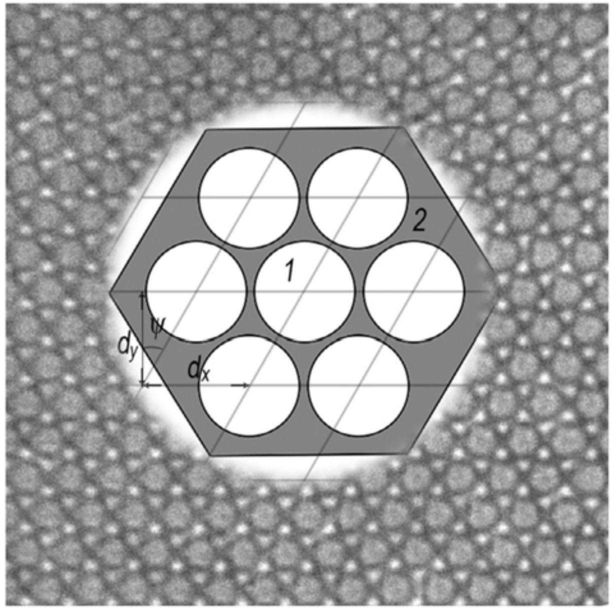
Microchannel plates (MCPs)







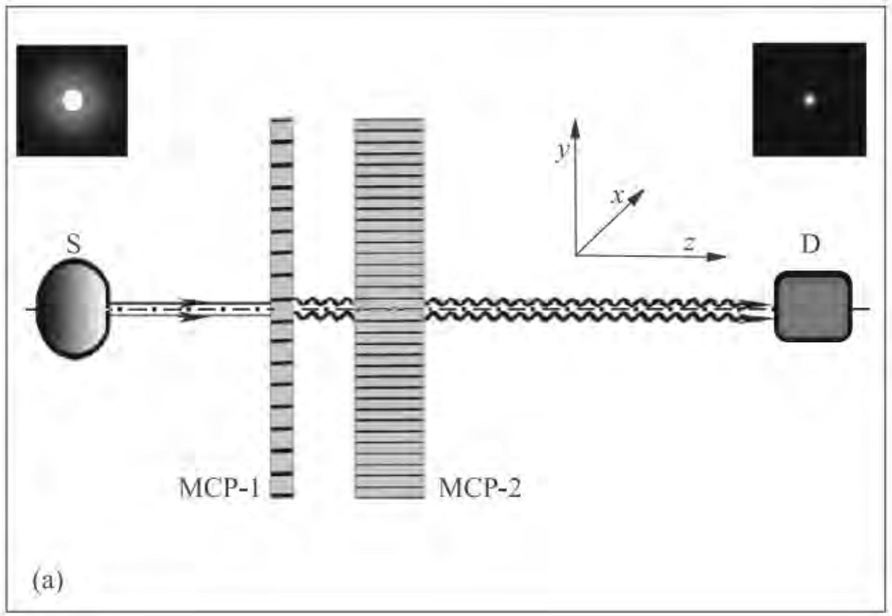




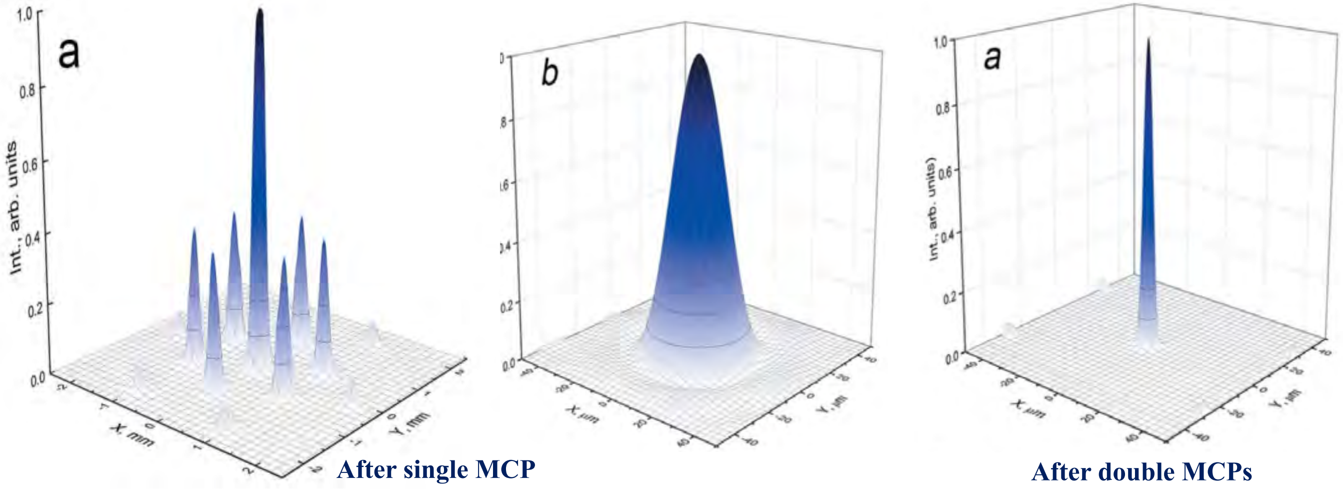
The focusing effect arises from total external reflection of radiation at the interior surfaces of the channels of a MCP. Point to point focusing was observed with flat and curved MCPs, and collimation from a point source to a quasi-parallel beam was observed with curved MCPs.

Working in Hard X-ray vs. soft X-ray and VUV

MCPs-based device



E_{ph}= 94 eV



Mazuritskiy, M. I., et al. J. of Synchrotron Radiation 29.2 (2022).

