

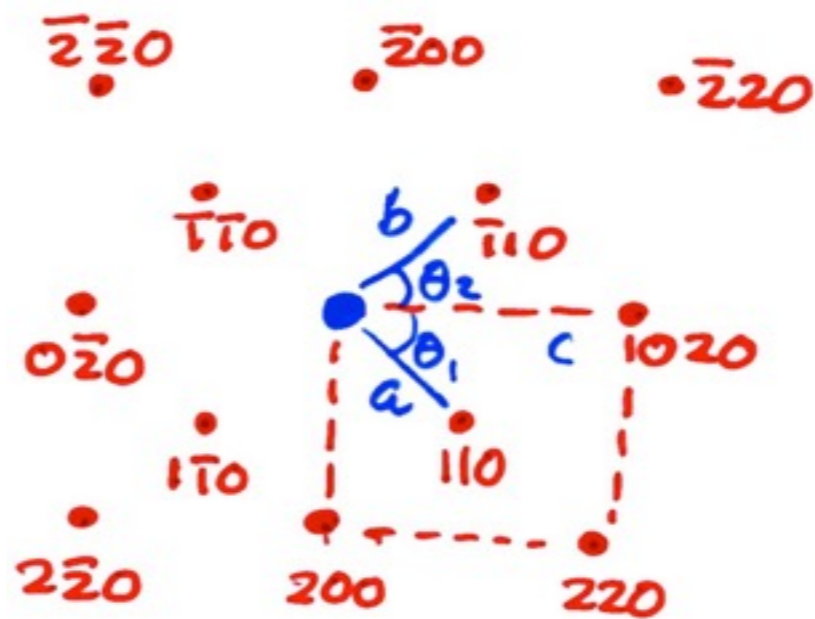
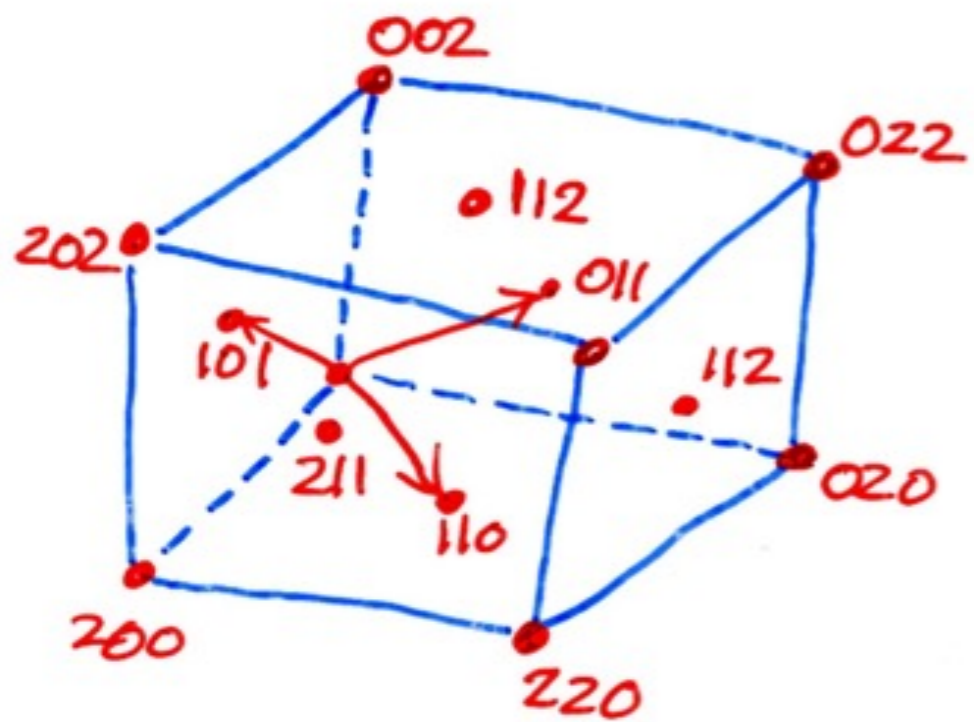
chapter 5

Analysis of Diffraction Pattern and Kikuchi Pattern

繞射花樣之建立與標定

(a) 體心立方晶體

晶軸 [001]



$$\frac{a}{b} = \frac{\sqrt{1^2+1^2+0}}{\sqrt{1^2+(-1)^2+0}} = 1 = \frac{1/d_{110}}{1/d_{\bar{1}\bar{1}0}}$$

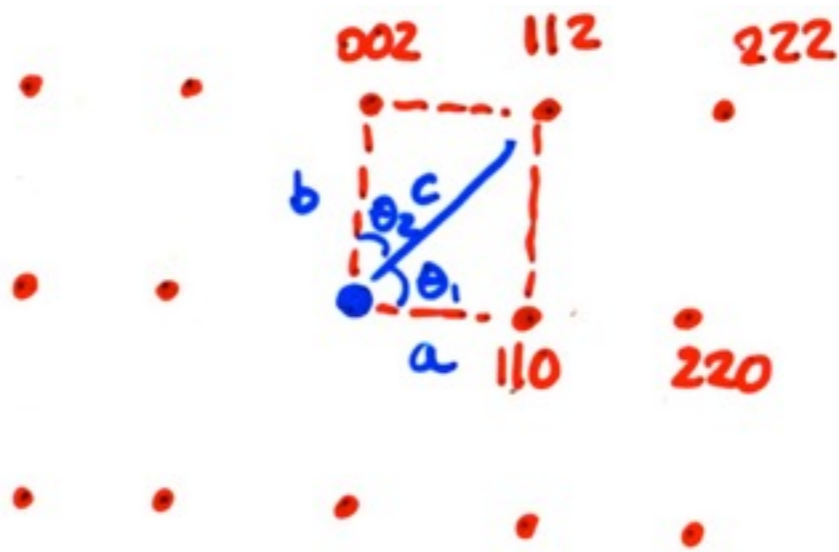
$\therefore a, b = 45^\circ$

晶軸 [-110]

$[uvw] \cdot \{hkl\} = 0$
晶軸 zolz

$[u \ v \ w] \cdot \{h \ k \ 1\} = 0$

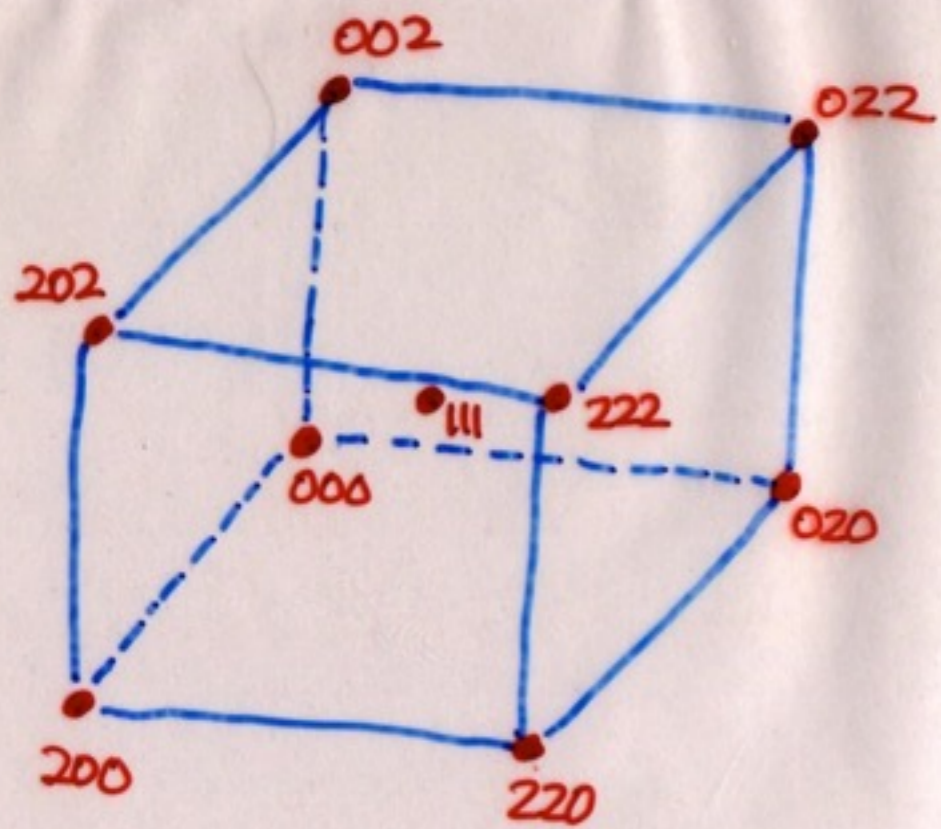
晶軸 zolz



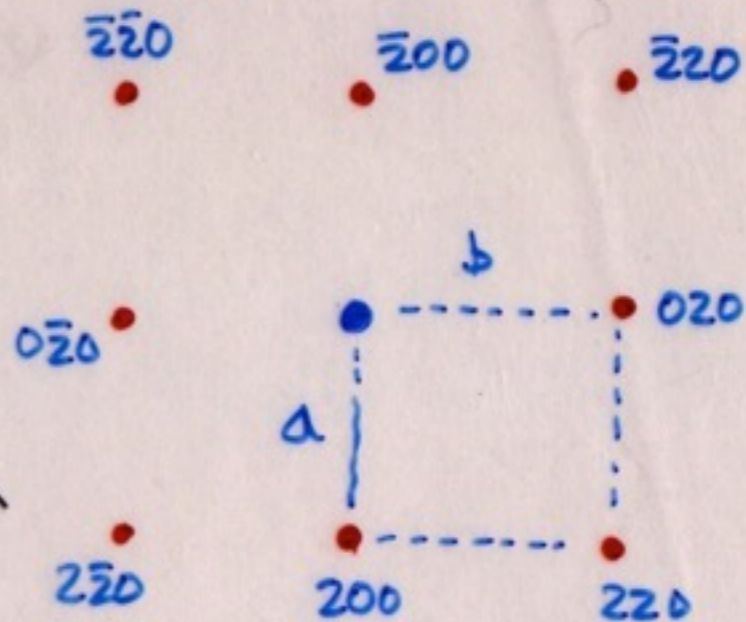
$$\frac{a}{b} = \frac{\sqrt{1^2+1^2+0^2}}{\sqrt{0^2+0^2+2^2}} = \frac{\sqrt{2}}{2} = \frac{1/d_{110}}{1/d_{002}}$$

$\therefore \theta(a,b) = 90^\circ$

面心立方



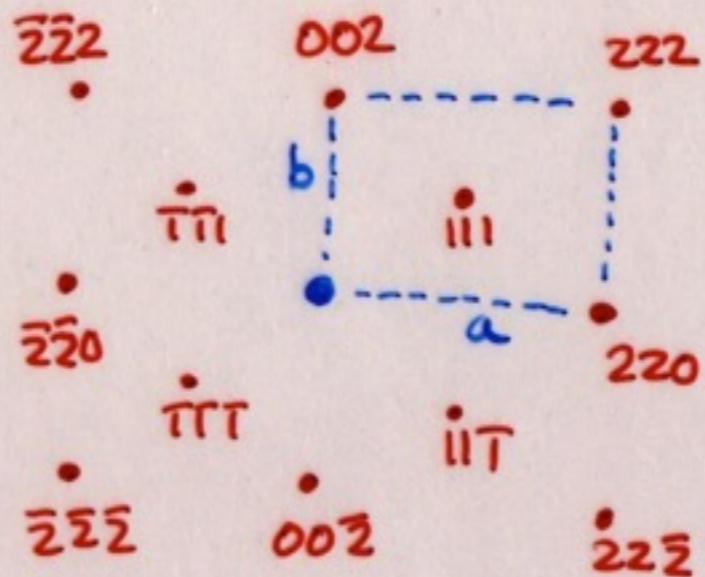
晶軸 [001]



$$\frac{a}{b} = \frac{\sqrt{2^2+0^2+0^2}}{\sqrt{0^2+2^2+0^2}} = 1$$

$$\angle \theta(a, b) = 90^\circ$$

晶軸 [110]



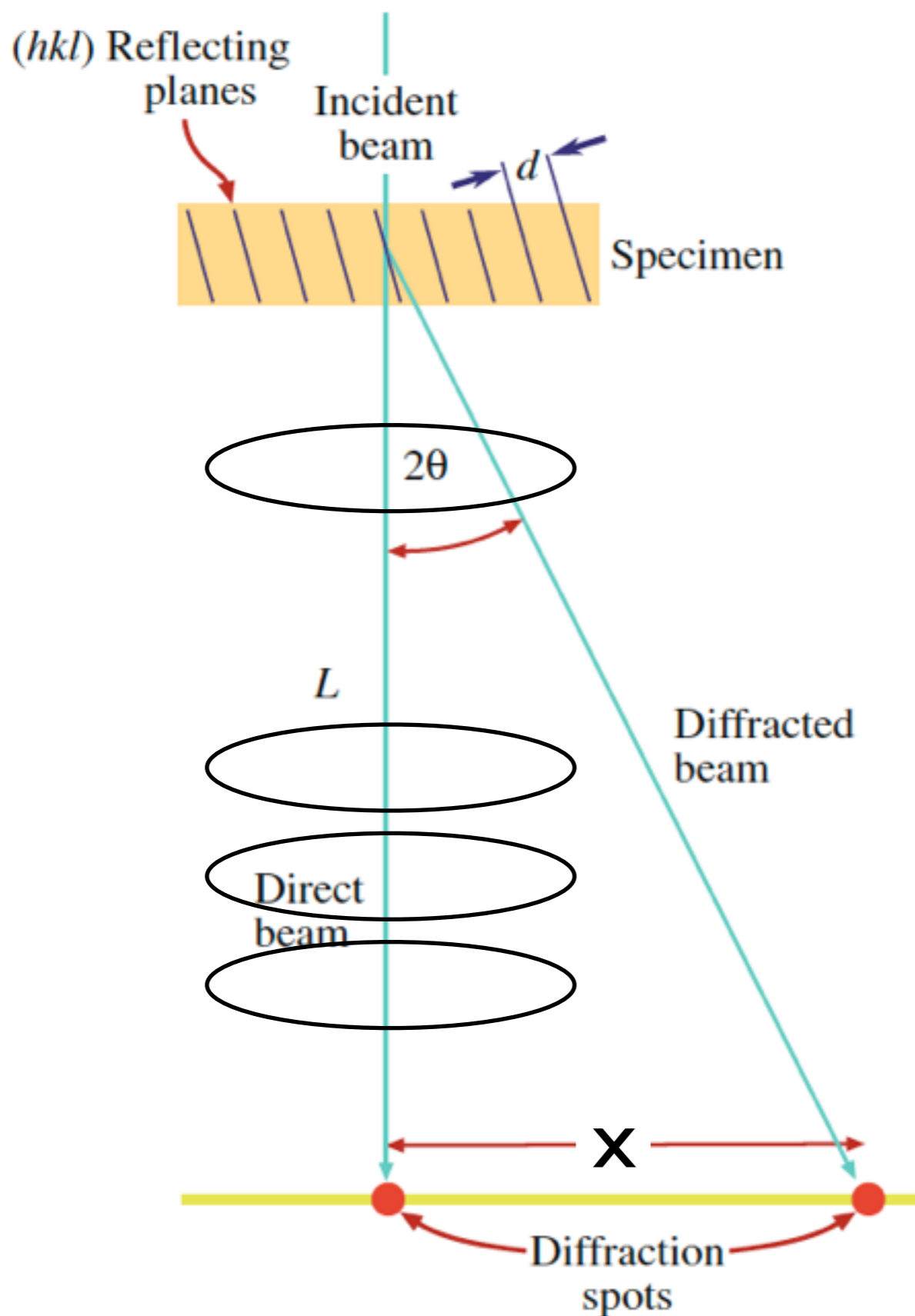
$$\frac{a}{b} = \frac{\sqrt{2^2+2^2+0^2}}{\sqrt{0^2+0^2+2^2}} = \frac{\sqrt{8}}{2} = \sqrt{2}$$

$$\angle \theta(a, b) = 90^\circ$$

$$[uvw] \cdot \{hkl\} = 0$$

晶軸 20L2

相機長度(Camera length)



$$2d\sin\theta=\lambda$$

$$2\theta\div\lambda/d$$

$$\tan 2\theta=x/L$$

$$2\theta\div x/L$$

$$x/L=\lambda/d$$

$$L\lambda=xd$$

L: Camera Length

$L\lambda$: Camera constant

未知繞射花樣之標定 (晶體結構分析)

(a) 選擇最短的三繞射點量測

$$\overline{oa}, \overline{ob}, \overline{oc}$$

$$\angle\theta(a,b), \angle\theta(b,c) \text{ \& } \angle\theta(c,a)$$

(b) 求出 $\frac{\overline{ob}}{\overline{oa}}, \frac{\overline{oc}}{\overline{oa}}, \frac{\overline{oc}}{\overline{ob}}$ $\longrightarrow d_a/d_b, d_a/d_c, d_c/d_b$

