X-ray Diffraction from Materials

2008 Spring Semester

Lecturer; Yang Mo Koo

Monday and Wednesday 14:45~16:00

8. Experimental X-ray Diffraction Procedures

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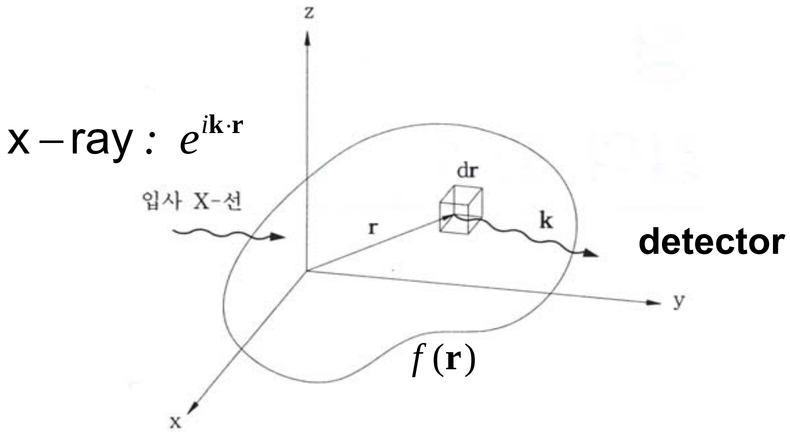


$$F(\mathbf{k}) = \frac{1}{(2\pi)^{3/2}} \int_{-\infty}^{+\infty} f(\mathbf{r}) e^{i\mathbf{k}\cdot\mathbf{r}} d\mathbf{r}$$

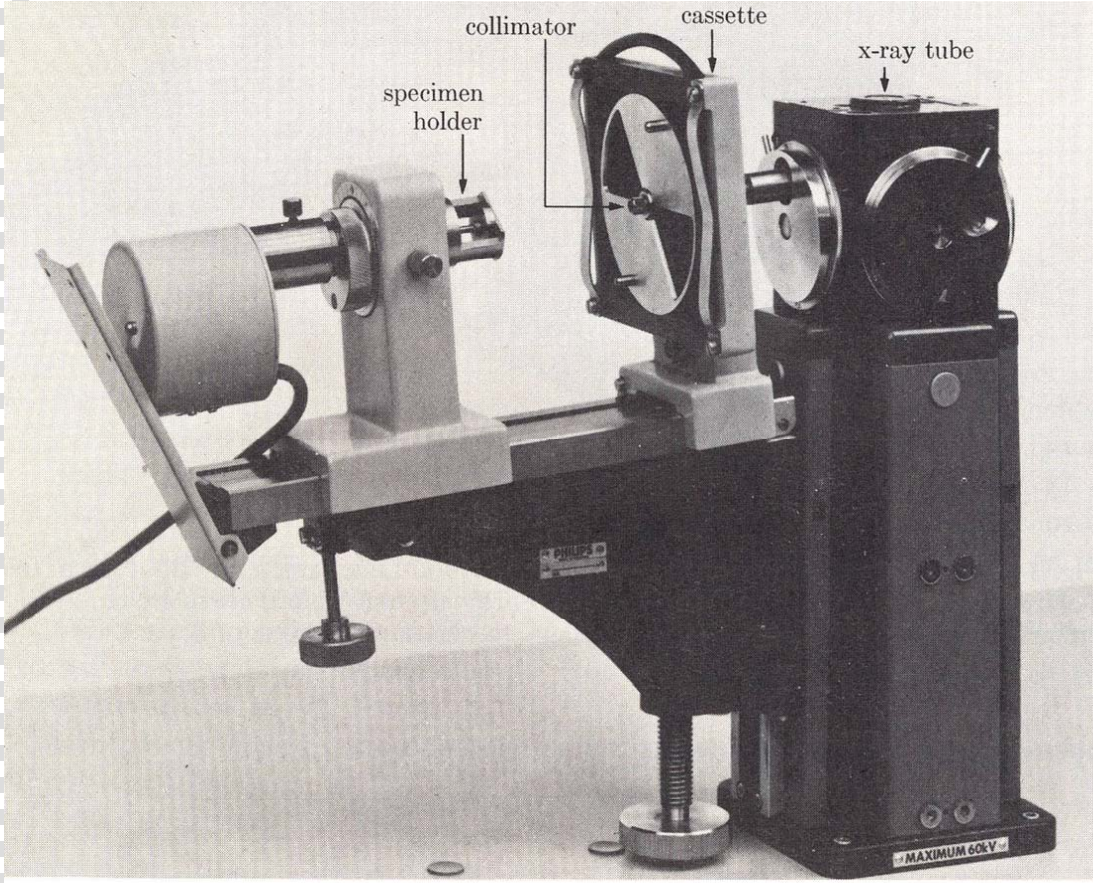
(i) X-ray sources
- vacuum sealed tube (chapter 2)
- synchrotron radiation (chapter 2)
- x-ray optical elements (chapter 3)

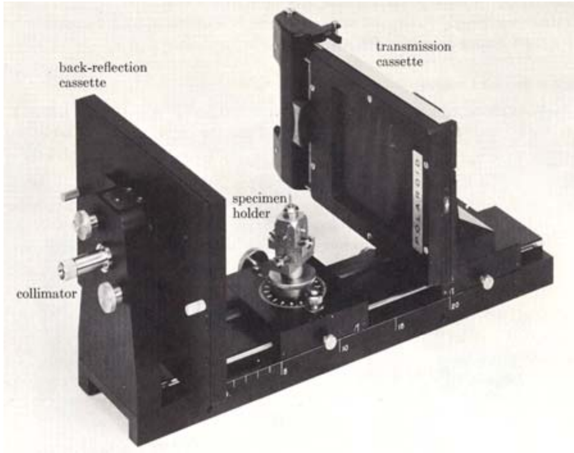
(ii) Crystals
- crystal structure and its symmetry (chapter 6)
- interaction between crystal and x-ray (chapter 7)
- crystal moving stage (chapter 8)

(iii) Detectors (chapter 4)
- film
- electronics

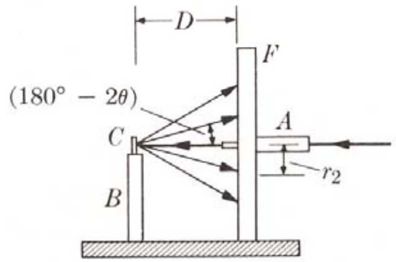
8.1 Diffraction Experiments using Films

8.1.1 Laue camera





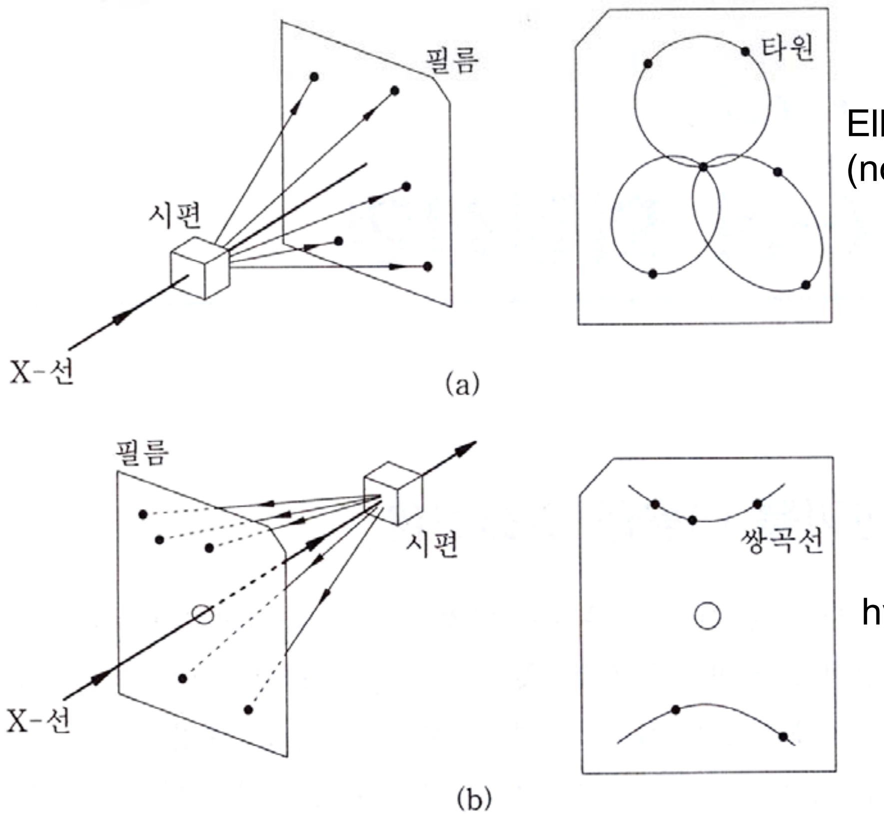
Polaroid cassette



Back-reflection camera

8.1 Diffraction Experiments using Films

Schematic diagrams of the diffraction pattern



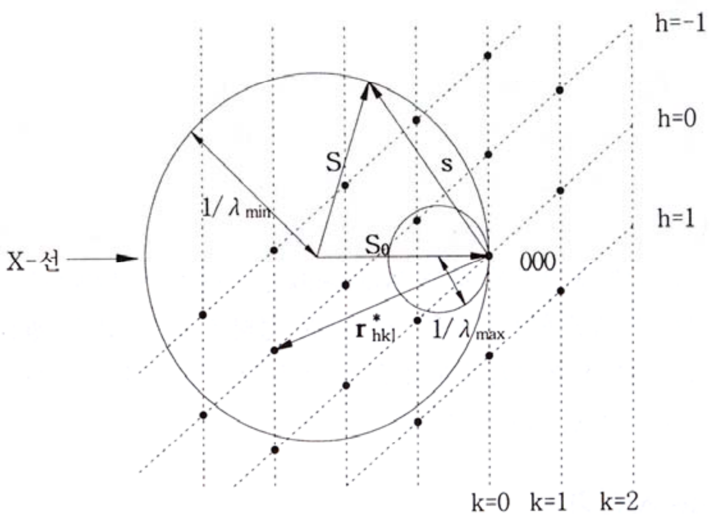
Elliptic curve shape
(not elliptic curve)