CROWN 2024

Confocal micRoscopy at sOft x-ray WaveleNghts

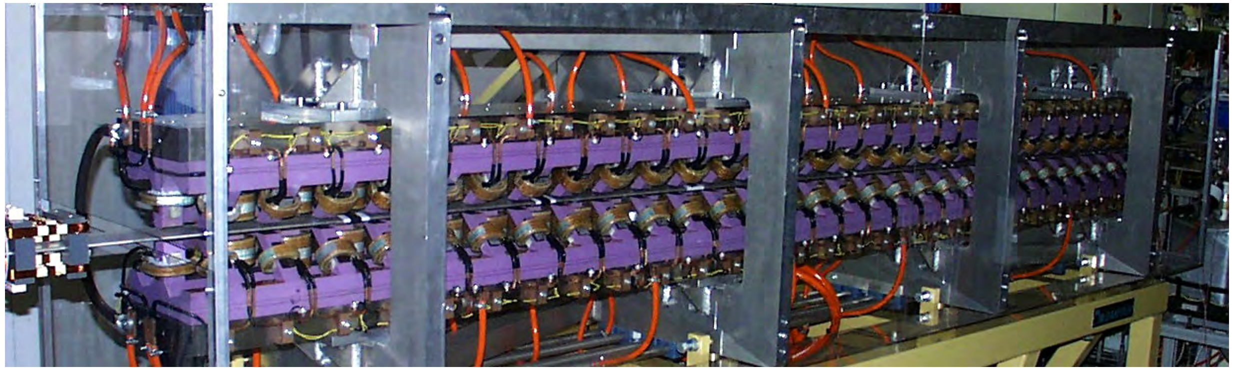
The invention of the confocal microscope is due to Marvin Minsky, who produced a working microscope in 1955. The principle of confocal imaging patented in 1957 is employed in all modern confocal microscopes that offers several advantages over conventional microscopy. The existing x-ray confocal microscopes use expensive special mirrors and requires intense x-ray fluxes.

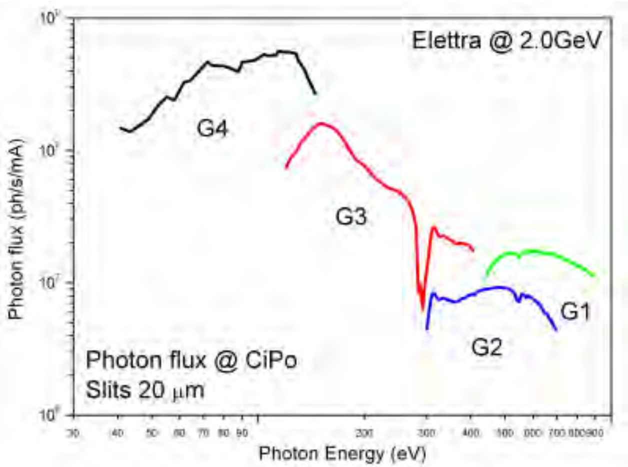
Meta-lens based confocal microscope

The present Concept/Principle relates to MCPs, i.e., optics based on a powerful optical technology that guarantees a superior capacity of generating high flux density of radiation, a high spatial resolution and a high temporal resolution. Actually, these meta-lenses (see Fig. 3a) may guide and shape a beam and control intensity, spot size, divergence

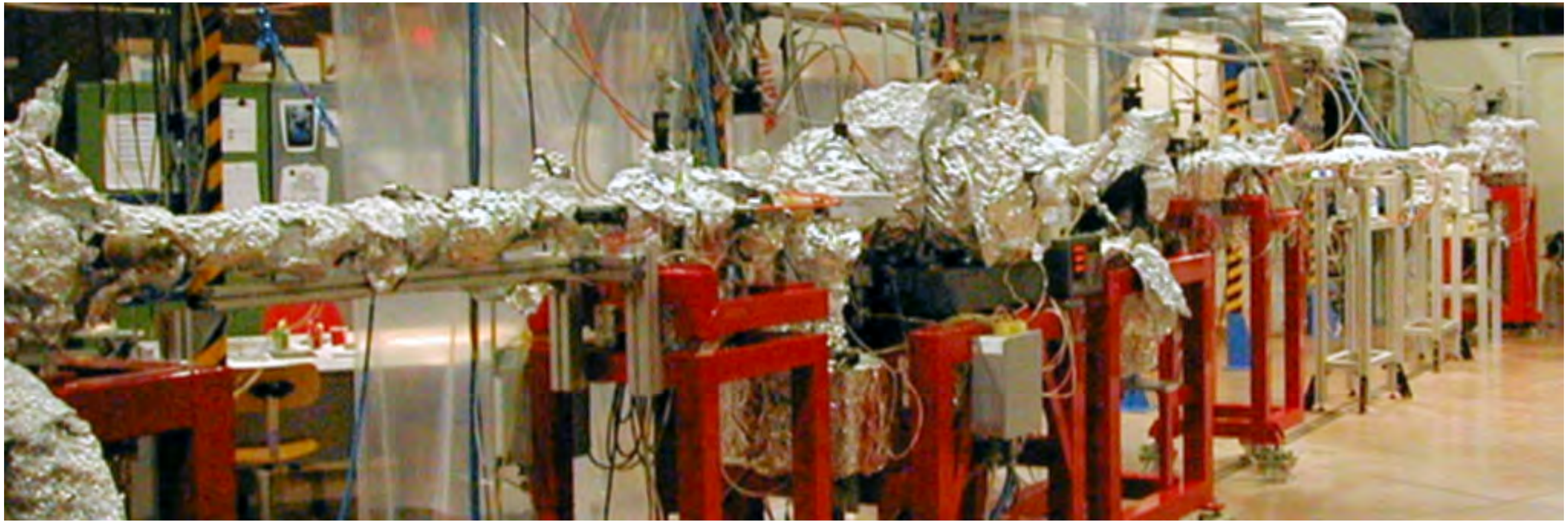
These compact and low weight optics are arrays of a large number of small hollow glass tubes of circular shape that work also as waveguides for radiation collecting radiation emerging from a source within a large solid angle.