(c) 查 d_1/d_2 表, $g_1/g_2(\theta)$ 表

(d) 標定繞射點 a, b 及 c

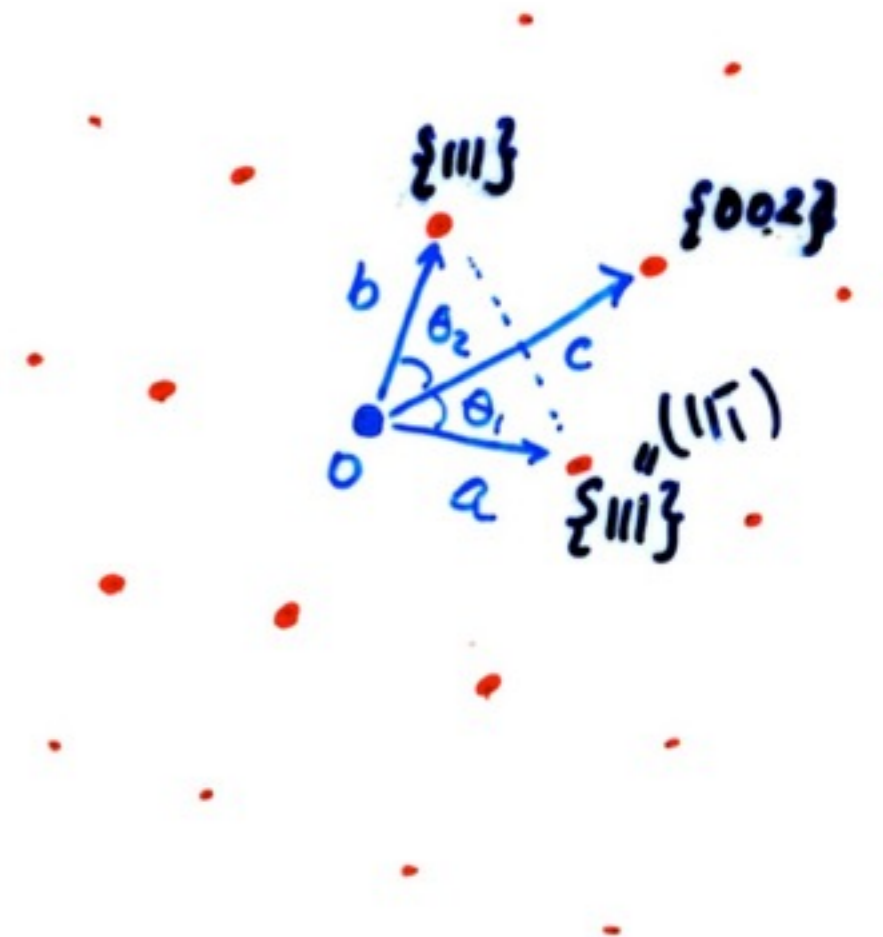
(e) 確認 $\overrightarrow{oc} = \overrightarrow{oa} + \overrightarrow{ob}$, 並標定其他繞射點 (以向量加法)

(f) 晶軸 = $\overrightarrow{oa} \times \overrightarrow{ob}$

(g) 以 $dX = L\lambda$ 求出晶體材料

$$\frac{a}{\sqrt{h^2 + k^2 + l^2}} = \frac{L\lambda}{|oa|}$$

$$a = \frac{L\lambda}{|oa|} \sqrt{h^2 + k^2 + l^2}$$



d-ratio for Cubic crystal

$d_1 \backslash d_2$	100	110	111	200	211	220	311
100	1						
110	1.414	1					
111	1.732	1.225	1				
200	2	1.414	1.155	1			
211	2.45	1.732	1.414	1.225	1		
220	2.828	2	1.633	1.414	1.155	1	
311	3.32	2.345	1.915	1.658	1.354	1.173	1

Interplanar Angle

$$\cos \phi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{(h_1^2 + k_1^2 + l_1^2)^{1/2} (h_2^2 + k_2^2 + l_2^2)^{1/2}}$$

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Cu ● IDLE

Angle / Plane Calculator

Lattice

Axes [Å]

A 4.0000

B 4.0000

C 4.0000

Angles [°]

Alpha 90.00

Beta 90.00

Gamma 90.00

Reflection 1

1 1 0

Length [1/Å] 0.3536

Plane Spacing 2.8284

Reflection 2

1 1 1

Length [1/Å] 0.4330

Plane Spacing 2.3094

Update Lattice parameters

Calculate/Update Angles

[h,k,l]	[h,k,l]	Angle	Zone
[1,1,0]	[1,1,1]	35.26	[1,-1,0]
[1,1,0]	[1,1,-1]	35.26	[-1,1,0]
[1,1,0]	[1,-1,1]	90.00	[1,-1,-2]
[1,1,0]	[1,-1,-1]	90.00	[-1,1,-2]
[1,1,0]	[-1,1,1]	90.00	[1,-1,2]
[1,1,0]	[-1,1,-1]	90.00	[-1,1,2]
[1,1,0]	[-1,-1,1]	144.74	[1,-1,0]
[1,1,0]	[-1,-1,-1]	144.74	[-1,1,0]
[1,-1,0]	[1,1,1]	90.00	[-1,-1,2]
[1,-1,0]	[1,1,-1]	90.00	[1,1,2]
[1,-1,0]	[1,-1,1]	35.26	[-1,-1,0]
[1,-1,0]	[1,-1,-1]	35.26	[1,1,0]
[1,-1,0]	[-1,1,1]	144.74	[-1,-1,0]
[1,-1,0]	[-1,1,-1]	144.74	[1,1,0]

Angle / Plane Calculator

Lattice

Axes [Å]

A 4.0000

B 4.0000

C 4.0000

Angles [°]

Alpha 90.00

Beta 90.00

Gamma 90.00

Reflection 1

1 1 1

Length [1/Å] 0.4330

Plane Spacing 2.3094

Reflection 2

1 1 1

Length [1/Å] 0.4330

Plane Spacing 2.3094

Update Lattice parameters

Calculate/Update Angles

[h,k,l]	[h,k,l]	Angle	Zone
[1,1,1]	[1,1,-1]	70.53	[-1,1,0]
[1,1,1]	[1,-1,1]	70.53	[1,0,-1]
[1,1,1]	[1,-1,-1]	109.47	[0,1,-1]
[1,1,1]	[-1,1,1]	70.53	[0,-1,1]
[1,1,1]	[-1,1,-1]	109.47	[-1,0,1]
[1,1,1]	[-1,-1,1]	109.47	[1,-1,0]
[1,1,-1]	[1,1,1]	70.53	[1,-1,0]
[1,1,-1]	[1,-1,1]	109.47	[0,-1,-1]
[1,1,-1]	[1,-1,-1]	70.53	[-1,0,-1]
[1,1,-1]	[-1,1,1]	109.47	[1,0,1]
[1,1,-1]	[-1,1,-1]	70.53	[0,1,1]
[1,1,-1]	[-1,-1,1]	109.47	[-1,1,0]
[1,-1,1]	[1,1,1]	70.53	[-1,0,1]
[1,-1,1]	[1,1,-1]	109.47	[0,1,1]

Example: JEOL 2000FXII, 200 KV, $\lambda = 0.0251 \text{ \AA}$, $L = 80 \text{ cm}$

$$\overline{oa} = 0.64 \text{ cm} \quad \angle \theta(a, b) = 110^\circ \quad L \lambda = 2.008 \text{ cm \AA}$$

$$\overline{ob} = 0.61 \text{ cm} \quad \angle \theta(a, c) = 54^\circ \quad \text{角度較長度準確}$$

$$\overline{oc} = 0.74 \text{ cm} \quad \angle \theta(b, c) = 54^\circ$$

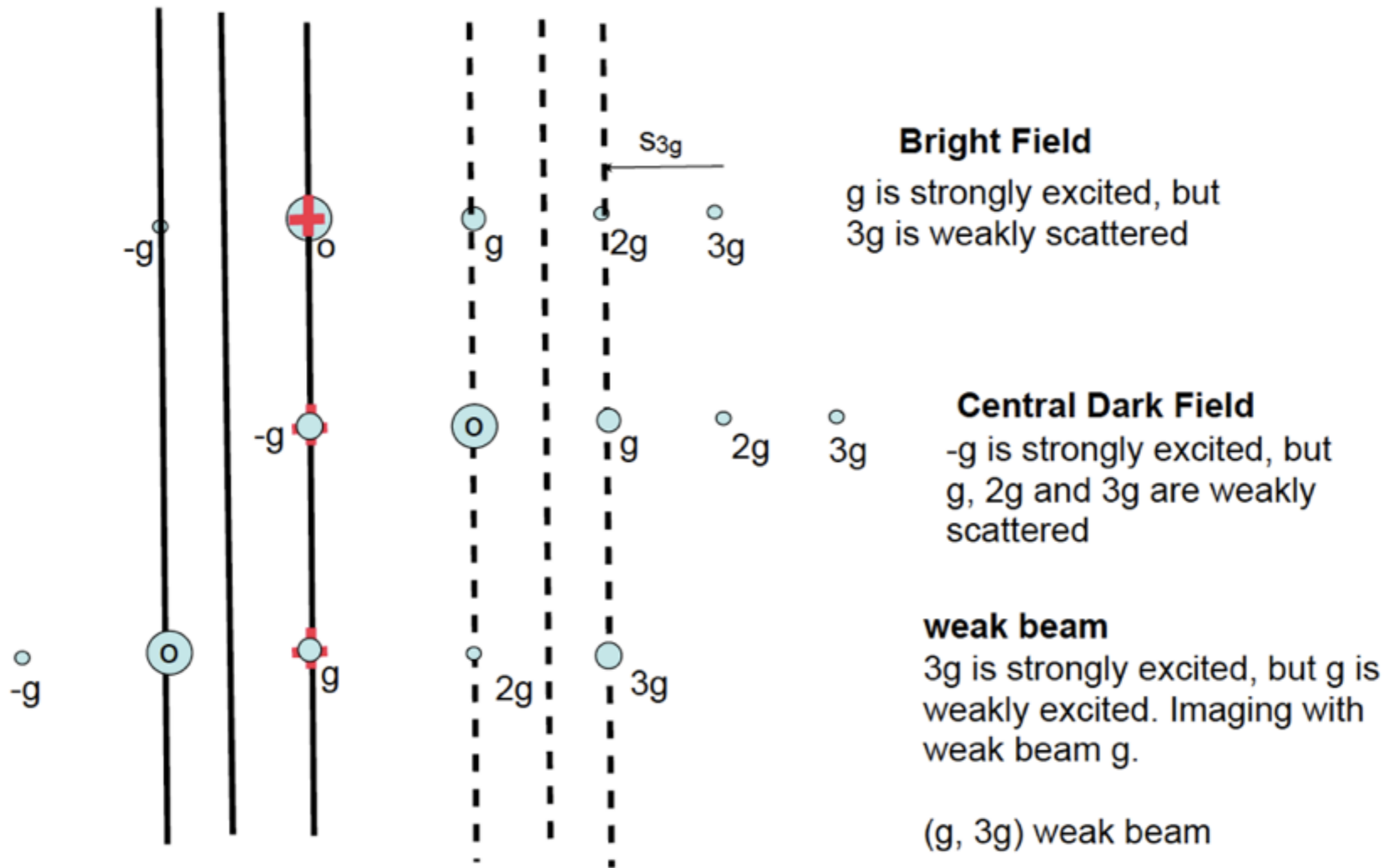
$$\frac{\overline{ab}}{\overline{oa} \rightarrow (111)} = 0.95, \frac{\overline{oc} \leftarrow (002)}{\overline{oa} \rightarrow (111)} = 1.15, \frac{\overline{oc} \leftarrow (002)}{\overline{ob}} = 1.21$$

— a: {111}, b: {111}, c: {200}

a \rightarrow (1-1-1), b \rightarrow (111), c \rightarrow (200) check 向量與角度的關係

晶軸 $\overrightarrow{oa} \times \overrightarrow{ob} = [01\bar{1}], a = \frac{L\lambda}{0.64} \times \sqrt{3} \cong 5.34 \text{ \AA} \rightarrow \text{Si}$

