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SMR/1270-20

SCHOOL ON SYNCHROTRON RADIATION

6 November – 8 December 2000

Miramare - Trieste, Italy

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Crystal Monochromators

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international centre for theoretical physics

School on Synchrotron Radiation

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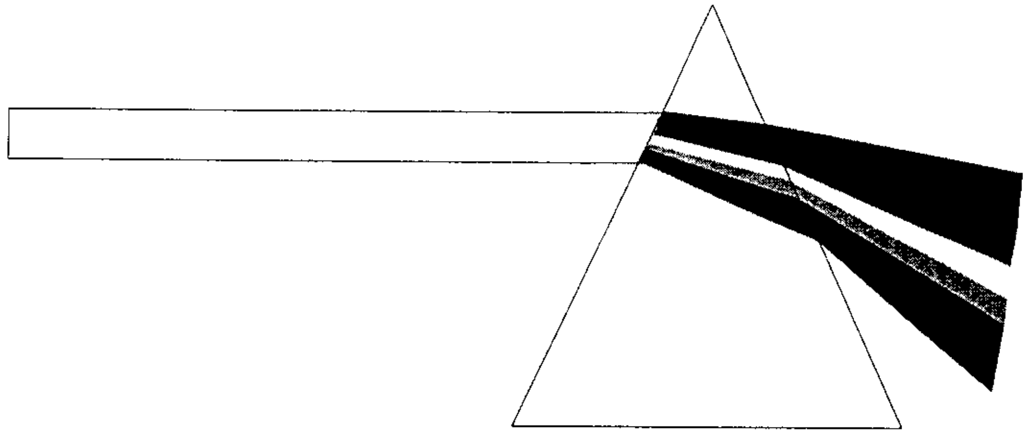
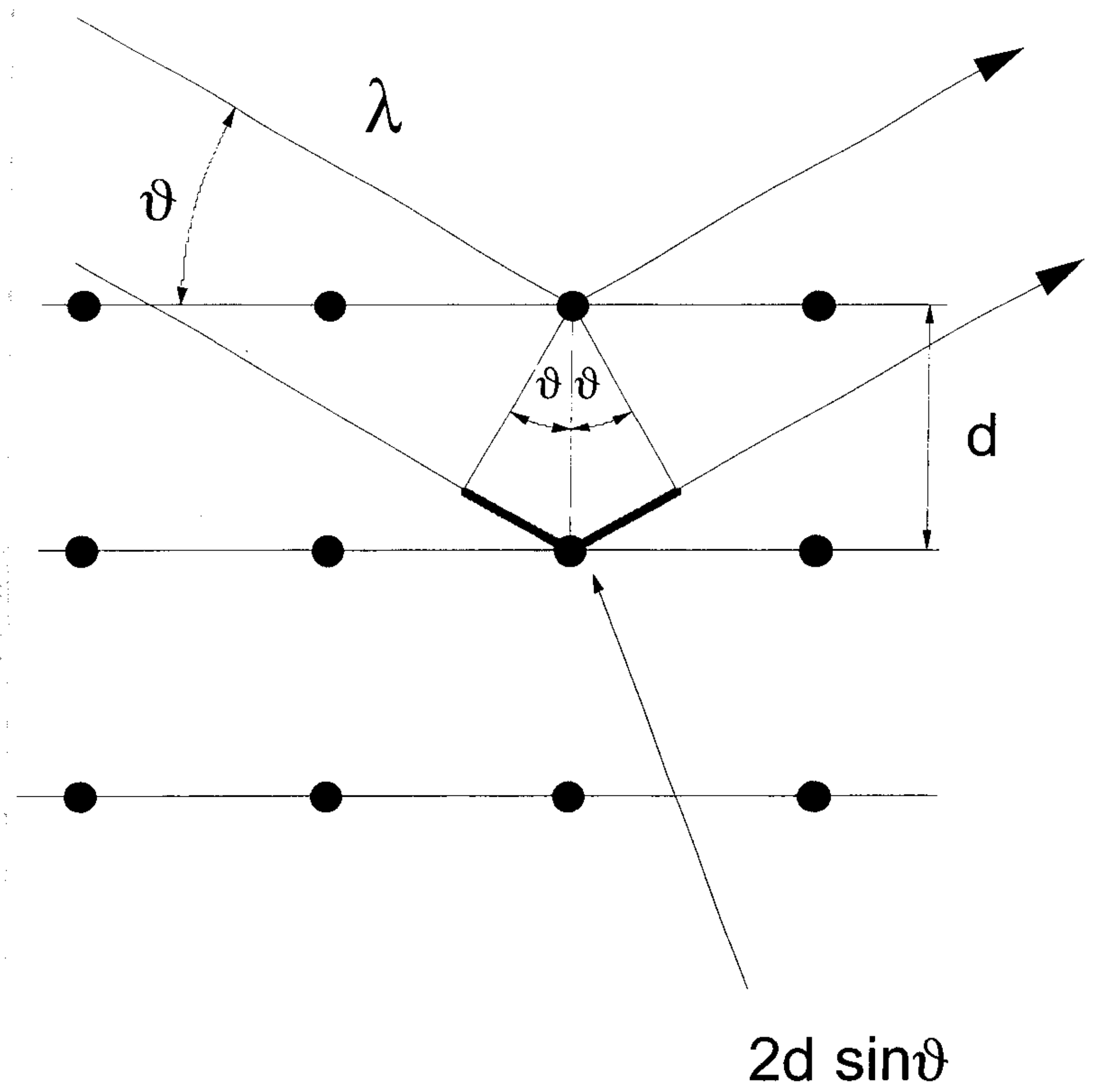
Crystal monochromators

Edoadro Busetto

elettra

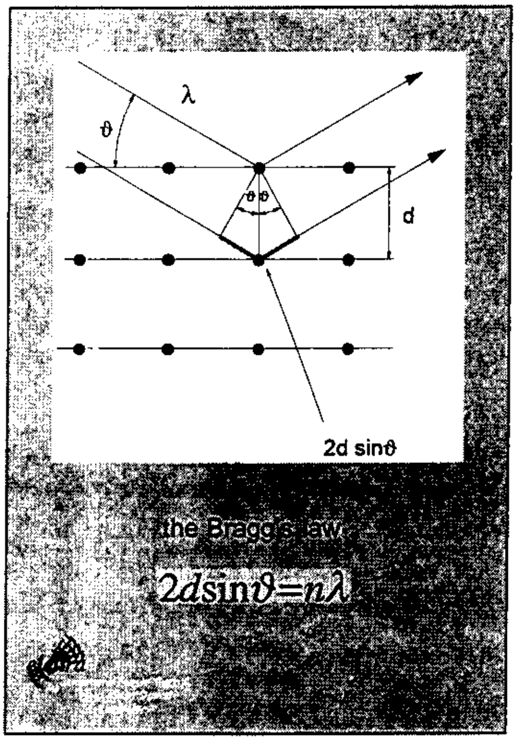


the optical prism is used to separate the components of the white visible light. sampling the out coming light with a slit it is possible to select a part of the spectrum with a spectral purity which depends on the distance and the slits aperture.



the Bragg's law

$$2d\sin\vartheta = n\lambda$$



A single crystal can be though, in the easier way, as the regular repetition of a point along the three space directions.

X-rays wavelengths are comparable with the interatomic distances; when x-rays scatter with a single crystal they produce diffraction under certain conditions .

The angular condition to achieve the diffracted wave of wavelength λ from a single crystal with **d** as crystal lattice planes distance is the Bragg law.

$$2d \sin\vartheta = n\lambda$$

$$2d\sin\vartheta = n\lambda$$

from the Bragg law

$$\sin\vartheta = 1 \;\Rightarrow\; \lambda_{max}$$

therefore

$$\lambda_{max} = 2d$$

and the Bragg angle is 90°

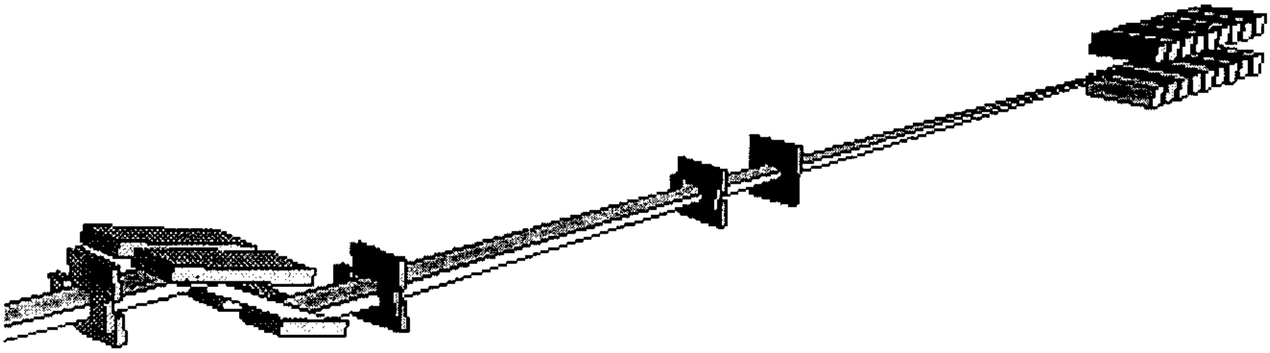
important properties for the x-ray monochromators

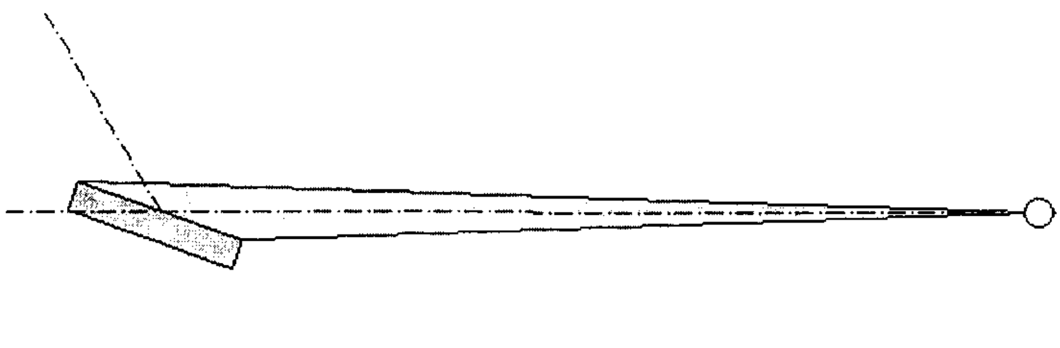
- *ENERGY RESOLUTION*

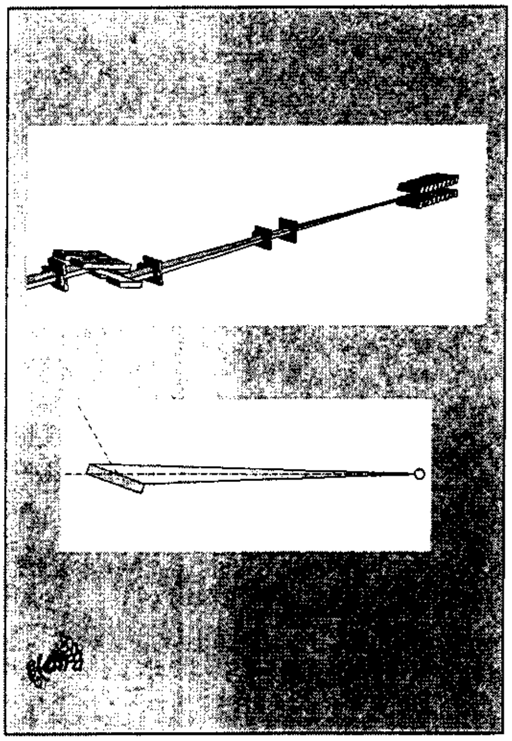
$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta E}{E} = \Delta\vartheta \cot g(\vartheta_B)$$

$\Delta\vartheta$ has two contribution :

- beam angular spread (optics)
- intrinsic reflection width of the monochromator (mosaic / single crystals)