hyperbola

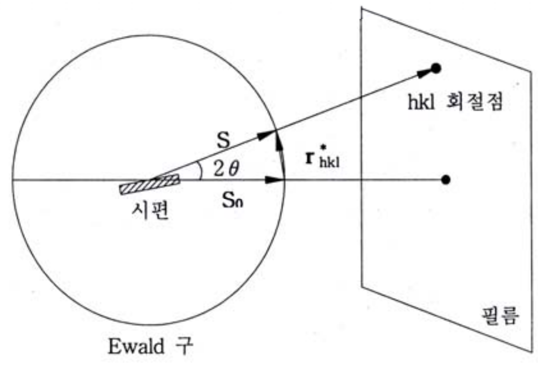
【그림 8-1】 (a) 투과 Laue 사진기, (b) 반사 Laue 사진기의 개략도와 필름에 나타나는 회절 무늬

8.1 Diffraction Experiments using Films

Diffraction condition for Laue geometry



【그림 8-2】 Laue 사진기에서의 회절 조건

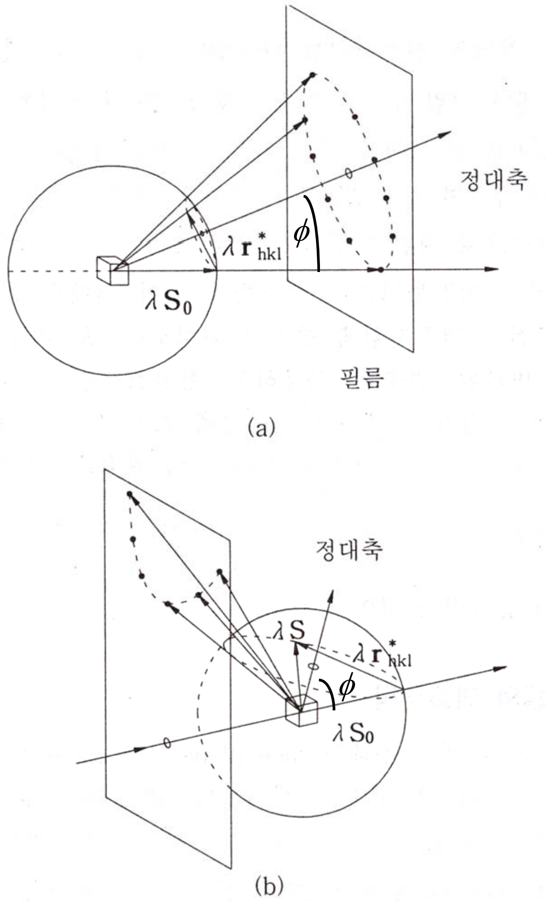


【그림 8-3】 Laue 사진기에서 회절 조건을 만족하는 역격자점 hkl이 필름 위에 투영되는 모양

If the reciprocal point hkl's locate inside of large Ewald sphere and outside of small Ewald sphere, diffraction spot of (hkl) hits the film.

8.1 Diffraction Experiments using Films

Intersection of a conical array of diffracted peaks makes a elliptic curve shape for transmission case and makes hyperbola for back reflection case.



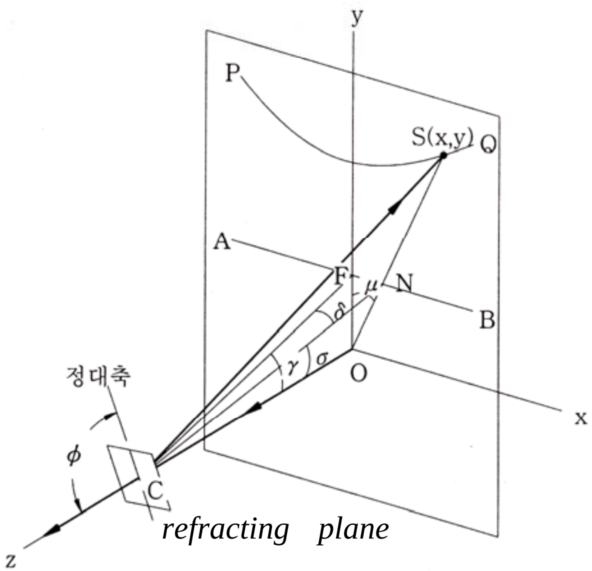
정대면들에 의한 회절 무늬: (a) 투과 Laue 사진기 (b) 반사 Laue 사진기

All of the planes of one zone reflected beams which lie on the surface of a cone whose axis is the zone axis and whose semi-apex angle is equal to the angle Φ at which the zone is inclined to the transmitted beam.

$0<\Phi<\pi/4$: transmission elliptic curve shape

$\pi/4<\Phi<\pi/2$: transmission hyperbola

8.1 Diffraction Experiments using Films



【그림 8-6】 반사 Laue 사진기에서 회절점의 위치

Reflecting plane: belong to a zone whose axis lies in yz-plane
CN: normal to the reflecting plane
PQ: trace of diffraction peaks
AN: trace of plane normal long to a zone.

Incident beam, plane normal, and diffracted beam are coplanar. Therefore, the direction of reflecting plane can be calculated if the position of N is estimated by diffraction spot S.

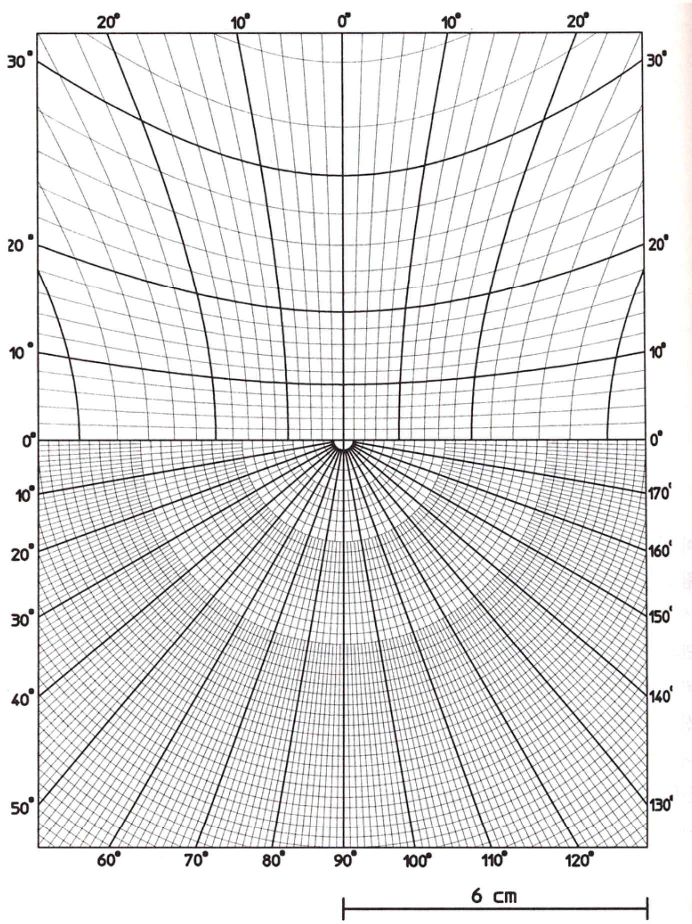
Crystal orientation can be obtained from the γ and δ of the diffraction spots.

$$\tan\mu = \frac{FN}{FO} = \frac{CF\tan\delta}{CF\sin\gamma} = \frac{\tan\delta}{\sin\gamma}$$

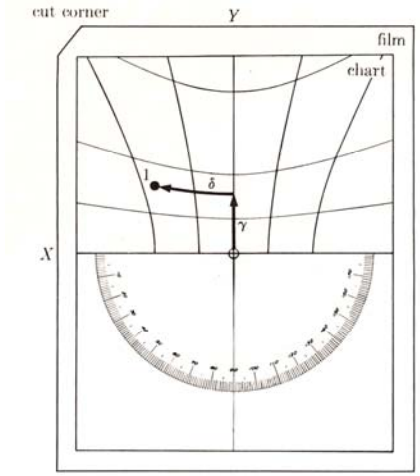
$$\tan\sigma = \frac{ON}{OC} = \left(\frac{FN}{\sin\mu}\right)\left(\frac{1}{CF\cos\gamma}\right) = \frac{\tan\delta}{\sin\mu\cos\gamma}$$

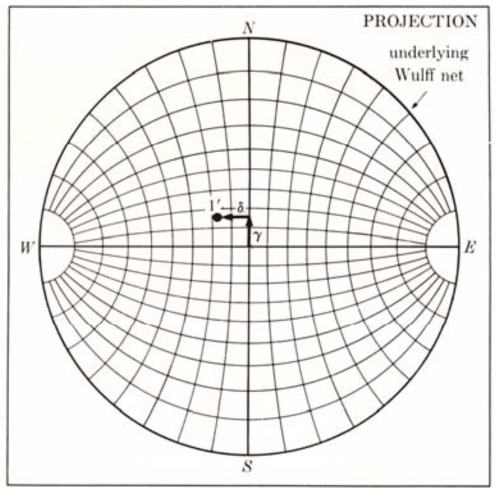
8.1 Diffraction Experiments using Films

Greninger chart: direct read of γ and δ from diffraction peaks.