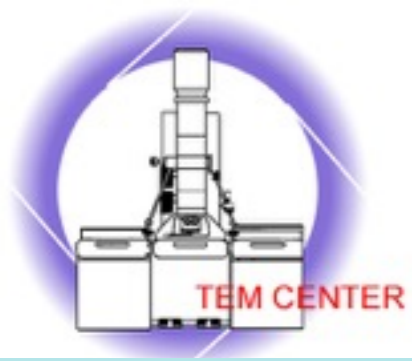
Dark Field and Weak Beam



Kikuchi Pattern

Deviation parameter s & Kikuchi pattern

Kikuchi Map in Zone axis



NTHU

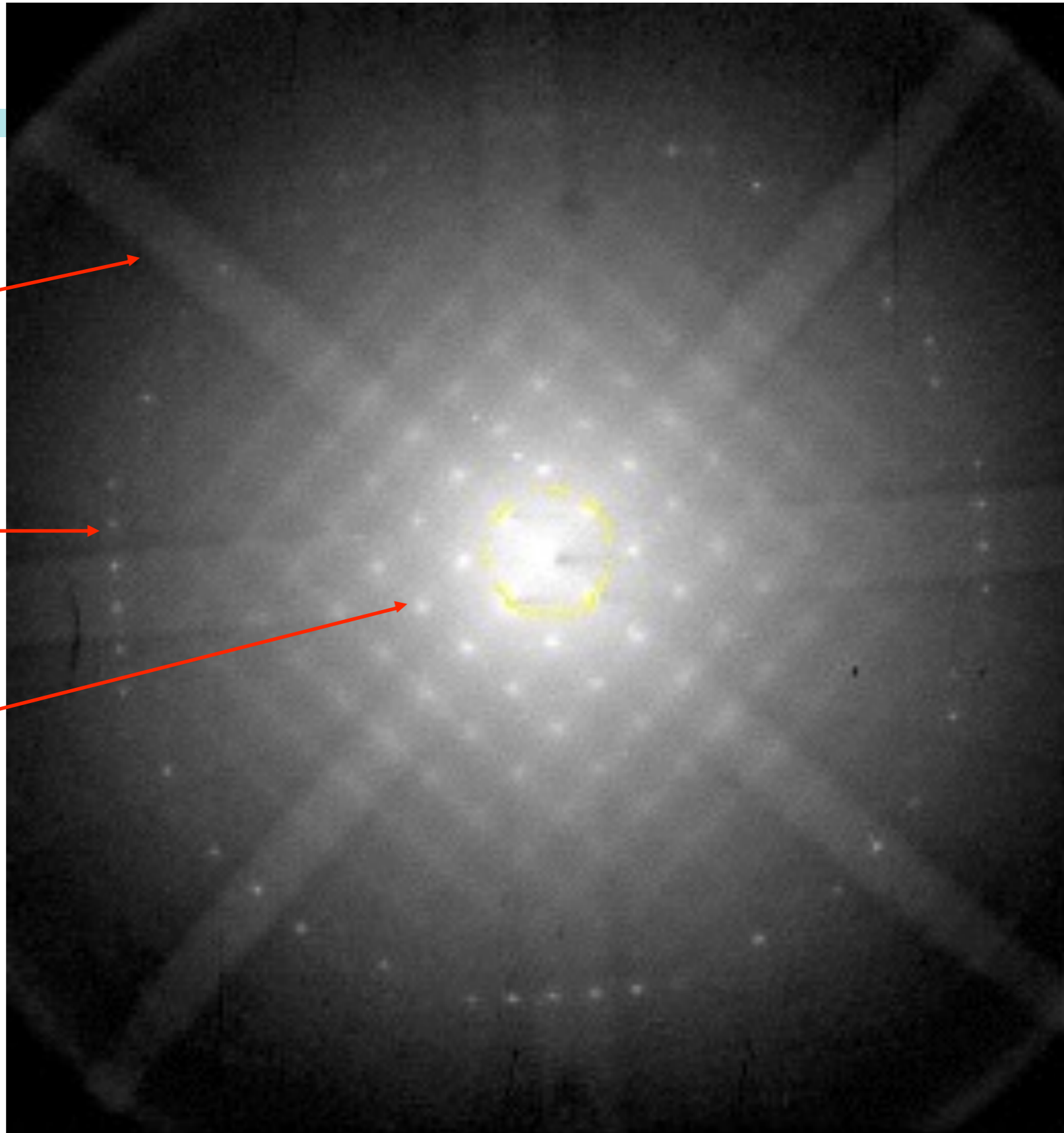
Kikuchi Lines



HOLZ



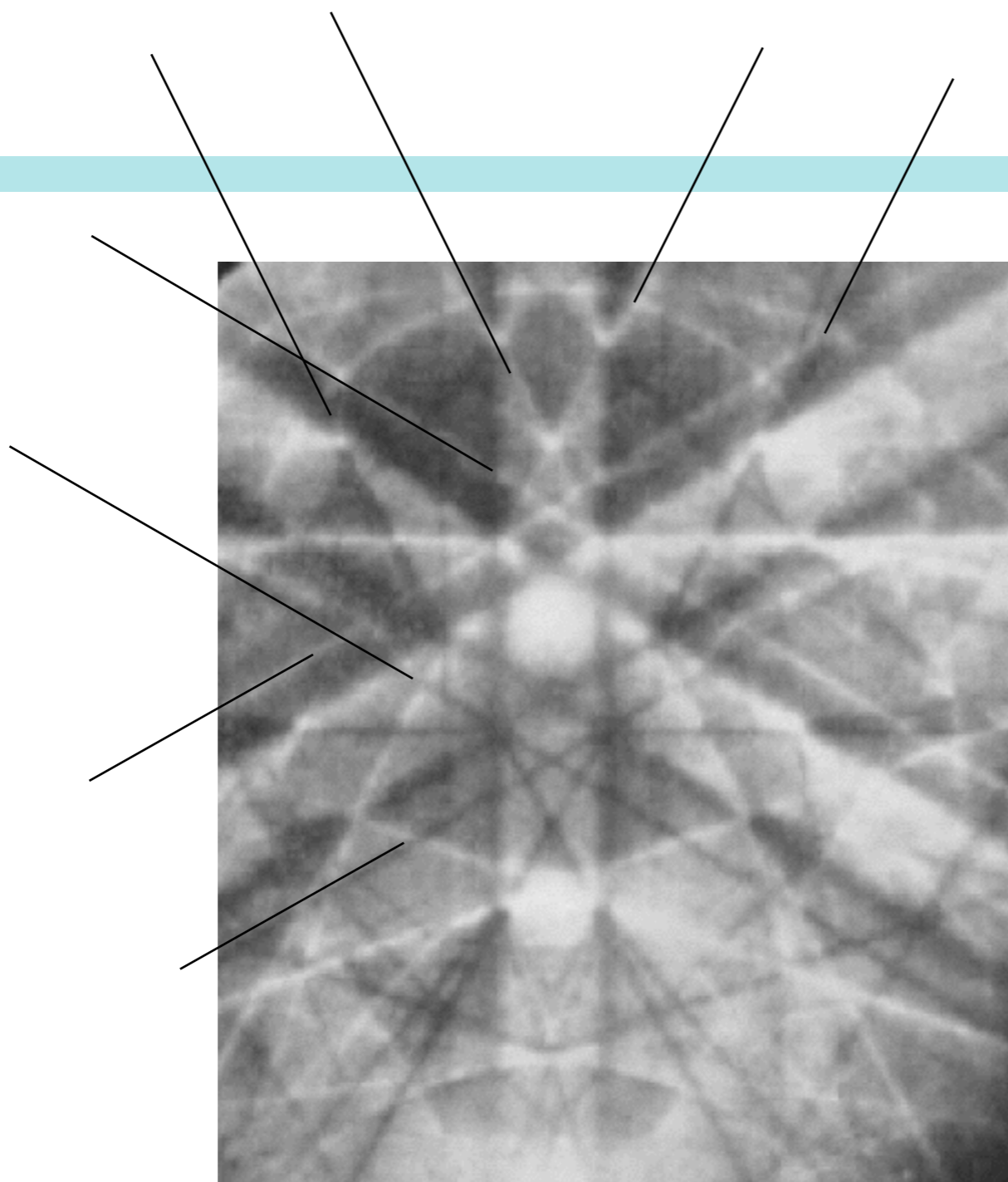
ZOLZ





Kikuchi Map not in Zone axis

NTHU



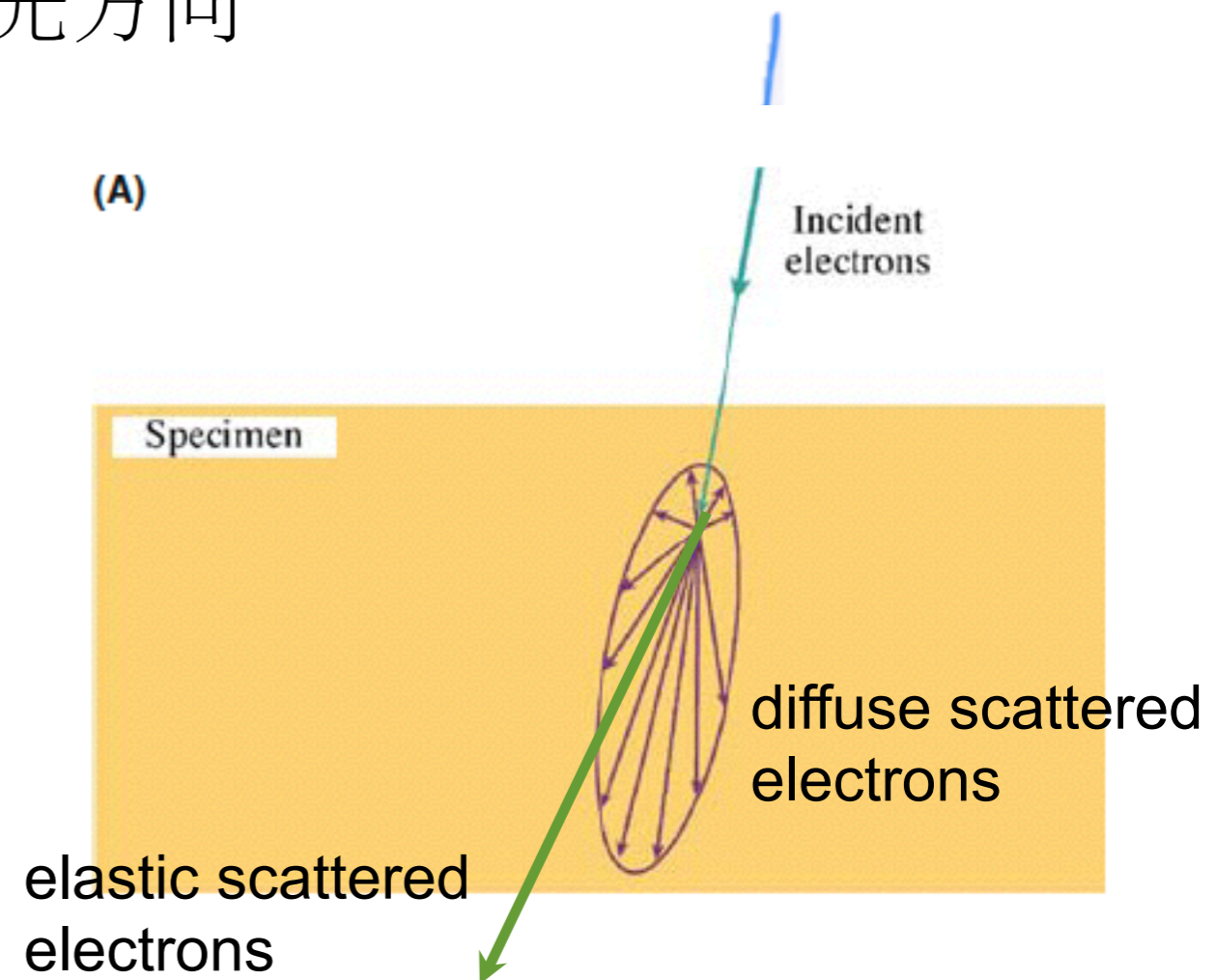
Kikuchi Pattern (菊池圖)

- 當樣品在是當厚度時（不太厚，不太薄）入射電子除了發生彈性散射外，亦有足夠的電子發生diffusely散射（往各方向）
- （因diffusely散射量比起入射電子與彈性散射小太多，樣品厚度必須大於某一程度才看得到，但不能太厚，會看不到。
- Diffusely散射包括一沒發生彈性散射零損電子，這些電子的相干性較差。
- 大部分的diffusely散射分佈在入射光方向

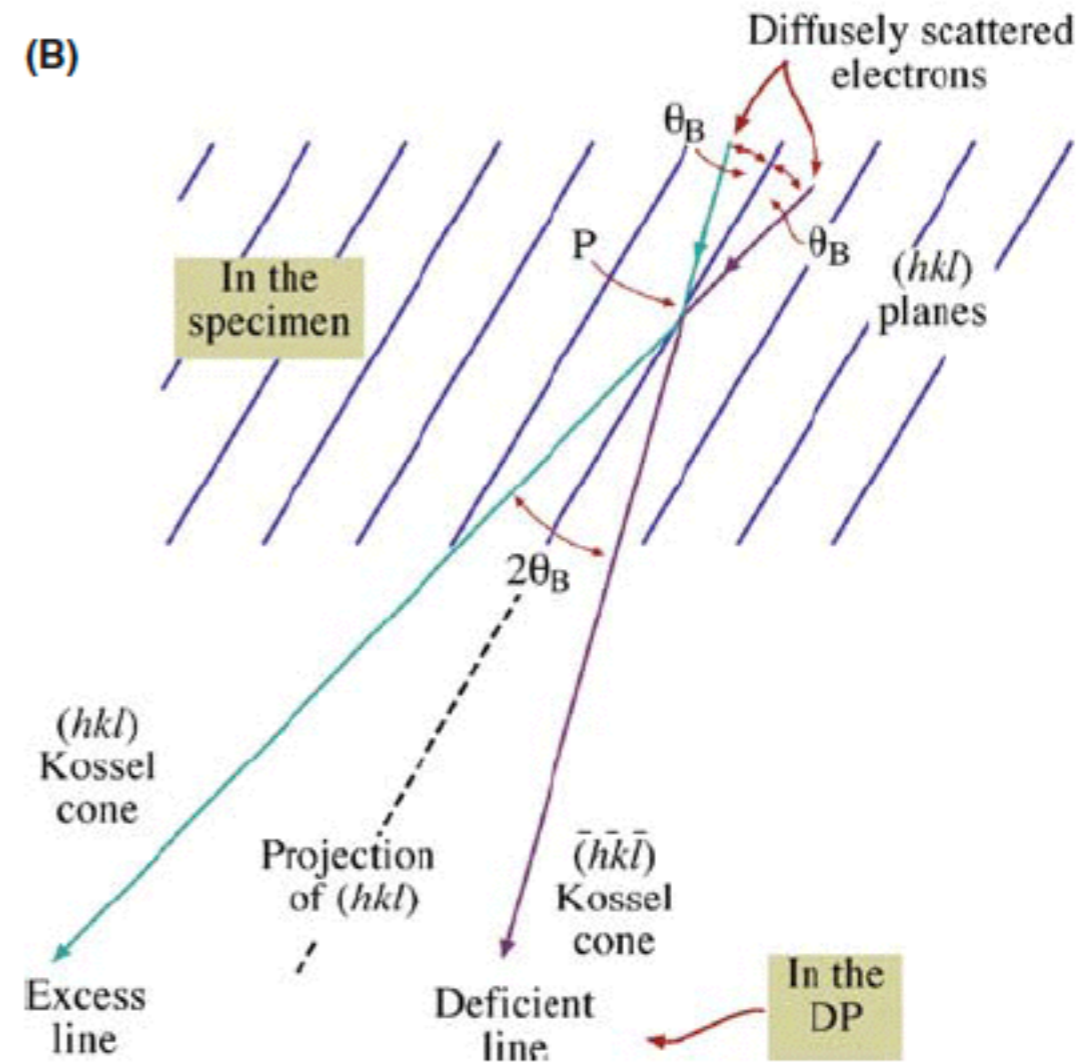
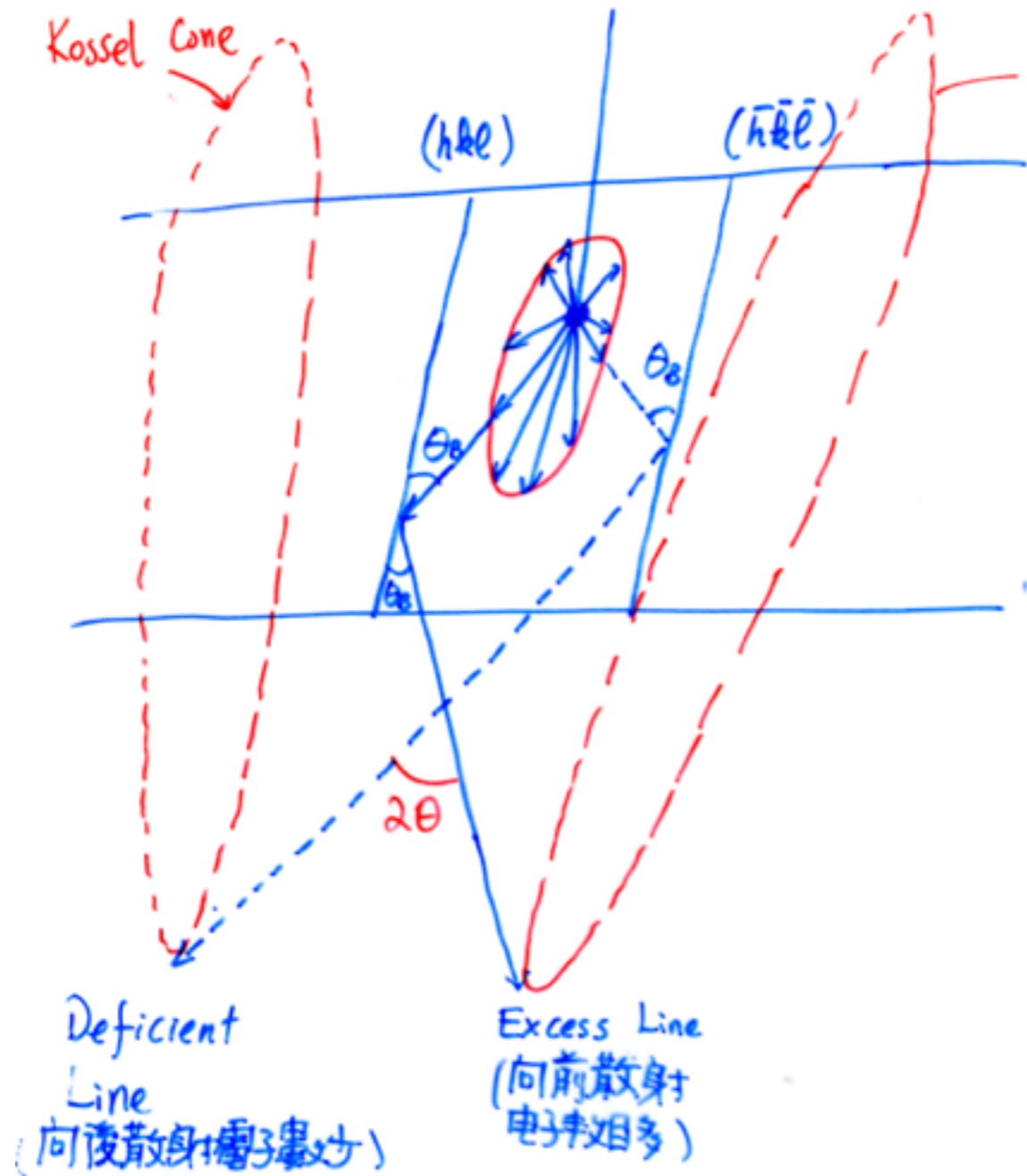
$$\theta_E = \frac{\Delta E}{2E_0}$$