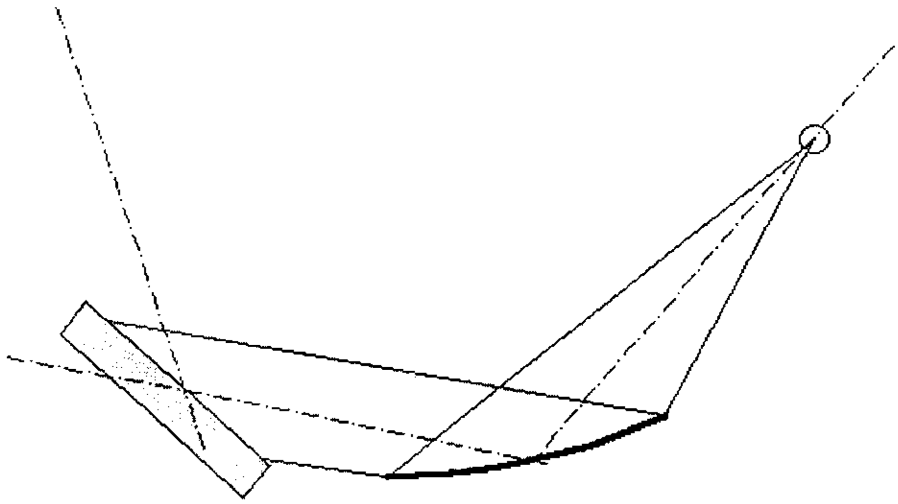
case of monochromators operating with beam divergence in the scattering plane

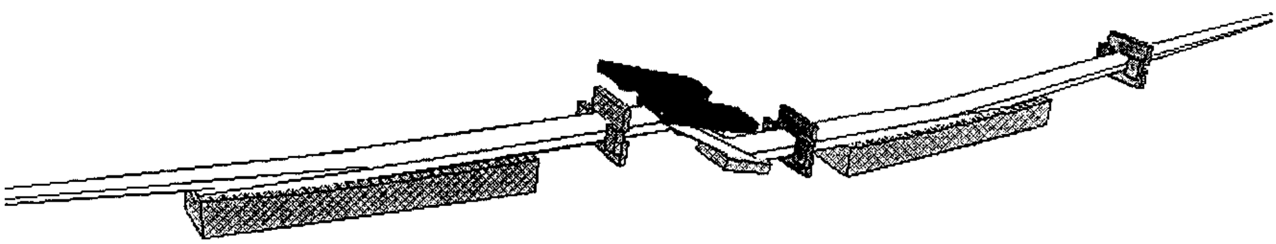
generally beam divergence is much more larger than the Darwin width of the monocrhomator . A factor 10 more in case of Si (111) has the consequence the energy resolution will decrease of the same factor.

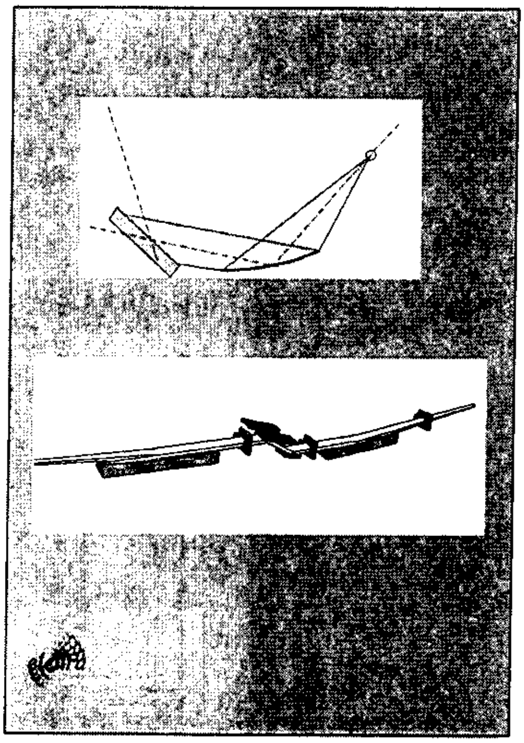
reasonable values for such a condition

Div.3mRad (≈0.02°), Dw(Si111,Cu)≈0.002°

resolving power less than 1000







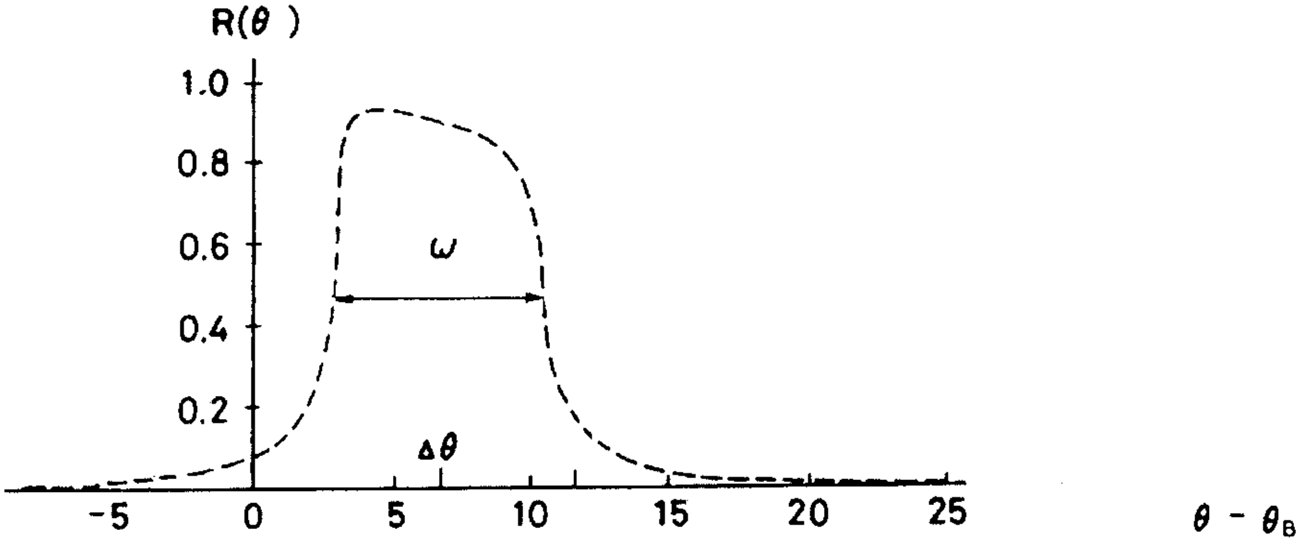
case of monochromators operating without beam divergence in the scattering plane.

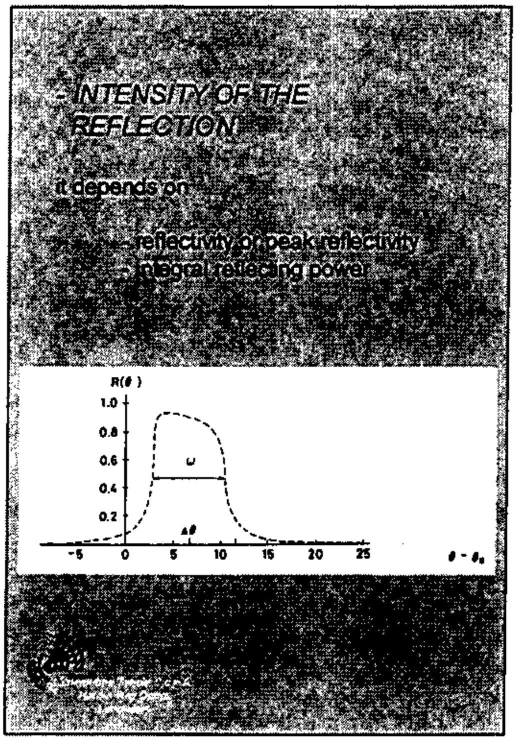
the beam is collimated by a cylindrical mirror and the resolving power of the monochromator could achieve the theoretical value ≈ 7000 in case of Si(111)

- INTENSITY OF THE REFLECTION

it depends on

- reflectivity or peak reflectivity
- integral reflecting power





the typical shape of the Darwin curve

-it describes the angular bandwidth of the diffracted beam when the crystal monochromator is operating in a divergent monochromatic beam

-we achieve the same result if the crystal is rocking around the Bragg angle in a collimated monochromatic beam

-the centre of the Darwin curve is shifted with respect to the origin of the coordinates because of the refraction effect

two models for the x-ray diffraction in single cristals

- kinematical model

scattering from each atom is considered only once:

$$F\left(\vec{q}\right)=\sum_{1}^{\infty} f_{i} \exp\left(i\vec{q}\bullet\vec{r}_{i}\right)$$

we can apply this model for:

- thin perfect crystals (no second interaction)
- distorted or mosaic crystals (loss of the phase condition)

according with Darwin model (1922) the mosaic crystal is defined by two general conditions:

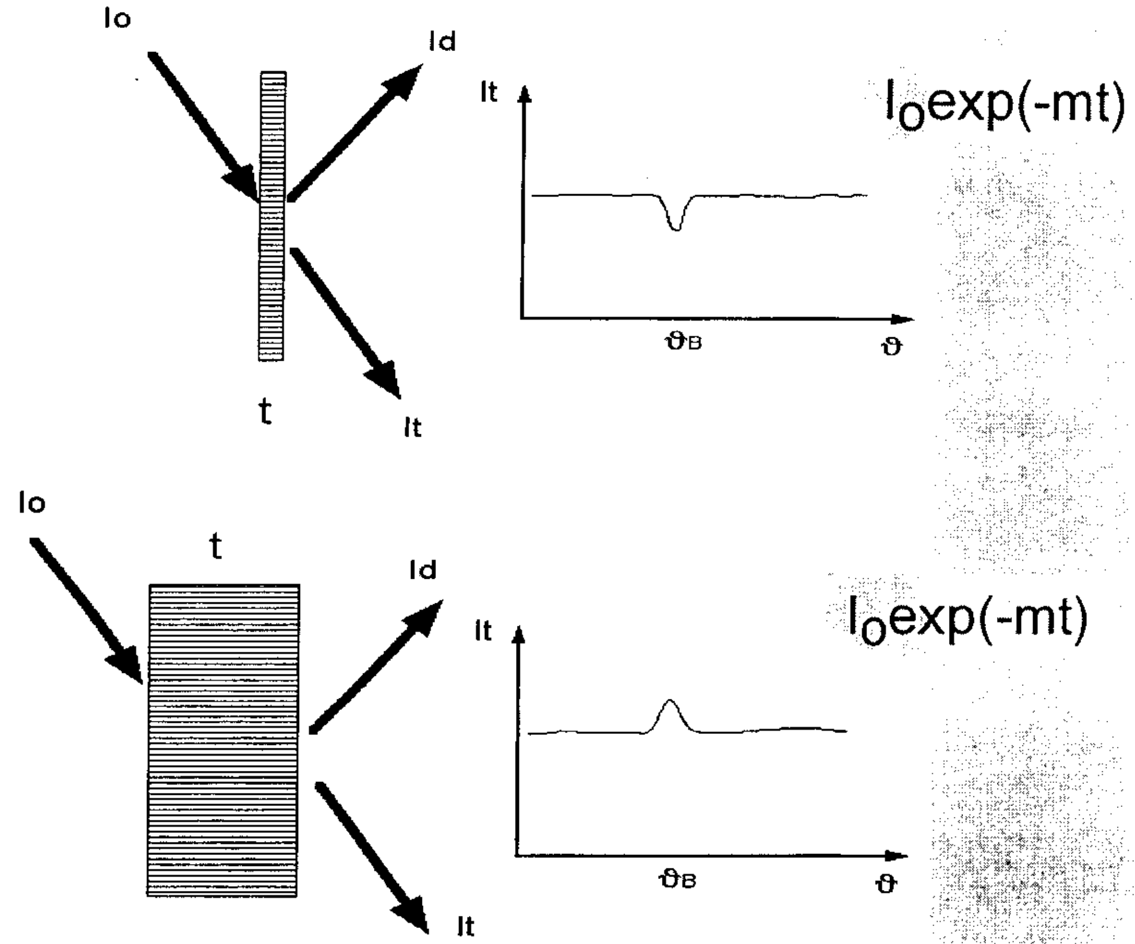
- crystallites have to be misoriented more than the Darwin with of the perfect crystal

- their dimensions have to be smaller than the extintion length of the considered radiation

A particular simple example of a dynamical effect:
the Borrmann effect

a)

b)



a) thin crystal: dip on the transmitted wave due to the aperture of the diffraction channel
b) thick crystal: contribution by transmitted and forward diffracted waves

