

НОВЫЕ ВОЗМОЖНОСТИ В ПРИМЕНЕНИИ РЕНТГЕНОВСКОЙ ПРЕЛОМЛЯЮЩЕЙ ОПТИКИ

Анатолий СНИГИРЕВ ESRF, Grenoble, France





Фонд некоммерческих программ «Династия» http://www.dynastyfdn.com/



Проф. Владимир Бушуев кафедра физики твердого тела физического факультета Московского государственного университета им. М. В. Ломоносова

Foundation Dynasty Prof. Vladimir Bushuev Moscow State University, Moscow, Russia

European Synchrotron Radiation Facility



SR Sources Worldwide



- New sources: Australia (Boomerang), Canada (CLS), China (???), France (SOLEIL), Germany (PETRA-III), Spain (ALBA), UK (DIAMOND),
- New projects: US (NSLS-II), Sweden (MAX-IV...
- The 3 large rings: APS (USA), ESRF (Europe), SPring-8 (Japan)

ESRF Members and Scientific Associates

Contribution to ESRF budget (and share of beam time)

Members

•	France	27.5%
•	Germany	25.5%
•	Italy	15%
•	UK	14%
•	Belgium/Netherlands	6%
•	Spain	4%
•	Switzerland	4%
•	Denmark/Norway/	4%
	Sweden/Finland	
		100%



Scientific Associates

Current discussions with:

- <u>Russia</u>
- Slovakia
- Estonia, Latvia, Lithuania
- Ireland (?)
- Greece (?)

MoU signed in Moscow at KCSR in 11 June 2008

Russian scientists and ESRF strengthen cooperation

PRESS RELEASE - The Kurchatov Institute in Moscow (Russia) and the European Synchrotron Radiation Facility (ESRF) in Grenoble (France) have made a step towards a closer collaboration between the scientific communities of these two institutes. A Memorandum of Understanding was signed on 11 June 2008 in Moscow to promote the different areas of this collaboration.



13.06.2008

The Kurchatov Institute in Moscow.

The Memorandum foresees a joint research and development programme as well as exchange of scientists and scientific expertise with the aim of pushing forward common projects. In this framework, the two institutes will also organise joint workshops and conferences.

In his speech at the signing ceremony, the Head of the Russian Federal Agency for Science and Innovations, Prof. Sergey N. Mazurenko, emphasised the importance of scientific and technological links between the two laboratories. This statement was echoed by Prof. Michael V. Kovalchuk, the Director of the Kurchatov Institute.

In his reply, the Director General of the ESRF, Prof. William G. Stirling, expressed his admiration for the achievements of Russian scientists using synchrotron light and his desire to see a longterm relationship develop with the Kurchatov Institute and Russian scientists.

The Principal Elements of the Upgrade Programme

- Reconstruction of ~ 1/3 of the beamlines: Improved performance and routine nano-focus capabilities
- Extension of ~1/3 of the Experimental Hall: 120 m long beamlines for nano-meter and nano-radian beams
- Upgrade of the accelerator complex: Very high brilliance and reliability of the X-ray source
- Development of new SR Instrumentation: To underpin the beamline and source improvements
- Enabling science-driven Partnerships: New science and applications

ESRF Council 22-25 November 2008 177 million Euros 2009-2015



Buildings and BLs movements

Many BLs involved: estimation of the cost of several scenarios



ESRF HIGHLIGHTS

		Even ID (ID02, ID06)	Odd ID (ID01, ID03)	Even BM (BM02, 4,) 3 mrad	Even BM (BM02, 4,) 9 mrad	Odd BM (BM01, 3,) 3 mrad	Odd BM (BM01, 3,) 9 mrad
Magnetid Field	[T]	Variable	Variable	0.4	0.85	0.4	0.85
Horiz. beta functions	[m]	37.5	0.3	1.3	0.9	2.1	1.6
Horiz. dispersion	[m]	0.144	0.033	0.059	0.042	0.088	0.073
Horiz. rms e- beam size	[µm]	415	51	95	75	131	112
Horiz rms e- divergence	[µrad]	10.3	108	115	111	102	97.4
Vert. beta functions	[m]	3.0	3.0	41.2	41.7	31.6	31.7
Vert. rms e- beam size	[µm]	8.6	8.6	32	32	28	28
Vert. rms e- divergence	[µrad]	2.9	2.9	1.3	1.3	0.9	0.9

Table 2: Beta functions, dispersion, rms beam size and divergence for the various source points.