ΔE : 損失之能量
 E_0 : 入射之能量

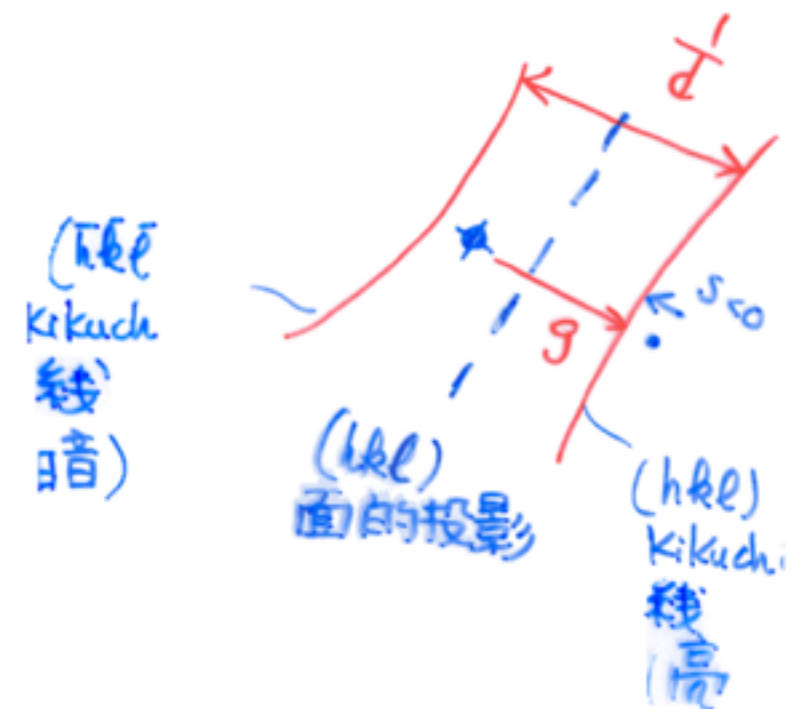
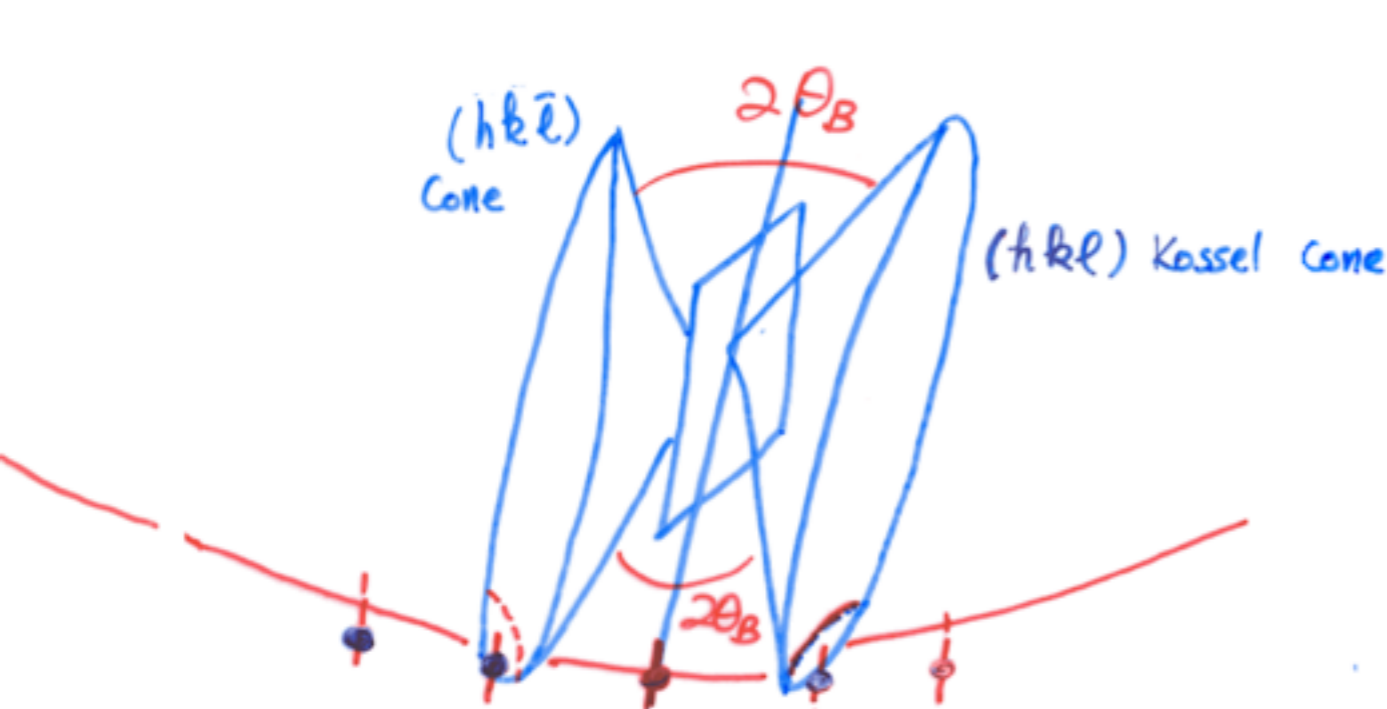
- 被散射往高角度方相的電子 數少

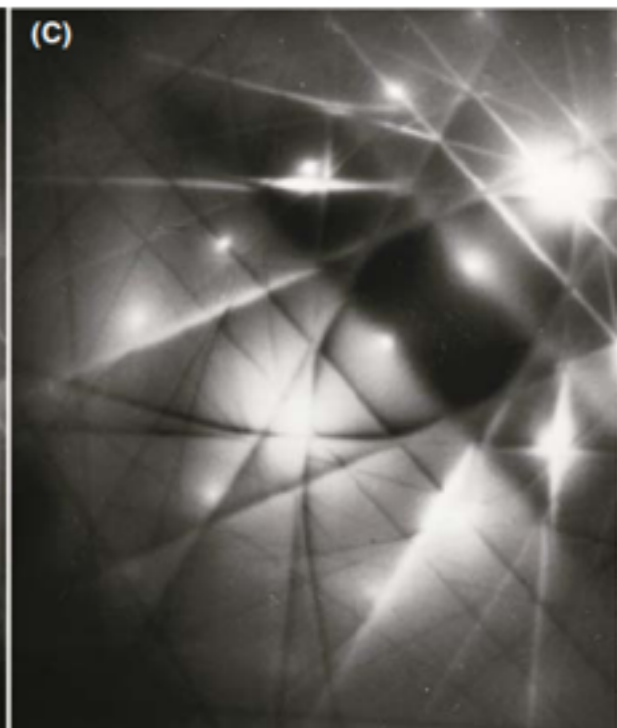
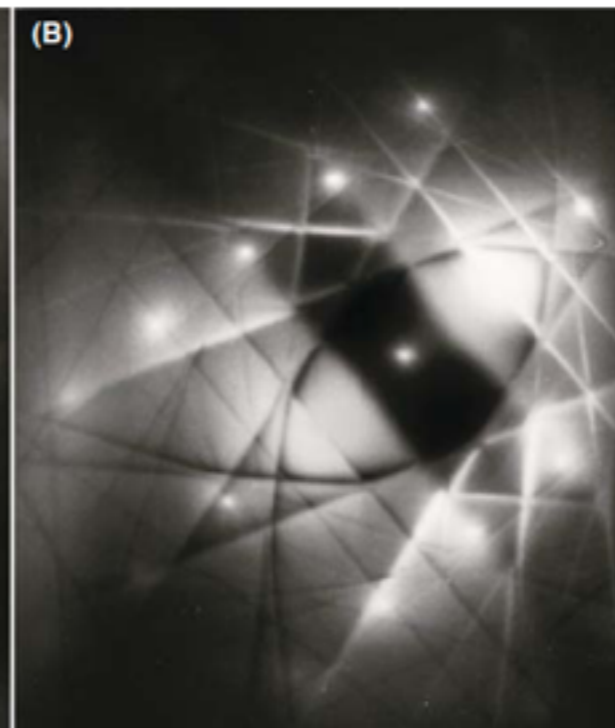
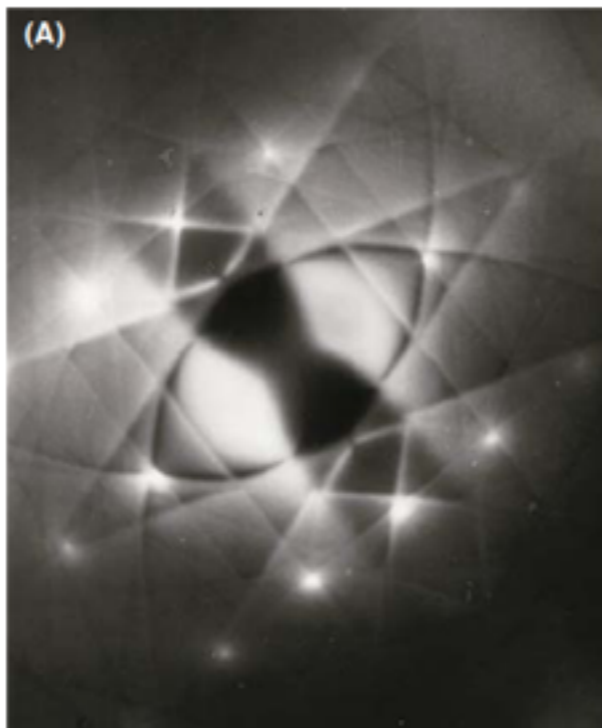
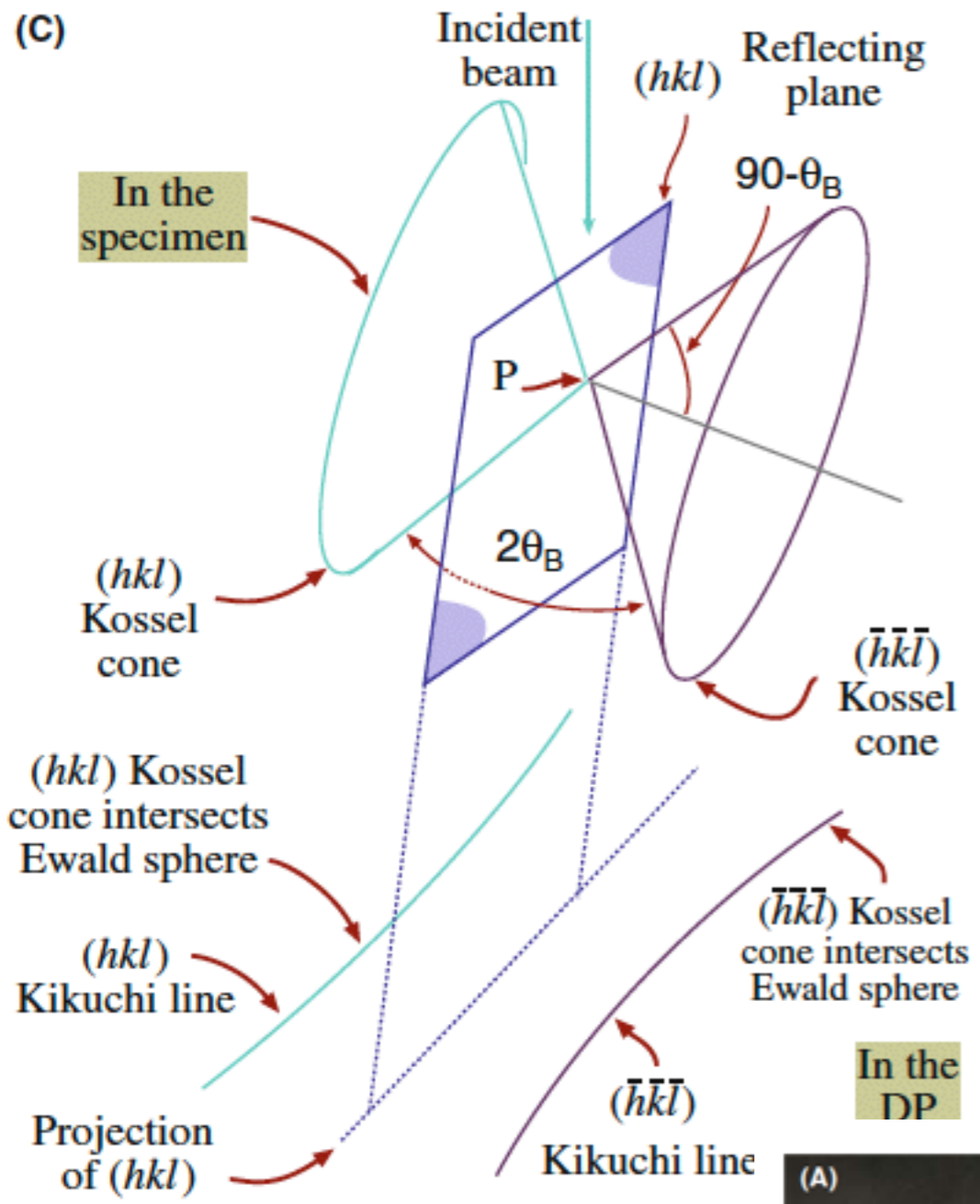


- 被 **diffusely** 散射的電子在樣品內類似一球形（拉長）的光源，接著被彈性散射
- 因為散射電子由各方向來（並非單一的 k 向量）而非是一範圍的 k 向量，因此形成一系列的繞射圓錐稱為 **Kossel cones**
- 繞射圓錐都是以一對出現，
 $\pm g, \pm 2g$