- dynamical model

for large and perfect crystal:

a) we can't longer consider single interaction. (extinction length)

b) we can't neglect, as well as in the kinematical model, the effect of the radiation absorption

perfect thick crystal with centre of symmetry
Bragg condition
linear polarization (KiKuta, 1971)

$$R_h(w) = L_h - \sqrt{\left(L_h^2 - 1\right)}$$

where $\mathbf{h} = (h,k,l)$

$$\mathbf{L_h} = \frac{\mathbf{1}}{\mathbf{1+k^2}}\left\{\mathbf{w^2+g^2+\left[\left(w^2+g^2-1+k^2\right)^2+4\left(gw-k^2\right)^2\right]^{1/2}}\right\}$$

w,**g**,**k** are function depending on the structure factor related to the particular reflection **h**(h,k,l)

elettra

$$w_h = \frac{1}{2}\left(\sqrt{b} + \frac{1}{\sqrt{b}}\right) \frac{F_{or}^{(n)}}{C\left|F_{hr}^{(n)}\right| e^{-M(n)}} + \frac{\sqrt{|b|}\pi V}{r_e}\left(\frac{n}{\lambda_1}\right)^2 \frac{\sin 2\vartheta_B\left(\vartheta - \vartheta_B\right)}{C\left|F_{hr}^{(n)}\right| e^{-M(n)}}$$

the diffracted wave

w : parameter representing the deviation of the angle ϑ_o from the Bragg angle ϑ_B

$\mathbf{n}$	order of the reflection
λ_1	wavelength of the fundamental
$e^{-M(n)}$	temperature factor
$\mathbf{V}$	volume of the unit cell
ϑ_B	Bragg angle
r_e	radius of the electron e^2/mc^2

$F_{or}^{(n)}$	real part of the structure factor related to the forward direction **h**(000)
$F_{hr}^{(n)}$	real part of the structure factor related to the diffracted direction **h**(h,k,l)

$$g_h = \frac{1}{2}\left(\sqrt{b} + \frac{1}{\sqrt{b}}\right) \frac{F_{oi}^{(n)}}{C \left|F_{hr}^{(n)}\right| e^{-M(n)}}$$

the part concerning the absorption

$$k_h = \frac{F_{hi}^{(n)}}{F_{hr}^{(n)}}$$ the correction for anomalous absorption

$F_{oi}^{(n)}$ imaginary part of the structure factor related to the forward direction **h**(000)

$F_{hi}^{(n)}$ imaginary part of the structure factor related to the diffracted direction **h**(h,k,l)

The structure factors $\mathbf{F}_{hr,i}$ are defined as Fourier sum over the reciprocal lattice vectors of the real and imaginary parts respectively of the total atomic scattering factor $\mathbf{f}_h$

$$F_{hr}^{(n)} = \sum_j \left(f_h^o + f_h' \right) e^{2\pi i \vec{h}\cdot\vec{r}_j}$$

$$F_{hi}^{(n)} = \sum_j f_h'' e^{2\pi i \vec{h}\cdot\vec{r}_j}$$

f' and **f'** represent the correction for the total anomalous dispersion in the region of the absorption edges.

the **b** parameter

defined as :

$$\mathbf{b}=\frac{\sin\left(\alpha-\vartheta_{B}\right)}{\sin\left(\alpha+\vartheta_{B}\right)}$$

α is the angle between the Bragg plane and the crystal surface

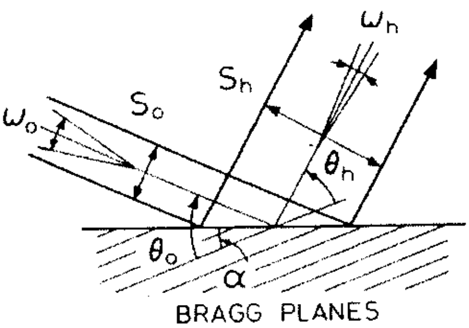


Fig. 3. Geometry of X-ray reflection by a perfect single crystal. θ_0: incidence angle; θ_h: reflection angle. For a non-zero asymmetry angle α ($0 < |\alpha| < \theta_B$), the angular width ω_0 for acceptance is not equal to the angular width ω_h for emergence. The figure is drawn for $b < 1.0$, where $\omega_0 > \omega_s > \omega_h$. Note also the change of beam cross sections, S_0 and S_h.

intrinsic width of the Bragg reflection

$$\omega_s=\frac{2}{\sin 2\vartheta_B}\frac{r_e\lambda^2}{\pi V}C\left|F_{hr}\right|e^{-M}$$

$$\omega_o=\frac{\omega_s}{\sqrt{b}}$$

the angular acceptance as function of the intrinsic width and the **b** parameter:

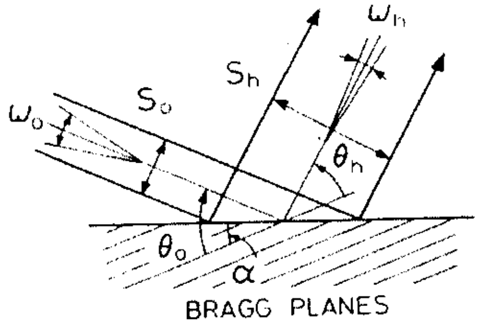


Fig. 3. Geometry of X-ray reflection by a perfect single crystal. θ_0: incidence angle; θ_h: reflection angle. For a non-zero asymmetry angle α ($0 < |\alpha| < \theta_B$), the angular width ω_0 for acceptance is not equal to the angular width ω_h for emergence. The figure is drawn for $b < 1.0$, where $\omega_0 > \omega_s > \omega_h$. Note also the change of beam cross sections, S_0 and S_h.

Bragg reflection width in case of asymmetric cut crystal is defined by:

$$\omega_h = \omega_s \sqrt{b}$$

$$\omega_h = b\omega_o$$

the angular acceptance as function of the Bragg reflection width :

also for the beams sections

$$S_h = \frac{S_o}{b}$$

combining the two formulas we have the well known Liouville's theorem

$$\omega_h S_h = \omega_o S_o$$

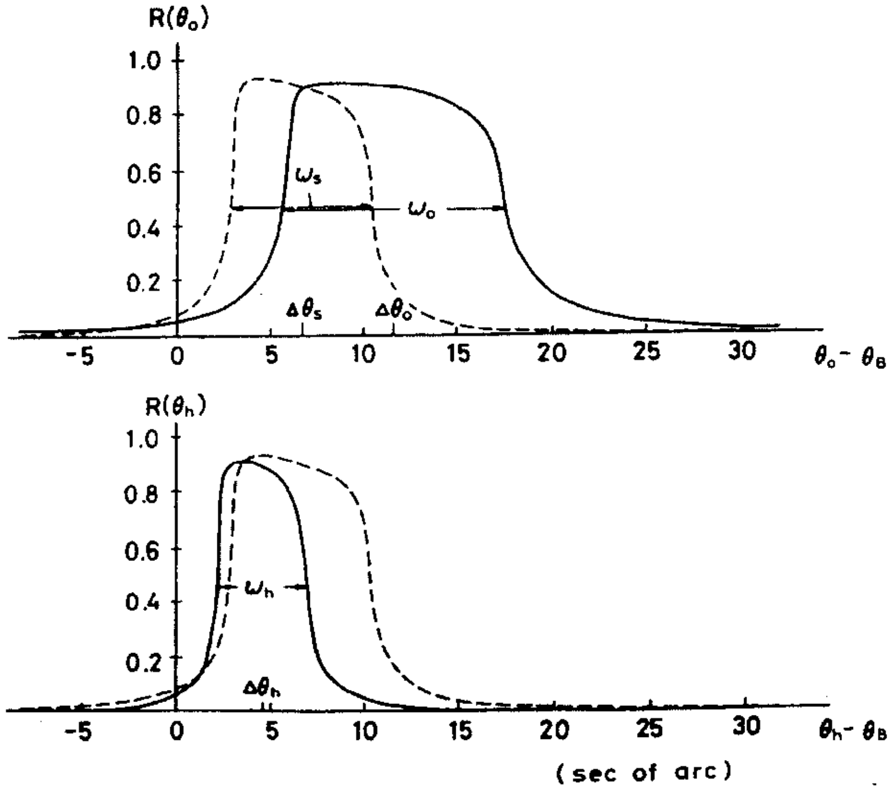


Fig. 4. Perfect-crystal reflection curves for the (111) reflection of silicon at 1.6 Å. $R(\theta_0)$ shows the reflectivity for the ideal plane wave as a function of the incidence angle θ_0, while $R(\theta_h)$ represents the intensity reflected at a reflection angle θ_h for a plane wave incident at θ_0, θ_0 and θ_h being related by $(\theta_h - \theta_B) = b(\theta_0 - \theta_B)$. The solid curves are calculated for an asymmetric case of $b = 0.4$, while the broken curves for the symmetric case ($b = 1.0$) where $R(\theta_0) \equiv R(\theta_h)$.

Table 2

Intrinsic Bragg reflection widths ω_s, energy resolutions $\Delta E/E$ and integral reflecting powers I of perfect crystals of silicon, germanium and α-quartz at 1.54 Å.

Crystal	*hkl*	ω_s (second or arc)	$\Delta E/E$ ($\times 10^5$)	I ($\times 10^6$)
Silicon	111	7.395	14.1	39.9
	220	5.459	6.04	29.7
	311	3.192	2.90	16.5
	400	3.603	2.53	19.3
	331	2.336	1.44	11.8
	422	2.925	1.47	15.5
	333 (511)	1.989	0.88	9.9
	440	2.675	0.96	14.0
	531	1.907	0.60	9.3
Germanium	111	16.338	32.64	85.9
	220	12.449	14.46	67.4
	311	7.230	6.92	37.1
	400	7.951	5.94	42.3
	331	5.076	3.34	25.4
	422	6.178	3.34	32.4
	333 (511)	4.127	2.00	20.2
	440	5.339	2.14	27.5
	531	3.719	1.33	17.7
α-quartz	100	3.798	10.00	18.8
	101	7.453	15.26	40.9
	110	2.512	3.69	12.2
	$10\bar{2}$	2.488	3.36	12.9
	200	2.252	2.81	11.5
	112	2.927	3.03	15.5
	202	2.072	1.93	10.6
	212	2.042	1.47	10.7
	$20\bar{3}$	2.430	1.74	12.9
	301	2.368	1.69	12.6

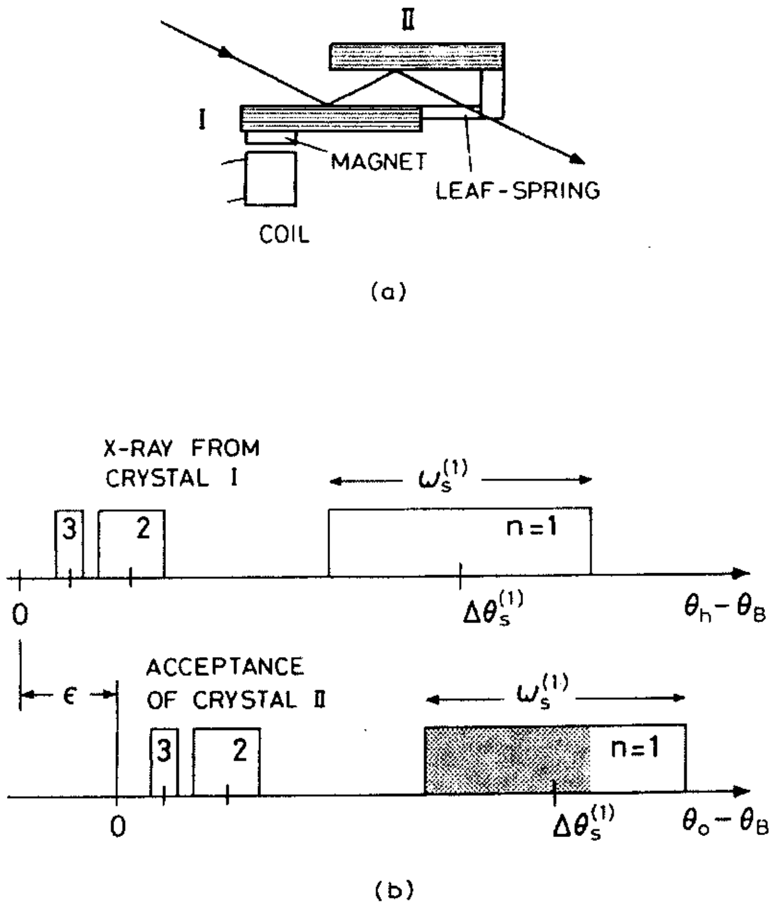


Fig. 33. An off-set harmonics-rejection monochromator. (a) Geometry of the monochromator. (b) The principle of harmonics rejection. Perfect-crystal reflection curves for the fundamental (n = 1) and the harmonics (n = 2, 3) are approximated by rectangular boxes. ε: off-set or misalignment angle. The shaded area represents delivered X-rays (Hart and Rodrigues 1978).

