united nations educational, scientific and cultural organization

international atomic energy agency

the
abdus salam
international centre for theoretical physics

SMR/1270-26

SCHOOL ON SYNCHROTRON RADIATION

6 November – 8 December 2000

Miramare - Trieste, Italy

Supported in part by the Italian Ministry of Foreign Affairs in connection with the SESEME project

Co-sponsors: Sincrotrone Trieste, Società Italiana di Luce di Sincrotrone (SILS) and the Arab Fund for Economic and Social Development

Optics for microscopy with synchrotron radiation

W. Jark
Sincrotrone Trieste
Italy

School on Synchrotron Radiation
ICTP, November 22nd, 2000

Optics for microscopy with synchrotron radiation

Werner Jark

Sincrotrone Trieste
Multilayers and Polarization
S.S. 14 km 163.5 in Area Science Park
34012 Basovizza (TS), Italy

With Synchrotron Radiation we want to see what we cannot see with classical visible light:

a) with detectors sensitive to very special characteristics ===> see other lectures later

b) in combination with the detectors from a) using resolutions below the limit of visible light at about 0.5 microns.

Can we also here do this by focusing with light optics (i.e. "lenses")?

How much can we learn from classical optics?

How much sweat do we have to put into completely new devices?

Two approaches for high resolution:

a) use a small sample beam (scanning)
b) magnify a well illuminated area (full field)

Todays state-of-the-art synchrotron radiation sources provide very small electron beam sizes (ca. 50 - 100 microns). If we succeed to produce a demagnified image of it we already have what we need for high resolution scanning techniques.

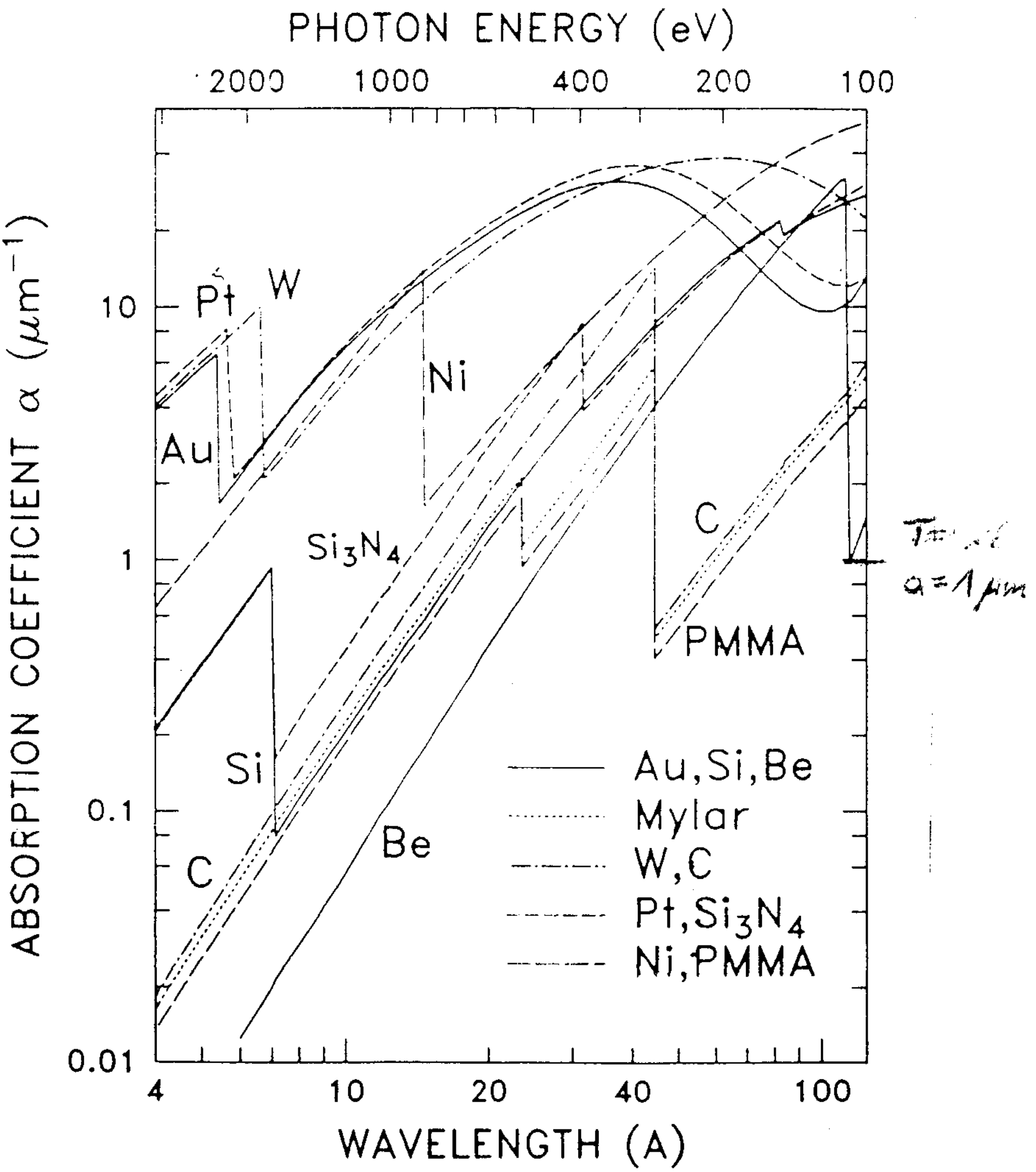
Important material boundary conditions:

Aspect	Spectral range		
	visible light E < 4 eV	VUV/soft x-rays 50 - 1000 eV	x-rays E > 2000 eV
transmission	yes, many	none	partly, many
refractive index	n > 1.5	0.9 < n < 1	0.999 < n < 1
reflectivity	for many high for all angles	high at grazing angles 2 - 20°	high at grazing angles < 0.5°
exceptions		multilayer	crystals

===>

The only impossible objects are:

classical refractive lenses for soft x-ray!


PHOTON ENERGY (eV)
2000
1000
400
200
100
ABSORPTION COEFFICIENT α (μm⁻¹)
10
1
0.1
0.01
Pt
W
Au
Ni
Si₃N₄
Si
C
Be
C
PMMA
Au,Si,Be
Mylar
W,C
Pt,Si₃N₄
Ni,PMMA
4
10
20
40
100
WAVELENGTH (A)
α = 1 μm

We will now distinguish between

classical objects:

- refractive lenses
- reflective mirrors at normal incidence

semiclassical objects:

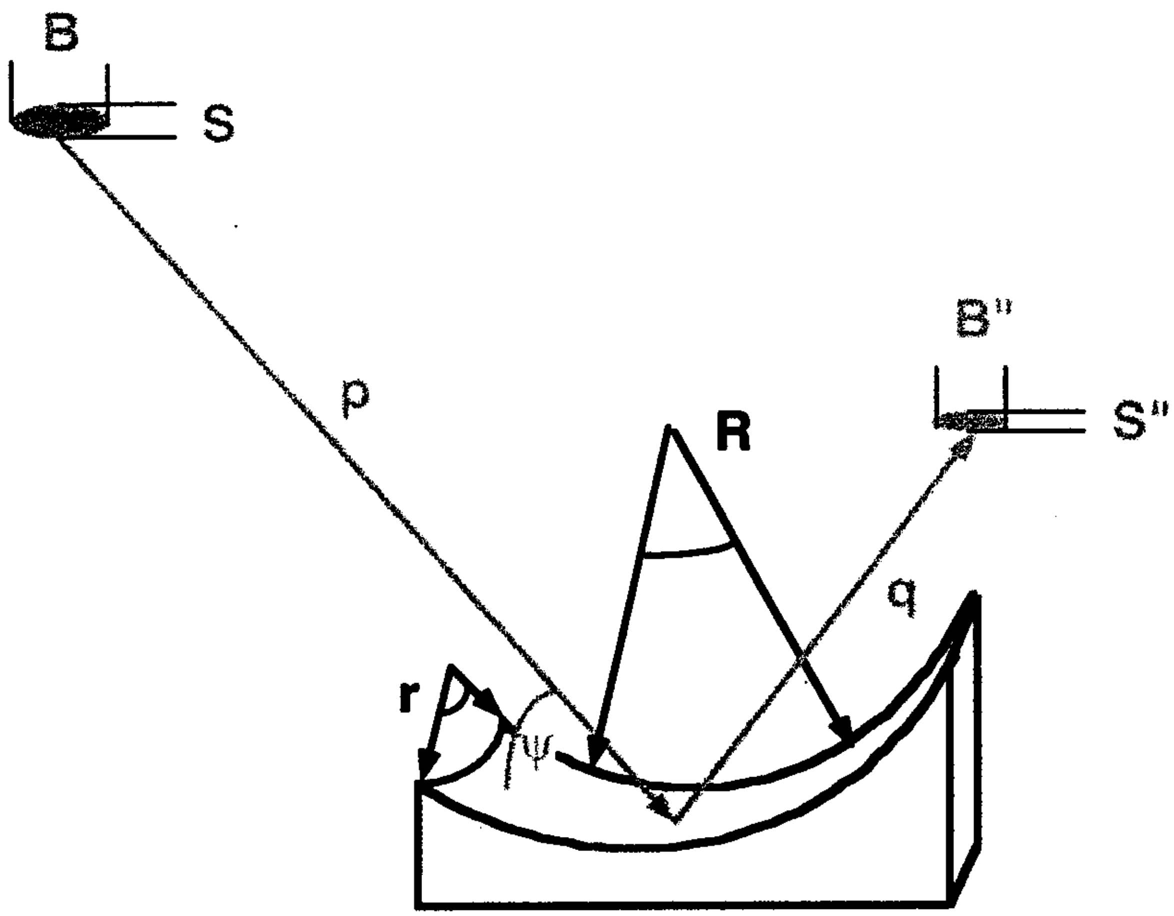
- reflective mirrors at grazing incidence
- diffractive optics

unusual application of classical objects:

- light guides
  (i.e. "fibers", waveguides)

presentation does not follow strictly the historical developments!

Some basic concepts



magnification:	M	= q/p
ideal image size:	S"	= M * S B" = M * B
focal length:	f	= q * p/(q+p)
		from [1/f=1/p+1/q]
radii of curvature:	r	= 2 * f * sin(ψ)
	R	= 2 * f/sin(ψ)

 1948

Formation of Optical Images by X-Rays

PAUL KIRKPATRICK AND A. V. BAEZ
Stanford University, Stanford, California
(Received March 12, 1948)

Several conceivable methods for the formation of optical images by x-rays are considered, and a method employing concave mirrors is adopted as the most promising. A concave spherical mirror receiving radiation at grazing incidence (a necessary arrangement with x-rays) images a point into a line in accordance with a focal length $f = Ri/2$ where R is the radius of curvature and i the grazing angle. The image is subject to an aberration such that a ray reflected at the periphery of the mirror misses the focal point of central rays by a distance given approximately by $S = 1.5Mr^2/R$, where M is the magnification of the image and r is the radius of the mirror face. The theoretically possible resolving power is such as to resolve point objects separated by about 70A, a limit which is independent of the wave-length used. Point images of points and therefore extended images of extended objects may be produced by causing the radiation to reflect from two concave mirrors in series. Sample results are presented.