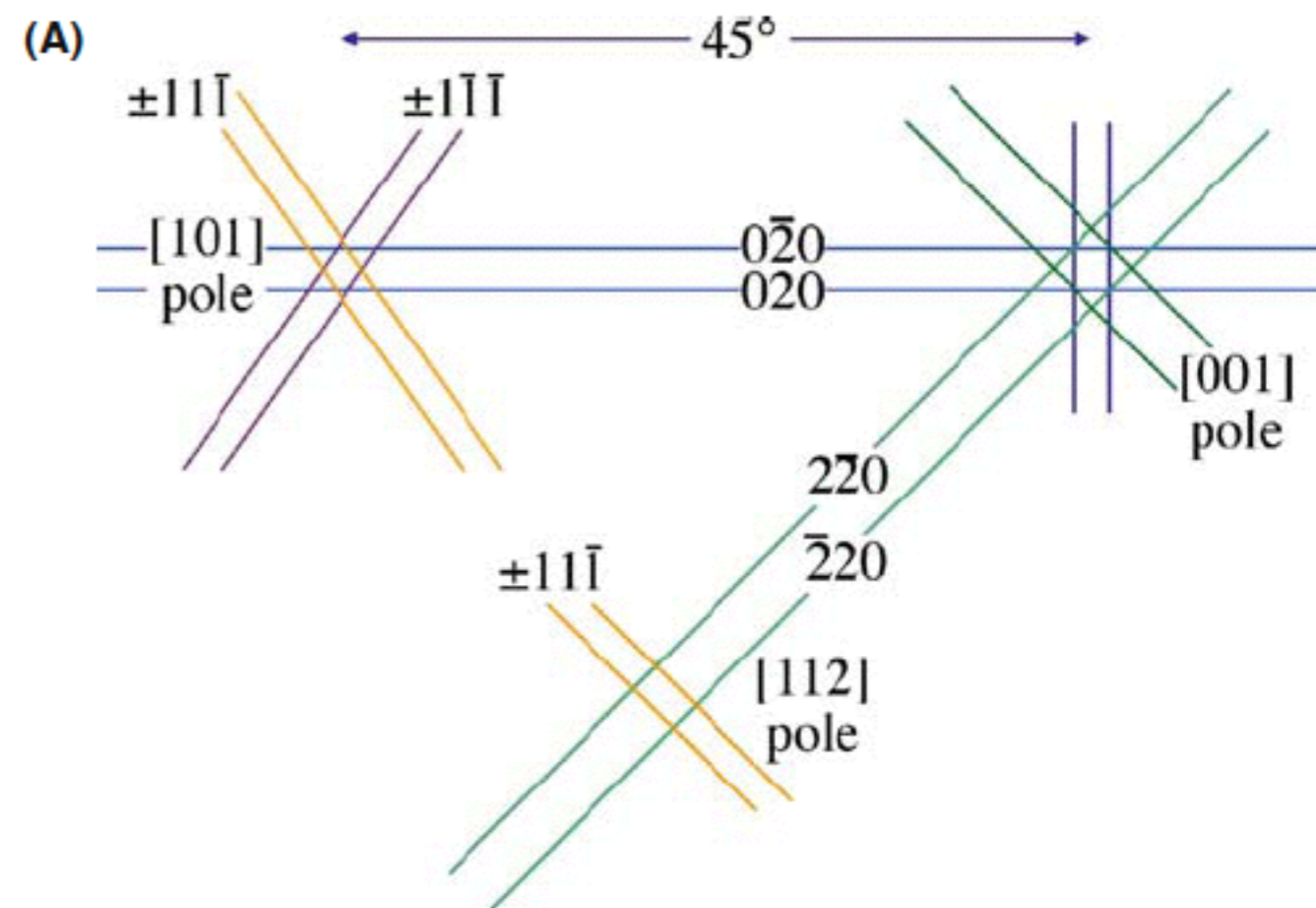
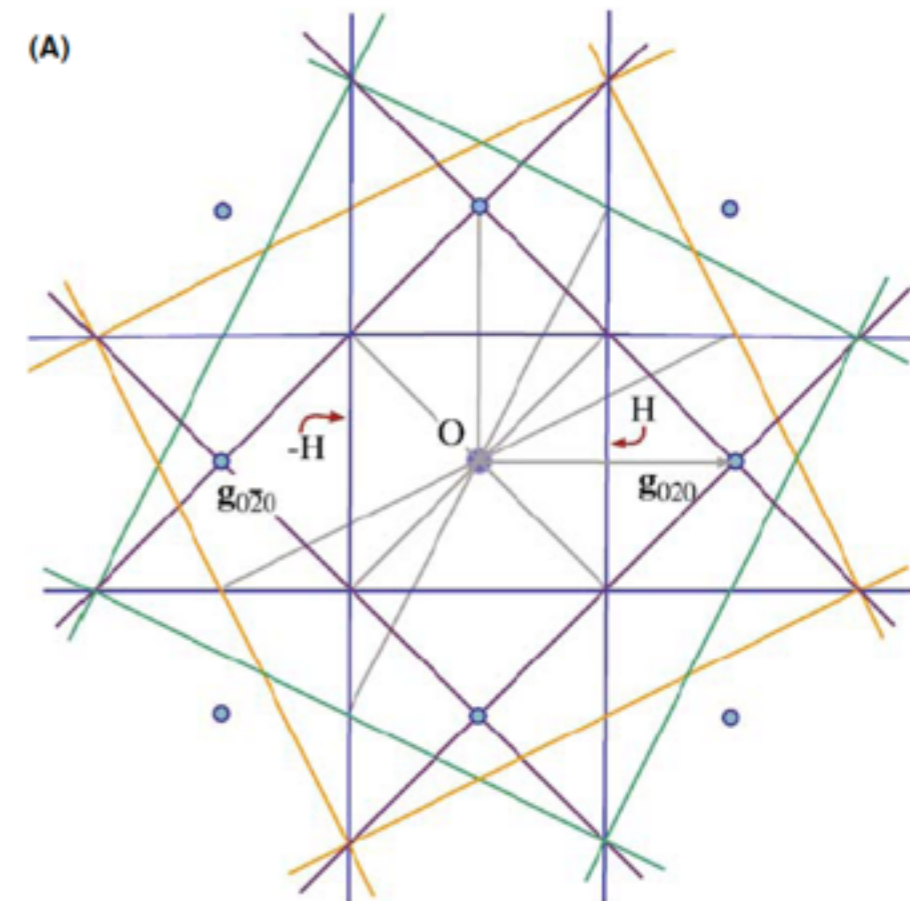
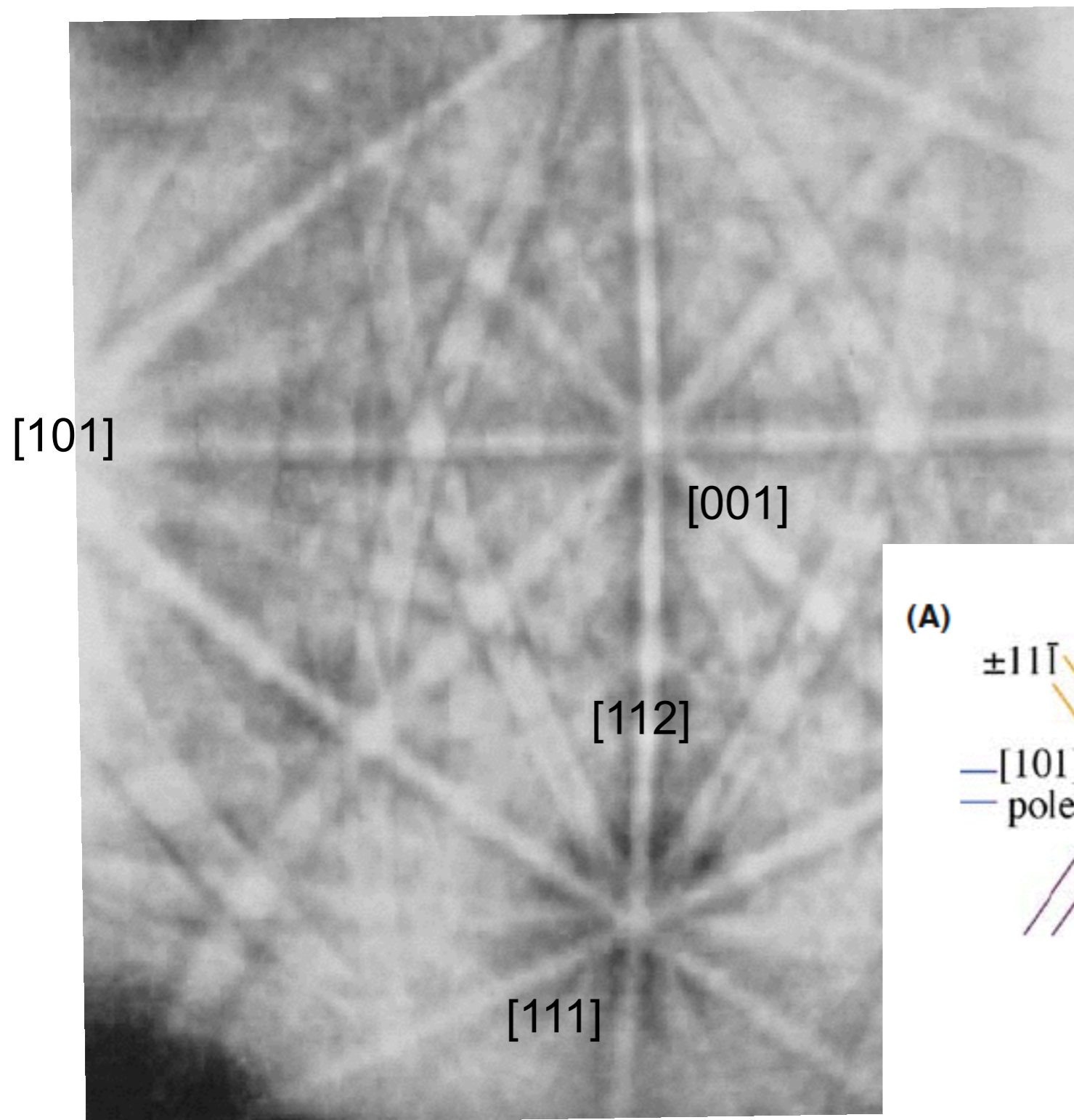


- 在繞射圖形（倒晶格空間看到的是Kossel cones看Ewald球的交線
- 因Ewald球近似平面，故交線是一對拋物線，而在靠近光軸時這對拋物線近似直線
- 我們通常稱為菊池現或菊池帶(Kikuchi lines or Kikuchi band)
- ~Kikuchi(1928)在電鏡還未發展之前就已解釋這現象
- Kikuch band的寬度 $\sim \frac{|g|_{hkl}}{d_{hkl}}$
- Kossel cone的角度 = $2\theta_B$
- Kikuchi lines在Bending極高應變的薄樣品中看不見
- 聚光束比平行光更清楚看到Kikuchi pattern

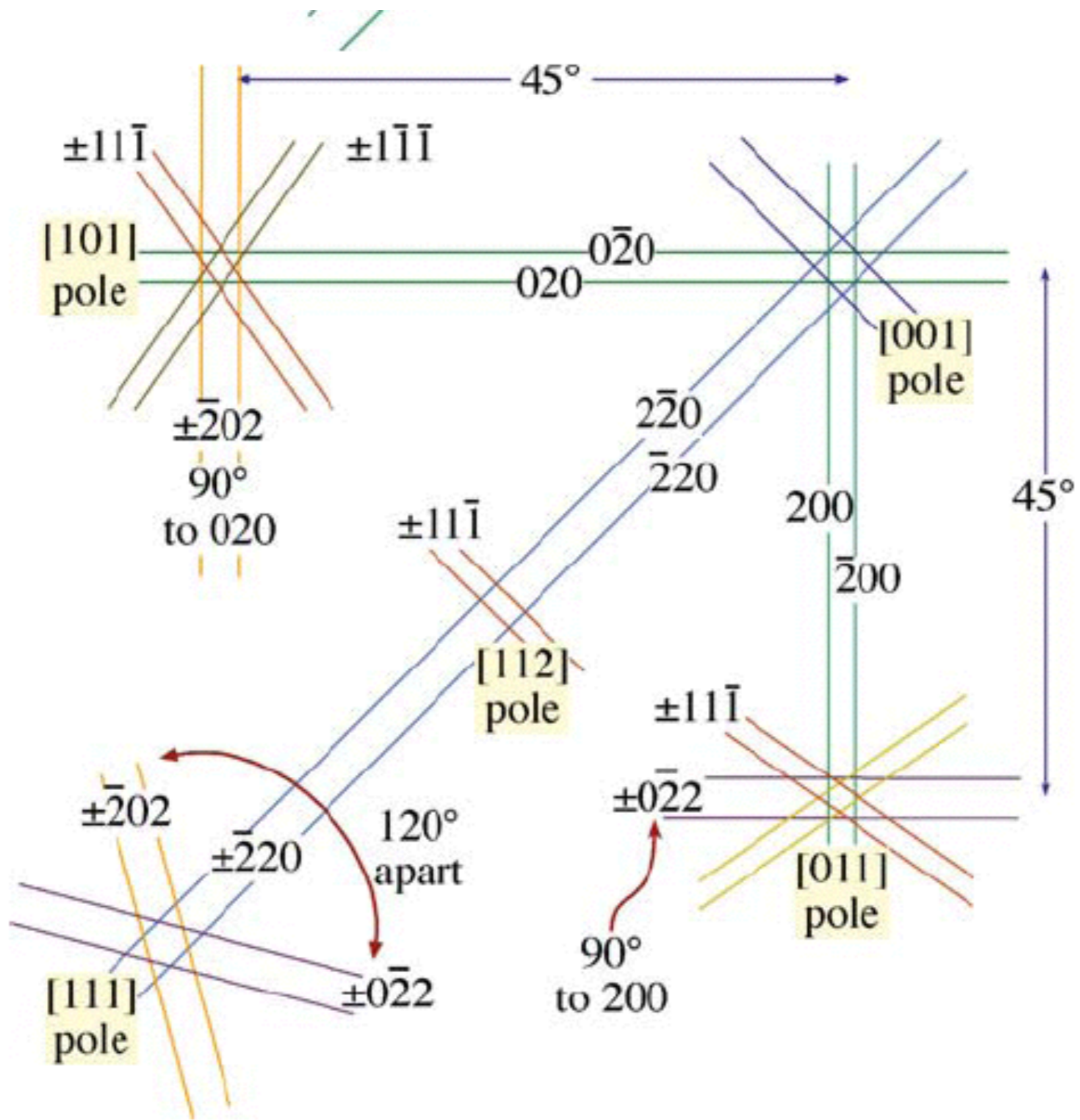




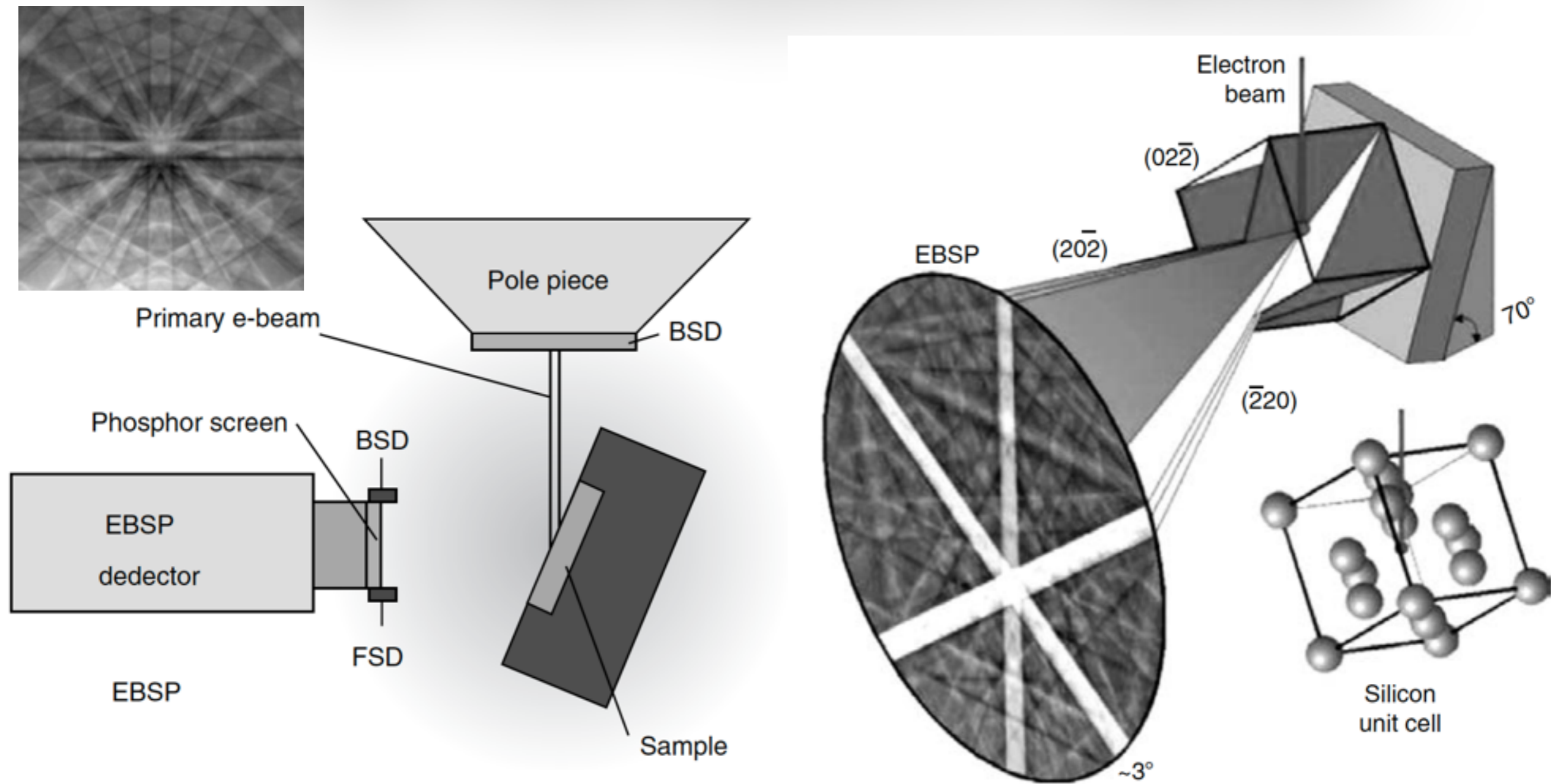
Kikuchi pattern at low camera length



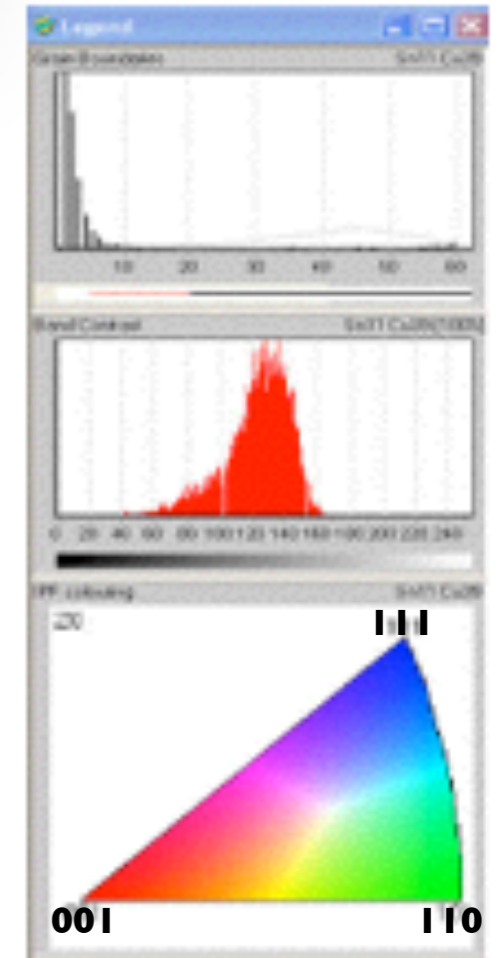
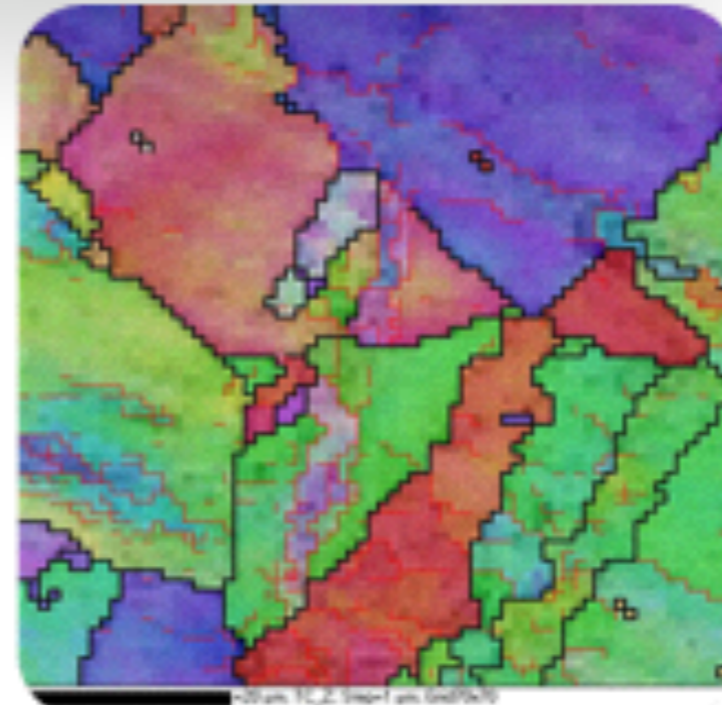
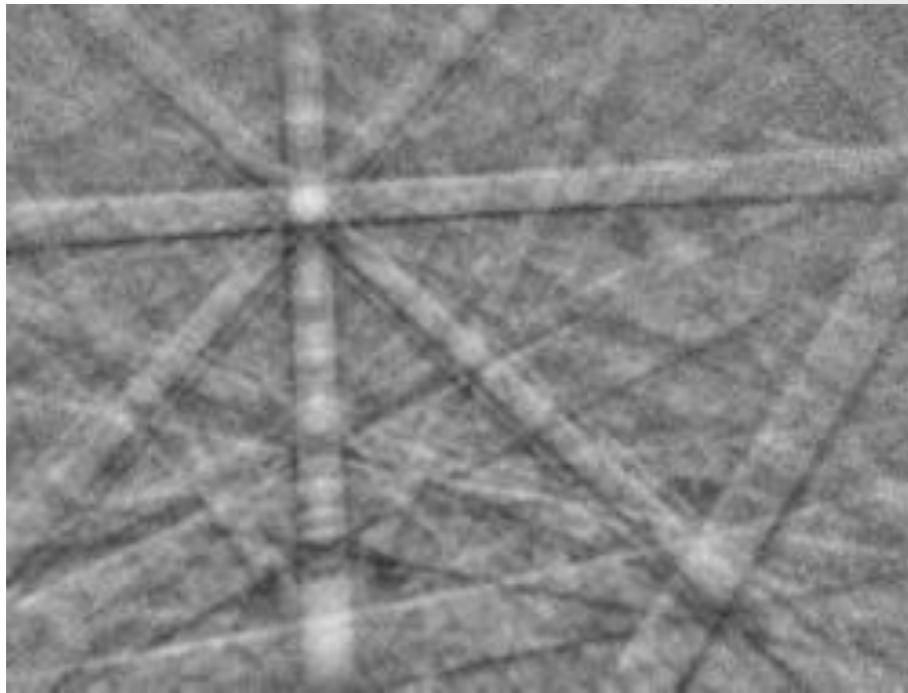
(B)