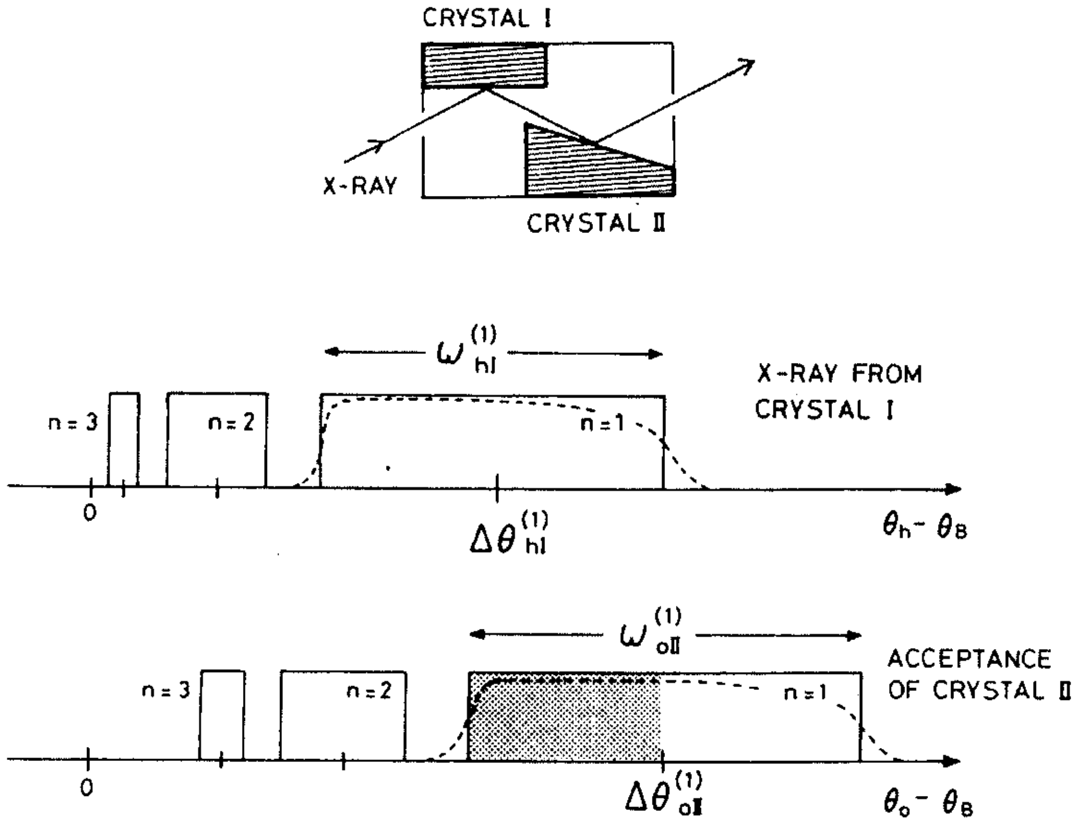
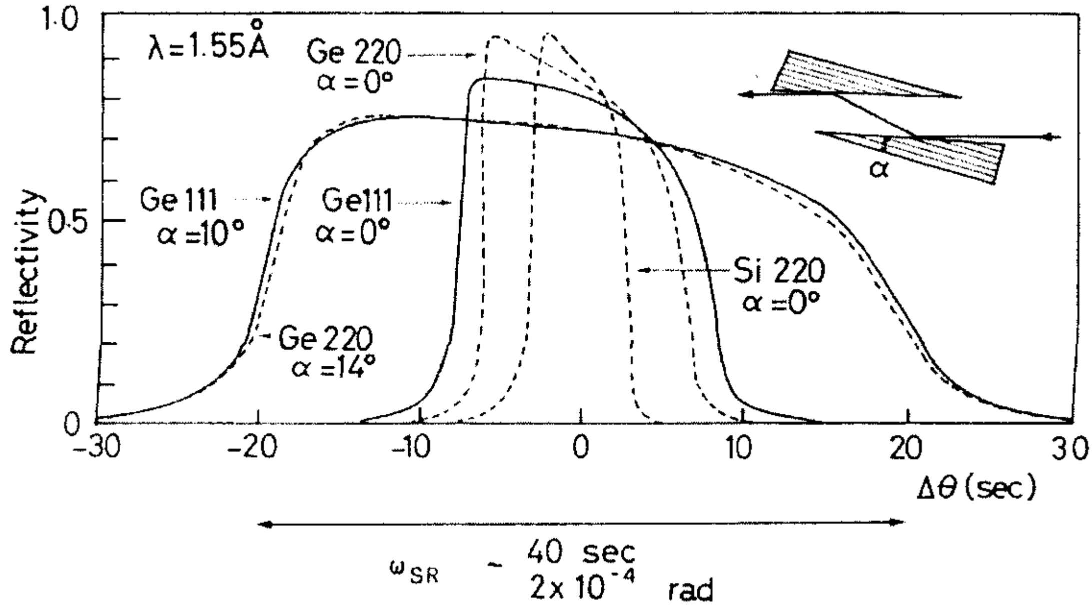


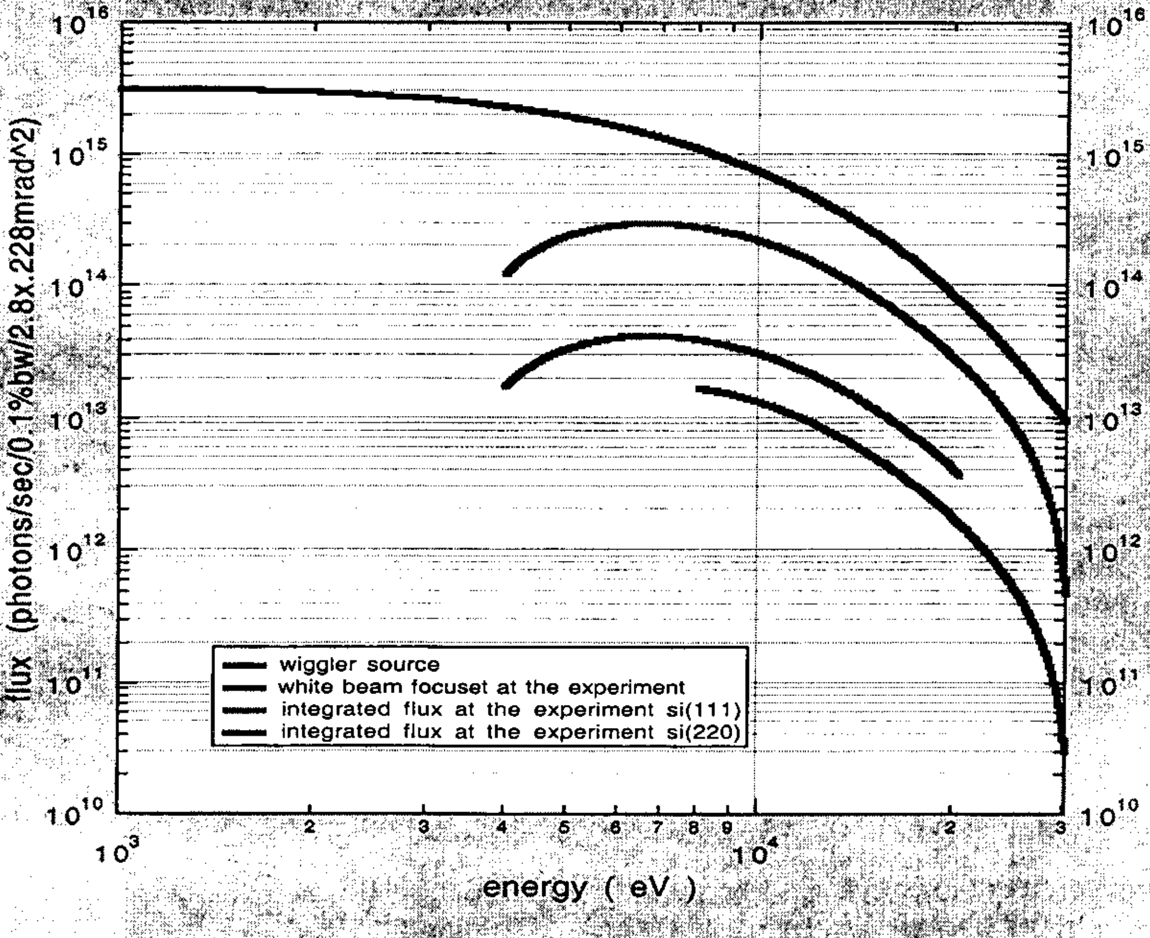
Fig. 34. A monolithic harmonics-rejection monochromator. (a) Crystals I and II of unequal asymmetry factors are built as two outstanding parts of a perfect single crystal. (b) The principle of harmonics rejection. Perfect-crystal reflection curves for the fundamental (n = 1) and the harmonics (n = 2, 3) are approximated by rectangular boxes. The broken curves show the real reflection curves for the fundamental. The shaded area represents delivered X-rays.



Calculated reflectivity curves of grooved monochromators using various asymmetric reflections of silicon and germanium for 1.55 Å X-rays (Kohra et al. 1978).