INTRODUCTION

THE literature of x-rays contains many passages deploring the supposed impossibility of focusing x-rays with lenses or mirrors. Both the difficulty and the regret are easily appreciated. A satisfactory x-ray microscopy would open up fields of investigation closed to the optical microscope because of its restricted resolution, and to the electron microscope because of the limited penetrating power of electrons. X-ray spectrometers and diffraction instruments would probably have evolved along simpler or more advantageous lines had their designers possessed the means of optical control available to workers with light in other spectral regions.

X-RAY LENSES

Roentgen's[1] first experiments convinced him that x-rays could not be concentrated by lenses; thirty years later his successors understood why. X-ray refractive indices are less than unity by an amount δ which for common solids and x-rays of general practice has a value of the order of 10^{-5}. It may readily be shown that the focal length f of a single refracting surface of radius R is approximately R/δ. For several surfaces in series, arranged cooperatively, we have $1/f = \delta(1/R_1 + 1/R_2 + \text{etc.})$. To make a successful lens we require a large δ and slight absorption. Unfortunately ma-

[1] W. C. Roentgen, *Sitzungsberichte der Würzburger Physikalischen-Medicinischen Gesellschaft* (1895).

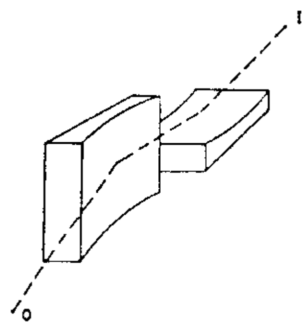
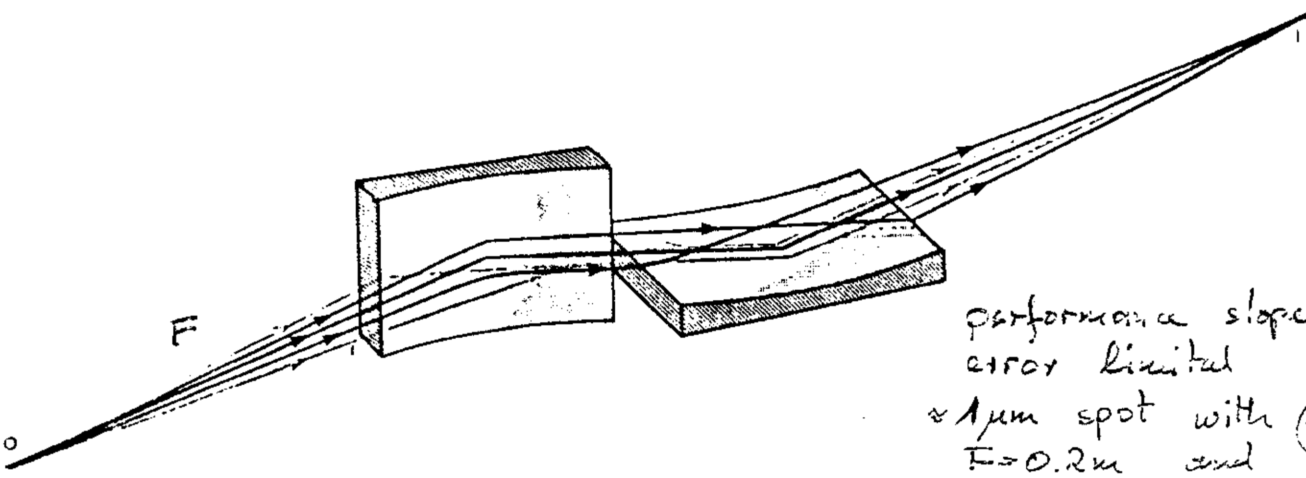


FIG. 11. Arrangement of concave mirrors to produce real images of extended objects with incidence at small grazing angles.

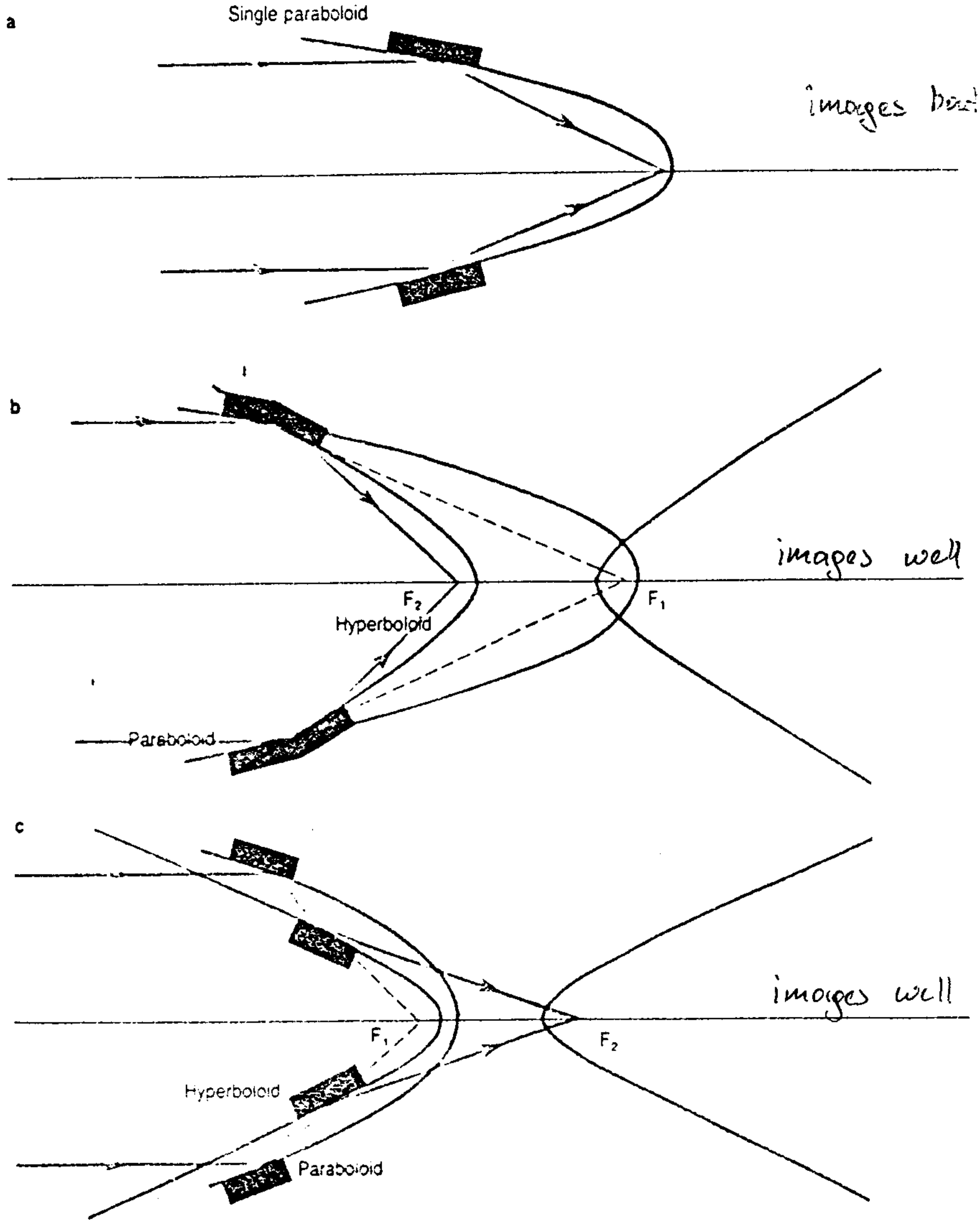
originally proposed for x-ray imaging
however,
today almost exclusively used for creation of small spots



performance slope error limited
≈1µm spot with F=0.2m and 0.25" rms (7)

Microscope principle that corrects the astigmatism associated with glancing-incidence spherical mirrors. Kirkpatrick-Baez microscopes based on this method of crossed mirrors are used to make x-ray photographs of the implosions of laser fusion capsules.
Figure 4

Wolter Optic



Glancing incidence optical systems based on conic sections. Hans Wolter proposed these systems for forming x-ray images. **a:** The single-paraboloid system, whose normal-incidence analog is the Newtonian telescope. **b:** Wolter's type I system, made up of an internal paraboloid and an internal hyperboloid. **c:** Wolter's type II system, which has an internal paraboloid and an external hyperboloid.

Figure 3

slope error limited

Schwarzschild objective

suggested for use with multilayer coatings in the soft x-ray range at DESY, Hamburg, Germany:

P.-P. Haelbich, W. Staehr and C. Kunz
Ann. N. Y. Acad. Sci., New York, 342, 148 (1980)

classical application: long distance objective for full field microscopy

here it is used in the reversed (demagnifying) orientation

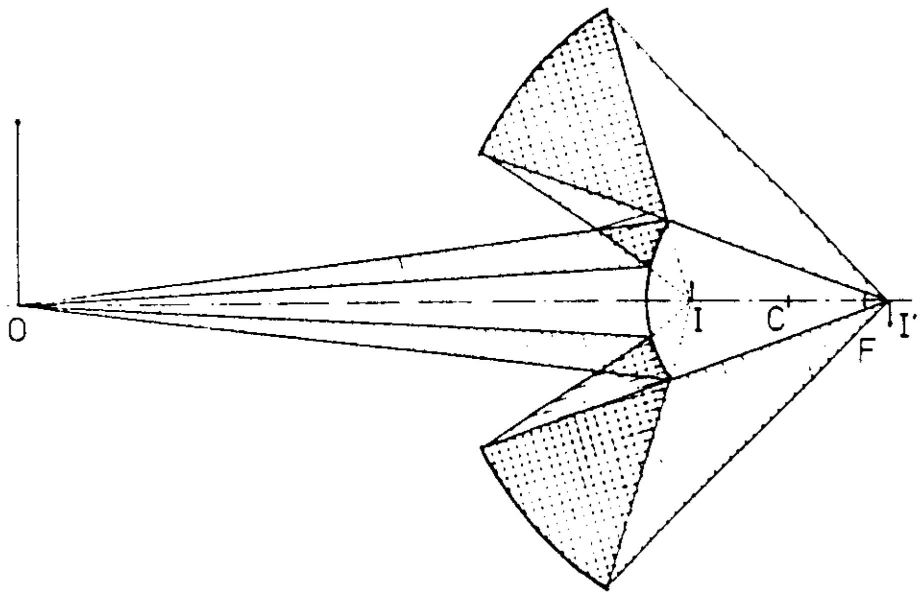
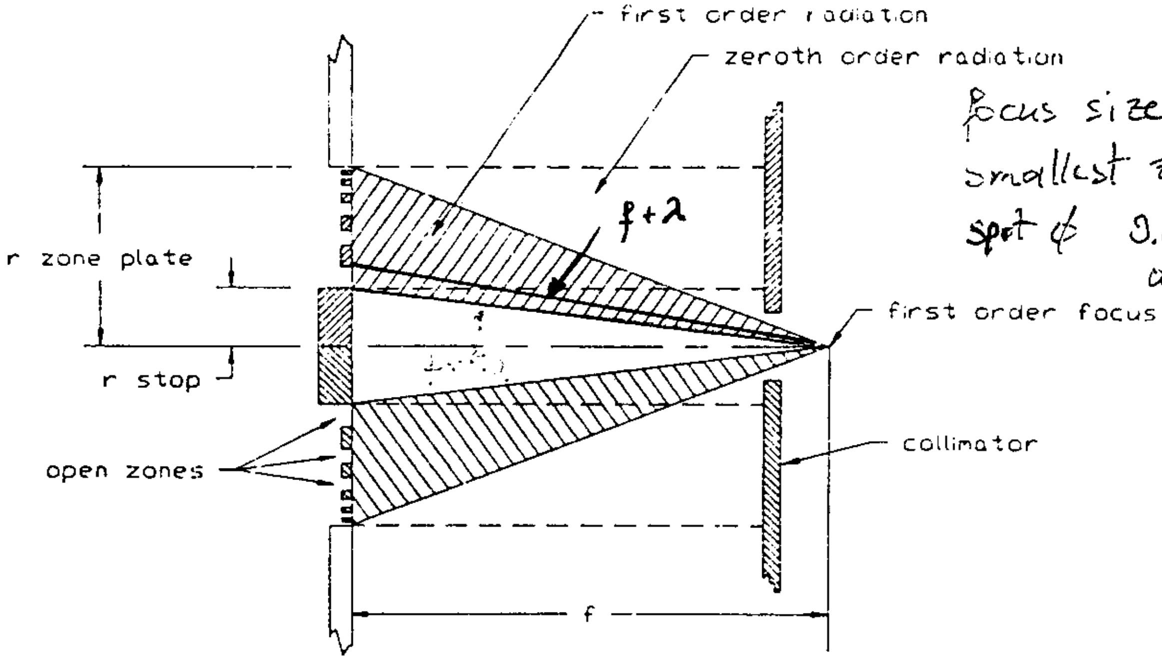