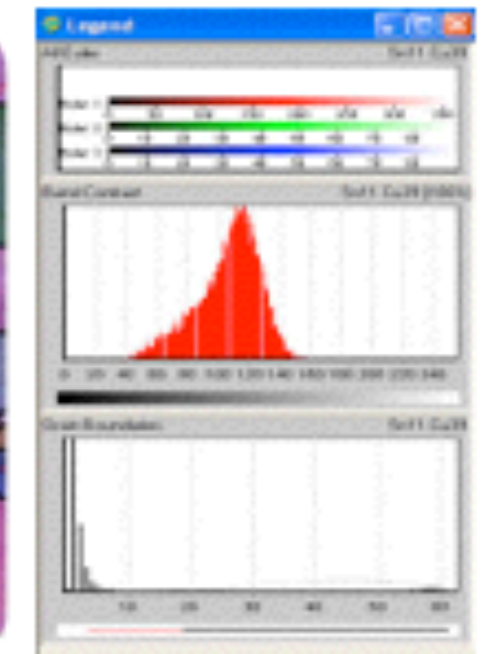
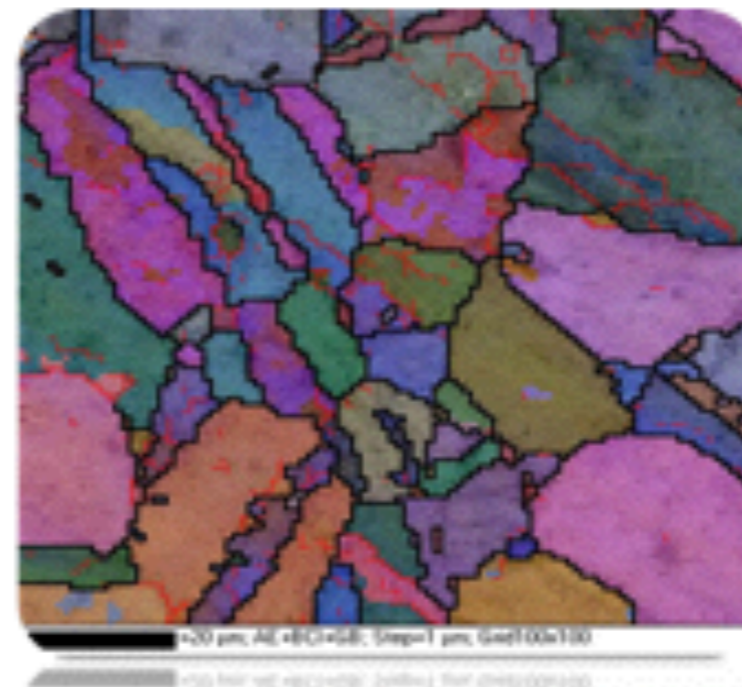
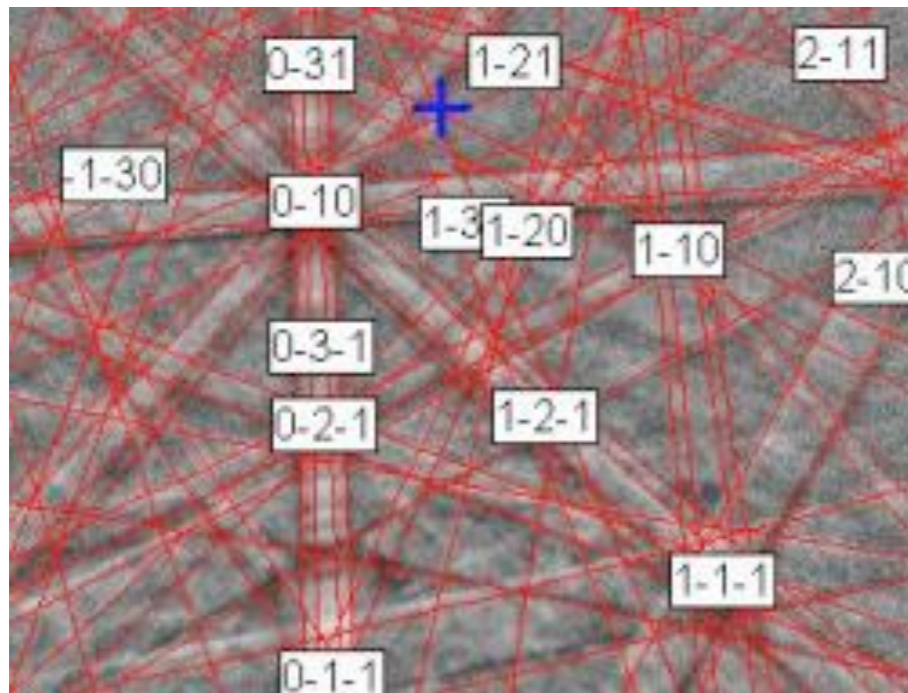
Back Scattering Electron Diffraction

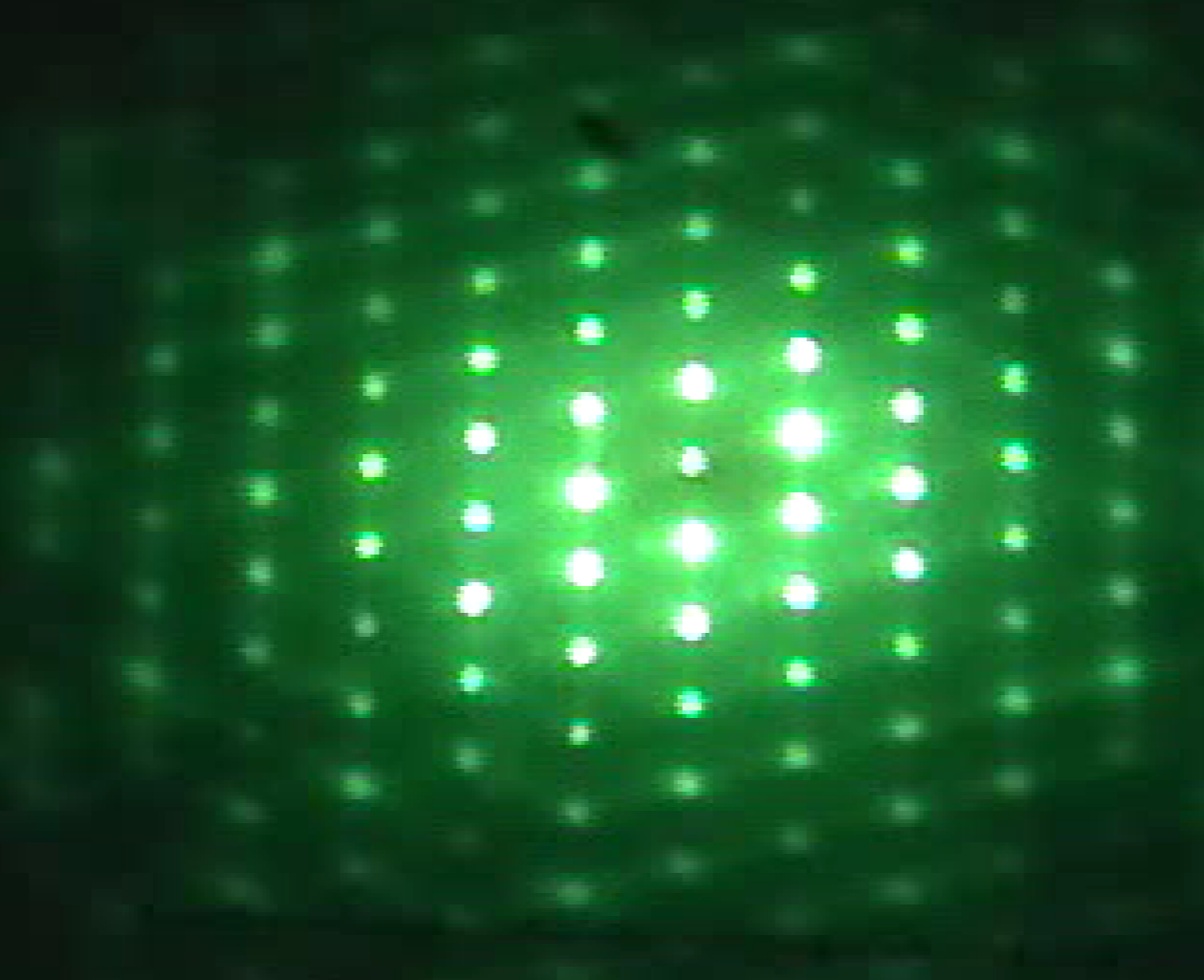


Crystallographic Map



▲ 由Z軸方向觀察的各晶面所佔的比例圖。
▲ 可估算出晶粒大小。

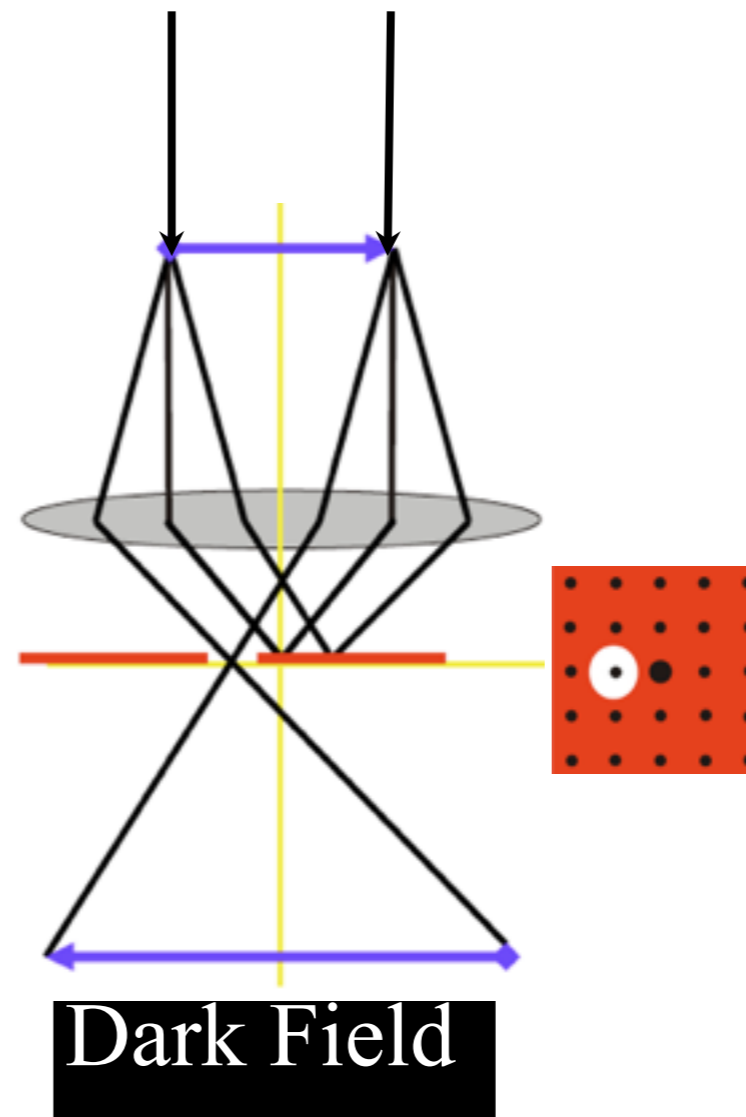
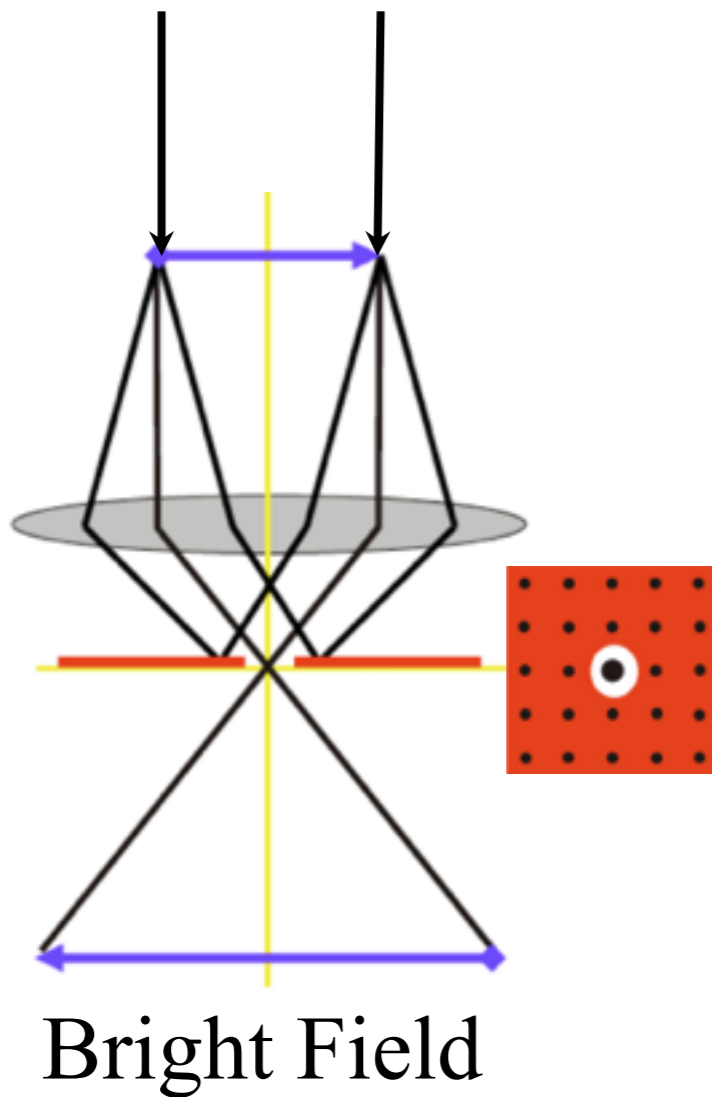