CiPo beamline@Elettra

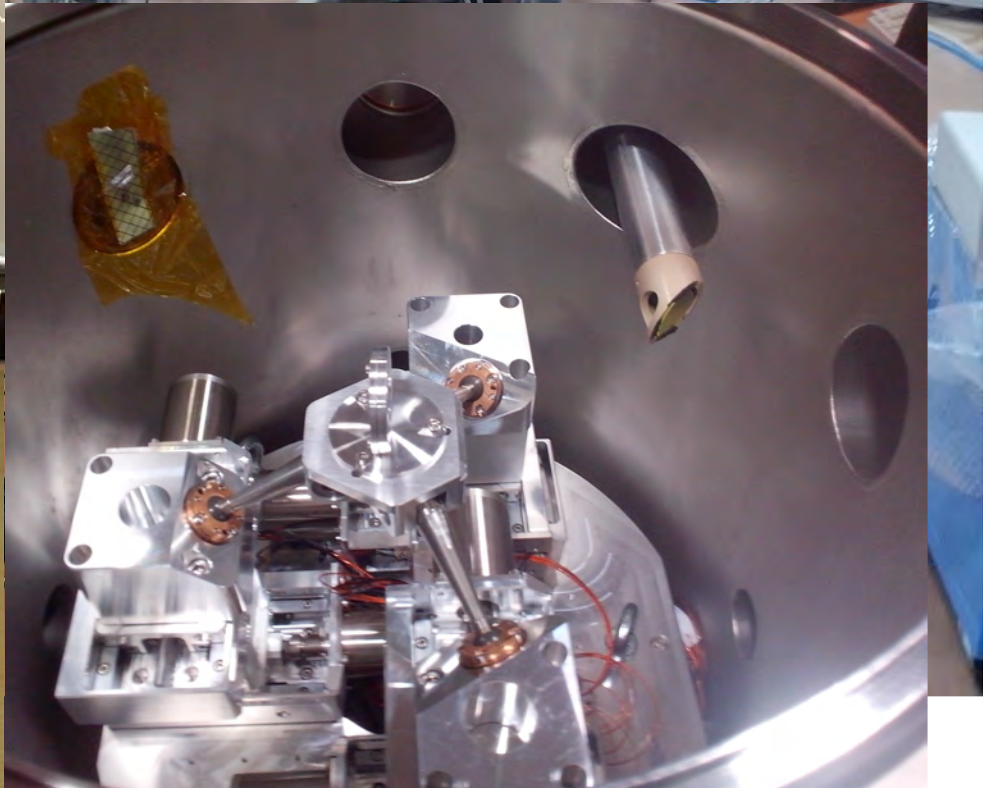




- *source: electromagnetic elliptical wiggler*
- *SGM monochromator (~40-900 eV)*
- *IRMA HV experimental chamber*




First Lasing of the European
UV/VUV FEL Project
at ELETTRA
CryoAlp
TXRF

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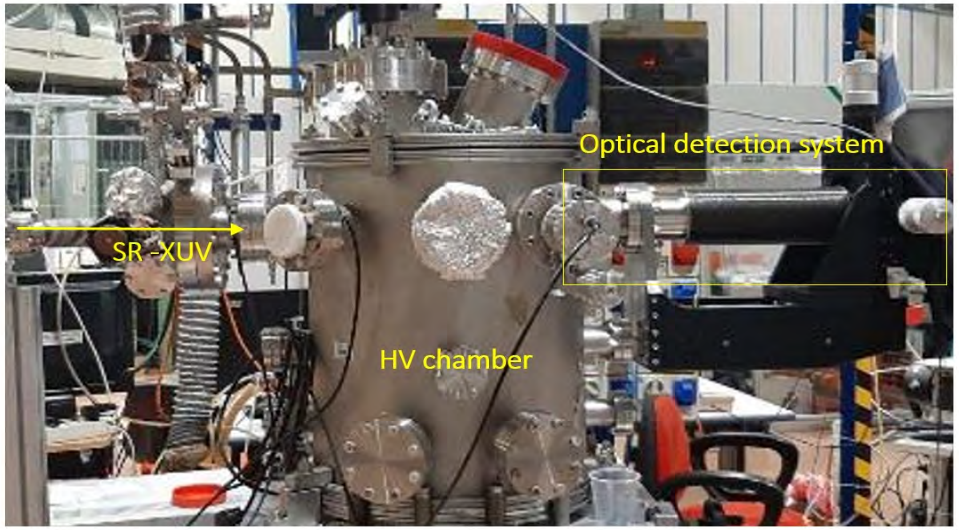
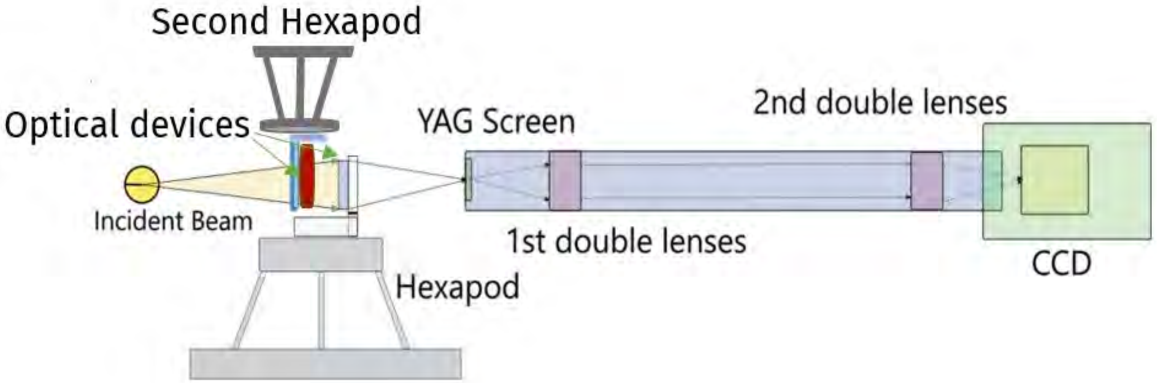
Confocal micRoscopy at sOft x-ray WaveleNghts