ESRF ID Source size (FWHM) :



Undulator X-ray beam





Coherence Characterisation by Holography / Boron fiber



$$I(x_{1}) = 1 + A(x_{1}) + 2A(x_{1})F(x_{1})$$

$$A(x_{1}) = \frac{f_{1}/2(x_{1})}{(1+f(x_{1})^{1/2}}, \quad f(x_{1}) = \frac{\alpha^{2}R}{(x_{1}-R)^{3}}, \quad \alpha = 2\delta z_{1}$$

$$F(x_{1}) = \cos\left(\pi \frac{(1-\beta^{2})}{\lambda z_{1}}(x_{1}-R)^{2}\right)\exp\left(-\frac{(x_{1}-R)^{2}}{\sigma_{mc}^{2}}\right),$$

50µm





V. Kohn, I. Snigireva, A. Snigirev. Phys. Rev. Let. 85, 13, 2745 (2000)

Techniques and Instrumentation Test Beamline ID06 @ ESRF

Science and technology programme

- White beam development
- Microoptics test bench
- Detector test bench
- Large Volume Press
- Pulsed magnetic fields



Microoptics test bench at ID6

A light for Science

C. Detlefs, T. Roth

Source: CPMU

White beam test station

Cinel mono (ESRF)



55m from source

Microoptics test bench

Detector test bench

Large Volume Press Pulsed magnetic fields

European Synchrotron Radiation Facility

Focusing Optics for Hard X-rays (E > 6 keV)

		reflectiv	diffractive	refractive		
	Kirkpatrick Baez systems		kpatrick Baez systems Capillaries Waveguides		Fresnel Zone plates	Refractive lenses
	mirrors Kirkpatrick Baez, 1948	multilayers Underwood Barbee, 1986	Kreger 1948	Feng et al 1993	Baez 1952	Snigirev et al, 1996
Energy	< 30 keV	< 80keV	< 20keV	< 20keV	< 30 keV (80)	<1 MeV
Bandwidth ΔE/E	w. b.	10 ⁻²	w.b.	10 ⁻³	10 ⁻³	10 ⁻³
resolution	25 nm @15keV Mimura 2006	41x45nm ² @24keV Hignette 2006	50 nm Bilderback 1994	40x25 nm ² Salditt 2004	30 nm @20 keV Kang, 2006 <u>17 nm</u> , 2007	50 nm@ 20keV Schroer, 2004 150nm@ 50keV Snigirev,2006

Introduction to Capillary Optics



Multi-Bounce Glass Capillary



Easy! to make small focal spot < 1 µm

50 nm! D. H. Bilderback, S. A. Hoffman, D. J. Thiel *Science, 1994, 263, 201.*

Short working distance (sub-mm scale) Low transmission

For Glass: θ_c (mrad) = 30 / E (keV) 2 mrad @ 15 keV 1 mrad @ 30 keV

One-Bounce Glass Capillary



Large working distance (cm scale) Near 100% transmission

Challenge! to make small focal spot < 1 µm

Short and compact – may fit in spot that is too short for KB mirror assembly!

First really useful elliptical x-ray optic -Balaic, Nugent, Barnea, Garrett, Wilkins, J. Synch. Rad. 1995; 2: 296.





2 step focusing using single-bounce ellipsoidal capillary combined with Fresnel zone plate.