Fig. 11. Cross section of a Schwarzschild objective with two spherical mirrors (from Haelbich et al. (1980)). Example for the actual parameters for a 10× objective with an aperture angle $\sin\frac{1}{2}\theta_a = 0.125$: diameter and radius of curvature for the big mirror, 4.4 and 13.409 cm, for the small mirror, 1 and 5.205 cm, mirror spacing 8.387 cm. (Example by R. Tibbetts, IBM.)

spot diameter < 0.1 μm achieved
very little tuning capabilities
performance limited by scattering producing significant tails

Fresnel Zone Plate

based on diffraction



focus size ≥ smallest zone width
spot ∅ 0.05 µm achieved

Amplitude FZP
alternate zones - opaque

efficiency ~ 10%

$\frac{1}{\pi^2}$

Phase FZP
alternate zones - phase shifting

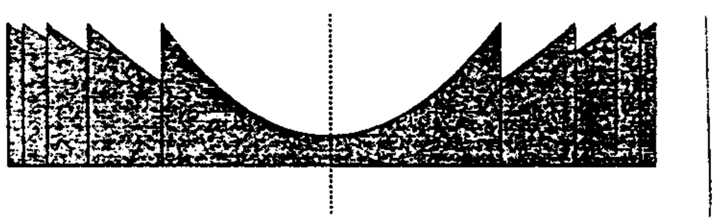
efficiency ~ 40%

$\frac{4}{\pi^2}$

Kinoform FZP
(sawtooth profile)

efficiency ~ 70 - 100%

ideal phase zone plate



Membrane support **(SI_3N_4, par exemple)**

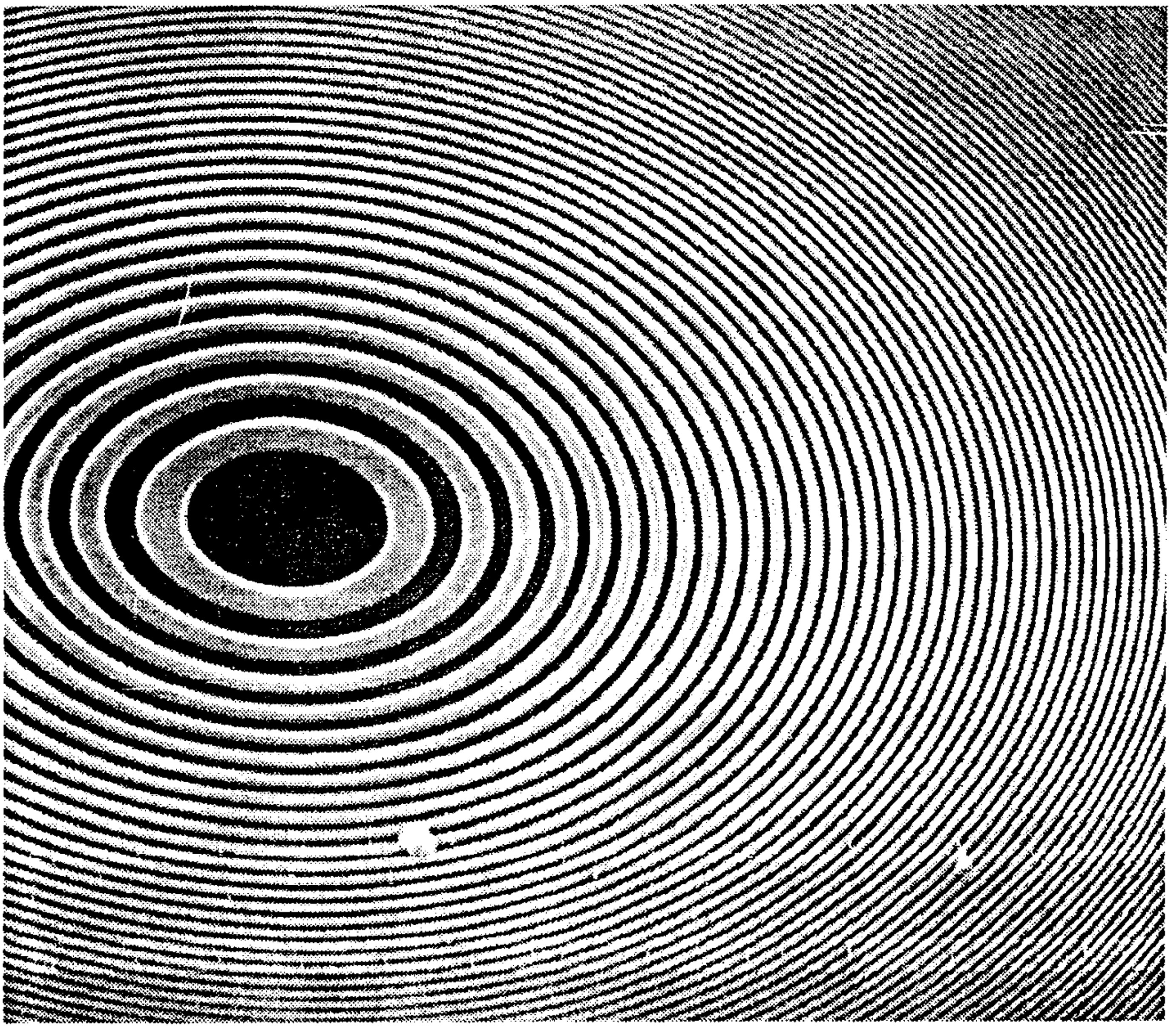
technical approximation
75% eff. achieved

Diffractive Optics

http://www-cxro.lbl.gov

Zone Plate Lenses

Highest spatial resolution in x-ray microscopy is achieved with zone plate lenses. The fabrication of zone plate lenses requires nano-fabrication techniques, because the required structures are of sub-micrometer scale.

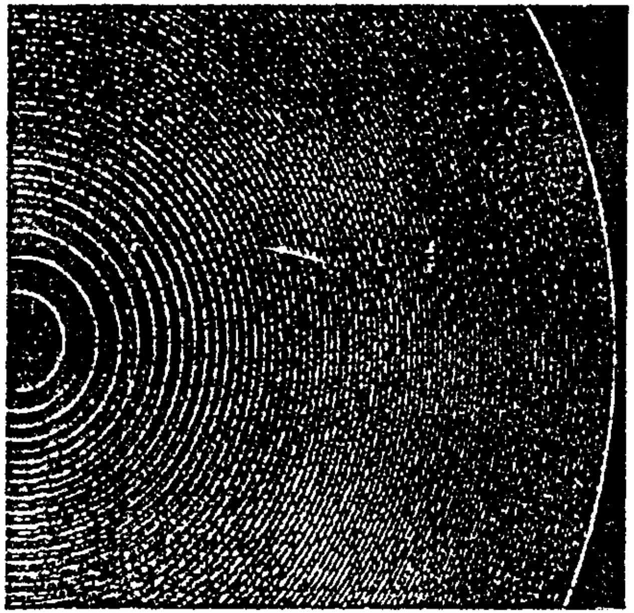


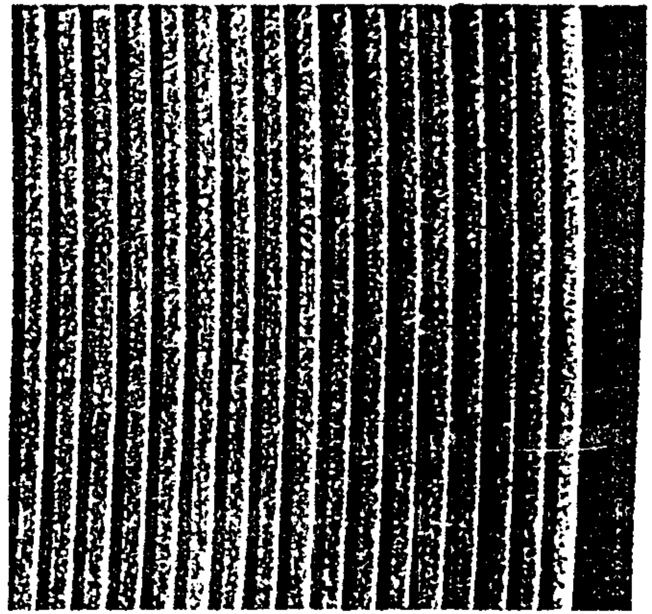
Scanning electron microscope image of the inner part of a Fresnel Zone Plate Lens. This lens has 318 nickel zones, a diameter of 45 μm, and the smallest (outermost) zone is 35 nm wide. A lens like this was used to obtain the images shown.