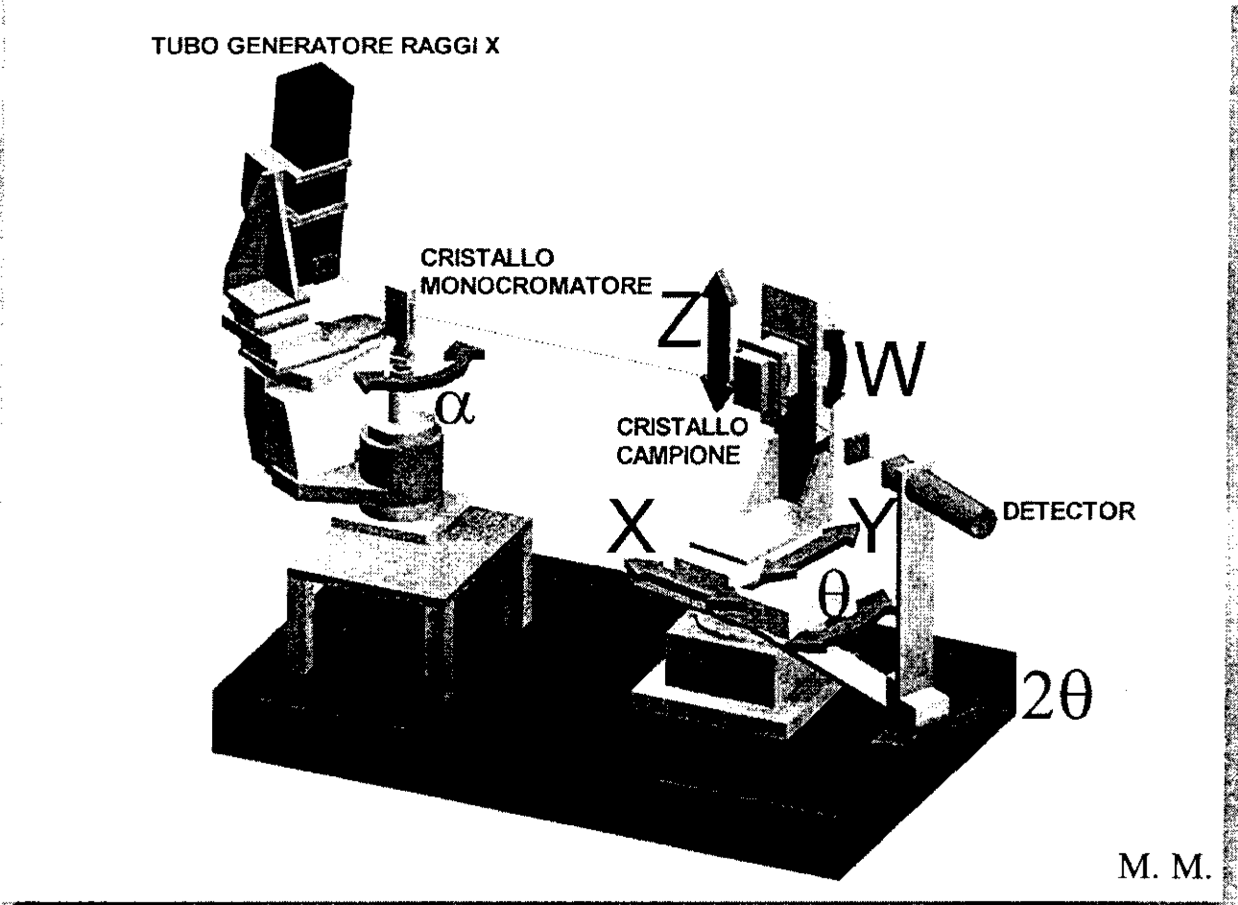
Diffraction 1

57 poles wiggler source at ELETTRA
400mA, 1.6T and 2GeV
total power: 8 kW

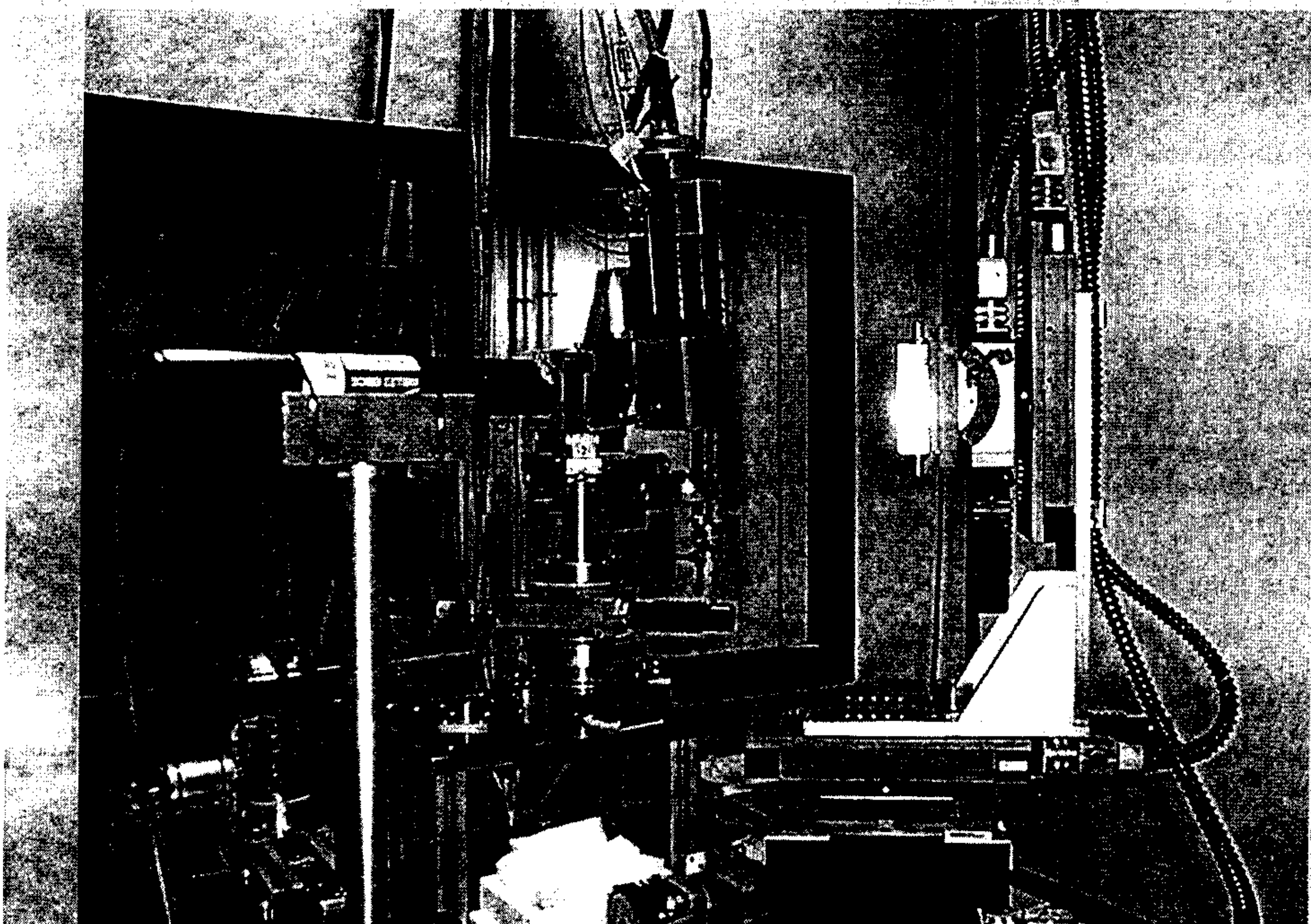


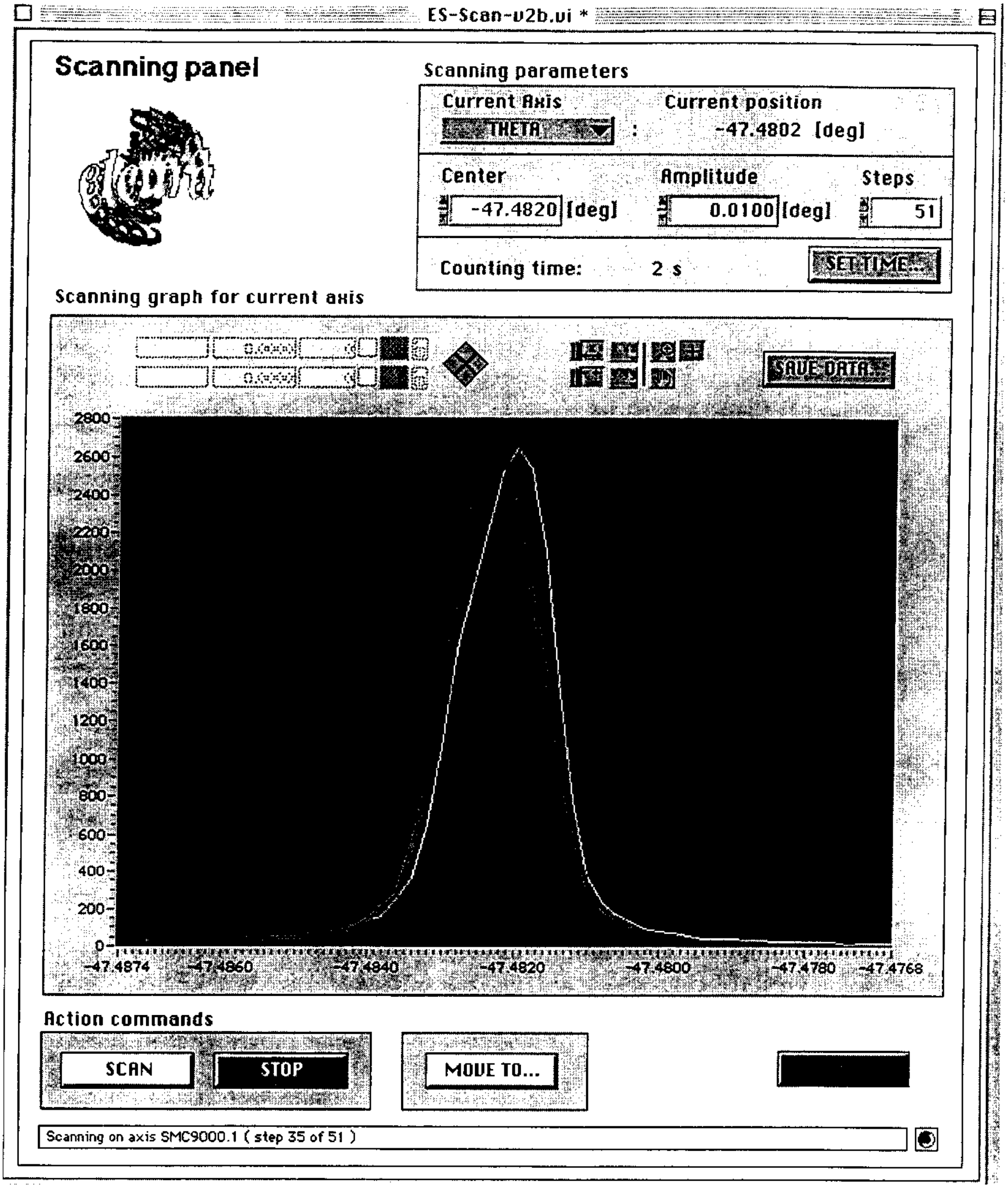
elettra

3D picture of the double ϑ-2ϑ equipment

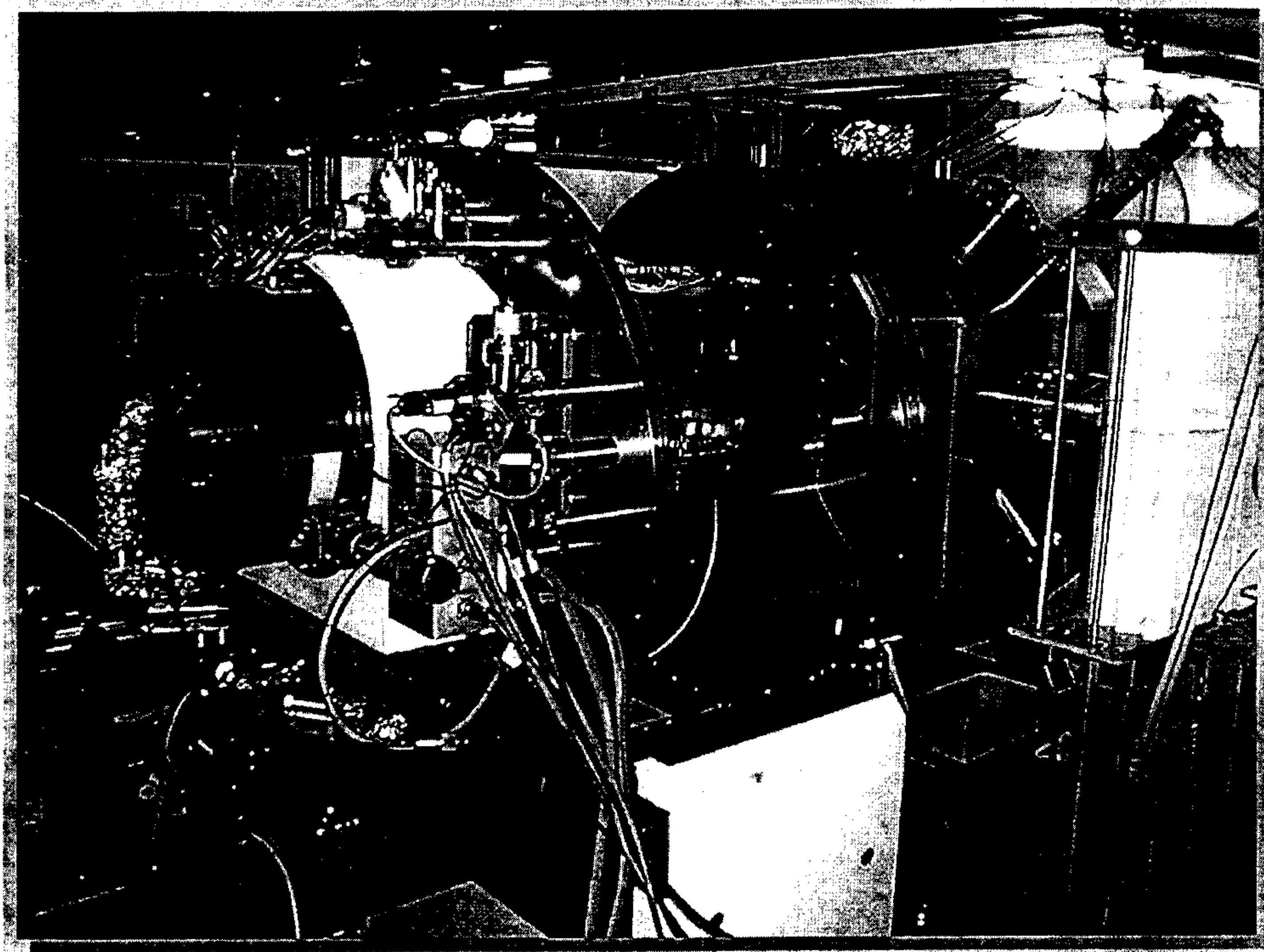


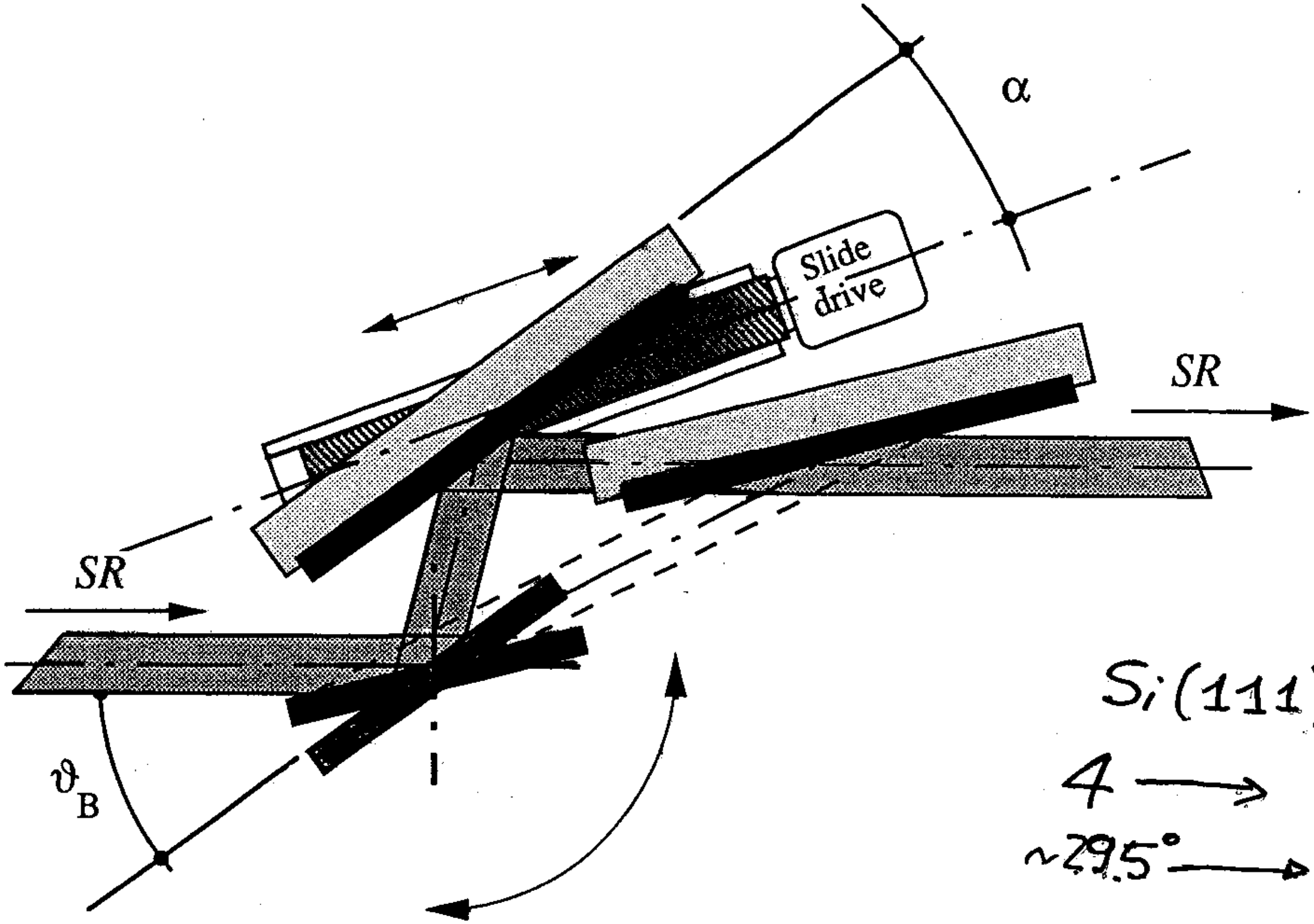
The double ϑ-2ϑ test station at the Hard X-ray Laboratory




ES-Scan-v2b.ui *
Scanning panel
Scanning parameters
Current Axis
THETA