 $l_1 = 27.5 \text{ mm}$ $l_2 = 2.5 \text{ mm}$

 α = 0.08 mrad M = 11 Δl = 5 mm

<u>0.25 μm !!!</u>

Fresnel Zone Plate (FZP)



FZP for hard X-rays (E > 5 keV)

APS:

Au FZP (collabor. With Wisconsin)outermost zone width $\sim 0.1 \ \mu m$ X-Radia: 30-50 nm20-50 k\$ML Laue FZPs

Spring-8:

Ta FZP (NTT AT) outermost zone width ~ 0.1- 0.2 μm Y. Kagoshima - 50 nm FZP at 10 keV jelly-rolled FZP (N. Kamijo) – imaging at 80 keV

ESRF:

Au, Ni etc. (collabor. PSI, Swiss and ELLETRA, Italy) outermost zone width ~ 0.1 - 0.2 µm Si FZPs outermost zone width ~ 0.4 µm





FZP Si chip

Chip	$T_m / h, \mu m$	E_{range} , keV	$\eta_{max}, \%$	E_{max} , keV
DOE-4	12/9	6 - 12	30	7.5
DOE-5	80 / 16	11 - 21	26	14
DOE-7	90 / 30	17 - 40	32	23





thickness of Si membrane maximum height of zones energy range where focusing efficiency >20% maximum focusing efficiency energy at which η_{max} maximum is achieved

aspect ratio ~50 !!!

X-ray Compound Refractive Lenses



N = 30

LETTERS TO NATURE



NATURE · VOL 384 · 7 NOVEMBER 1996

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

<u>Refractive optics</u> after 10 years development

standard tool at SR beamlines worldwide.

~ 50% of ESRF beamlines use refractive lenses

the most versatile and adaptable X-ray optics

- energy range
- focal length
- -from a few keV to hundreds of keV -from a few millimeters to tens of meters
- focal spot
- -from tens of nanometers to tens of microns
- microradian collimation
- high stability and low cost

applications: microdiffraction, microfluorescence and imaging, standing wave microscopy etc.

Russian collaborators:

Kurchatov Institute, Moscow IMT RAS, Chernogolovka

The first AL CRL



Si parabolic lens





50-100 m

Refractive optics:

Condensers/collimators	F ~ 10 m	10 μm
Micro-optics	F ~ 1 m	1 μ m
Nano-optics	F ~ 10-100 mm	10 – 100 nm

Energy range: CRL 10 - 100 - (1000) keV

Reftractive optics / Materials







	/		
Í	1		
		SU	8/Ni

	material	mode	E, keV	F, m	resolution, μm
	Al	microfocus	18	0.3	0.48
		Imaging/tomo	25	1	0.3/0.4
	Ве	microfocus	12	0.5	1
		imaging	12	0.5	0.11
	PMMA	microfocus	12	1	10
	SU8	microfocus	14	0.2	0.27
	Ni	microfocus	40-120	0.5	0.8
	diamond	microfocus	9	0.5	2.2
	glassy carbon	microfocus	25	0.03	1.4
	Si planar	microfocus	14	0.25	0.6
i	Si nanolens	microfocus	15	0.03	0.05







Parabolic Compound Refractive Lenses

 $\frac{R}{2N\delta}$

Collab. B.Lengeler, C.Schroer, *RWTH, Aachen, Germany*

stack of lenses: compound refractive lens (CRL)

Al, Be

R = 0.2 mm $2R_0 = 0.9 \text{mm}$ $d \approx 5 \mu \text{m}$

single lens

R = 0.5mm $2R_0 = 2-3 mm$

variable number of lenses: N = 10...300



CRL transfocator Energy range 10 -100 keV



Cinel X-ray Refractive Lens Transfocator



cartridge and lenses

cartridges with Be lenses



(in-vacuum/white beam) ID11

lens and cartridges assembly5.12.08chamber assembly8.12.08

installation at ID11 test / commissioning January 2009 Jan-Feb 2009



32 + 64 = 96 Al lenses 1 + 2 + 4 + 8 + 16 + 32 = 63 Be lenses



vacuum chamber



actuators

aboard view









X-ray High Resolution Diffraction Using Refractive Lenses



E = 28 keV Al CRL, N = 112 F = 1.3 m

HR CCD det $2 \mu m$ resolution

resolution is limited by angular source size: s/L ~ 1 μrad

Si photonic crystal pitch - $4 \ \mu m$



M. Drakopoulos, A. Snigirev, I. Snigireva, J. Schilling, Applied Physics Letters, 86, 014102, 2005.



Microradian x-ray diffraction



2D detector

A versatile tool to study novel functional materials with nano- to micro-scale structure.