【그림 8-7】 반사 Laue 사진기를 위한 Greninger 망 (시편과 필름의 거리 3cm)

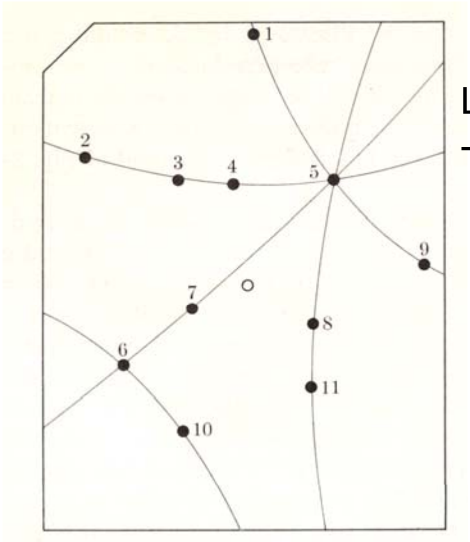




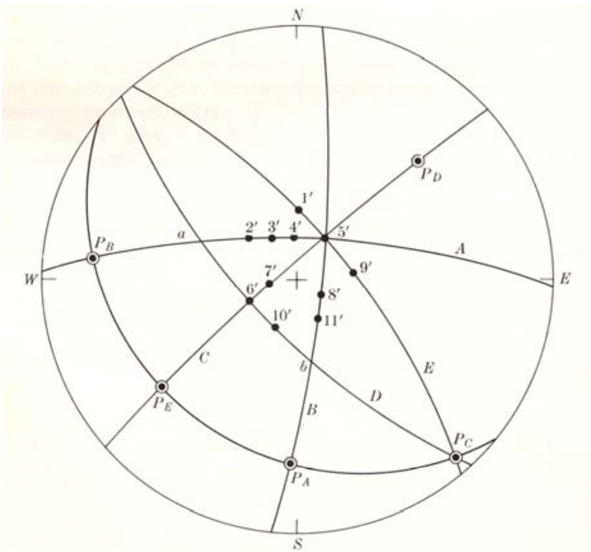
Leonhardt chart: transmission Laue experiment

8.1 Diffraction Experiments using Films

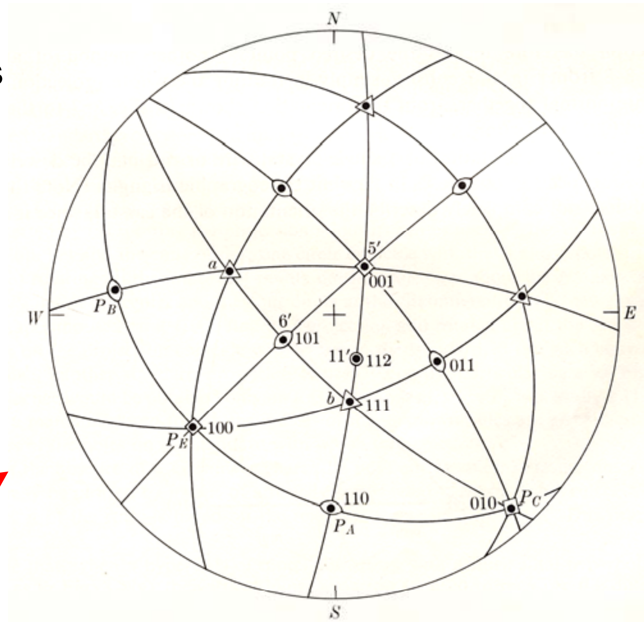
Crystal orientation determination



Laue back reflection;
-measure γ and δ of spots



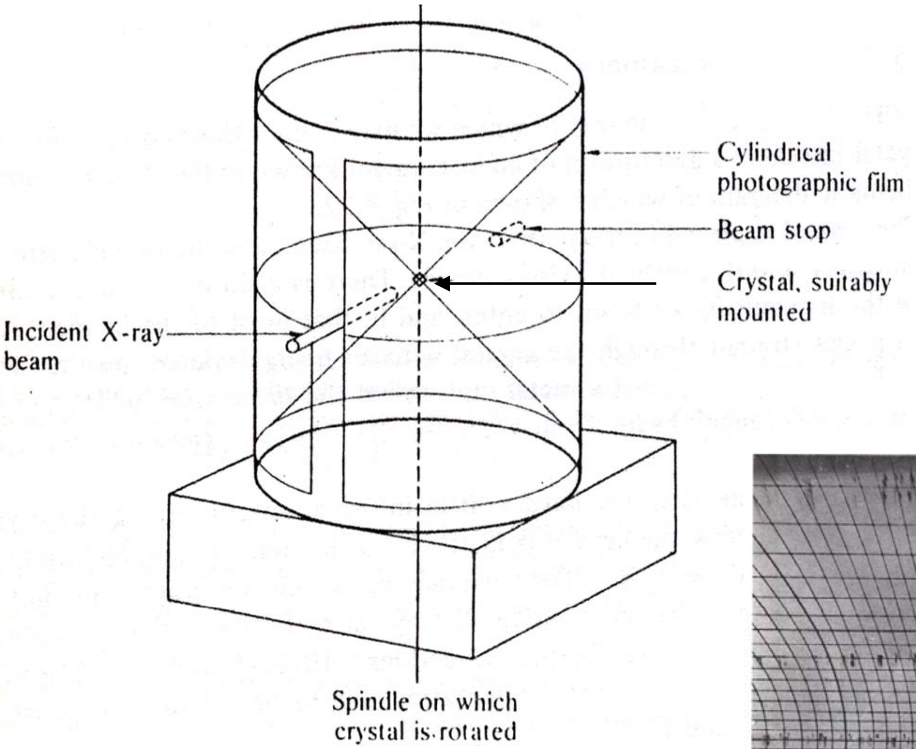
Plot of reflecting plane normal at Wulff net using γ and δ.