Current position
-47.4802 [deg]
Center
-47.4820 [deg]
Amplitude
0.0100 [deg]
Steps
51
Counting time:
2 s
SET TIME...
Scanning graph for current axis
SAVE DATA...
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-47.4874
-47.4860
-47.4840
-47.4820
-47.4800
-47.4780
-47.4768
Action commands
SCAN
STOP
MOVE TO...
Scanning on axis SMC9000.1 (step 35 of 51)

The double crystal monochromator at the **diffraction1** beamline



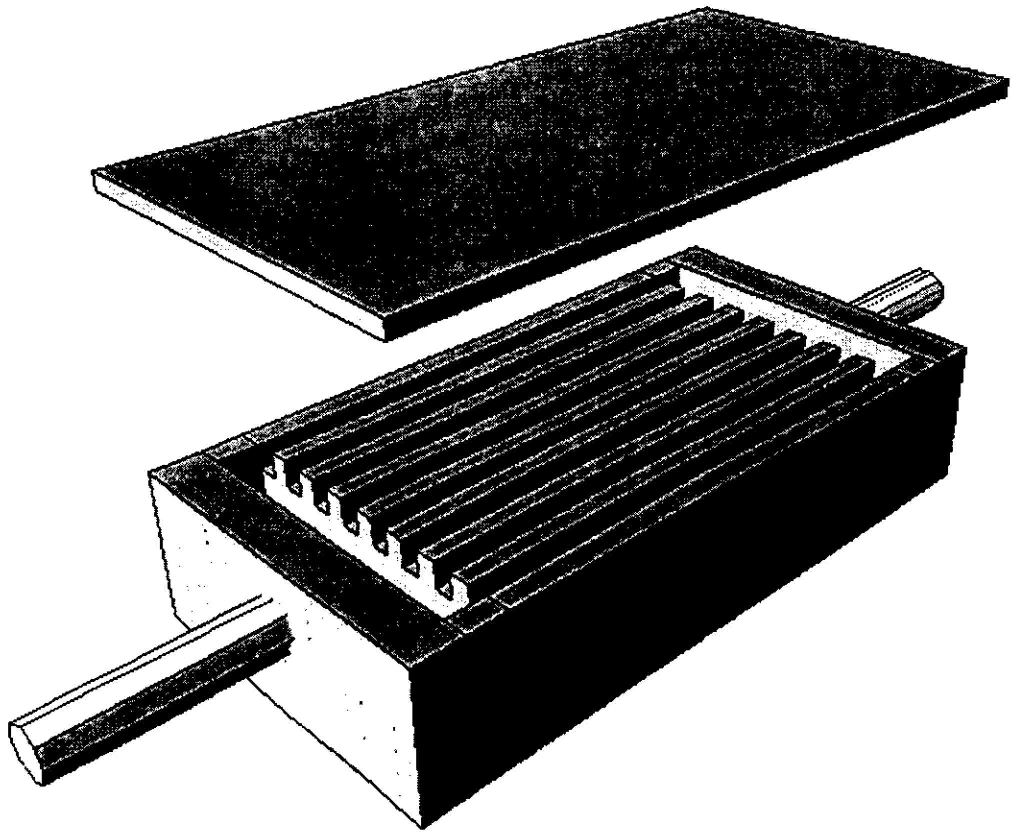


Si(111)