It allows

- 3D structure determination on the scale of 10s nm 10s microns
- probing positional order on distances up to 100 microns
- quantitative characterization of various types of disorder
 (stacking disorder, domain size, microstrain, etc)
- to identify the superstructure built by pores in the membrane
- to measure the width of the Bragg peaks, thus quantitatively probing the long-range order.
- to study the spatial distribution of the crystalline grains along the substrate surface by taking measurements at different lateral positions in the samples and to construct the domain maps.



Diffraction patterns of colloids made from polystyrene spheres (425 nm)

A light for Science



Clear face-centred cubic structure

- Presence of sliding defects
- These defects are crucial in generating ...ABCABC... stacking order

Panel A displays a full diffraction pattern while a zoom into one quarter is given in B along with the crystallographic assignment of the peaks. Again, the relative weakness of the 002, 222 and 224 reflections can be understood on the basis of Fig. 1C. A closer look on the central part of the pattern reveals presence of the Bragg rods, which are caused by the presence of the stacking disorder.



European Synchrotron Radiation Facility

A light for Science



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Spotlight on science Stacking faults in colloidal photonic crystals revealed by microradian Xray diffraction



24-11-2009

The presence of a network of intersecting stacking faults in self-assembled colloidal crystals was demonstrated with X-ray diffraction with microradian resolution. These defects

can seriously affect the optical properties of



Obit

200

ESR

photonic materials fabric ... Read More...



protein from that sequence. Ensuring that the

media



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European Synchrotron Radiation Facility

Lens chip design

F = 10cm @ E = 10 - 50 keV





22 mm

NN	Energy	Single lens	Number of	Radius of parabola	Total lens
	🔓 (keV)	length (µm)	lenses	apex (µm)	length (µm)
4	10	50	12	6.25	620
12	10	100	6	3.13	614
2	45	50	28	6.25	1436
Z	15	100	14	3.13	1422
2	20	50	52	6.25	2660
J	20	100	26	3.13	2634
4	25	50	80	6.25	4088
4	25	100	40	3.13	4048
5	30	50	116	6.25	5924
3		100	58	3.13	5866
6	35	50	160	6.25	8168
o		100	80	3.13	8088
7	40	50	208	6.25	10616
		100	104	3.13	10512
8	45	50	264	6.25	13472
0		100	132	3.13	13340
۵	50	50	324	6.25	16532
9	50	100	162	3.13	16370
10	55	50	392	6.25	20000
10	55	100	196	3.13	19804



10 lenses per set

7 sets ~ 70 CRLs !

Схема изготовления Si преломляющих линз



Si nanolenses



Принцип "Bosch" процесса травления кремния



Si пластина с защитной SiO₂ маской

Изотропное травление кремния в плазме SF₆

Осаждение полимера в плазме C₄F₈

(новый цикл) Изотропное травление кремния в плазме SF₆

Уменьшение амплитуды шероховатости

Оптимизация цикла травления



Стандартный цикл травления

Уменьшенный цикл травления



Chip 01 Bosch process W4 Chip 07 Bosch process W5

Scallop height ~40 nm Scallop length 400 nm

LEO 1530	Mag = 154.45 K X	EHT =	5.00 kV	Signal A = InLens
Interpreter to resolute Serial No. = LEO 1530-21-90	100nm* HI	WD =	6 mm	Output To = Default Printer



EHT = 5.00 kV

WD = 6 mm

Signal A = InLens

Output To = Default Printer

Mag = 154.43 K X

200nm*

LEO 1530

remer to resolute Serial No. = LEO 1530-21-90

Scallop height ~20 nm Scallop length 250 nm

Date :2 Nov 2006

Time :10:52:29

Gun Vacuum = 1.15e-009 Torr

Noise Reduction = Line Int. Done

Si planar lens

ID6, Sept. 2008 F = 40 mm E = 31 keV



Mag = 73 X EHT - 20.00 kV Signal A - SE2 Date :11 Aug 2008 Gun Vacuum - 2.96e.009 mBar 200 µm WD - 17 mm Output To - Default Printer Time :16:31:18 Noise Reduction - Line Int. Done