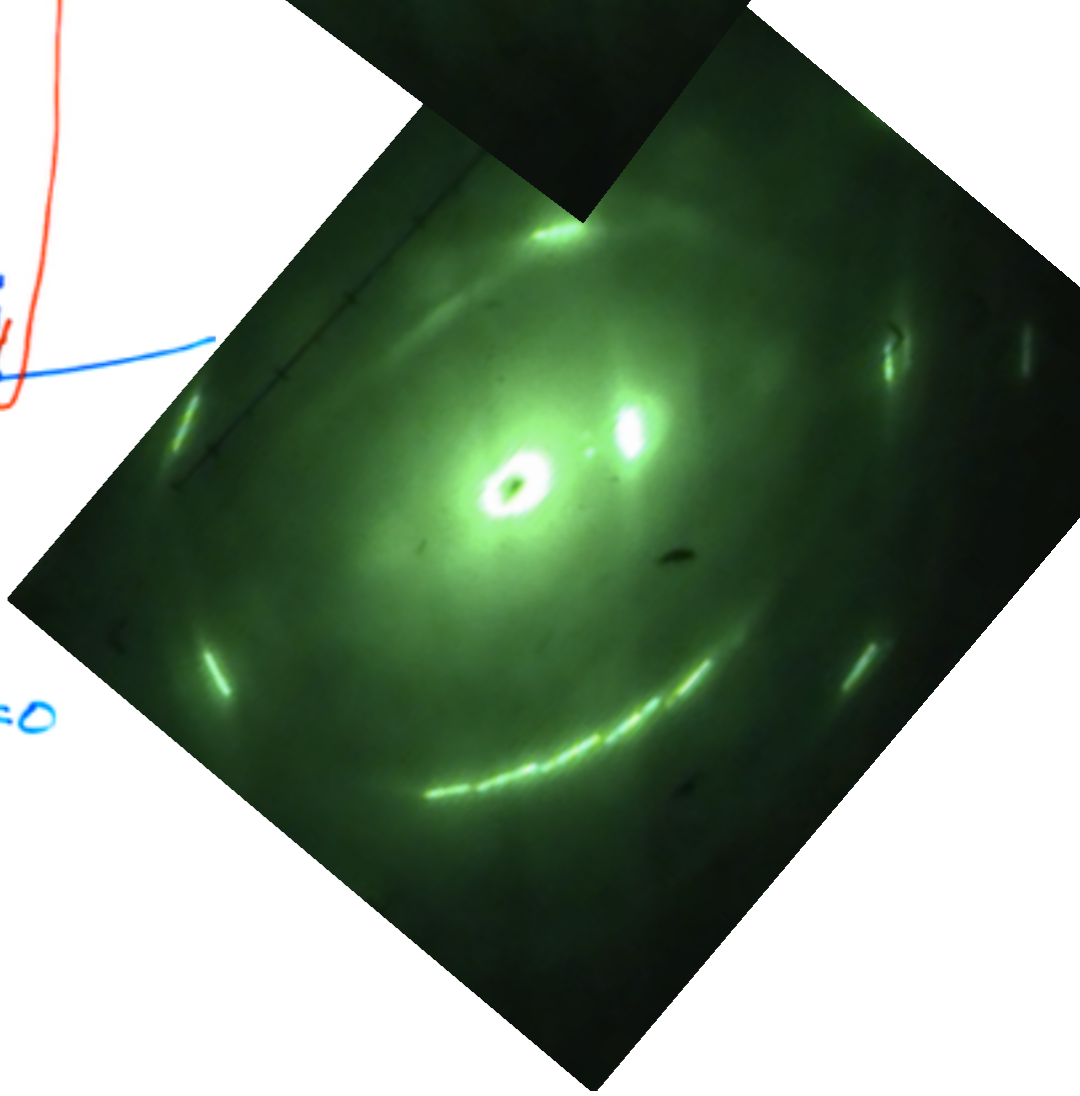
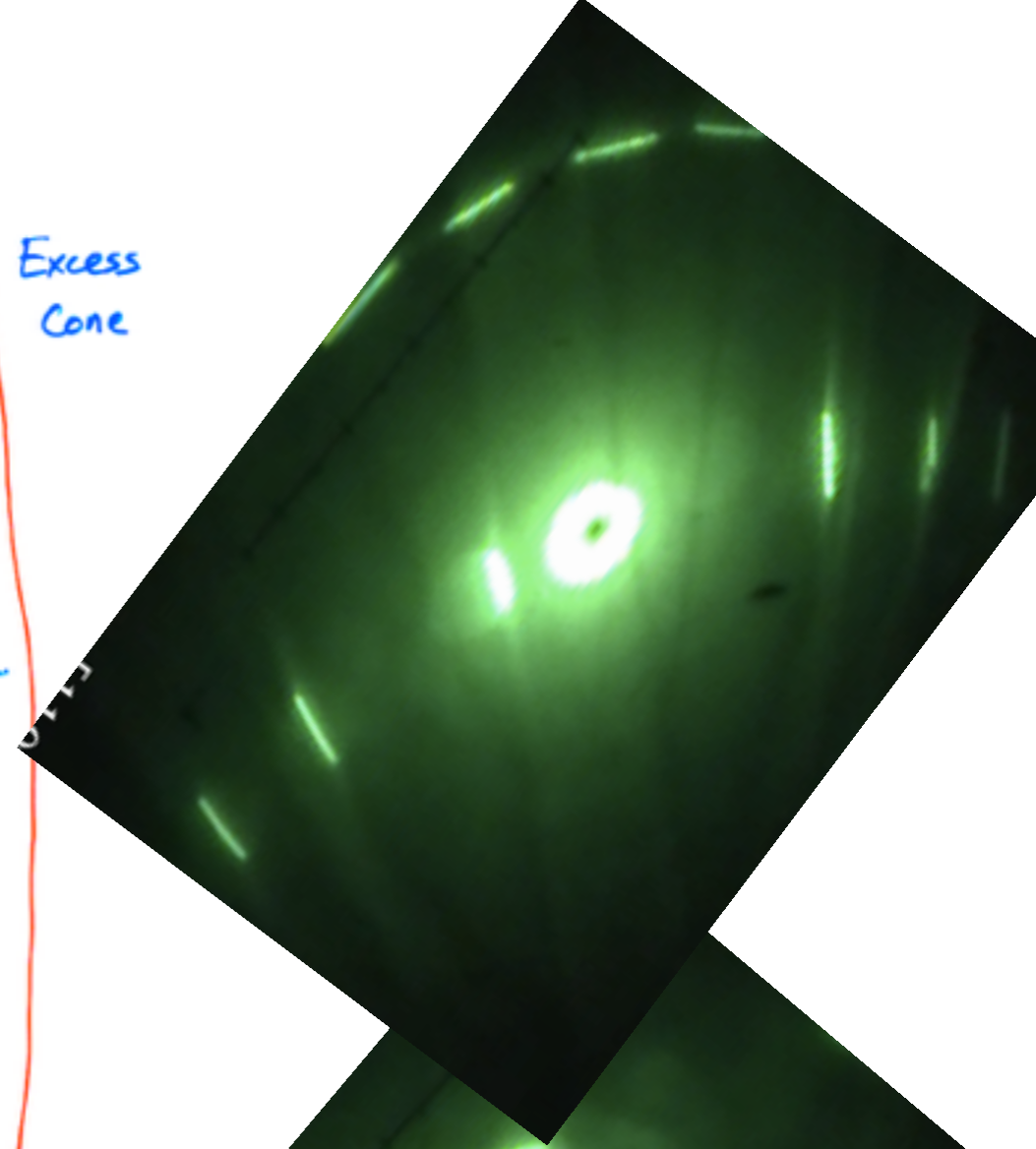
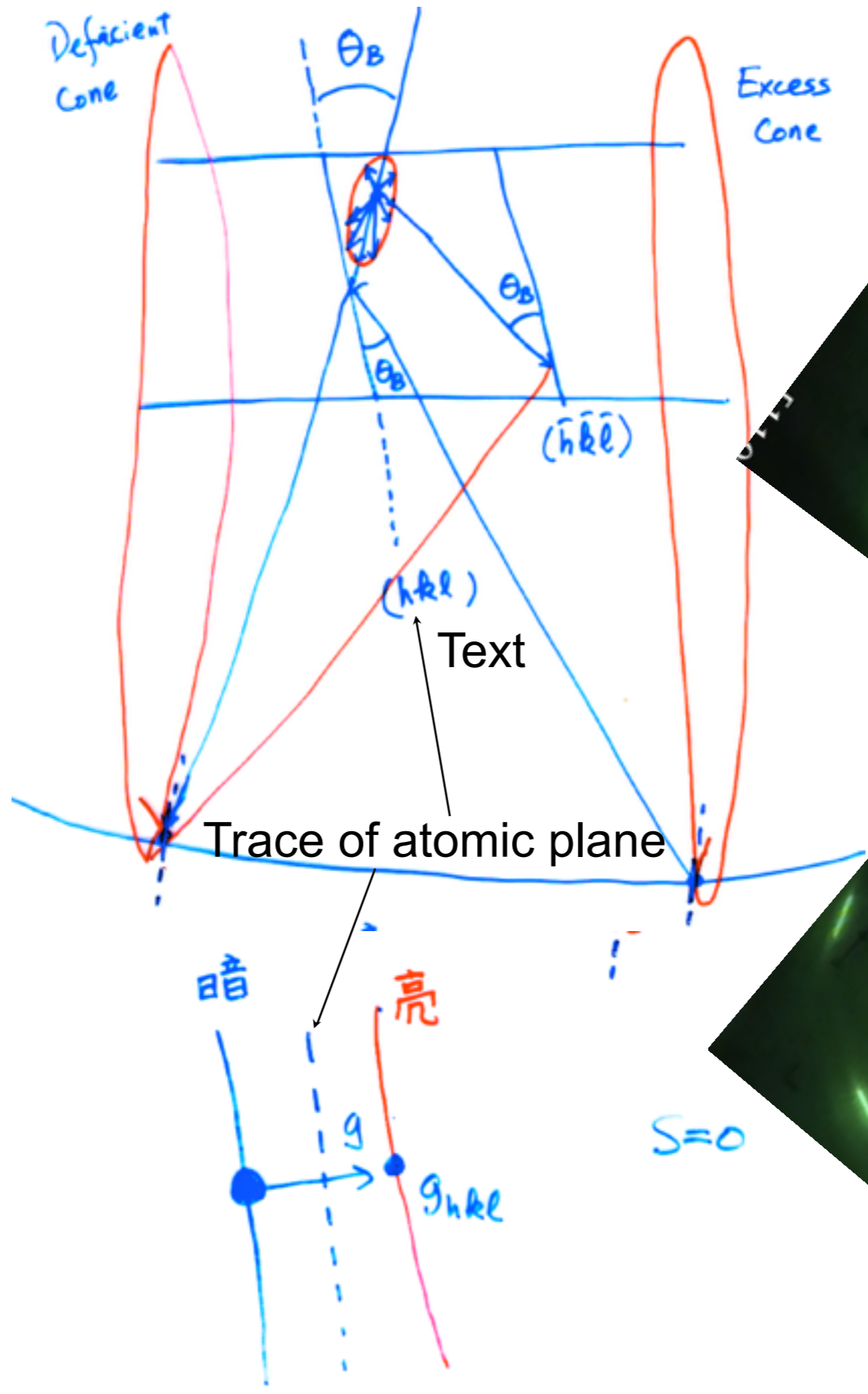


[110]Si

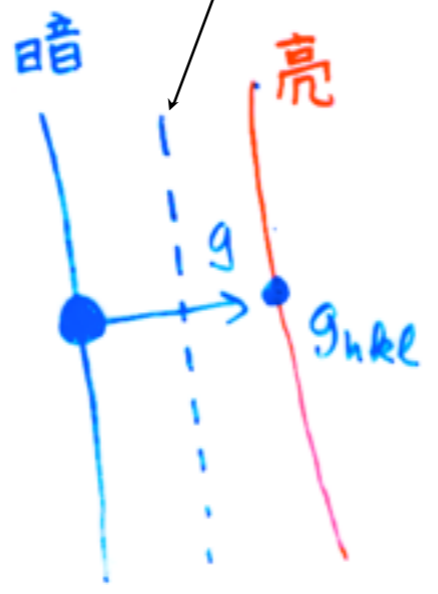
Kinematic Contrast

- mass/ thickness contrast
- Bragg (orientation) contrast

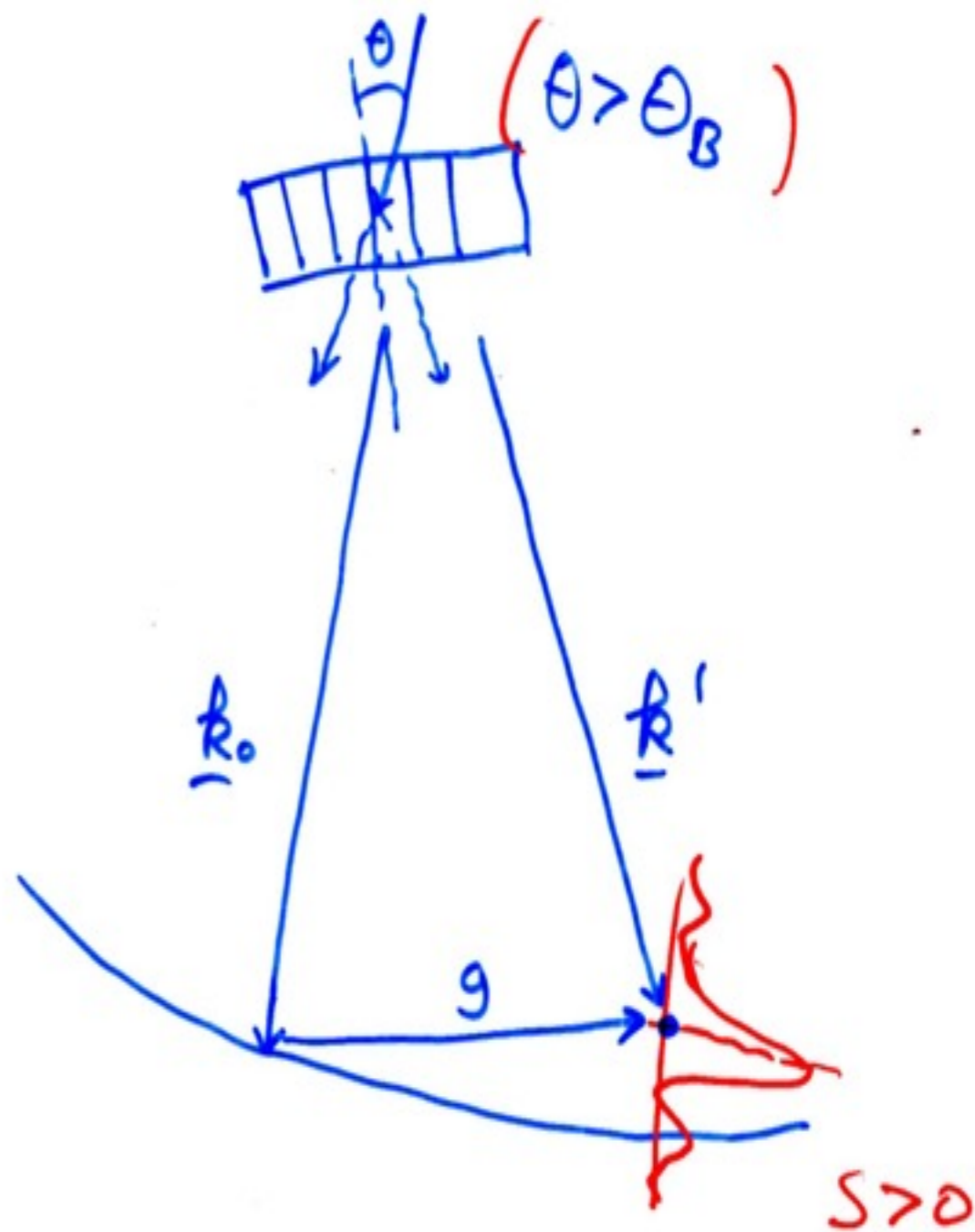
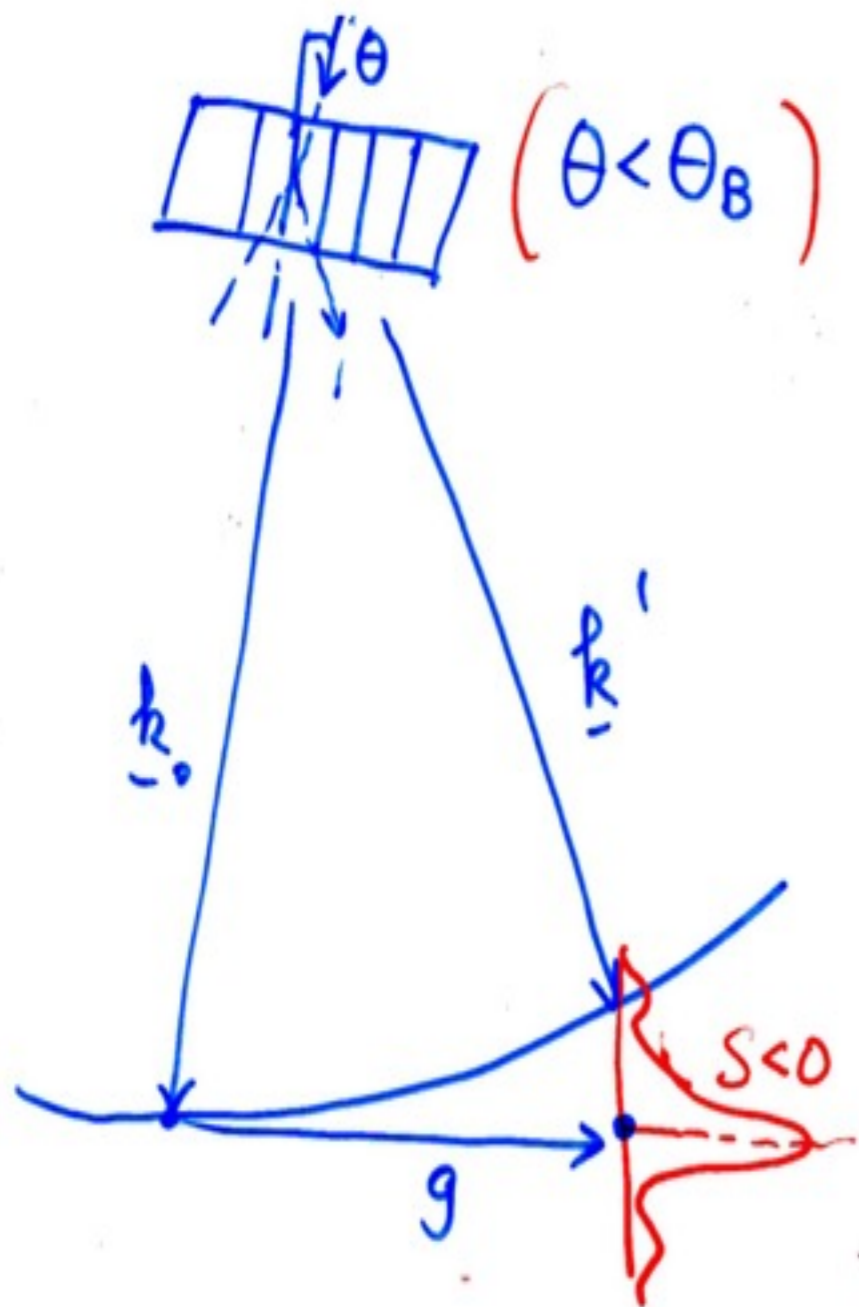