required positioning accuracy for last ring $\frac{1}{2000}$ of its diameter

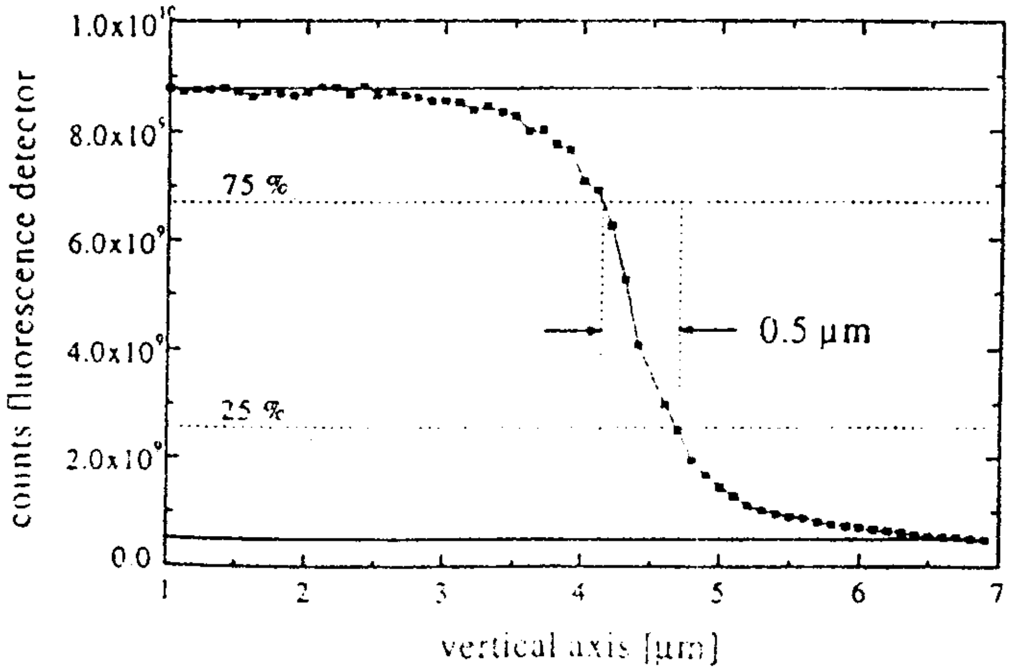
FZP

E.Di Fabrizio,
M. Gentili,
IESS, CNR, Rome, Italy





Cr-mask scan to resolve the spot size: 0.5 μm (V), 6 μm (H), flux 10^{10} ph/s

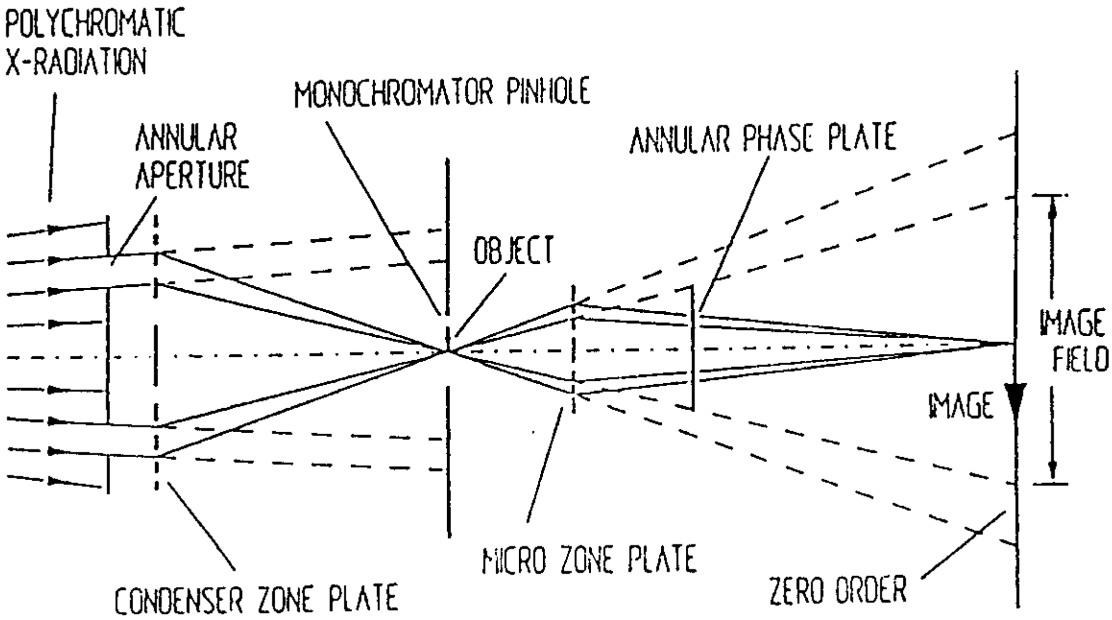


<u>Fresnel zone plate parameters</u>

169 zones, radius first zone: 7.8 μm
radius last zone: 0.3 μm
zone material: gold, thickness: 1.15 μm
substrate: SiN, thickness: 2 μm
aperture: 200 μm
focal length 8 keV: 400 mm
12 keV: 600 mm

A zone plate can image well with resolution < 0.1 μm (better than visible light)

X-ray optical arrangement of the X-ray microscope at BESSY



The annular condenser in combination with the annular phase plate in the back focal plane of the micro zone plate allows phase contrast imaging

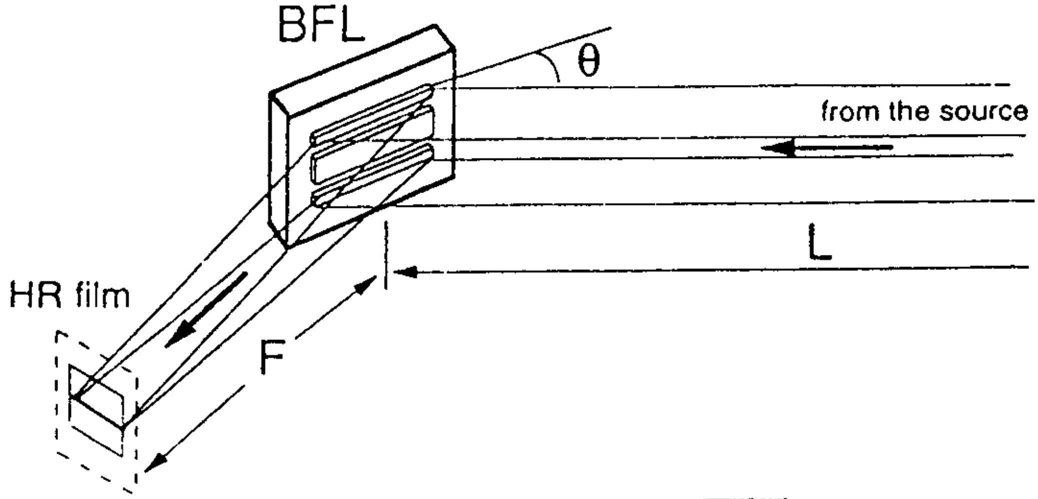
The annular aperture can easily be removed, thus allowing imaging in amplitude contrast

Bragg-Fresnel Optics:

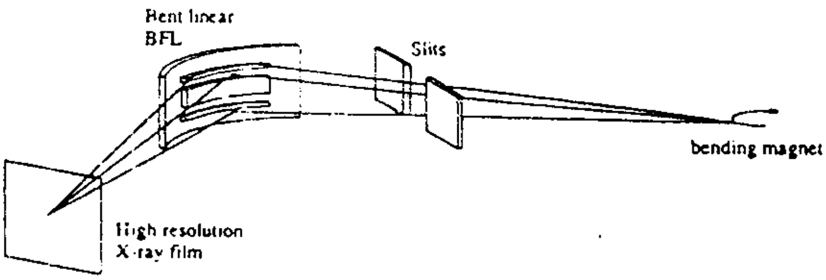
Development at IMT (Institute for Microelectronics Technology, RAS, 142432 Cernogolovka, Moscow district, Russia):

V.V. Aristov, A. A. Snigirev Yu. A. Basov, A. Yu. Nikulin
X-ray Bragg Optics
AIP Conference Proc. 147, 253 (1986)

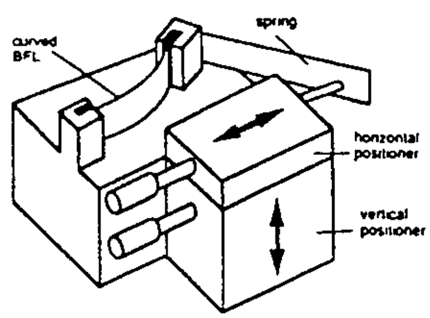
V. V. Aristov, A. V. Gaponov, V. M. Genkin, Y. A. Gorbatov, A. I. Erko, V. V. Martinov, L. A. Matveeva, N. N. Salaschenko
Focusing properties of profiles X-ray multilayer mirrors
JETP Lett. N. 44, 265 (1986)



TWO DIMENSIONAL X-RAY MICROFOCUSING WITH CURVED LINEAR BRAGG-FRESNEL LENS
Snigirev A., Snigireva I., Freund A. *(ESRF, Grenoble)*
Hartman Ya. *(IMT, Chernogolovka)*

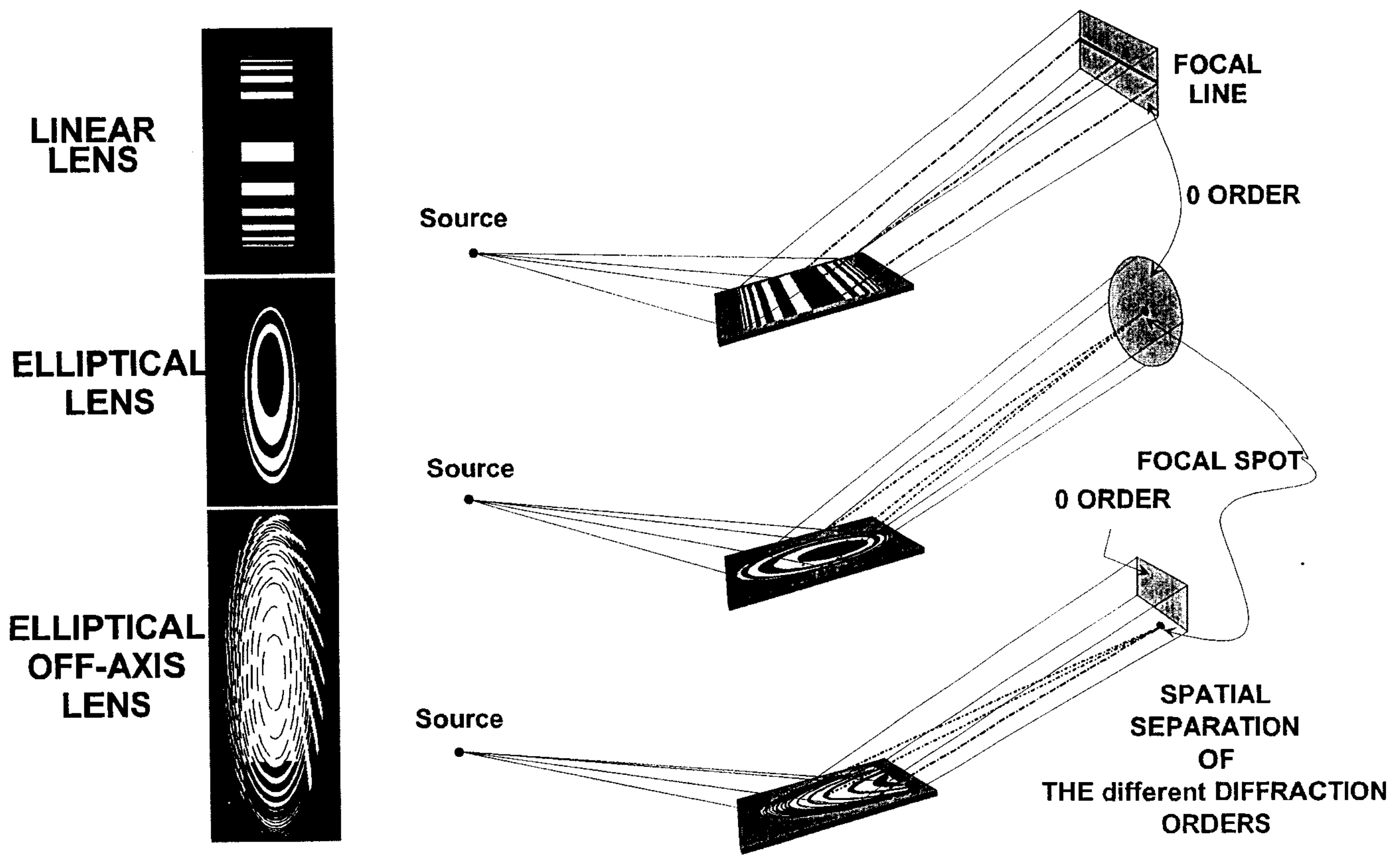


BFL:	linear 150μm(V)*10mm(H)
Energy	15-20keV
Radius of curvature	7-12m
Focal distance	0.4-0.6m
Focus spot	5*10μm2
Gain in flux density (compare with flat BFL)	**x100**