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Wave propagation and focusing of soft X-rays by spherical bent microchannel plates

M. I. Mazuritskiy,[a]* A. M. Lerer,[a] A. Marcelli,[b,c,d] S. B. Dabagov,[b,e,f] M. Coreno,[d] A. D'Elia[g] and S. J. Rezvani[b,g]

[a]Physics Department, Southern Federal University, 344090 Rostov-on-Don, Russian Federation, [b]INFN – Laboratori Nazionali di Frascati, via Enrico Fermi 54, 00044 Frascati, Italy, [c]RICMASS – Rome International Center for Materials Science Superstripes, 00185 Rome, Italy, [d]Istituto Struttura della Materia, CNR, Via del Fosso del Cavaliere 100, 00133 Rome, Italy, [e]RAS P. N. Lebedev Physical Institute, 119991 Moscow, Russian Federation, [f]NR Nuclear University MEPhI, 115409 Moscow, Russian Federation, and [g]IOM-CNR, Laboratorio Nazionale TASC, Basovizza SS-14, km 163.5, 34012 Trieste, Italy. *Correspondence e-mail: mazurmik@gmail.com

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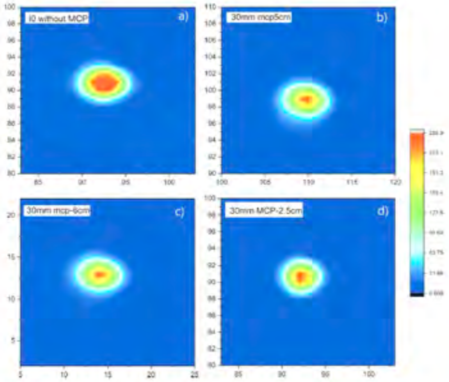


Figure 4
Maps obtained translating the MCP along the z-axis (*i.e.* changing the distance between the MCP and the CCD). The color bar refers to the radiation intensity. The area in each panel is constant (20 × 20 pixels) and the xy-axes are expressed in pixels. The map of the incident beam is top left while other maps correspond to distances of 25, 50 and 60 mm along the z-axis.

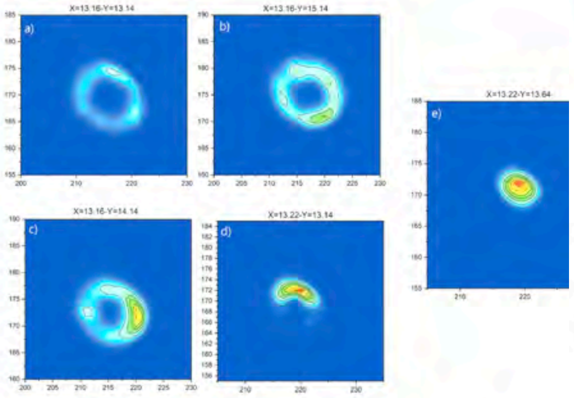
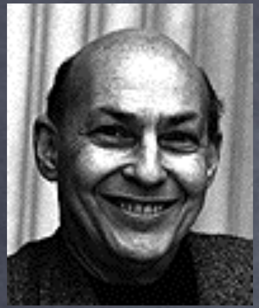
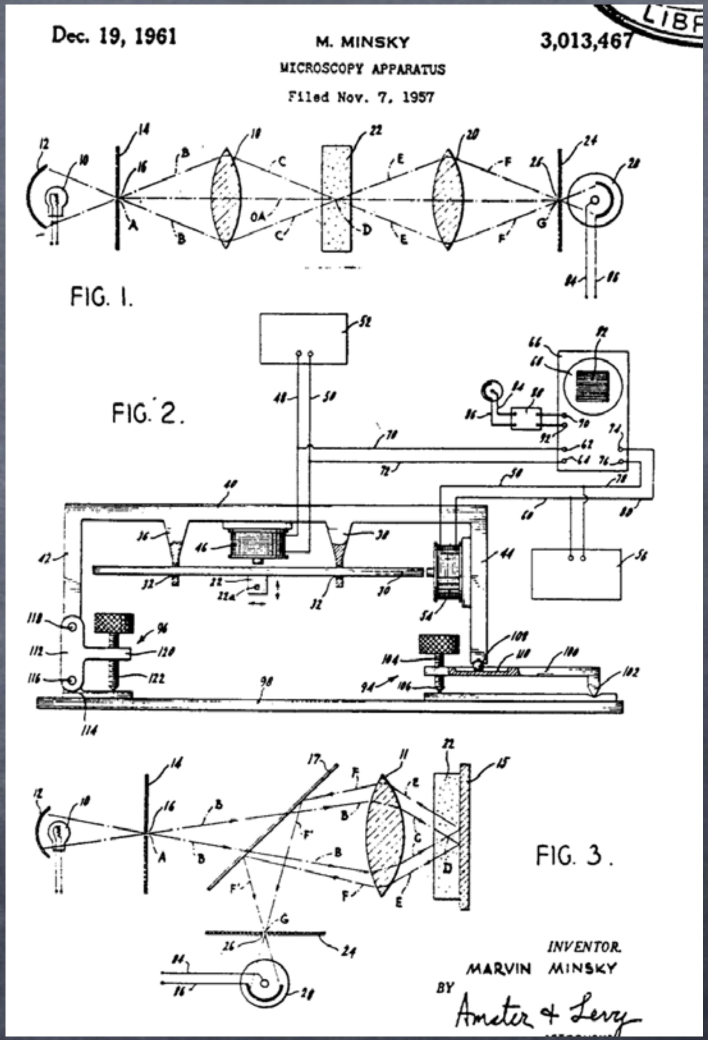