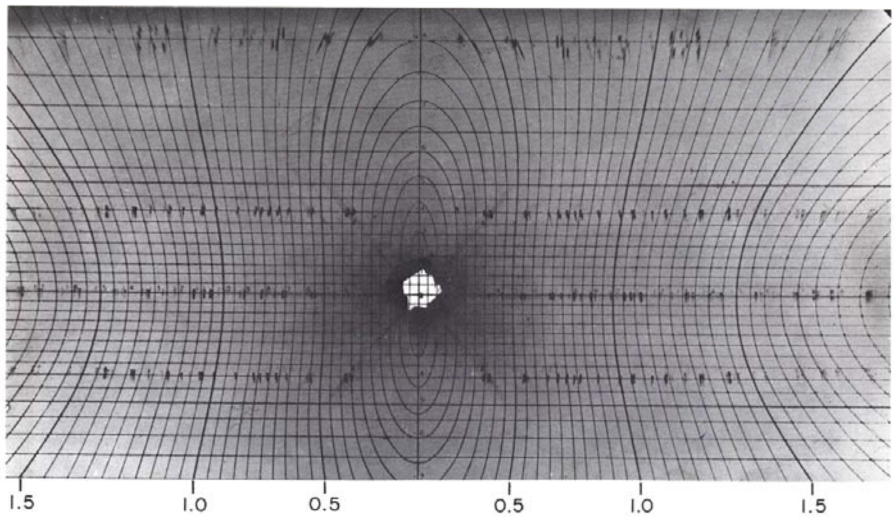


Crystal orientation

8.1 Diffraction Experiments using Films

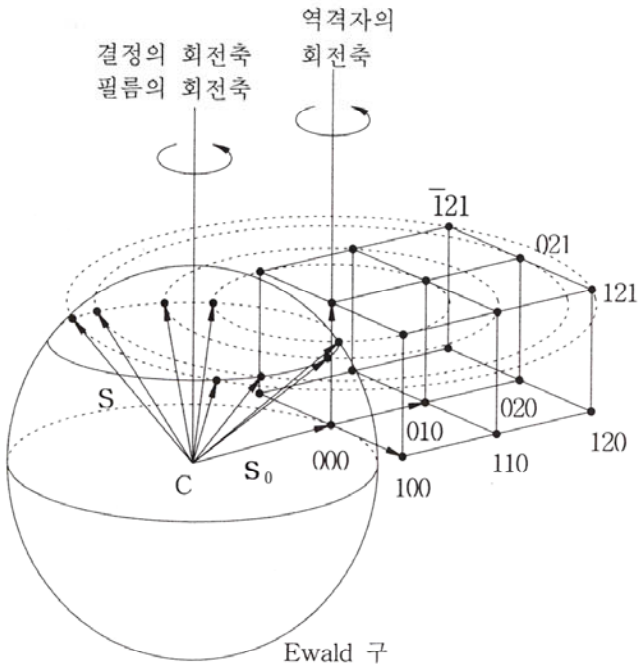
Rotating Crystal Camera





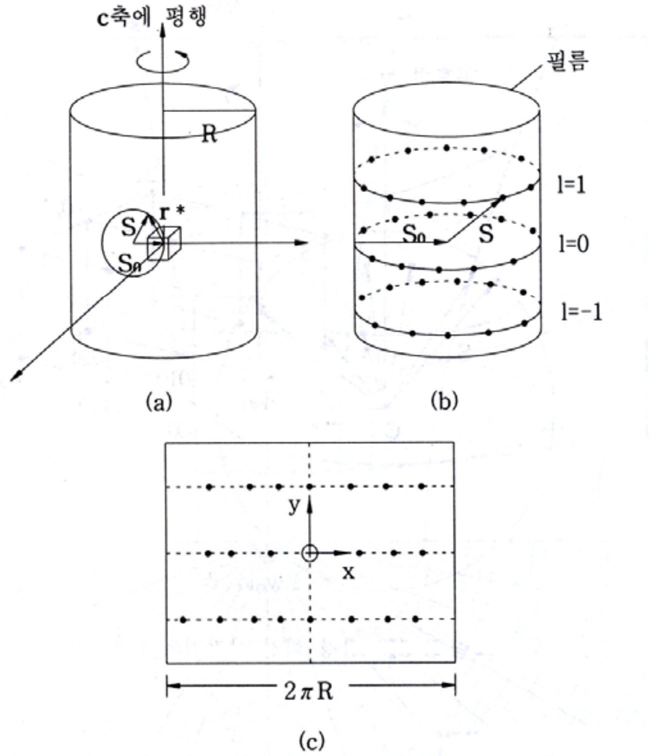
8.1 Diffraction Experiments using Films

Diffraction condition



【그림 8-8】 결정 회전 사진기의 회절 조건

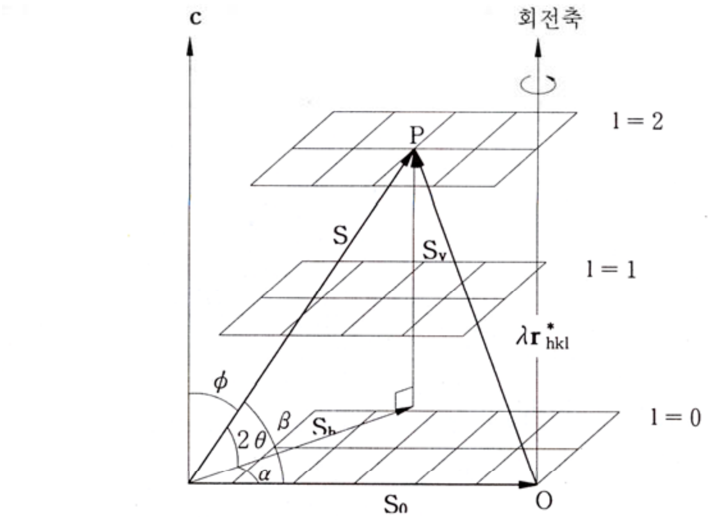
Diffraction pattern



【그림 8-9】 (a) 결정 회전 사진기의 장치도 (b) 필름 상에 나타나는 회절 무늬

8.1 Diffraction Experiments using Films

Indexing of diffraction peaks



【그림 8-10】 결정 회전 사진기의 필름 좌표와 격자 상수의 관계

$$(\mathbf{S}-\mathbf{S}_0)\cdot\mathbf{c}=\mathbf{r}^*_{hkl}\cdot\mathbf{c}=l$$

since $|\mathbf{S}|=1/\lambda$ and $\mathbf{S}_0\cdot\mathbf{c}=0$

$$\mathrm{S}\cdot\mathrm{c}=\mathrm{l}\ \rightarrow\ \frac{\mathrm{c}}{\lambda}\sin\beta=l$$

$\therefore$ the lattice parameter c becomes

$$\mathrm{c}=\frac{\lambda l}{\sin\beta}=\frac{\lambda l}{\cos\phi}$$

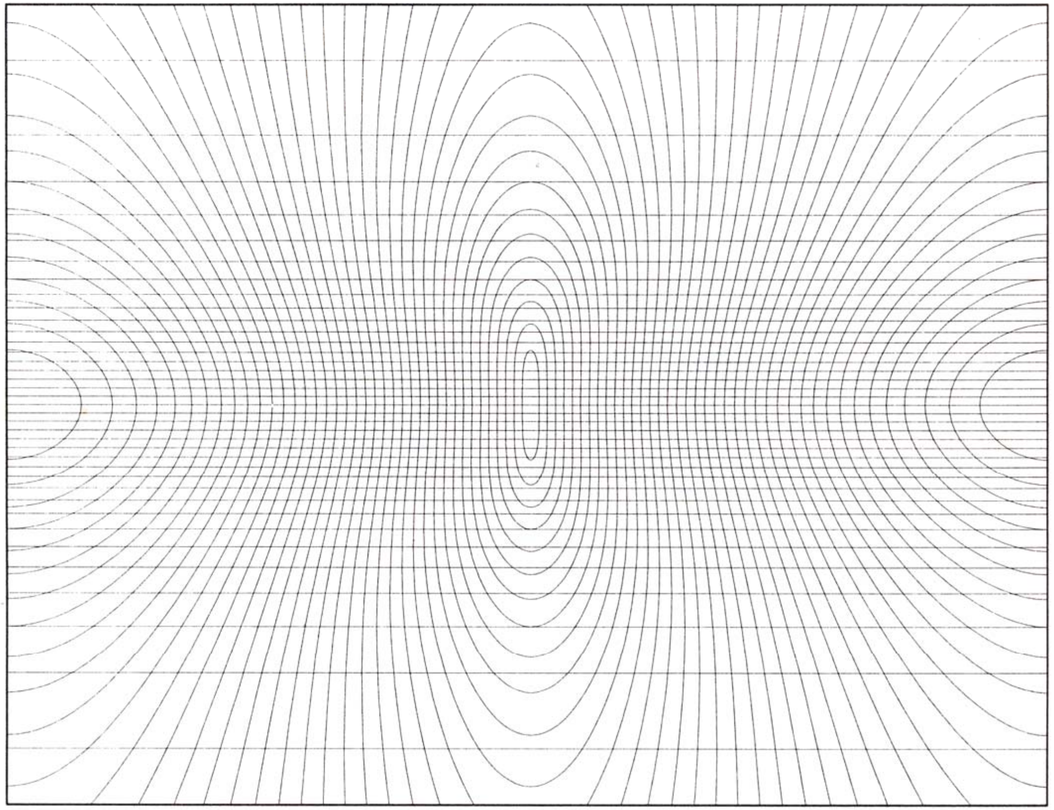
Indexing of h and k

$$\sin^2\beta+\lambda^2\left|\mathbf{r}^*_h\right|^2=2(1-\cos\alpha\cos\beta)$$

where $\left|\mathbf{r}^*_h\right|=\left|h\mathbf{a}^*+k\mathbf{b}^*\right|$

8.1 Diffraction Experiments using Films

Bernal chart: read of $\lambda|\mathbf{r}_v^*|$ and $\lambda|\mathbf{r}_h^*|$ from film.

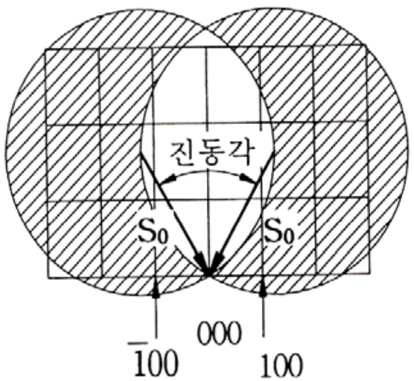


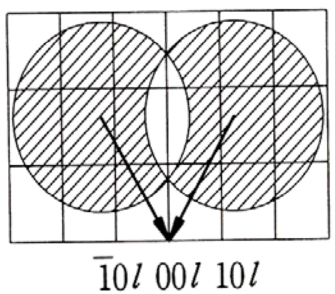
【그림 8-11】 Bernal 망 $\lambda|\mathbf{r}_v^*|$ 및 $\lambda|\mathbf{r}_h^*|$는 각각 0.05 간격으로 그려져 있음.