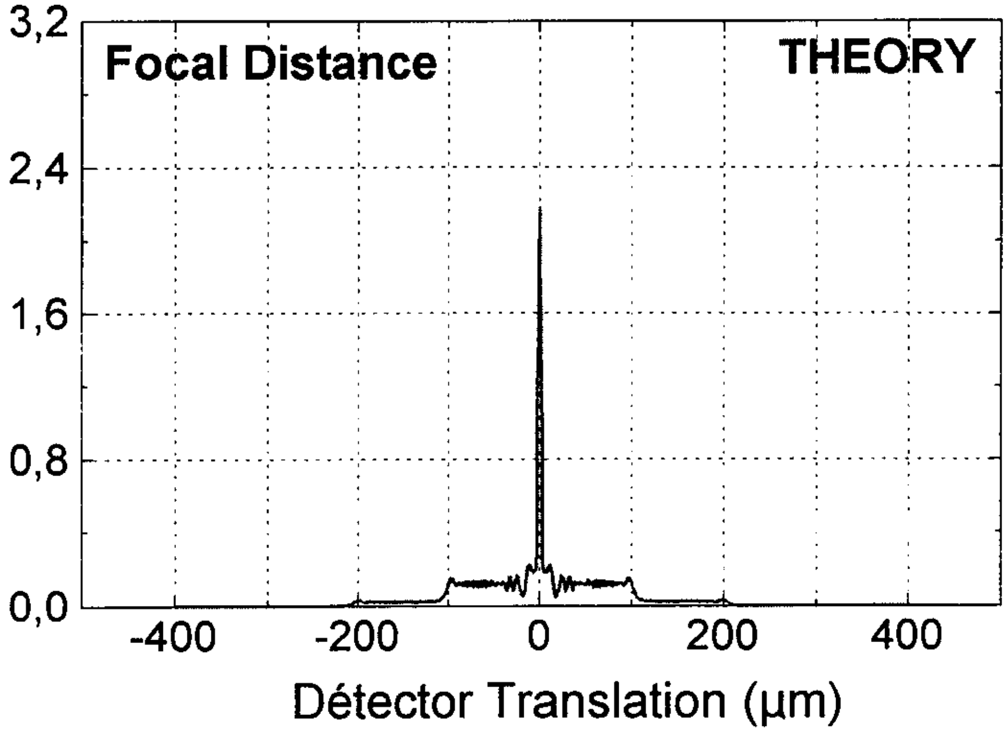


0.5μm spot achieved, however, overlapping 0.th order be...

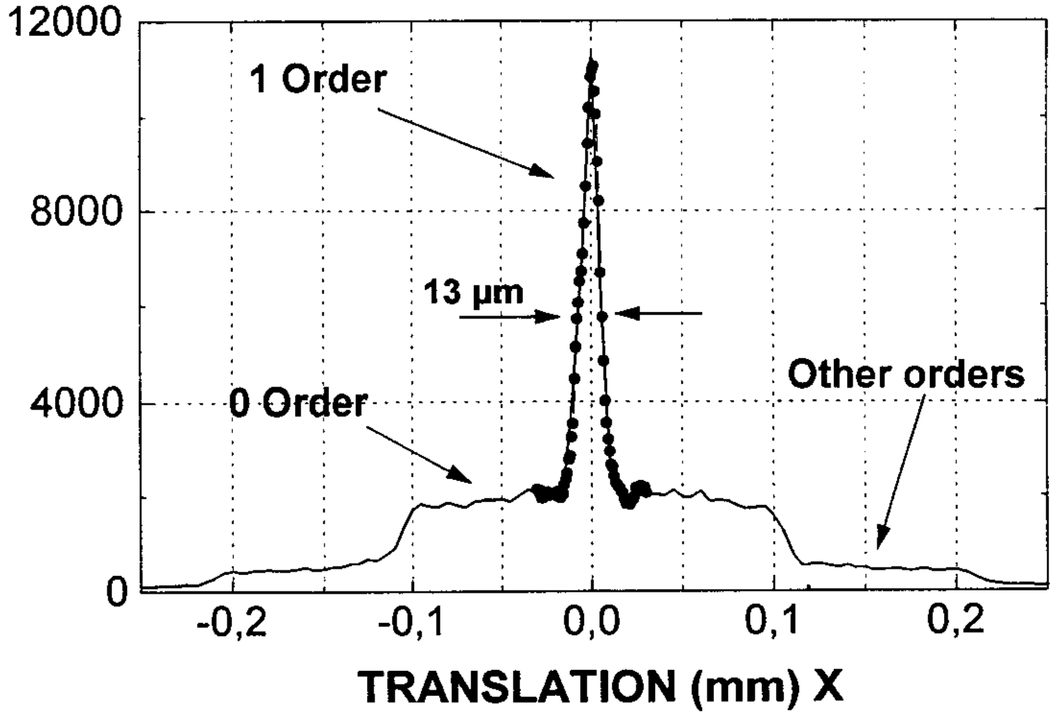
NEW KINDS of FOCUSING and IMAGING OPTICS for the X and XUV ENERGY RANGES



[redacted]

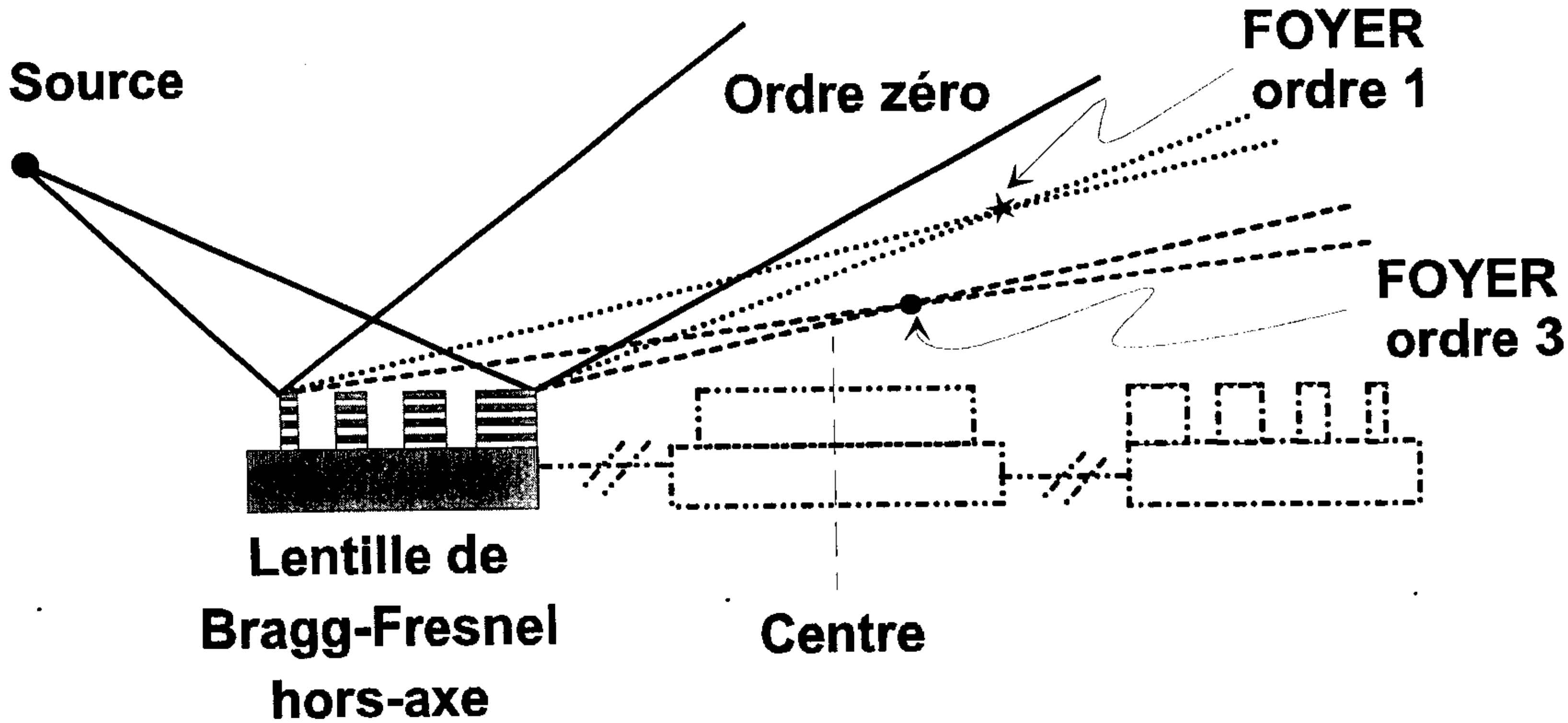


Focal Distance
THEORY
3,2
2,4
1,6
0,8
0,0
-400
-200
0
200
400
Détector Translation (µm)



12000
8000
4000
0
1 Order
13 µm
0 Order
Other orders
-0,2
-0,1
0,0
0,1
0,2
TRANSLATION (mm) X