Figure 3
Radiation distribution patterns of the MCP with bending radius R = 30 mm at z = 15 mm. The color bar refers to the radiation intensity. Maps have been collected moving the MCP in the x direction from ~13 to ~14 mm and in the y direction from ~13 to ~15 mm. The xy coordinates of each map are shown at the top. The area in each panel is constant (30 × 30 pixels) and the numbers on the xy-axis are pixels.

Confocal scanning microscope



In 1961 Marvin Minsky proposed the construction of a confocal scanning microscope. This concept was later extensively developed by Egger and Davidovits at Yale, from Shepherd and Wilson at Oxford and by Brakenhoff and colleagues at Amsterdam.



...... An ideal microscope would examine each point of the specimen and measure the amount of light scattered or absorbed by that point. But if we try to make many such measurements at the same time then every focal image point will be clouded by aberrant rays of scattered light deflected points of the specimen that are not the point you're looking at. Most of those extra rays would be gone if we could illuminate only one specimen point at a time. There is no way to eliminate every possible such ray, because of multiple scattering, but it is easy to remove all rays not initially aimed at the focal point; just use a second microscope (instead of a condenser lens) to image a pinhole aperture on a single point of the specimen. This reduces the amount of light in the specimen by orders of magnitude without reducing the focal brightness at all. Still, some of the initially focused light will be scattered by out- of-focus specimen points onto other points in the image plane. But we can reject those rays, as well, by placing a second pinhole aperture in the image plane that lies beyond the exit side of the objective lens. We end up with an elegant, symmetrical geometry: a pinhole and an objective lens on each side of the specimen. (We could also employ a reflected light scheme by placing a single lens and pinhole on only one side of the specimen - and using a half-silvered mirror to separate the entering and exiting rays.) This brings an extra premium because the diffraction patterns of both pinhole apertures are multiplied coherently: the central peak is sharpened and the resolution is increased. (One can think of the lenses on both sides of the microscope combining, in effect, to form a single, larger lens, thus increasing the difference in light path lengths for point-pairs in the object plane.)

The price of single-point illumination is being able to measure only one point at a time. This is why a confocal microscope must scan the specimen, point by point and that can take a long time because we must add all the time intervals it takes to collect enough light to measure each image point. ......

http://web.media.mit.edu/~minsky/

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- **Application Areas:**

Soft X-ray/VUV imaging: the applications area of this technology are: materials science, biology, biomedicine, geophysics, cultural heritage and industrial characterizations.

- **European Framework:**

Industrial Leadership - Nanotechnologies, Advanced materials, Biotechnology, Advanced manufacturing and processing.

This instrument is based on a multi- disciplinary technology. It may enable processes, goods and service innovation. It will provide a technology brick suitable to enable a wide range of product applications, improving energy and align the second meta-lens in the confocal geometry.

- **IP Rights:**

The X-ray confocal microscope with meta-lenses is an invention to be protected by a patent. This product/apparatus is novel, show new characteristics; it is a step beyond the average knowledge, and it is capable of industrial applications.

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Confocal micRoscopy at sOft x-ray WaveleNghts

- **Status**

We have assembled at Elettra/CiPo an experimental end-station to test optical layouts based on MCPs and meta-lenses for 3rd and 4th generation radiation sources. We tested flat and bent devices working in the normal incidence geometry in transmission, at different energies. Tests with coherent XUV radiation are being conceived also to pursue holographic applications with novel light sources (FERMI FEL, Elettra 2.0).

- **Market Opportunity**

”The global x-ray imaging market accounted for revenue of 9 G$ in 2015, and it is expected to grow by ~5% during 2016-2022”. [www.psmarketresearch.com/market-analysis/x-ray-imaging-market]

The patent trend in x-ray and microscopy area are continuously increasing, still no patents for design x-ray confocal microscopes using meta-lenses exist. Those using Fresnel lenses works with monochromatic radiation and require high x-ray flux at present are not compatible with conventional sources. This instrument may work with conventional x-ray sources, is compact and use low cost meta-lenses. It makes possible industrial applications and commercial applications interested to soft x-ray imaging using compact laboratory sources.

- **Competitors**

In the field of x-ray optics there are many players and competitors like Canon, Carl Zeiss, Matsushita, Philips, Nikon, At&t, Sumitomo, Xerox, Rigaku, Xenocs, CEA, etc. with hundreds of patents in the area and potentially interested to this technology. Carl Zeiss is probably the main player interested to a licensing. A full search has to be performed.