Hard X-ray Interferometers



Double slit

W. Leitenberger, S. Kuznetsov, A. Snigirev, Interferometric measurements with hard X-rays using a double slit Optics Communications **191**, 91-96 (2001)



 $\Lambda \sim 1 - 10 \ \mu m$

Double mirror

K. Fezzaa, F. Comin, S. Marchesini, R. Coisson, M. Belakhovsky, X-ray interferometry at ESRF using two coherent beams from Fresnel mirrors Journal of X-Ray Science and Technology **7**, 12-23 (1997)

Talbot interferometer

P. Cloetens, J. P. Guigay, C. De Martino, J. Baruchel, and M. Schlenker, Fractional Talbot imaging of phase gratings with hard x rays, Opt. Lett. **22**, 1059-1061 (1997)

Grating interferometer

T. Weitkamp, B. Nohammer, A. Diaz, C. David, E. Ziegler, X-ray wavefront analysis and optics characterization with a grating interferometer Appl. Phys. Lett., 86, 054101 (2005)

Billet split lens

Professeur Felix Billet (1808 -1882) la Faculté des sciences de Dijon d<u>epuis 1843</u>



Billet split lens







far-field interference

X-ray bi-lens interferometer

50







20 µm





 $\Lambda_5 - \Lambda_4 = 5 \text{ nm !}$

10 μm

APS » Journals » Physical Review Letters » Covers » Vol. 103, Iss. 6



Scanning electron microscope images of silicon bilenses carefully designed to create a novel type of x-ray interferometer.

PRL 103, 064801 (2009)

PHYSICAL REVIEW LETTERS

week ending 7 AUGUST 2009

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X-Ray Nanointerferometer Based on Si Refractive Bilenses

A. Snigirev,¹ I. Snigireva,¹ V. Kohn,² V. Yunkin,³ S. Kuznetsov,³ M. B. Grigoriev,³ T. Roth,¹ G. Vaughan,¹ and C. Detlefs¹

¹ESRF, B.P. 220, 38043 Grenoble, France ²Russian Research Center "Kurchatov Institute," 123182, Moscow, Russia ³IMT RAS, 142432 Chernogolovka, Moscow region, Russia (Received 28 April 2009; published 3 August 2009)

We report a novel type of x-ray interferometer employing a bilens system consisting of two parallel compound refractive lenses, each of which creates a diffraction limited beam under coherent illumination. By closely overlapping such coherent beams, an interference field with a fringe spacing ranging from tens of nanometers to tens of micrometers is produced. In an experiment performed with 12 keV x rays, submicron fringes were observed by scanning and moiré imaging of the test grid. The far field interference pattern was used to characterize the x-ray coherence. Our technique opens up new opportunities for studying natural and man-made nanoscale materials.





Applications

- Coherence / Optics characterization
- Interferometry phase contrast
- Standing wave technique
- Moiré radiography
- Fourier holography
- Double slit topography

Crystal based Bi-lens interferometer Grating based 0.1 – 10 nm 10 – 1000 nm >1000 nm

to be published in PRL, July 31 2009



Principles of Optics

7th (expanded) edition

Max Born and Emil Wolf

Electromagnetic Theory of Propagation, Interference and Diffraction of Light



1st edition in 1959 !!

t for Science

Solution of the Phase Problem in the Theory of Structure Determination of Crystals from X-Ray Diffraction Experiments

Emil Wolf*

Department of Physics and Astronomy and the Institute of Optics, University of Rochester, Rochester, New York 14627, USA (Received 6 May 2009; published 10 August 2009)

We present a solution to a long-standing basic problem encountered in the theory of structure determination of crystalline media from x-ray diffraction experiments; namely, the problem of determining phases of the diffracted beams.

Incident beam

FIG. 1. Illustrating notation relating to Young's interference experiment.