8.1 Diffraction Experiments using Films

Oscillation crystal method:

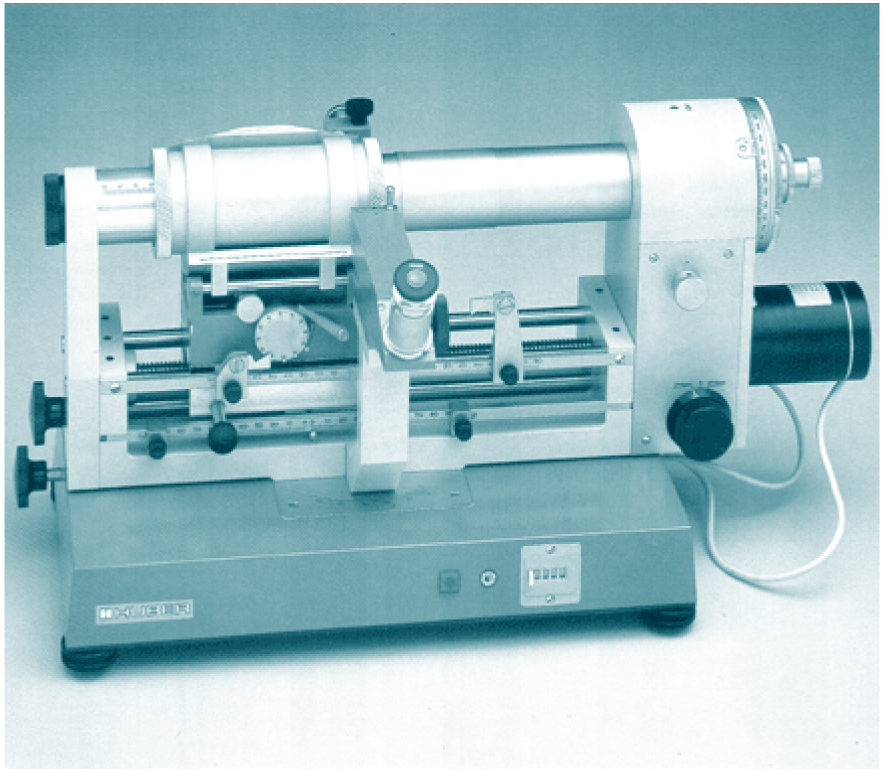
There are a few combination which satisfies the condition; $|\mathbf{r}_h^*| = |h\mathbf{a}^* + k\mathbf{b}^*|$
It causes the overlapping of different diffraction peaks. One can reduce overlapping by oscillating crystal or moving film (Weissenberg camera)

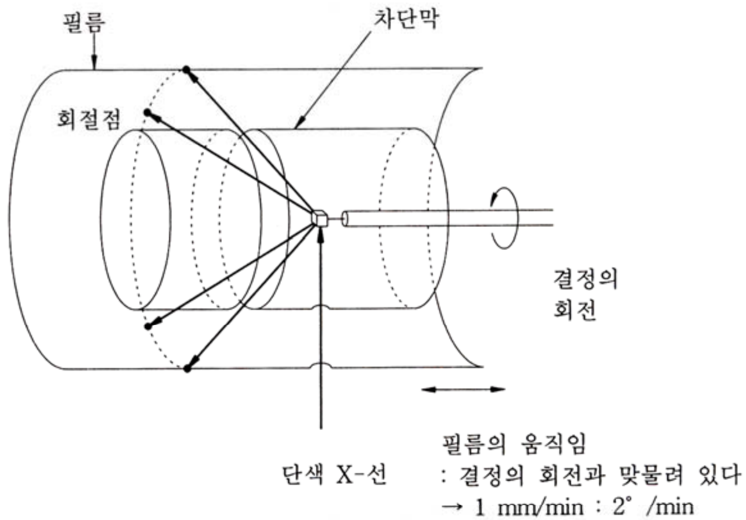




【그림 8-12】 결정 진동시의 회절 조건 (a) l = 0 (b) l ≠ 0

Weissenberg camera

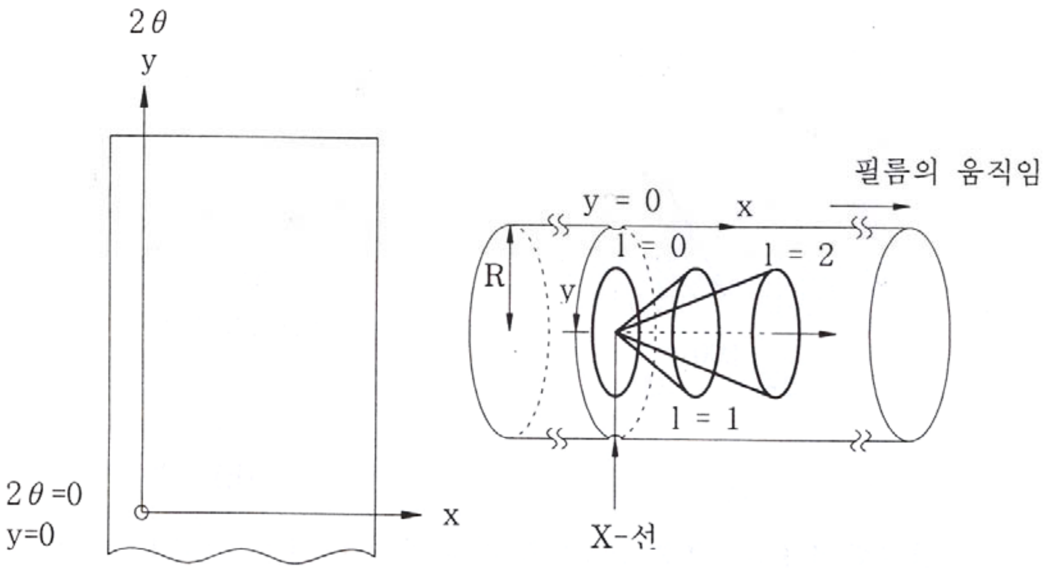




【그림 8-13】 Weissenberg 사진기의 구조

8.1 Diffraction Experiments using Films

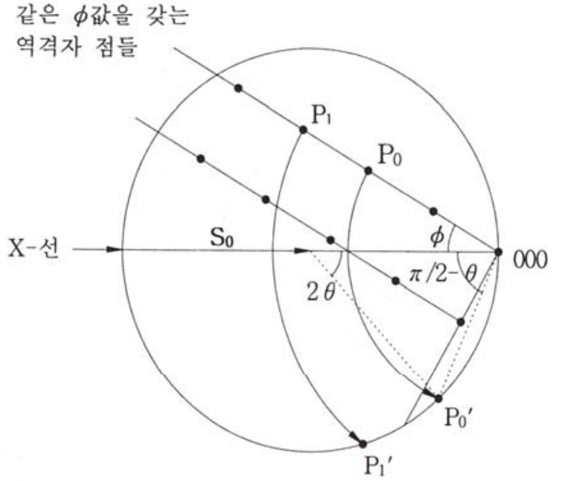
Film coordinate and diffraction condition



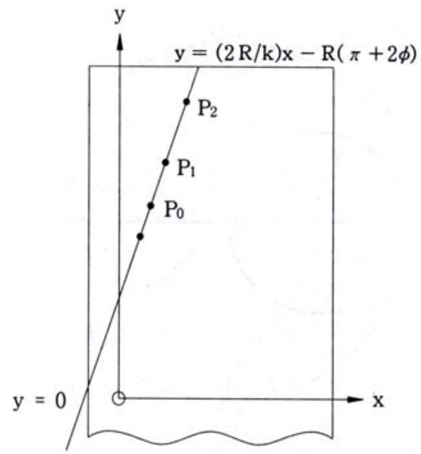
【그림 8-14】 Weissenberg 사진기의 회절 조건과 필름 상의 좌표

x: film moving direction
y: rotation direction
ω: rotation angle of crystal
k: the ratio between x/ω ~ 0.5mm/min

8.1 Diffraction Experiments using Films



【그림 8-15】 결정이 회전할 때 역격자점이 Ewald 구와 만나는 회전각



【그림 8-16】 같은 φ를 가진 역격자점이 Weissenberg 사진기의 필름에 나타난 모양

Relationship between x and y for l=0

$$x = k\omega$$

$$y = R2\theta$$

Let's see the diffraction condition for P_0. To occur diffraction at P_0, rotation angle of crystal would be

$$\omega = \frac{\pi}{2} - \theta + \phi$$

8.1 Diffraction Experiments using Films

Coordinate of diffraction peak of P_0 at film;

$$x = k\left(\frac{\pi}{2} - \theta + \phi\right)$$

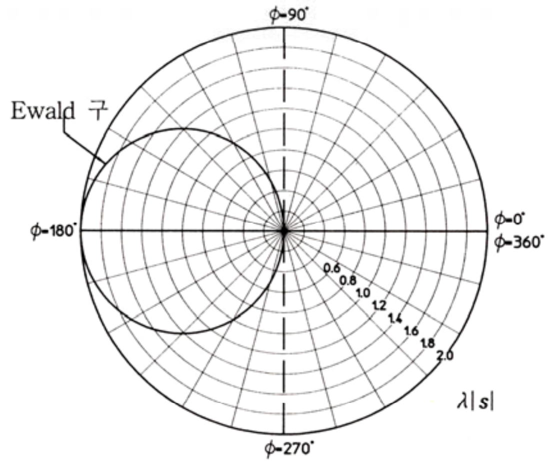
and

$$y = -\frac{2R}{k}x + R(\pi + 2\phi)$$

The reciprocal point which has the same ϕ lies on straight line.