CROWN – 4.5 FTE

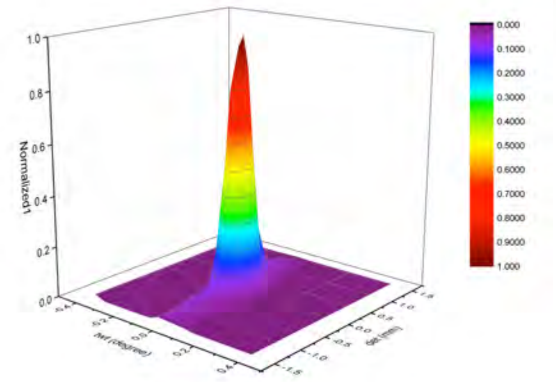
Participants LNF 3.0 FTE

- Augusto Marcelli (CN) 30%
- Salvatore Macis 30%
- Paola De Padova 30%
- Javid S. Rezvani 30%
- Zeinab Ebrahimpour 30%
- Giancarlo Della Ventura 20%
- Francesca Tombolini 20%
- Sultan Dabagov 10%
- PhD Univ. Camerino. 50%

- Marcello Coreno (CL) 30%
- Monica De Simone 30 %
- Fabio Zuccaro (T) 30 %
- Awad Mohamed 30 %
- Gabriele Bonano 30%

Collaborazioni

- CNR - ISM & IOM
- Universita’ di Roma Tre
- Universita’ di Camerino
- Universita’ di Modena
- Sincrotrone Trieste



Focal spot

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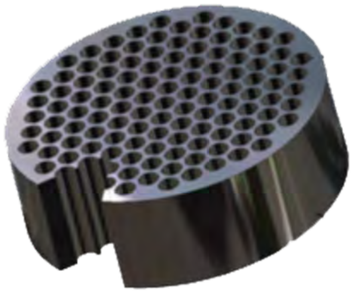