4 → 20.5 keV

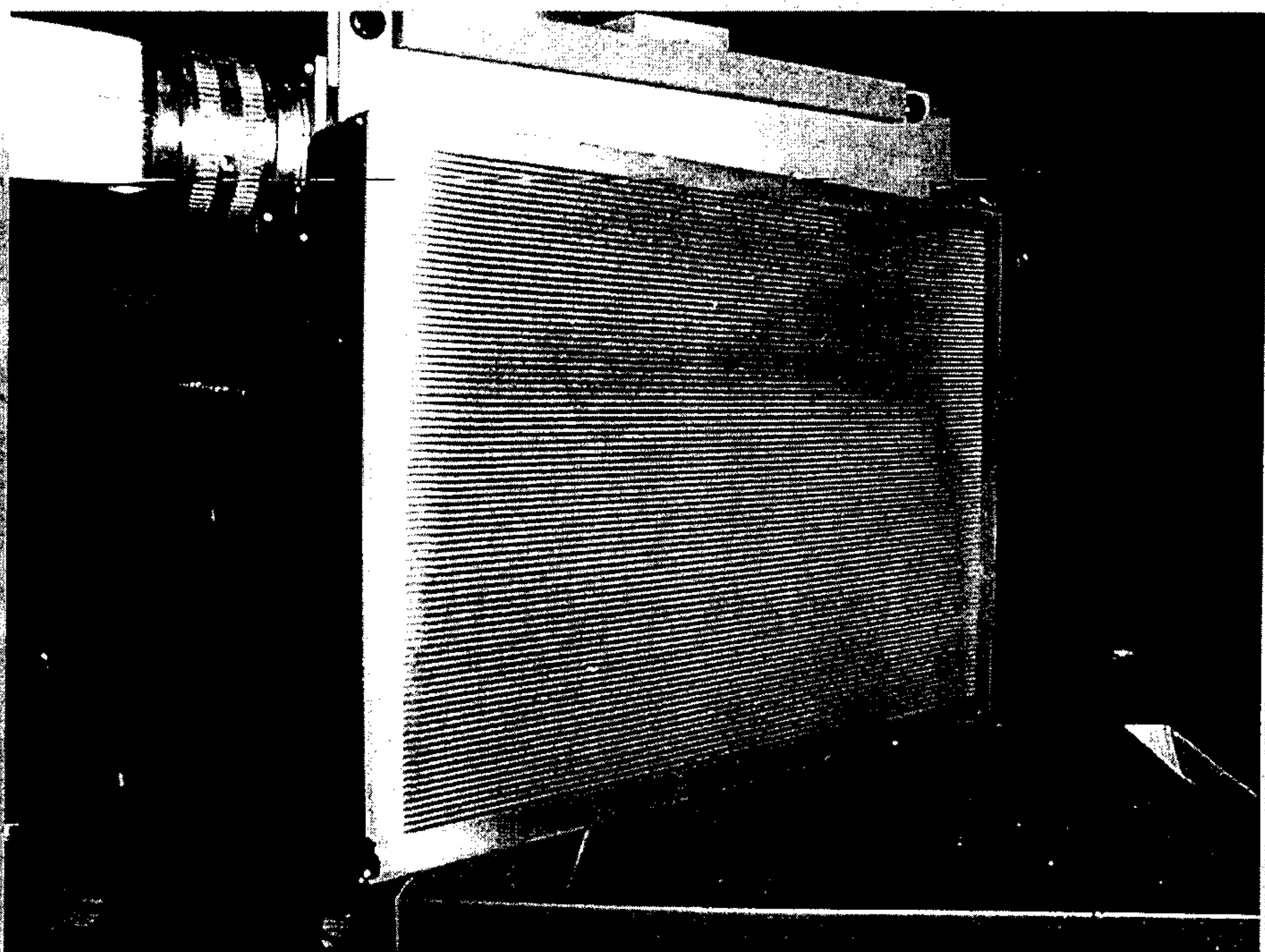
~29.5° → 5.5° θ_B

Conceptual design of an internally water cooled crystal



Sincrotrone Trieste
Hard X-Ray

The two Si components before the Si-Si brazing

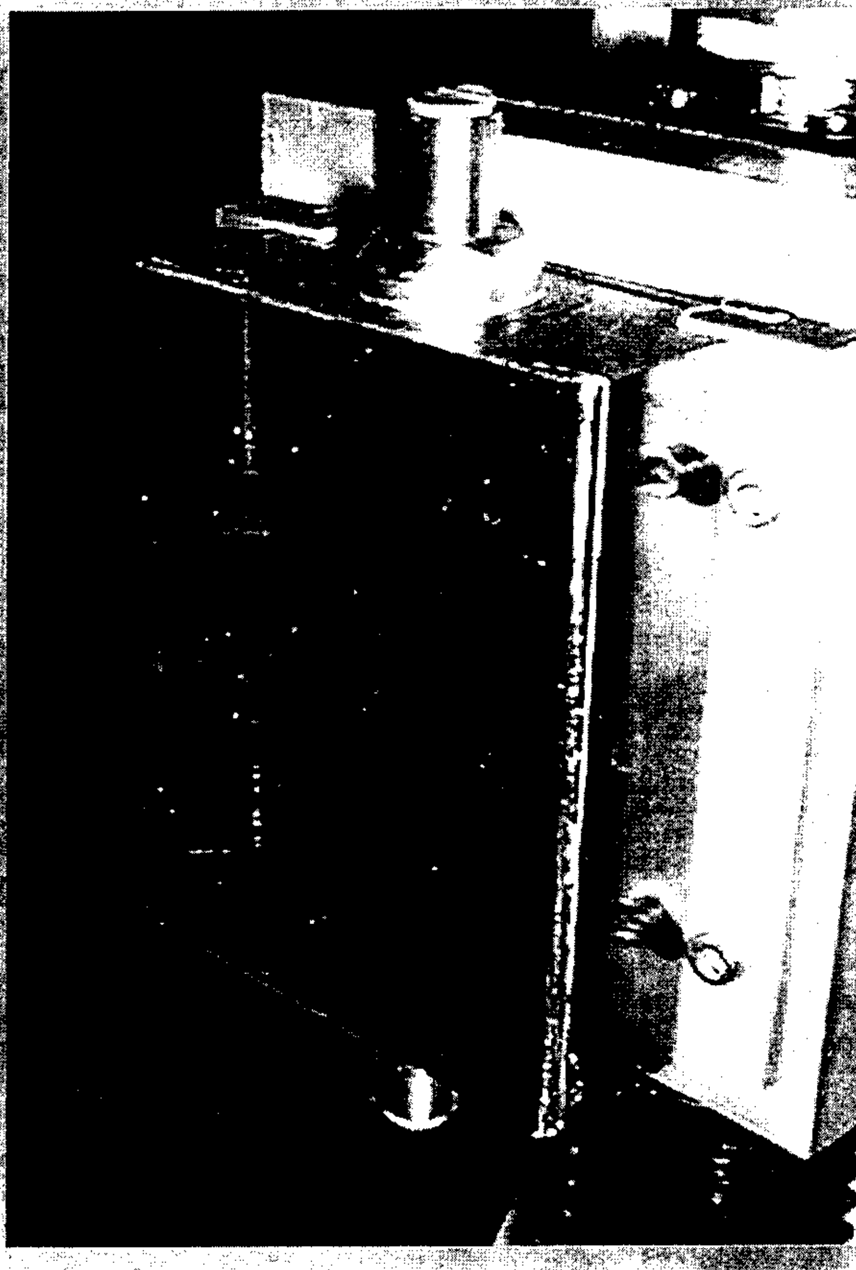


channels: thickness: 300μm
depth: 2mm

Diffraction 1:
first optical element in the beam
Si(111) internally water cooled

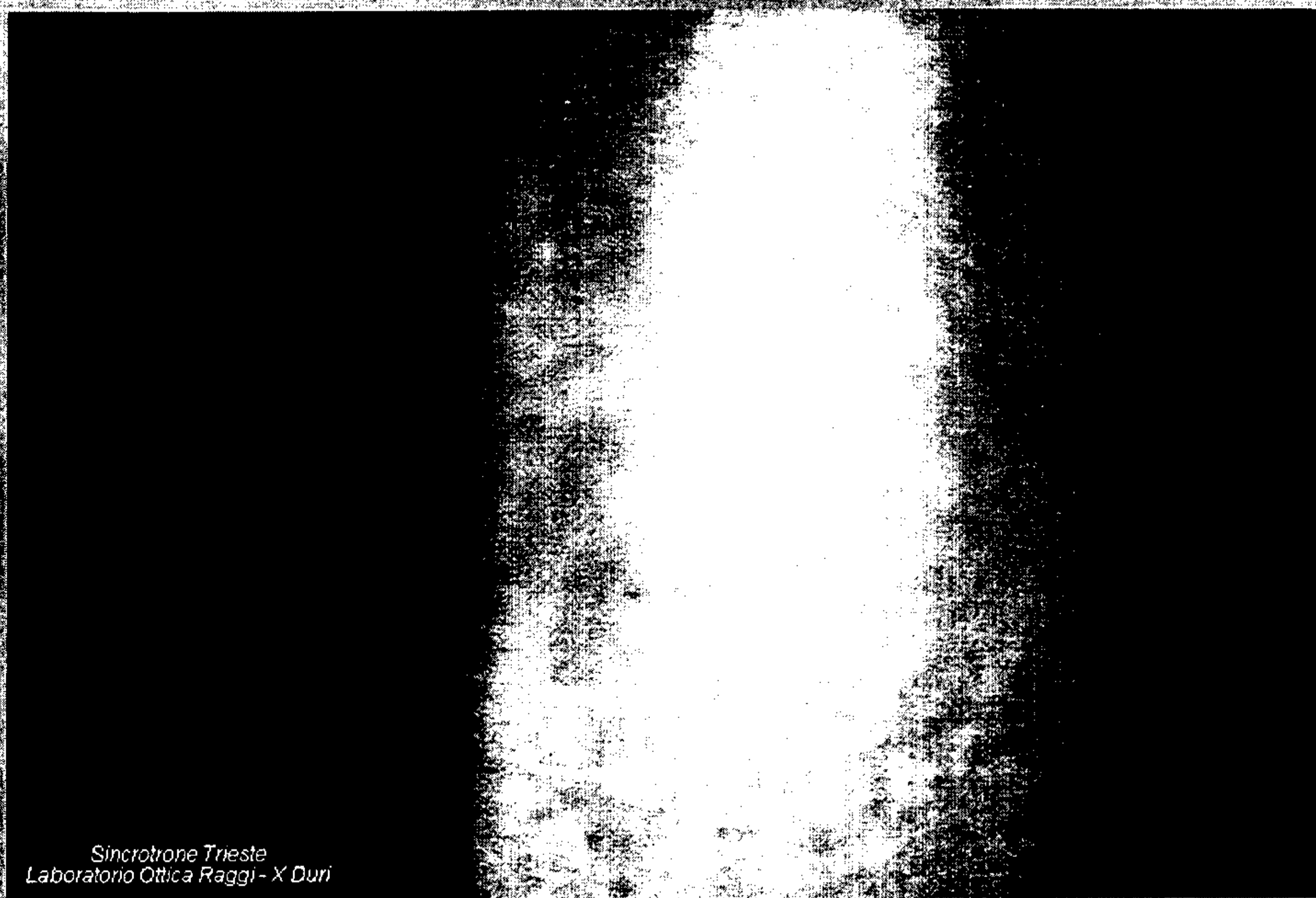
total power absorbed 0.5 kW*

* $1.5 \times 0.28\ mrad^2$

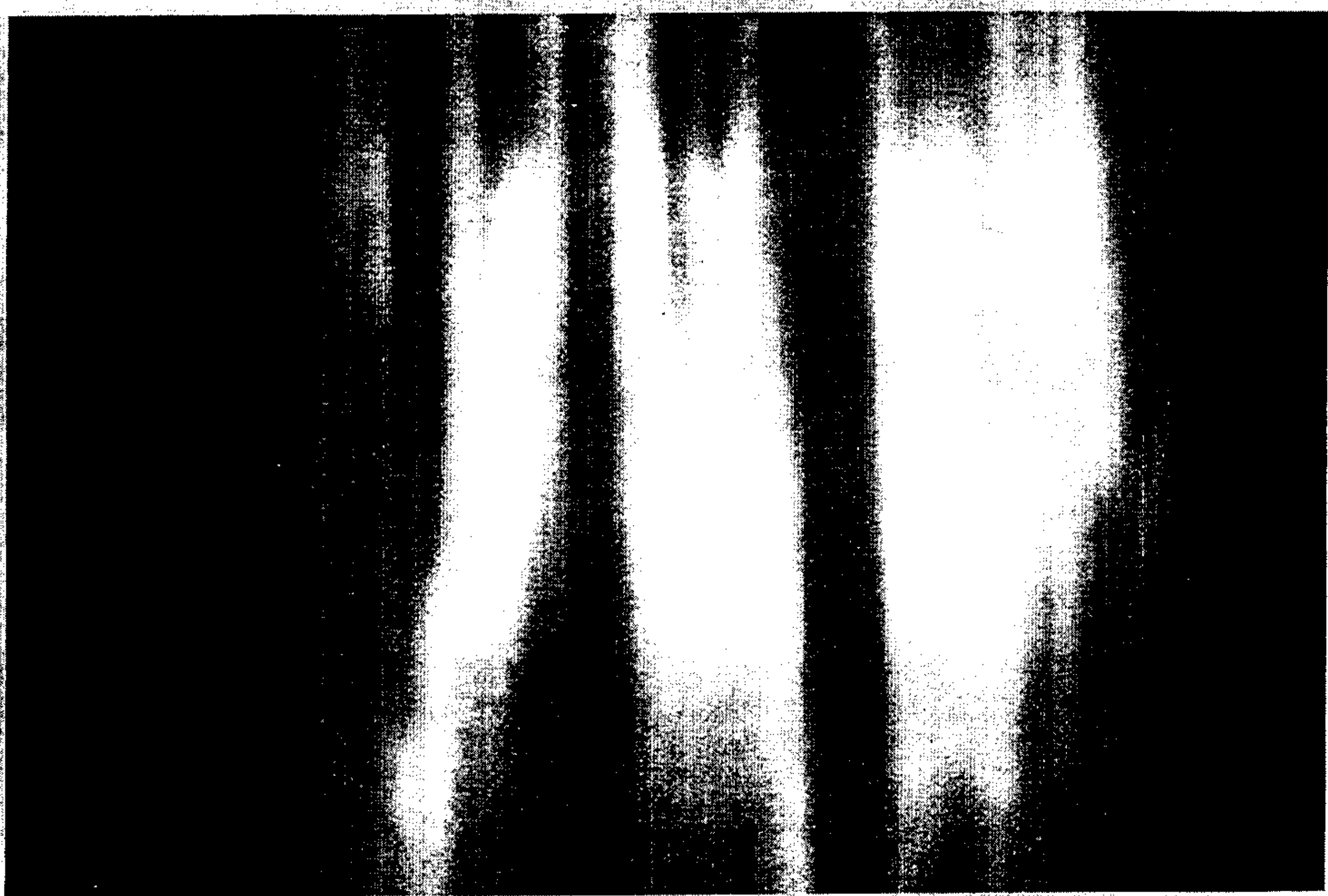


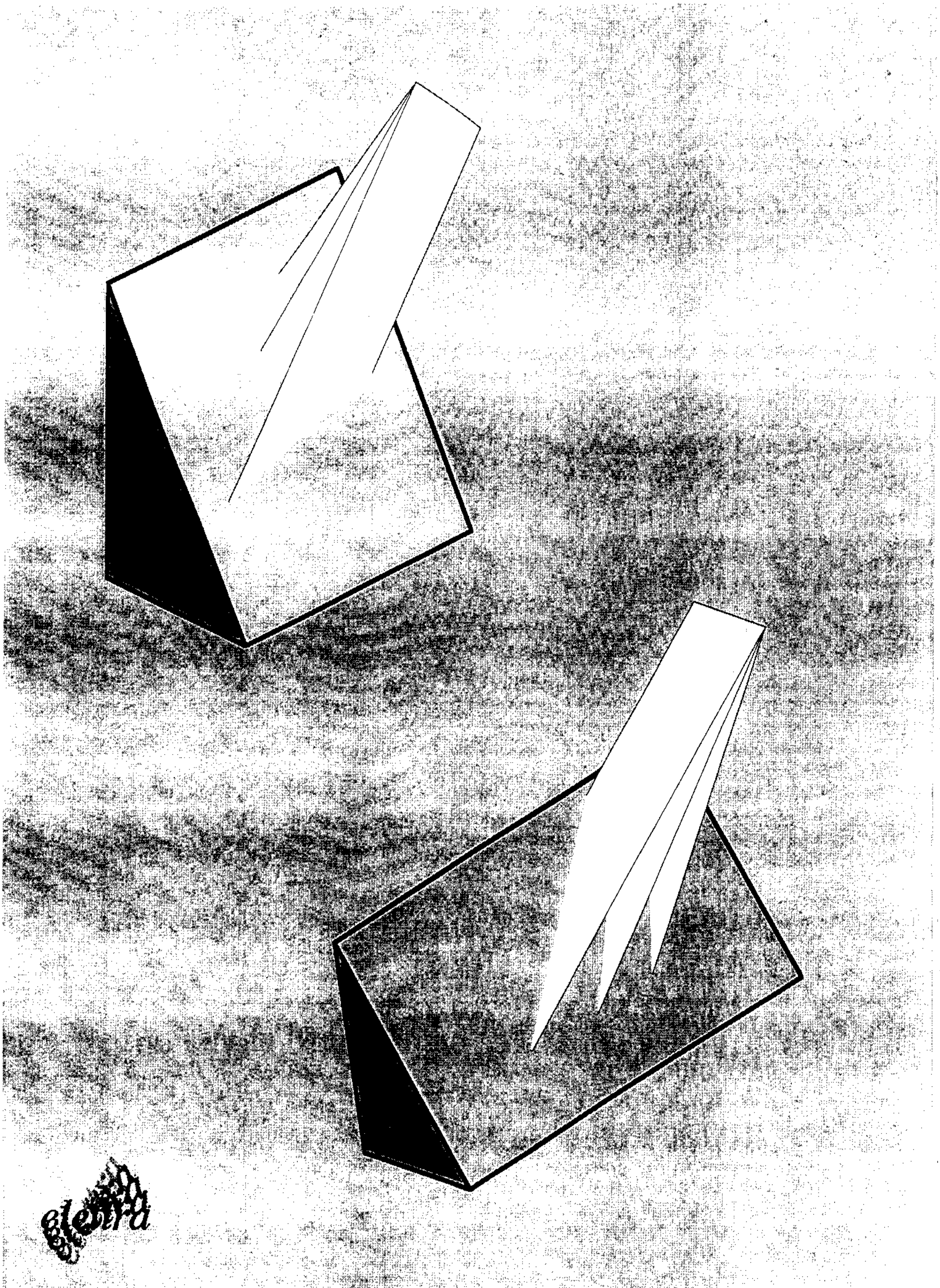
Topography of the internal cooled Si-crystal with channels perpendicular to the scattering plane