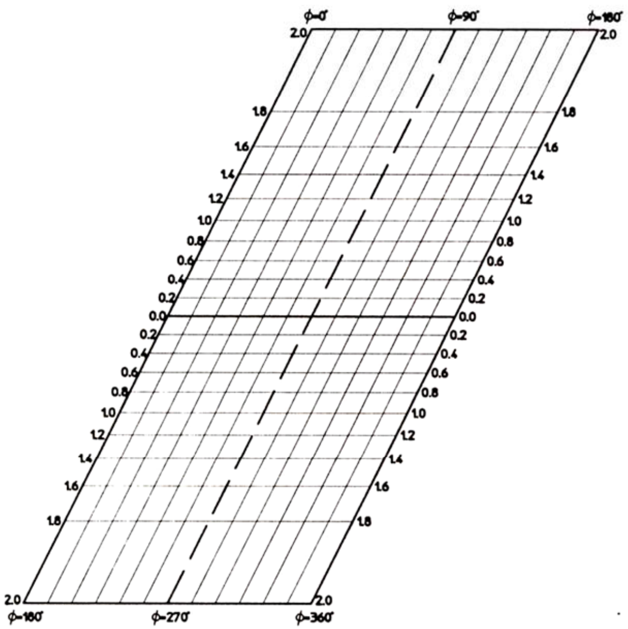
Since a monochromatic x-ray is used in Weissenberg camera, the reciprocal points lie inside of large circle can be diffract by crystal rotation.

When the polar mesh of the large circle transforms to film using the above equation;

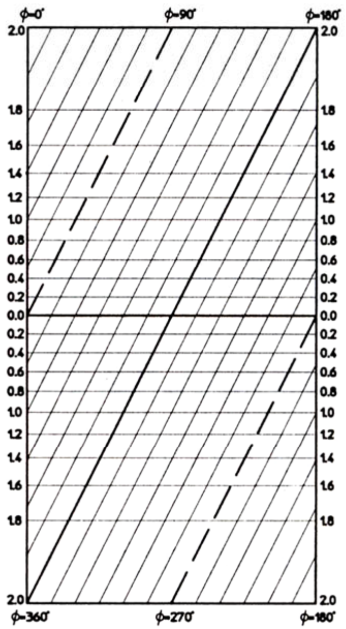


【그림 8-17】 극좌표계로 나타낸 회절 가능한 역공간

8.1 Diffraction Experiments using Films



(a)

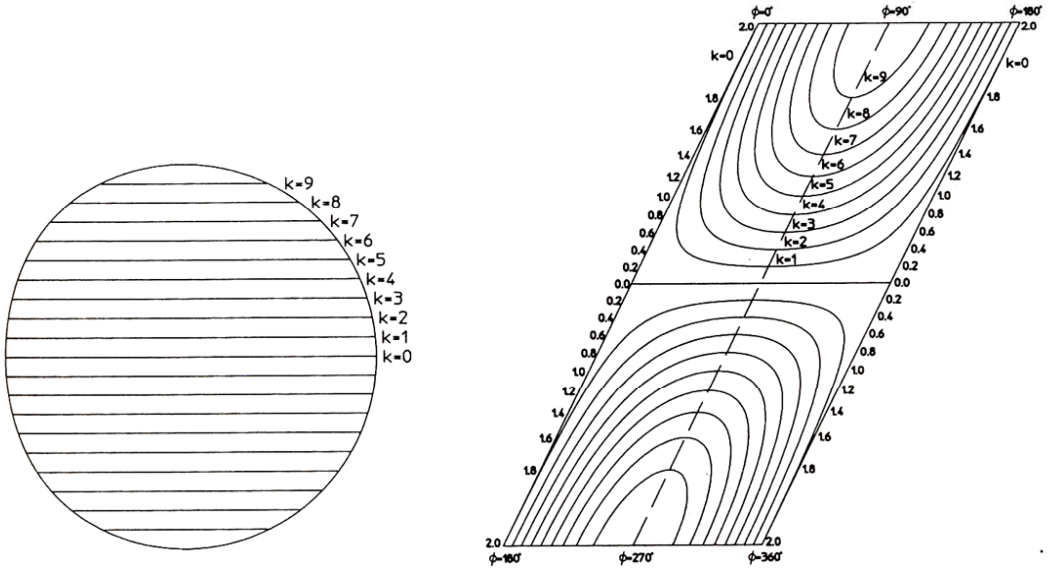


(b)

【그림 8-18】 역공간의 극좌표와 필름좌표에 나타난 모양 (a) 그림 8-17의 극좌표를 변환한 좌표 (b) 표준 Weissenberg 필름에 나타낸 좌표

8.1 Diffraction Experiments using Films

When this large circle is divided by latitude such as k=1, 2, 3, , the shape of transformed latitude becomes

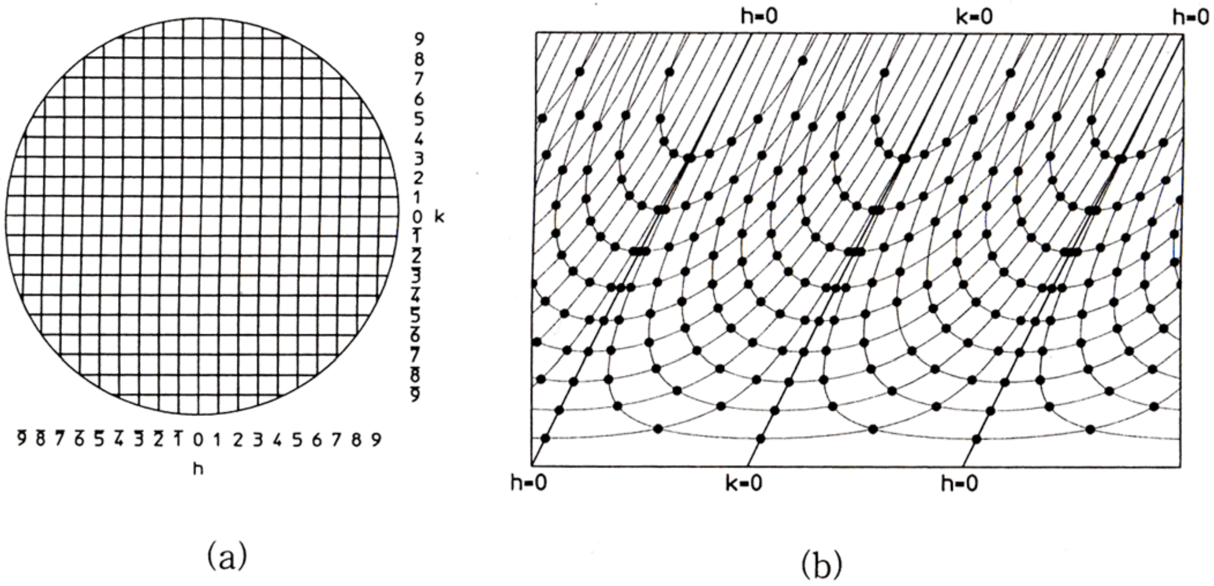


(a) (b)

【그림 8-19】 역공간 직선의 변환 (a) 역공간에 나타낸 등간격 ($\lambda|s|=0.2$) 선 (b) (a)의 등간격선이 Weissenberg 필름에 나타난 모양