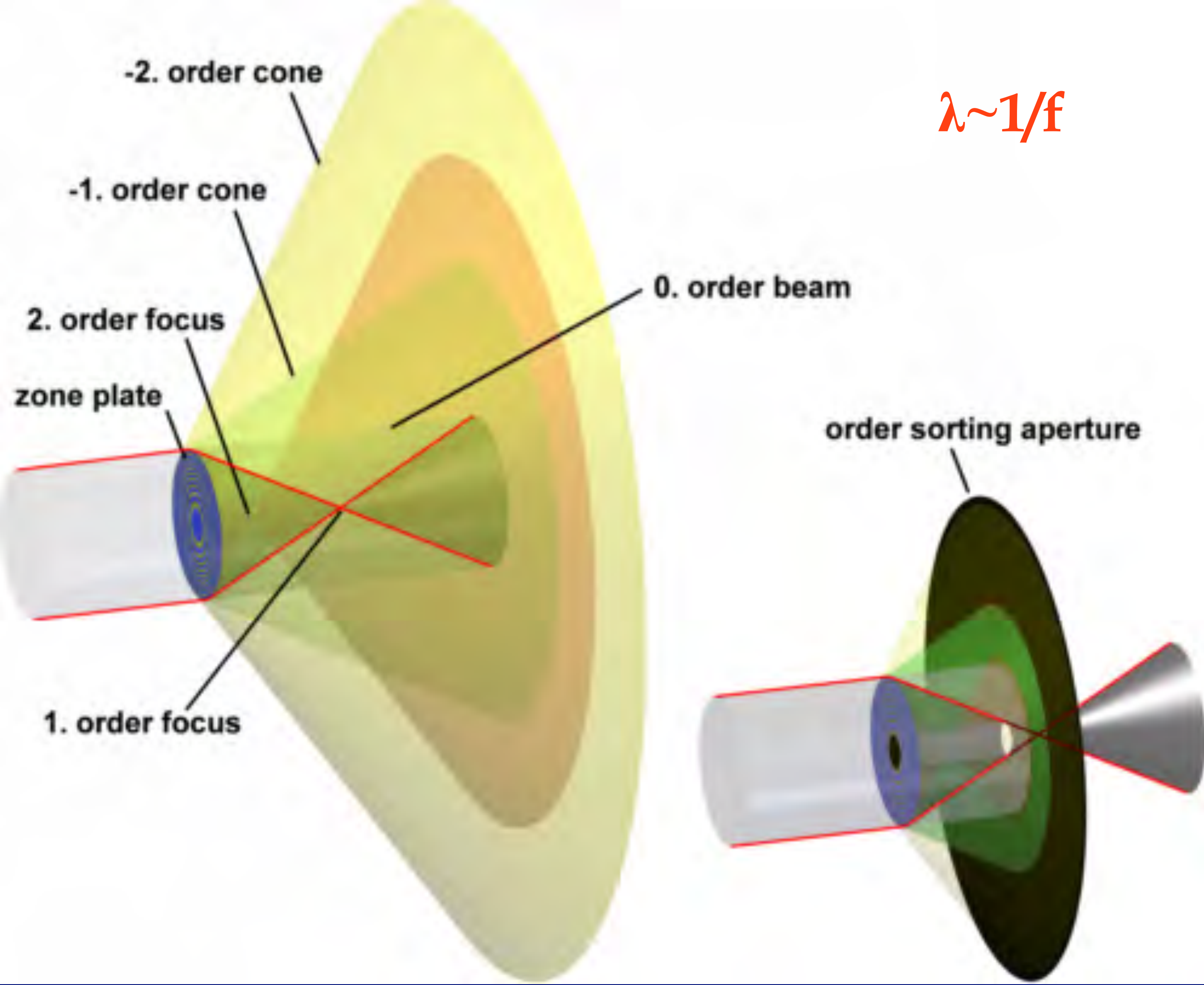
MCP optics

Hexapod for the alignment of the meta-lenses

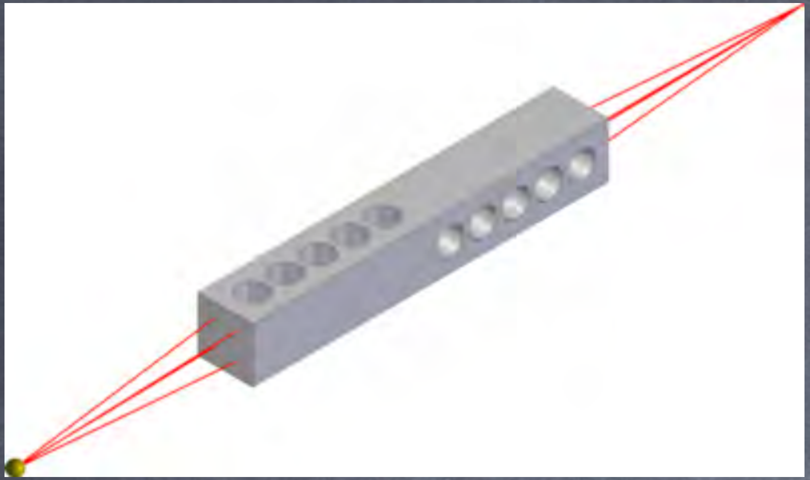
X-ray focusing: Fresnel zone plates

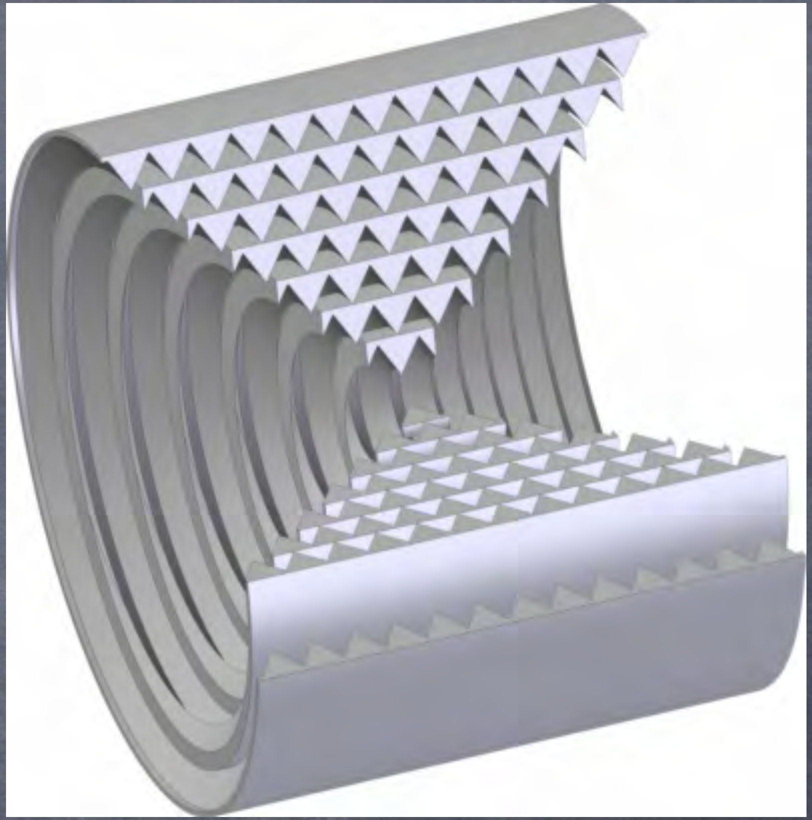
- Diffractive optics: radially varied grating spacing
- Largest diffraction angle is given by outermost (finest) zone width δ_{rN} as $\theta=\lambda/(2\delta_{rN})$
- Rayleigh resolution is then $\delta_t=0.61\ \lambda/(\theta)$

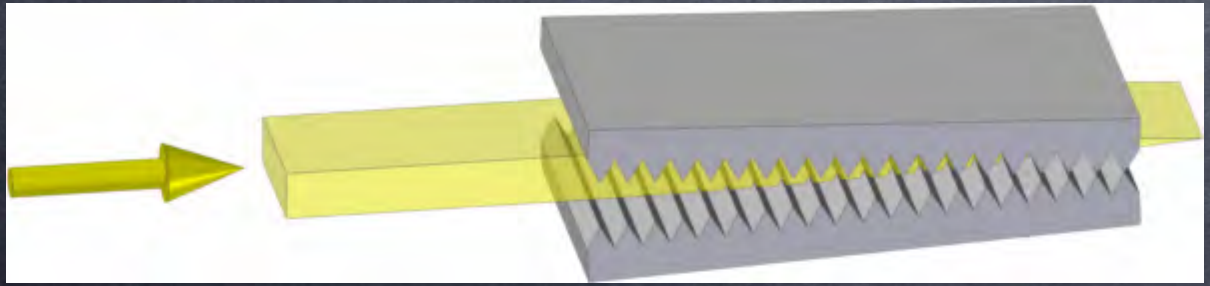
  $=1.22\ \delta_{rN}$
- Zones must be positioned to ~1/3 width over diameter (10 nm in 100 μm, or 1:10^4)