Same topography but with channels in the same direction of the scattering plane



surface of a Si(111) crystal with a evident stressed structure induced by a back-side machining and not removed by chemical hatching

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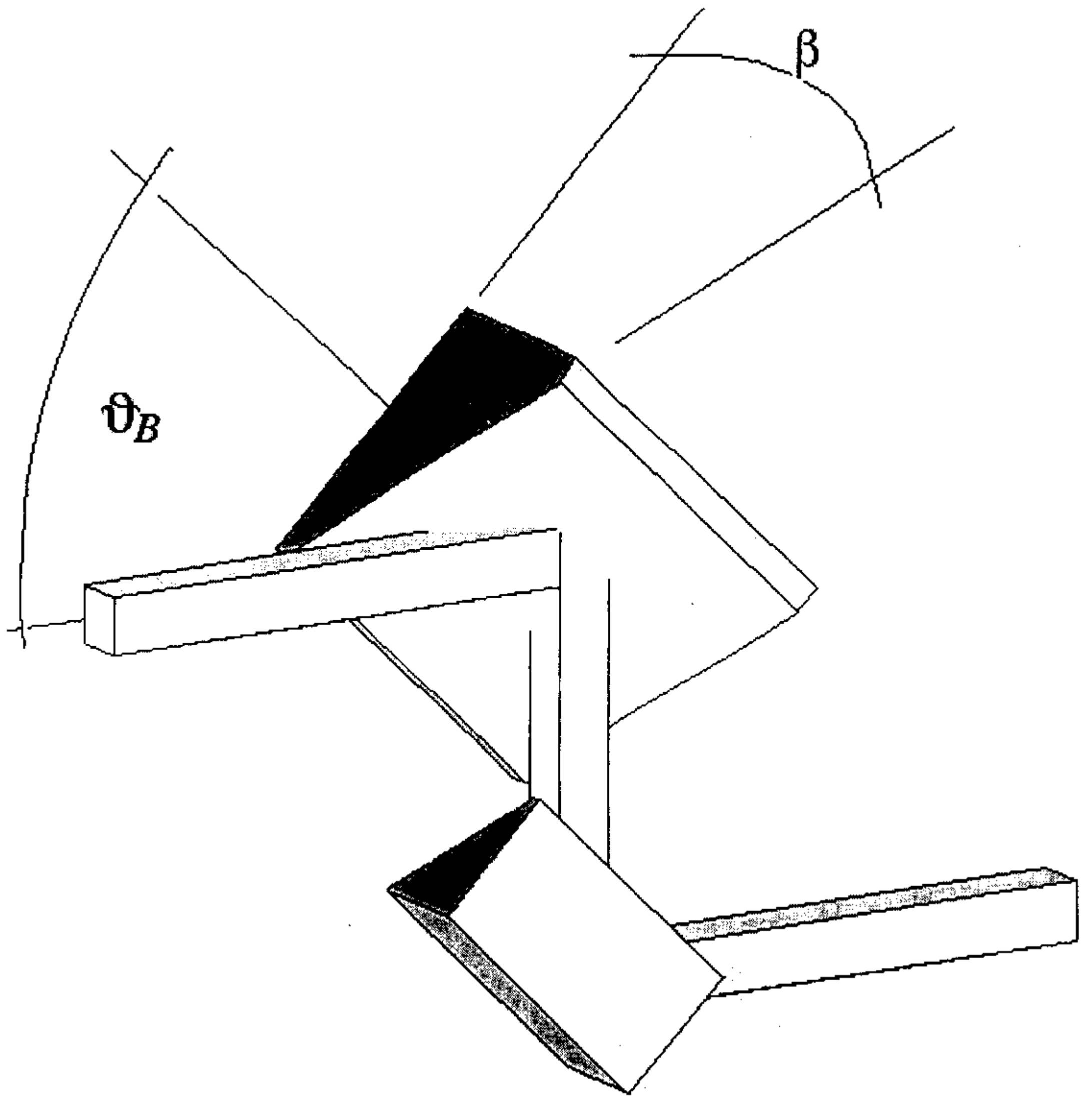
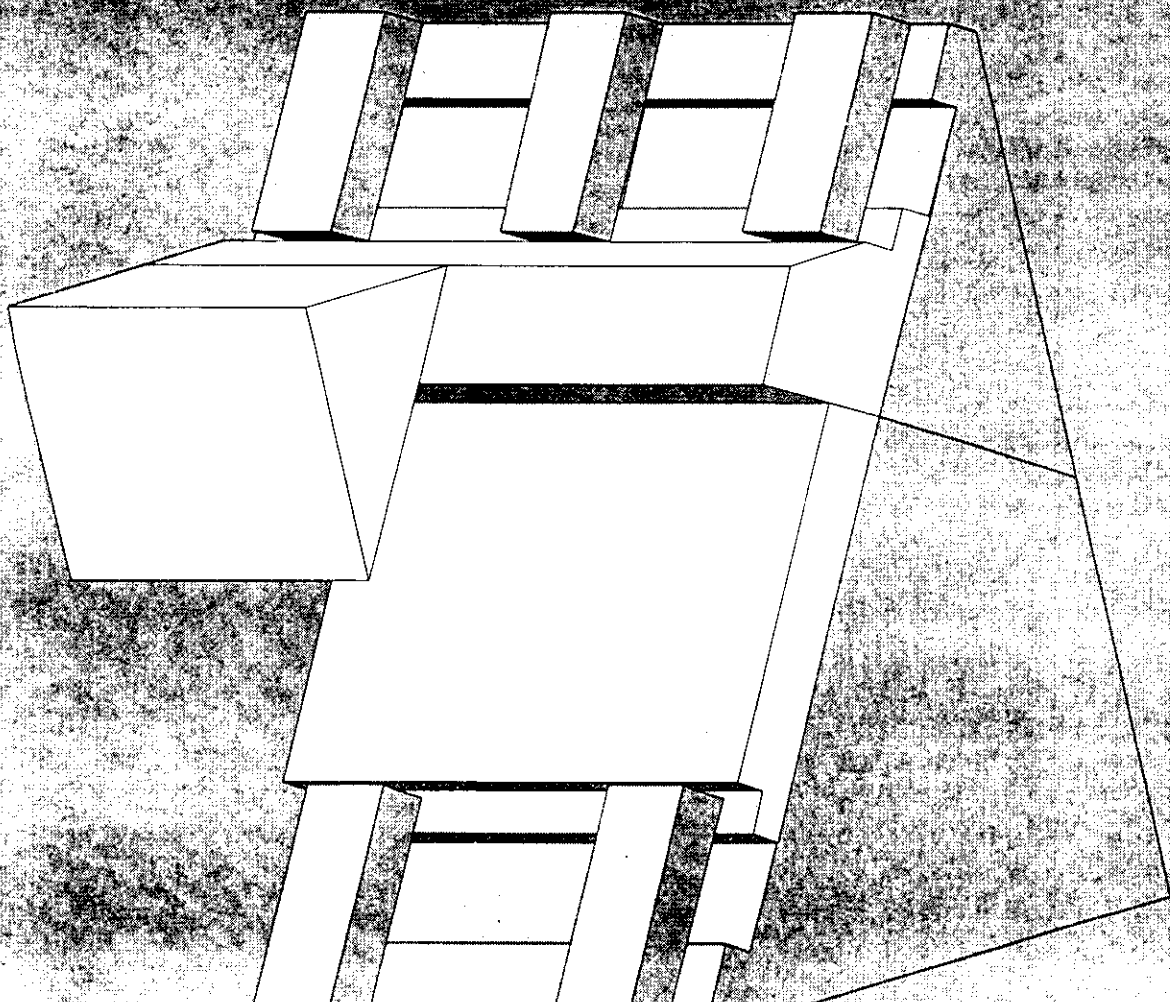
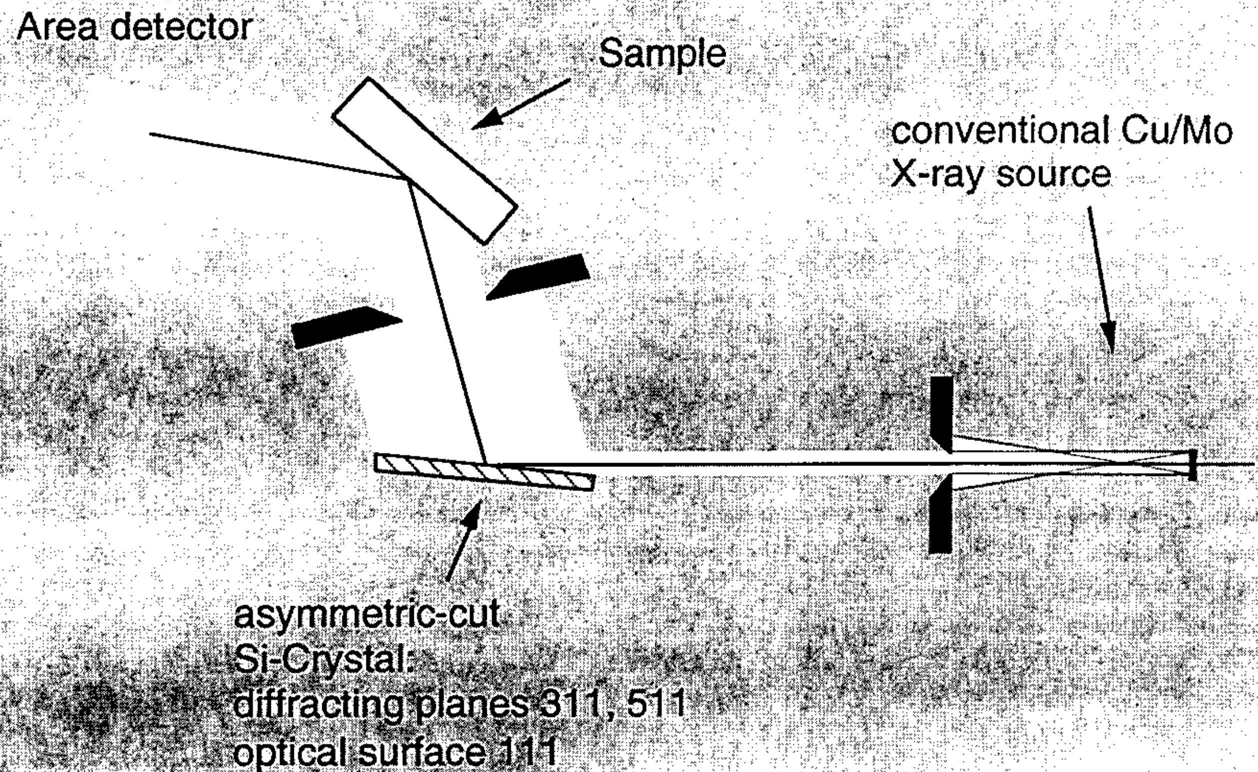


Sincrotrone Trieste S.C.p.A
Hard-X Ray Optics
Laboratory



Sincrotrone Trieste S.c.p.A.

improvements



The equipment consists on two vertical axis diffractometer. The beam will be enlarged horizontally by the use of asymmetric-cut crystals

second crystal movement tests at the micromechanics laboratory

M.Miculin