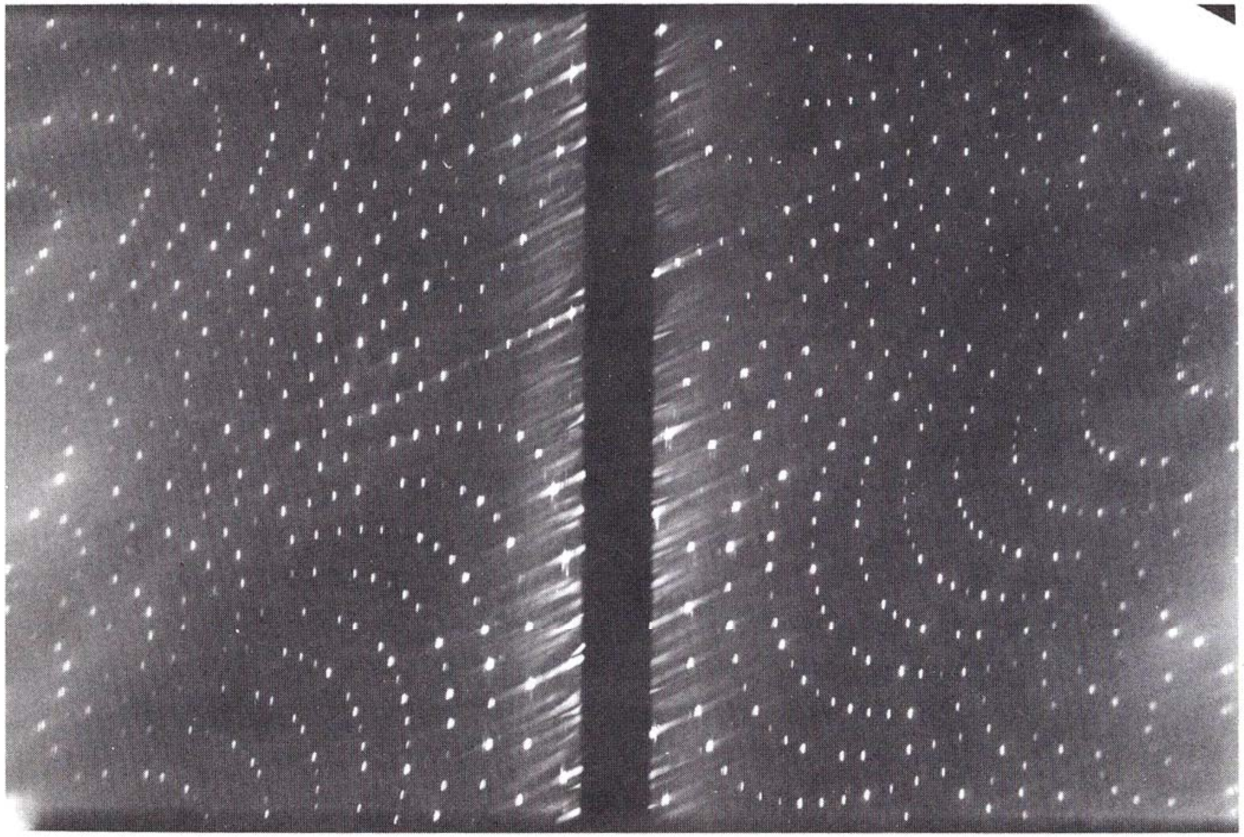
8.1 Diffraction Experiments using Films

When this large circle is divided by two perpendicular line mesh such as k=0, 1, 2, 3, … and h=0, 1, 2, 3, …   the shape of transformed mesh becomes



【그림 8-20】 l = 0 역격자면의 회절 무늬 (a) l = 0 면의 역격자점들과 색인 (b) (a)의 역격자점들의 Weissenberg 회절 무늬

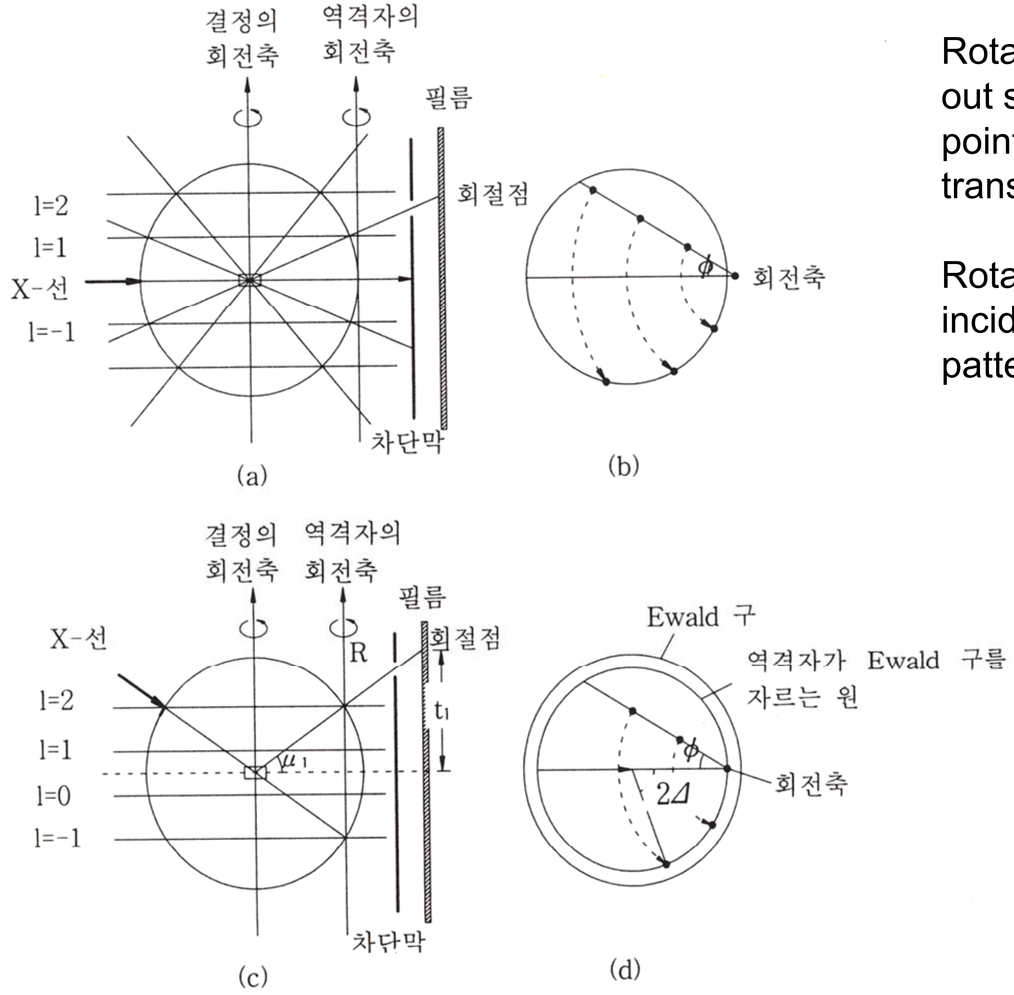
8.1 Diffraction Experiments using Films



A typical zero-level Weissenberg film, reproduced by permission of Prof. J. Ibers of Northwestern University.

8.1 Diffraction Experiments using Films

Equi-inclination method for l≠0



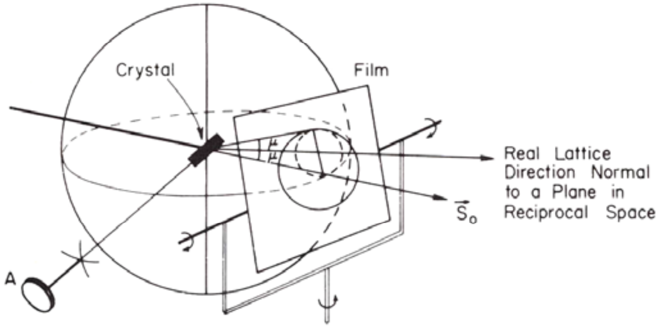
【그림 8-21】 등경사법에 의한 1≠0 면의 회절 조건

Rotation axis of reciprocal lattice locates out side of circle of Ewald sphere. The points of which ϕ is same will not transform straight line.

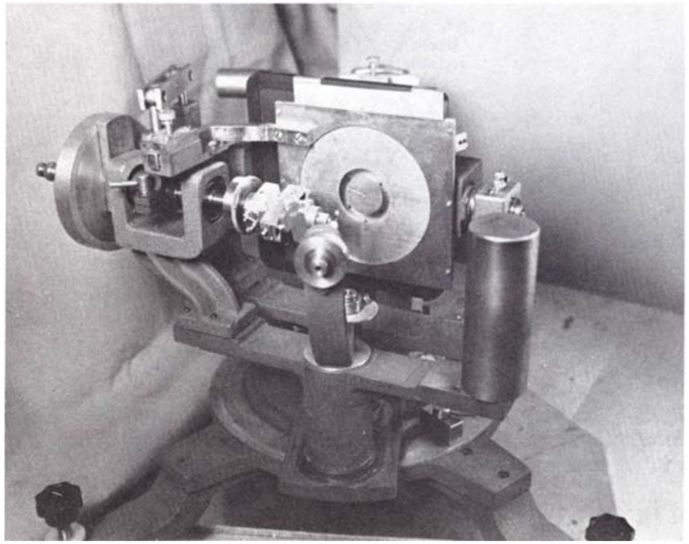
Rotation axis can be adjusted by inclining incident x-ray. → same shape of diffraction pattern for l=0.