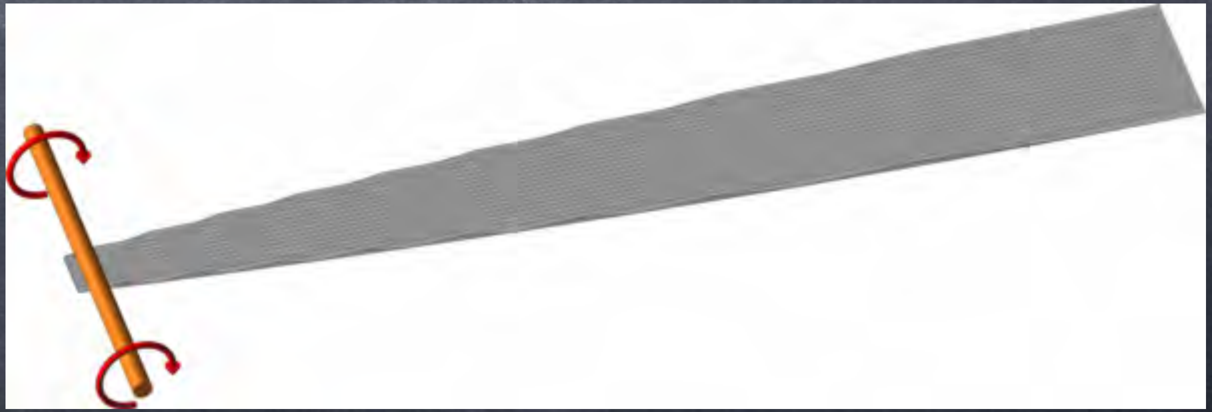


x-ray lenses

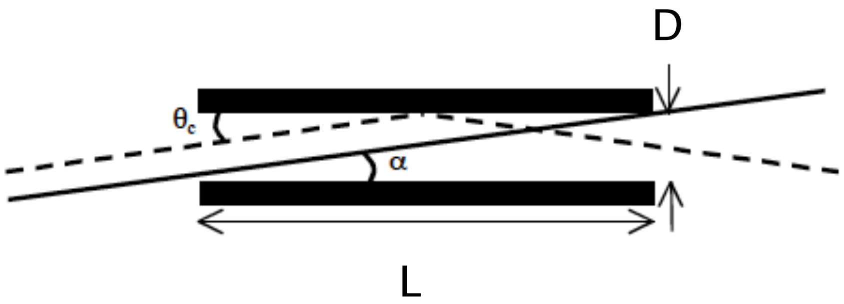


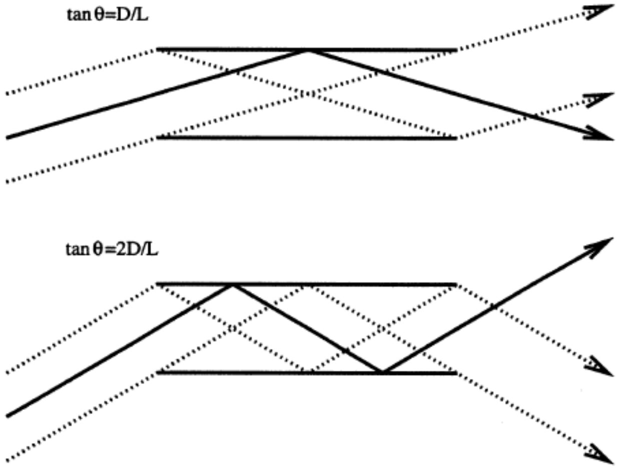


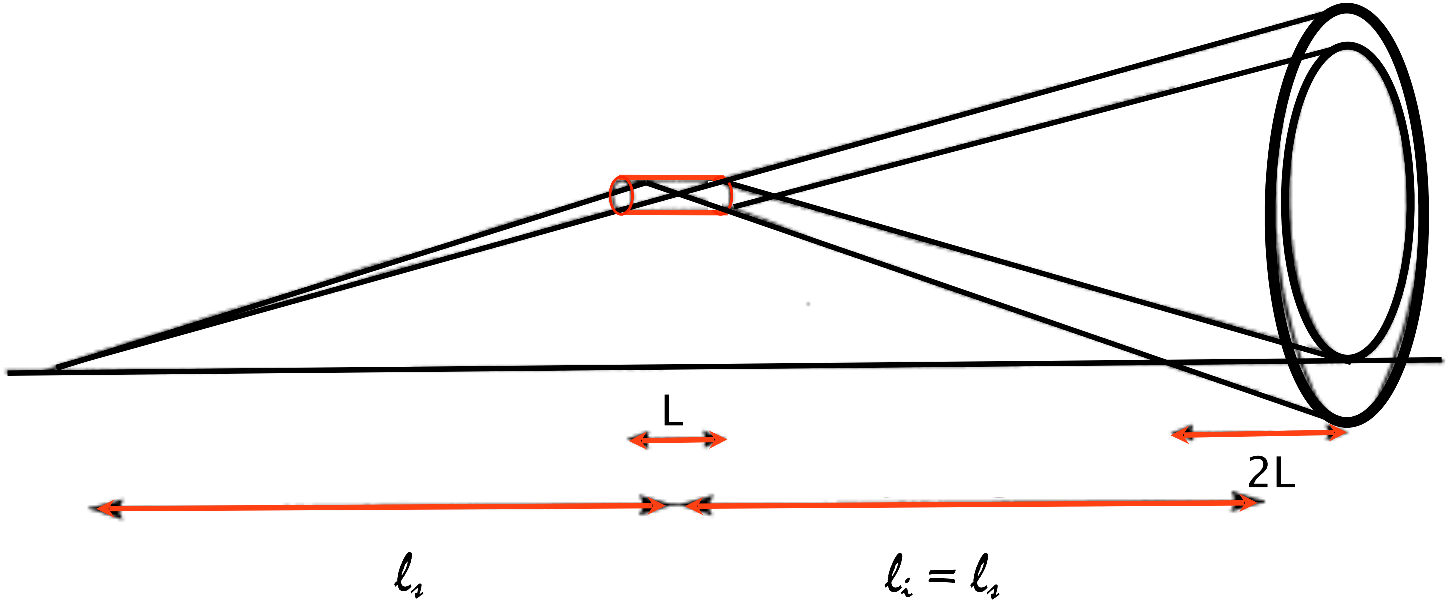




Focusing efficiency







$M = \ell_i / \ell_s$

Imaging of object inner structure using passing through of soft x-ray radiation and coded aperture approach