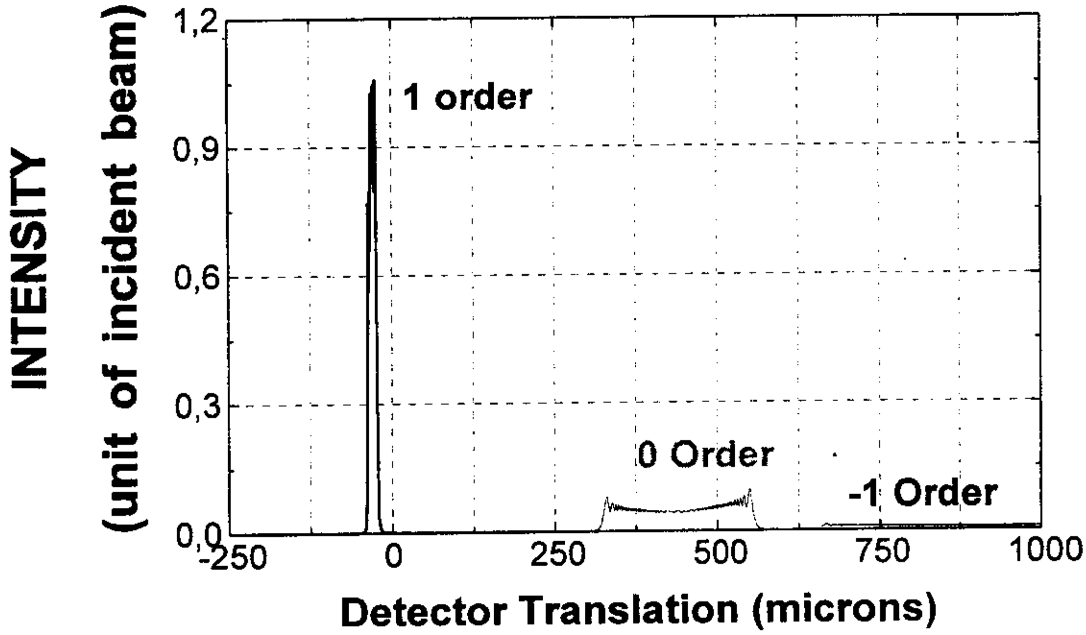
[redacted]



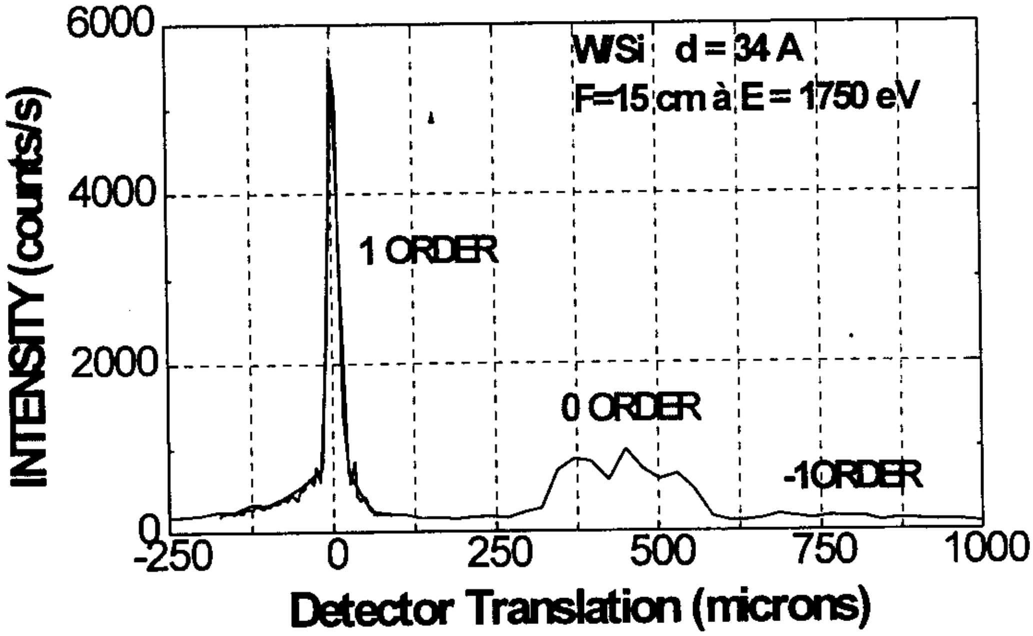
Source
Ordre zéro
FOYER
ordre 1
FOYER
ordre 3
Lentille de
Bragg-Fresnel
hors-axe
Centre

[redacted]

THEORY



EXPERIENCE



INCIDENCE ANGLE : 6.1 DEG.

A compound refractive lens for focusing high-energy X-rays

A. Snigirev*, V. Kohn†, I. Snigireva* & B. Lengeler*‡

* European Synchrotron Radiation Facility, BP220, F-38043 Grenoble Cedex, France
† Kurchatov, I. V., Institute of Atomic Energy, 123182 Moscow, Russia

THE development of techniques for focusing X-rays has occupied physicists for more than a century. Refractive lenses, which are used extensively in visible-light optics, are generally considered inappropriate for focusing X-rays, because refraction effects are extremely small and absorption is strong. This has lead to the development of alternative approaches[1,2] based on bent crystals and X-ray mirrors, Fresnel and Bragg–Fresnel zone plates, and capillary optics (Kumakhov lenses). Here we describe a simple procedure for fabricating refractive lenses that are effective for focusing of X-rays in the energy range 5–40 keV. The problems associated with absorption are minimized by fabricating the lenses from low-atomic-weight materials. Refraction of X-rays by one such lens is still extremely small, but a compound lens (consisting of tens or hundreds of individual lenses arranged in a linear array) can readily focus X-rays in one or two dimensions. We have fabricated a compound lens by drilling 30 closely spaced holes (each having a radius of 0.3 mm) in an aluminium block, and we demonstrate its effectiveness by focusing a 14-keV X-ray beam to a spot size of 8 μm.

The index of refraction for X-rays in matter can be written as $n = 1 - \delta + i\beta$, where β is the absorption index and δ is the

‡ Present address: Physikalisches Institut, RWTH Aachen, 52056 Aachen, Germany

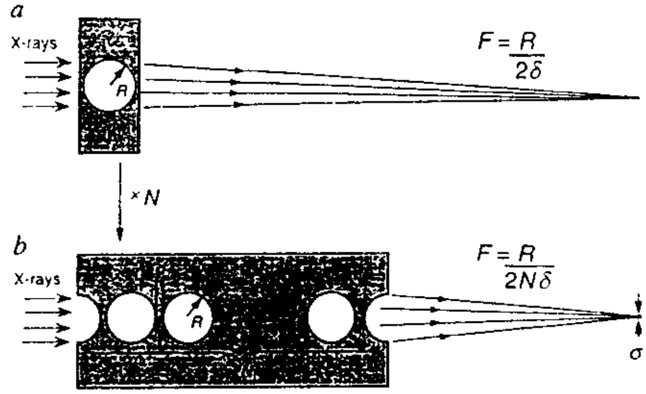


FIG. 1 Schematic diagram showing the principles of X-ray focusing by a compound refractive lens (CRL). As $(1 - \delta)$ is smaller than 1 (where δ is the decrement of the refractive index), a collecting lens for X-rays must have a concave shape. *a*, A simple concave lens fabricated as a cylindrical hole in the material. *b*, A CRL consisting of a number (N) of cylindrical holes placed close together in a row along the optical axis, focuses the X-rays at a distance that is N times shorter compared to a single lens. R is the radius of the holes, d is the spacing between the holes, λ is the X-ray wavelength, and F is the focal distance for a parallel input beam.

refractive index decrement. Refraction being very small (δ is typically between 10^{-5} and 10^{-7}), all attempts to date to build refractive lenses for X-rays have been unsuccessful. Recently, the discussion about refractive lenses has been revived. Suehiro, Miyaji and Hayashi[3] have proposed a refractive lens of high-atomic-number (high-Z) material for focusing X-rays. Michette[4]

other improvements

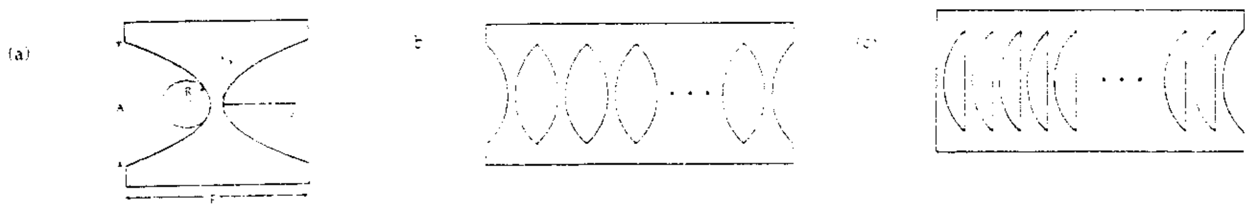


Fig. 3. Schematic view of the parabolically shaped single and compound refractive lenses: R, radius of curvature; A, aperture of the lens; p, length of the single lens or the distance between the centers of the two neighboring holes for a compound lens. (a) Single parabolic refractive lens. (b) compound refractive lens with parabolically shaped holes. (c) compound refractive lens with parabolically shaped half-holes.

Table 1. Calculated CRL Parameters for Boron and Aluminum[a]

Lens Material, Hole Radius, R Focal Distance, F	Energy, E (keV)	Number of Holes, N	Effective Aperture, A (μm)	Resolution σ (μm)	Real Gain, g	Length, L (mm)
	5	13	251	1	190	13
B	10	55	211	0.6	186	55
R = 500 μm	20	222	177	0.4	168	224
F = 1 m	30	501	160	0.3	166	506
	40	892	149	0.2	176	901
Al	10	45	116	1	6	45
R = 500 μm	20	184	163	0.4	36	186
F = 1 m	30	417	160	0.3	60	421
	40	743	149	0.2	80	750

[a] Calculations were made in the following conditions: source size, 50 μm; source–lens distance, 50 m, lens focal distance, F = 1 m; spacing between holes, d = 10 μm. G is the ideal gain in the intensity at the focus for a point source. The real gain g takes into account the finite source size and the attenuation of the x rays owing to absorption in the material between the holes.

19

Focusing hard X-rays with old LPs

A vinyl long-playing record can be used to form a cheap, aberration-free refractive lens.

Björn Cederström*, Robert N. Cahn†, Mats Danielsson*, Mats Lundqvist*, David R. Nygren†

**Department of Physics, Royal Institute of Technology, SE-104 05 Stockholm, Sweden*
e-mail: ceder@particle.kth.se
†Physics Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA

NATURE | VOL 404 | 27 APRIL 2000 | www.nature.com

In the small-angle approximation, a ray traverses the saw-tooth refractive lens in a straight line parallel to the optical axis. A ray separated by y from the optical axis traverses a thickness of material given by

$$x(y) = y^2 N/(y_g \tan\theta)$$

This is a parabola, with radius of curvature

$$R = y_g \tan\theta/2N$$

The total length of the lens is $L = 2Ny_s/\tan\theta$, and so $R = (y_g/2N)(2y_sN/L) = y_g y_s/L$.

Thus we can treat the saw-tooth refractive lens as a single parabolic lens with a focal length of $f = R/\delta$, where δ is the decrement of the real part of the index of refraction from unity (typically 10^{-6} for hard X-rays). The focusing properties are independent of the fixed free parameter θ.