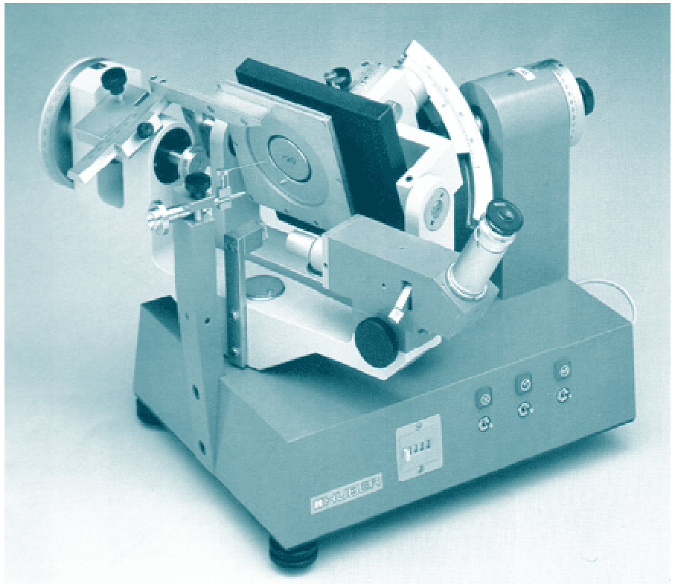
8.1 Diffraction Experiments using Films

Precession (Buerger) camera



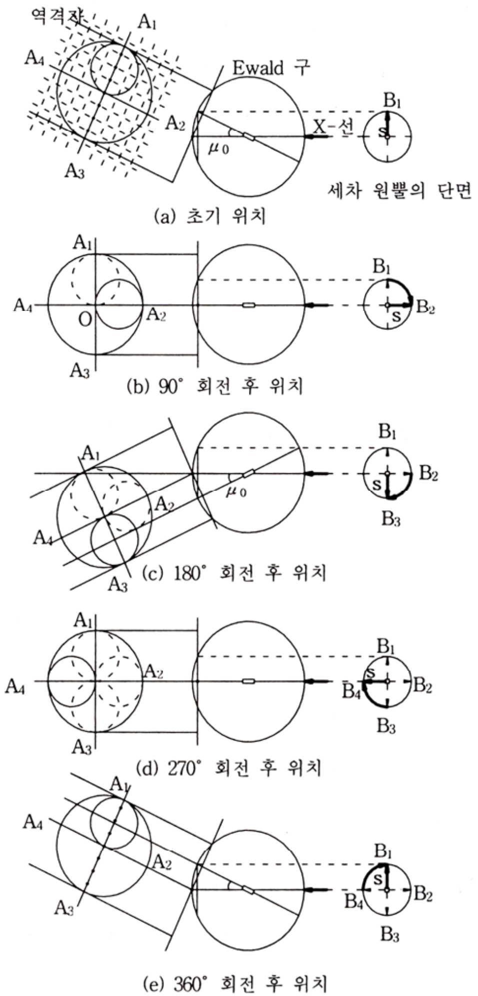
The precession camera. The small circle around the lattice direction precesses around S_0. (After Henry, N. F. M., Lipson, H., and Wooster, W. A. "The Interpretation of X-Ray Diffraction Photographs." MacMillan, London, 1951.)





8.1 Diffraction Experiments using Films

Precession motion and diffraction pattern



【그림 8-22】 세차운동을 하는 동안 산란 벡터와 역격자 및 차단막의 움직임

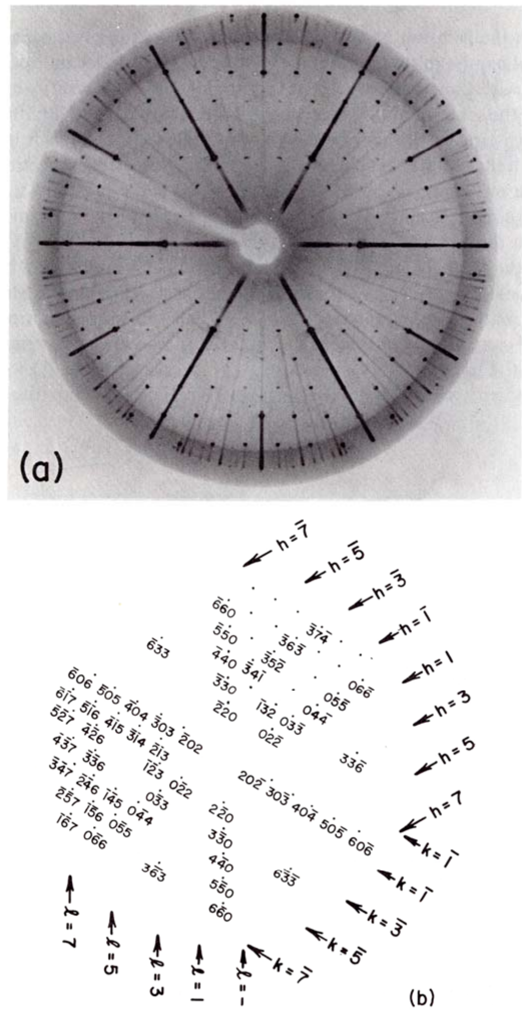
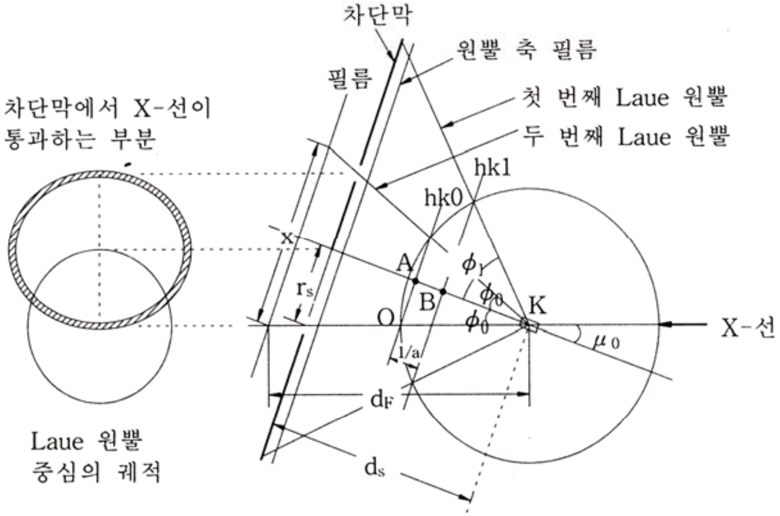


FIG. 5-33. A typical precession film, taken by C. Fairhurst. The film is the zero-layer obtained by precessing the [111] axis of a crystal of cubic Ag–Hg; Mo K_α, Zr filter, $\mu = 20°$, $r_s = 15$ mm, crystal to film, 42 mm (perpendicular to film). This pattern was over-exposed to show streaking from white radiation. The white region is the shadow of the beam stop.

8.1 Diffraction Experiments using Films

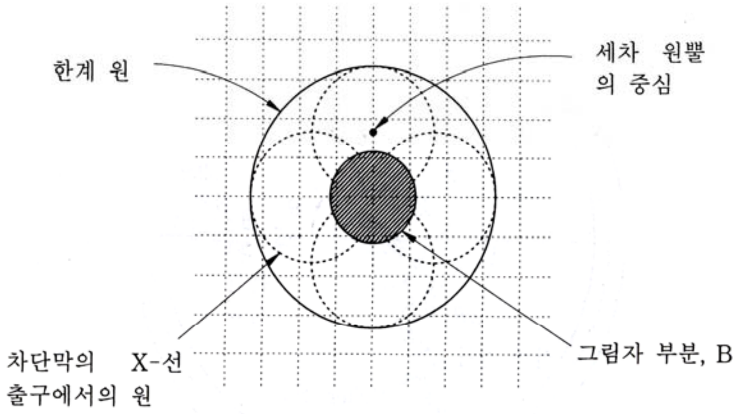
Precession motion and diffraction condition for l=0



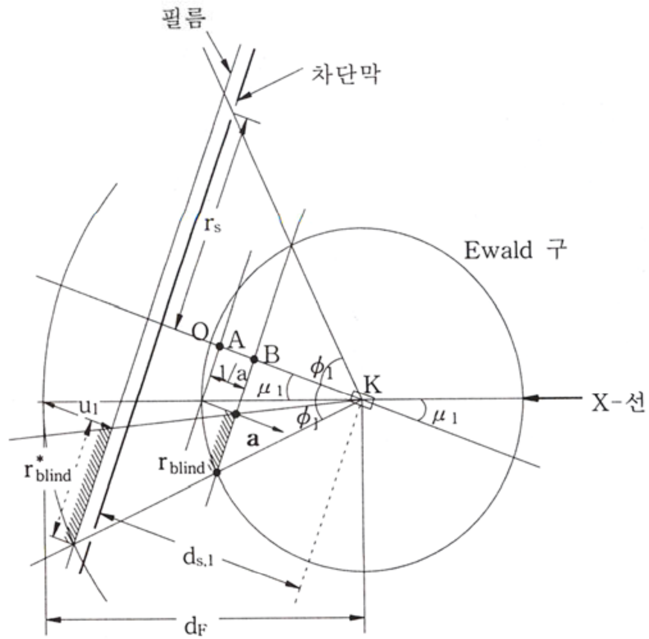
【그림 8-23】 세차운동사진기의 회절 조건

8.1 Diffraction Experiments using Films

Precession motion and diffraction condition for l≠0



【그림 8-25】 l = 1 역격자 층에 대한 차단막의 세차 운동과 회절되는 역격자점과 회절되지 않는 영역



【그림 8-24】 l = 1 역격자 층의 회절을 위한 세차 운동