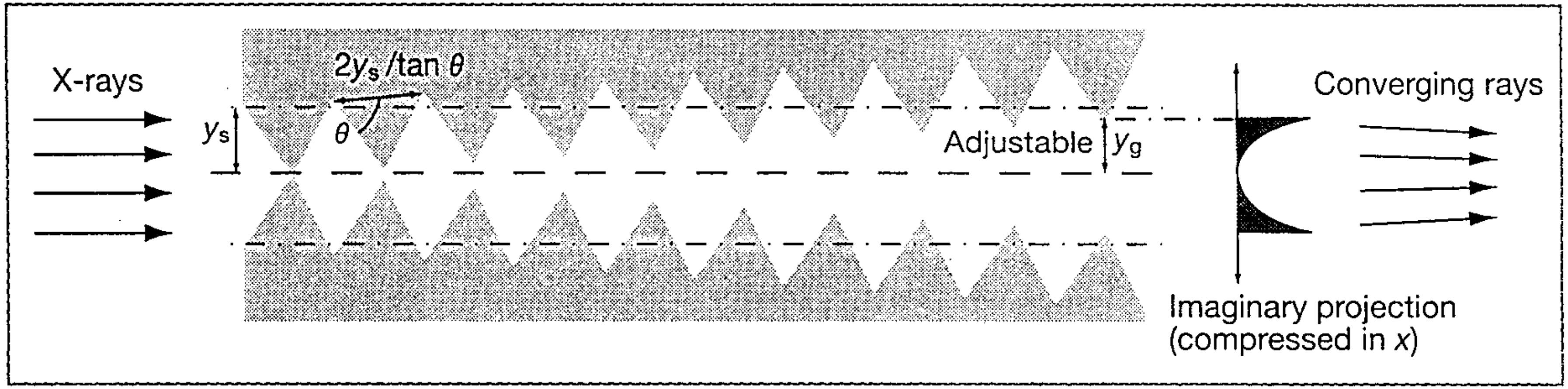


Figure 1 The saw-tooth refractive lens for hard X-rays. Only ten of 300 teeth are shown. The lens bears a striking resemblance to phonograph records, the groove pitch of which is about 180 μm. Measurements of the profile indicate that the depth of the groove is insufficient (about 25μm) and that there is a large amount of material between the grooves, however, resulting in unnecessary X-ray attenuation. We therefore had a dedicated master cut with a groove depth of 90 μm, from which a vinyl record was pressed. Two 60-mm long sections were cut out to form the lens. With 180 μm separation at the end, the focal length is 218 mm for 23-keV X-rays.

Nanometer Spatial Resolution Achieved in Hard X-ray Imaging and Laue Diffraction Experiments

Donald H. Bilderback,* Stephen A. Hoffman, Daniel J. Thiel

Tapered glass capillaries have successfully condensed hard x-ray beams to ultrasmall dimensions providing unprecedented spatial resolution for the characterization of materials. A spatial resolution of 50 nanometers was obtained while imaging a lithographically prepared gold pattern with x-rays in the energy range of 5 to 8 kiloelectron volts. This is the highest resolution scanning x-ray image made to date with hard x-rays. With a beam 360 nanometers in diameter, Laue diffraction was observed from the smallest sample volume ever probed by x-ray diffraction, 5×10^{-3} cubic micrometers.

routinely used with good gain 1 μm

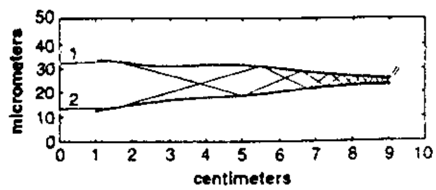


Fig. 1. Profile of the inner diameter (ID) of a capillary measured with an optical microscope. The entering ID is 22 μm and the exit ID is 3 μm. The calculated trajectories of two rays from a parallel x-ray beam are shown. Ray 1 undergoes 12 successive bounces with a net throughput of 57%, as calculated by a two-dimensional ray-tracing program that includes the x-ray reflectance for each bounce. Ray 2 undergoes 11 reflections with a net throughput of 61%. The average reflectivity per bounce exceeds 95%, and the total deflection angles are 2.3 and 2.2 mrad, respectively.

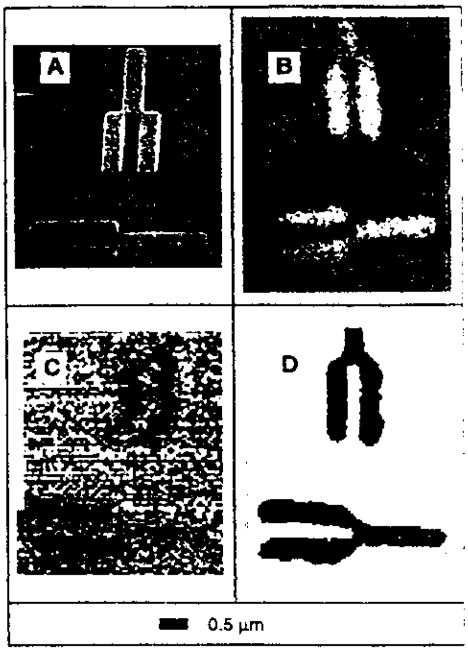


Fig. 2. Various images of a lithographically prepared test sample consisting of a 100-nm-thick gold pattern deposited on a 200-nm thick Si_3N_4 substrate. The line widths of the features are 300 nm. **(A)** Scanning electron micrograph. **(B)** Optical image obtained with a visible-light microscope with a numerical aperture of 0.9. The image is blurred because the structure has features below the resolving power of the microscope. **(C)** Unprocessed x-ray absorption image. The image was formed from a two-dimensional scan consisting of 50 nm by 50 nm pixels. **(D)** X-ray image after processing. The data in each row were horizontally shifted to compensate for the effects of spatial drift (thermal, air currents, electronic, and so on). A median processor was then applied, which averages all pixel intensities located in a circle of radius 2 pixels.

J. Synchrotron Rad. (1994). 1, 37–42

X-ray Applications with Glass-Capillary Optics

D. H. Bilderback,[a,b] D. J. Thiel,[a,c] R. Pahl[a] and K. E. Brister[a]

[a]Cornell High Energy Synchrotron Source (CHESS), [b]School of Applied and Engineering Physics, and [c]Department of Biochemistry, Cell and Molecular Biology, Cornell University, Ithaca, NY 14853, USA

(Received 23 May 1994; accepted 23 June 1994)

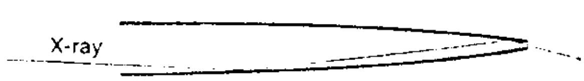


Figure 1
An X-ray beam can efficiently be transmitted through a capillary concentrator if reflection from the smooth walls takes place at angles less than the critical angle of reflection. For hard X-rays and typical borosilicate glasses, the critical angle for 10 keV X-rays is about 3 mrad or 0.2°.

Journal of Synchrotron Radiation
ISSN 0909-0495 © 1994

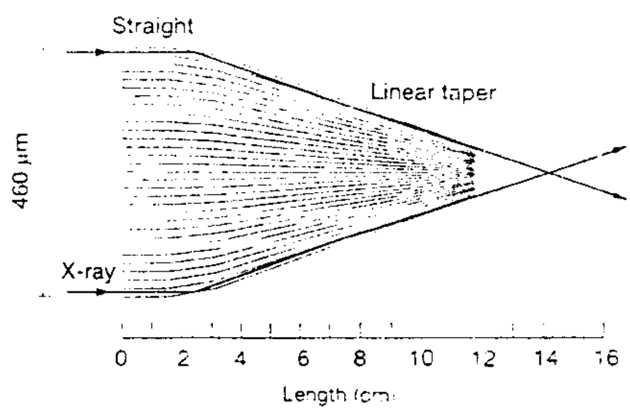


Figure 2
Schematic diagram of X-rays passing through a perfect polycapillary concentrator. A device has been used to condense 6 keV X-rays to a diameter of 68 μm while enhancing the intensity (flux/area) by a factor of 5 (Hoffman *et al.*, 1994b). Before pulling, the concentrator consisted of 330 parallel tubes of 18 μm inner diameter and 2 μm wall thickness (Gibson, 1994).

THIN FILM WAVEGUIDES FOR APPLICATIONS IN X-RAY MICROSCOPY WITH SUBMICRON RESOLUTION

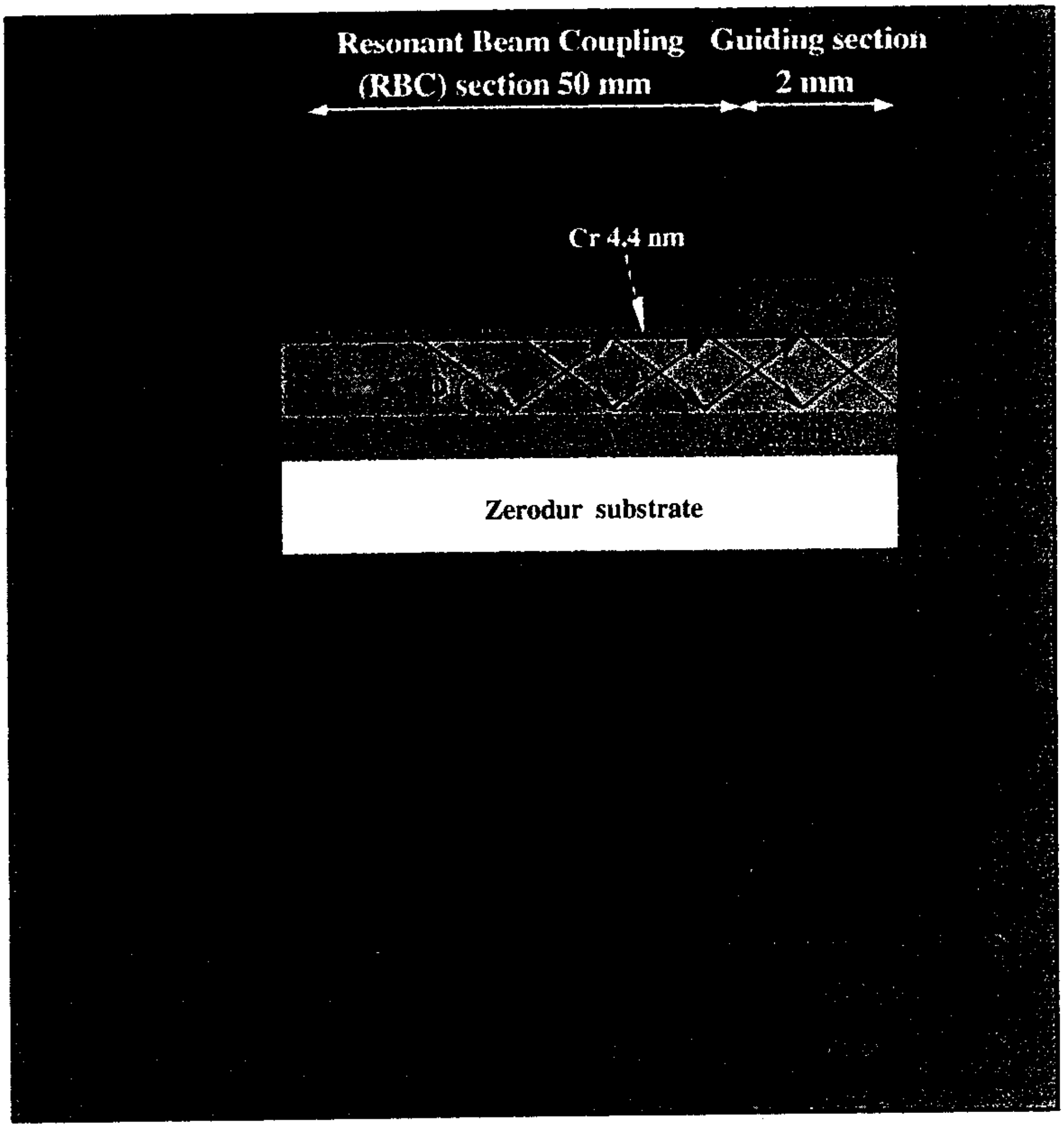
W. JARK	SINCROTRONE TRIESTE
	(WERNER.)JARK@ELETTRA.TRIESTE.IT
S. DI FONZO	SINCROTRONE TRIESTE
A. CEDOLA	IESS-CNR / ESRF
S. LAGOMARSINO	IESS-CNR

http://www.elettra.trieste.it/departments/experiments/multilayer/

With participation of:

B. R. Mueller	SINCROTRONE TRIESTE
C. Riekel	ESRF
P. Engstroem	ESRF
P. Cloetens	ESRF
E. Di Fabrizio	IESS-CNR
A. Brahm	ESRF
J. Pelka	Pol. Acad. Sciences
A. Freund	ESRF
O. Hignette	ESRF
G. Soullie'	SINCROTRONE TRIESTE
M. Mueller	ESRF
C. Giannini	PASTIS-CNRSM
L. De Caro	PASTIS-CNRSM

Microbeam properties



KEY REFERENCES:

- **FIRST X-RAY WAVEGUIDE: BEAM PASSES AN OBSTACLE,**

E. Spiller and A. Segmuller, APL 24, 60 (1974),

Y. P. Feng et al., PRL 71, 537 (1993). APL 64, 930 (1994)

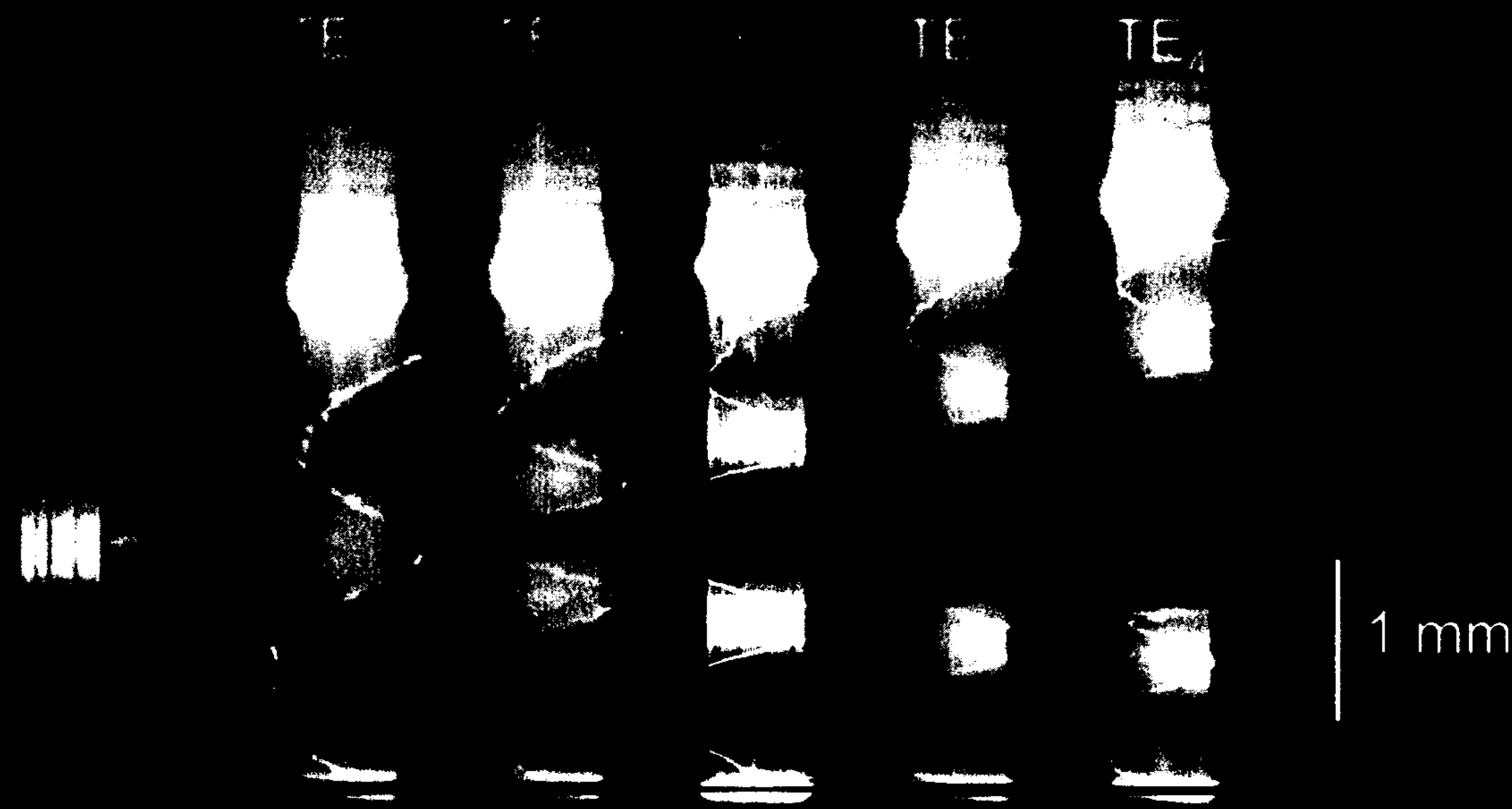
FIRST EXITING BEAM:

S. Lagomarsino et al., JAP 79, 4471 (1996), Y. P. Feng et al., APL 67, 3647 (1995), W. Jark et al., JAP 80, 4831 (1996)

FIRST SUBMICRON RESOLUTION PHASE CONTRAST MICROSCOPE:

S. Lagomarsino et al., APL 71, 2557 (1997), A. Cedola et al., Proc. SPIE 3154, 123 (1997), S. Di Fonzo et al., J. Synch. Rad. 5, 376 (1998)

Fraunhofer Diffraction



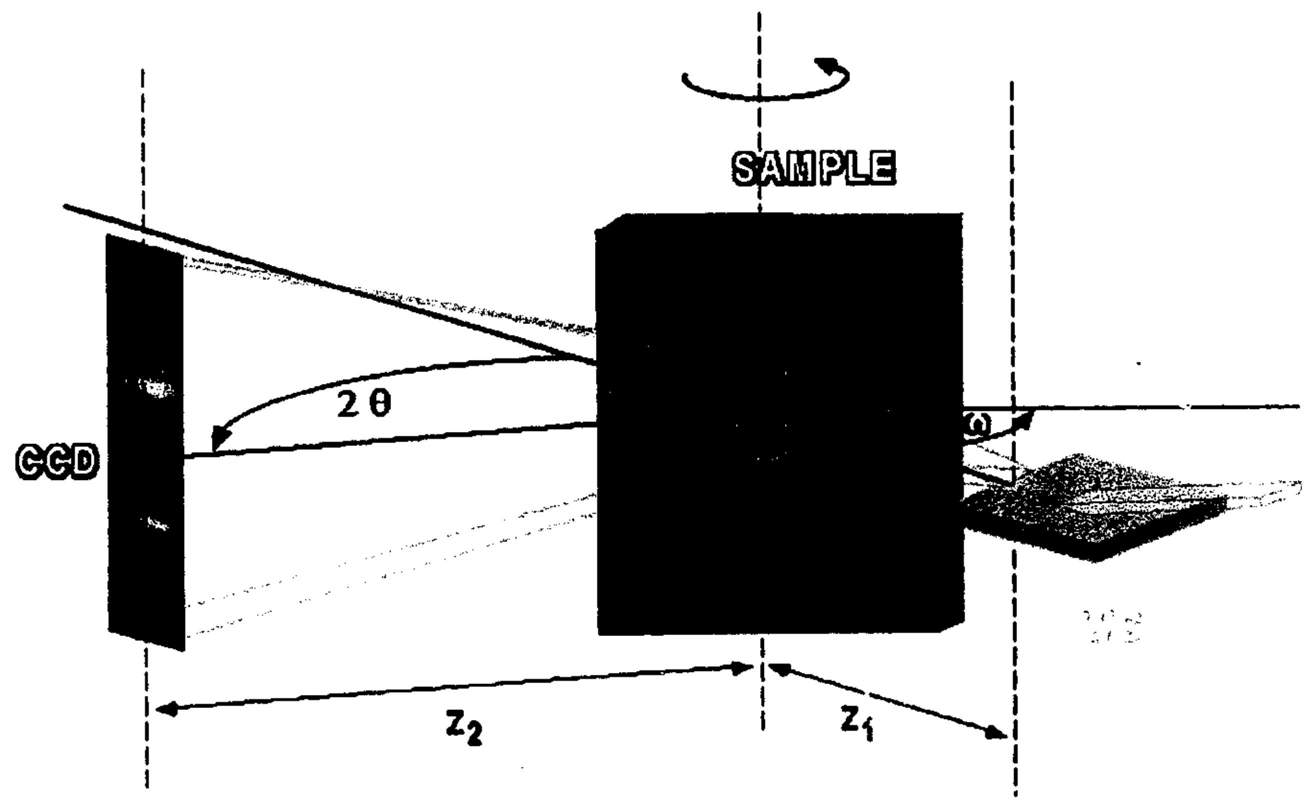
CCD camera images (10 μm pixel size) at 840 mm distance for 13 keV

X-ray diffraction imaging

THE WAVEGUIDED BEAM IS :

- [illegible]D IN THE HORIZONTAL PLANE [illegible] AND DIVERGENT [illegible]

MAGNIFIED VIEW IN THE VERTICAL PLANE OF THE DIFFRACTION PROPERTIES OF CRYSTALLINE SAMPLES



Microscope properties:

- VERTICAL SOURCE SIZE:

≤ 80 nm

- **TYPICAL PARAMETERS**

$Z_1 \leq 5$ mm

$M \geq 120$

$Z_2 = 600$ mm

$\sigma_{VERT} = 1$ mrad $\rightarrow$ spot at sample $\leq$ 15 μm

LATERAL RESOLUTION

≤ 83 nm

with CCD pixel size ≤ 10 μm

Suggestions for the projector:

Do not only look at the public relations data of an objective!

Decide first, what you really want to do!

Then decide for the minimum requested spot size at the sample. Here you cannot intervene anymore by putting an aperture.

Look carefully:

- do you need photon energy tuning or not?
- can you accept higher harmonics (photon energies) at your sample or not?
- can you accept intense tails or not?
- what is your beam size at the objective position?
- can you get the "dream" objective from somewhere?