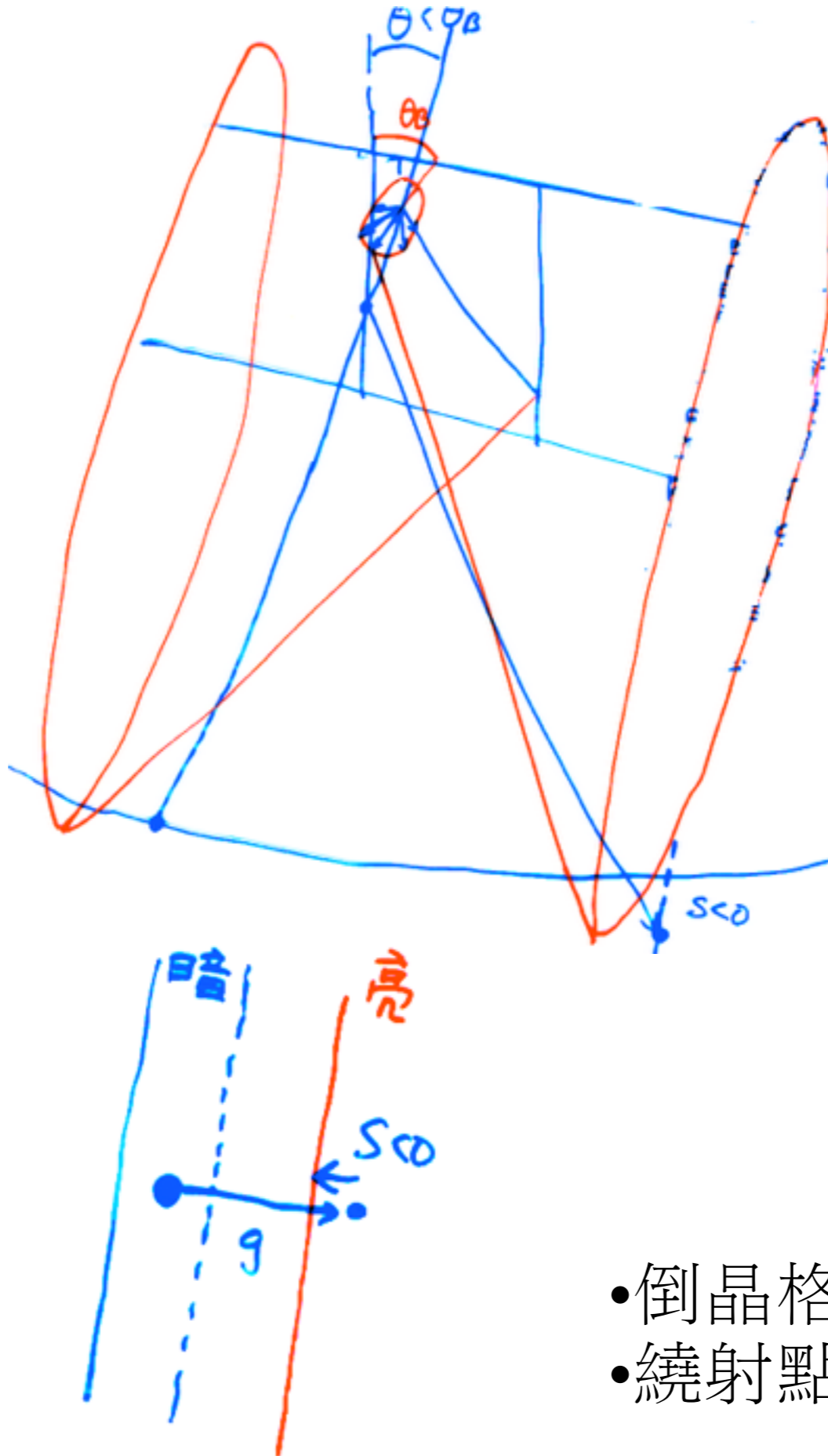
當 $S=0$ 時穿透光
及繞射光各在
deficient and
Excess line上



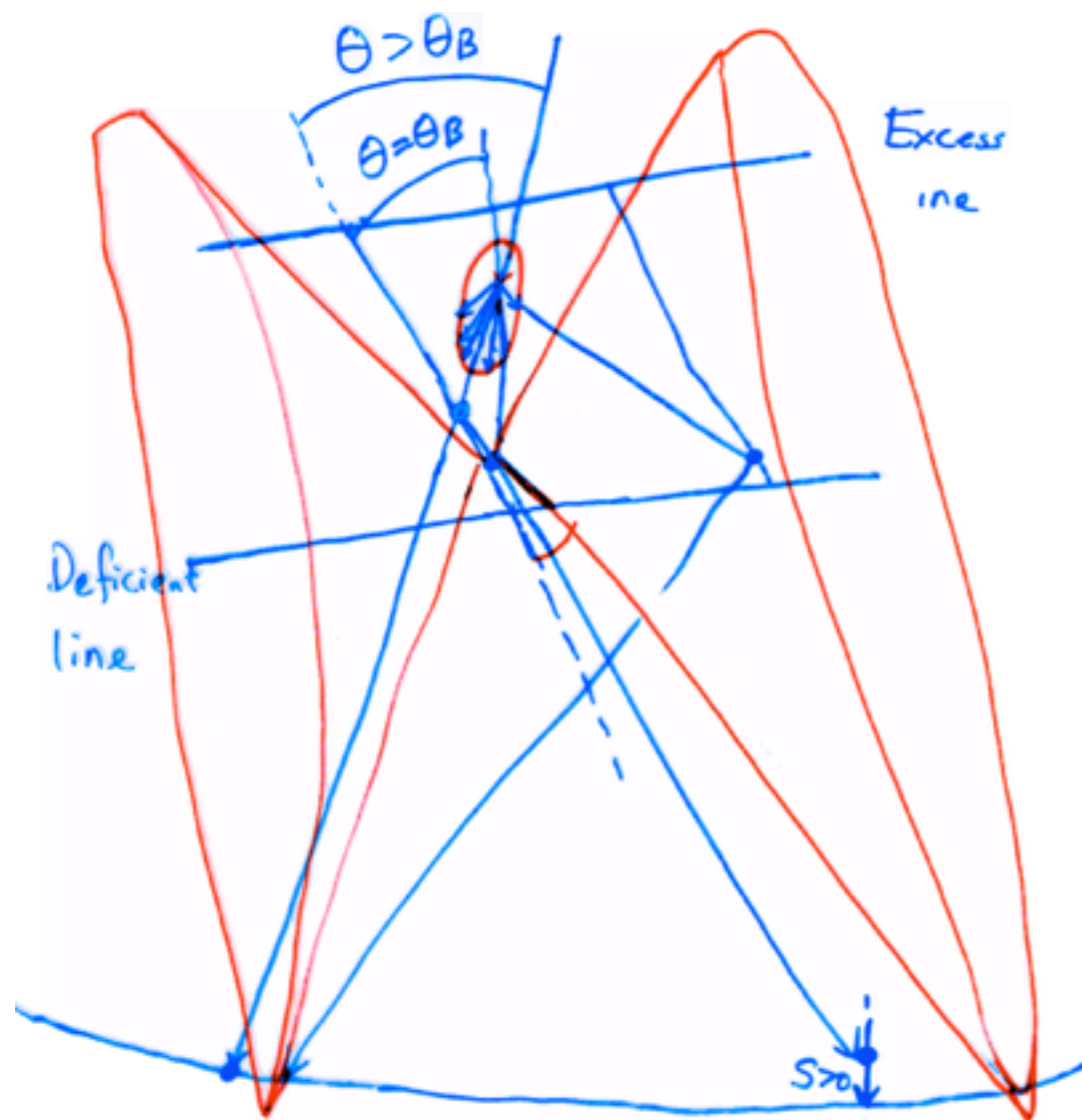
習慣上， $S > 0$ 倒晶格點在 Ewald 球內部； $S < 0$ 倒晶格點在 Ewald 球外部



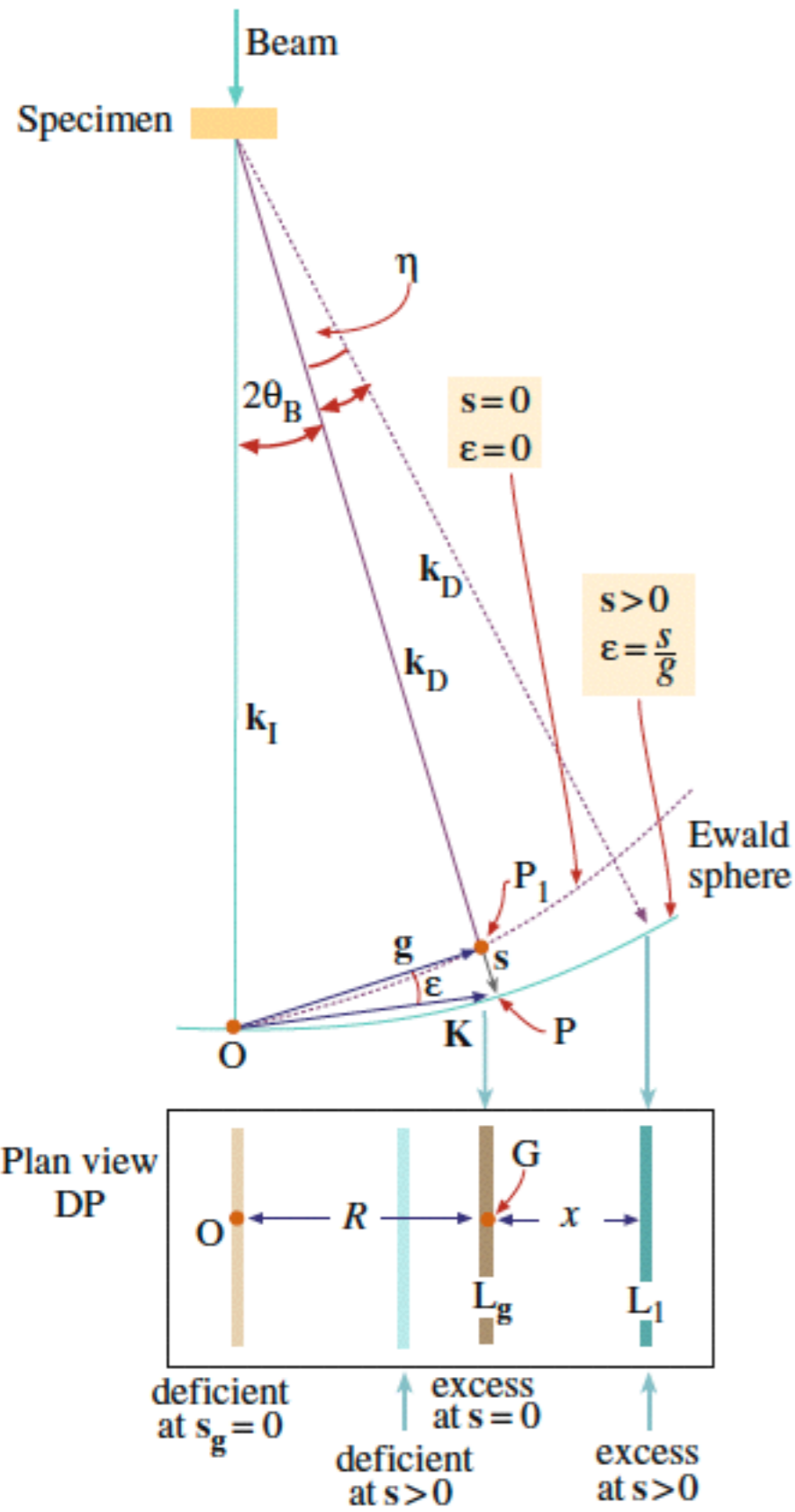
- **Kossel Cone**
好比被釘在
晶體平面上
- 晶體被傾轉
Kossel Cone及
倒晶格亦隨著
傾轉
- 因倒晶格沿著
樣品表面鬆弛，
故即使傾轉在10
度上下，內繞射
點僅是強度變化
，而位置不變
- 但**Kikuchi line**對
晶向的改變就很
敏感



- 倒晶格點在Ewald
- 繞射點在Excess line 外



- $S > 0$ 時，倒晶格點在Ewald球之內側
- 繞射點在Excess line之內側



Again, with our small-angle approximation, the distance between the excess and deficient Kikuchi lines, R (the distance g measured on the DP), is equivalent to $2\theta_B L$. So, using Bragg's law, we have

$$\frac{R}{L} = 2\theta_B = \frac{\lambda}{d} \quad (19.4)$$

Hence the expression for s is

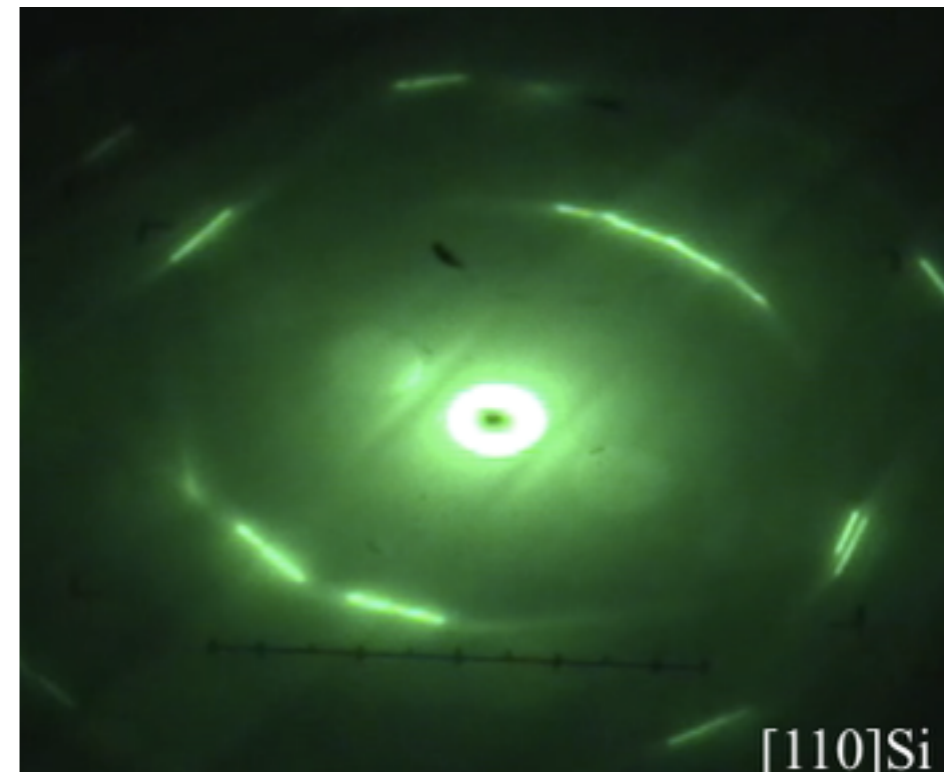
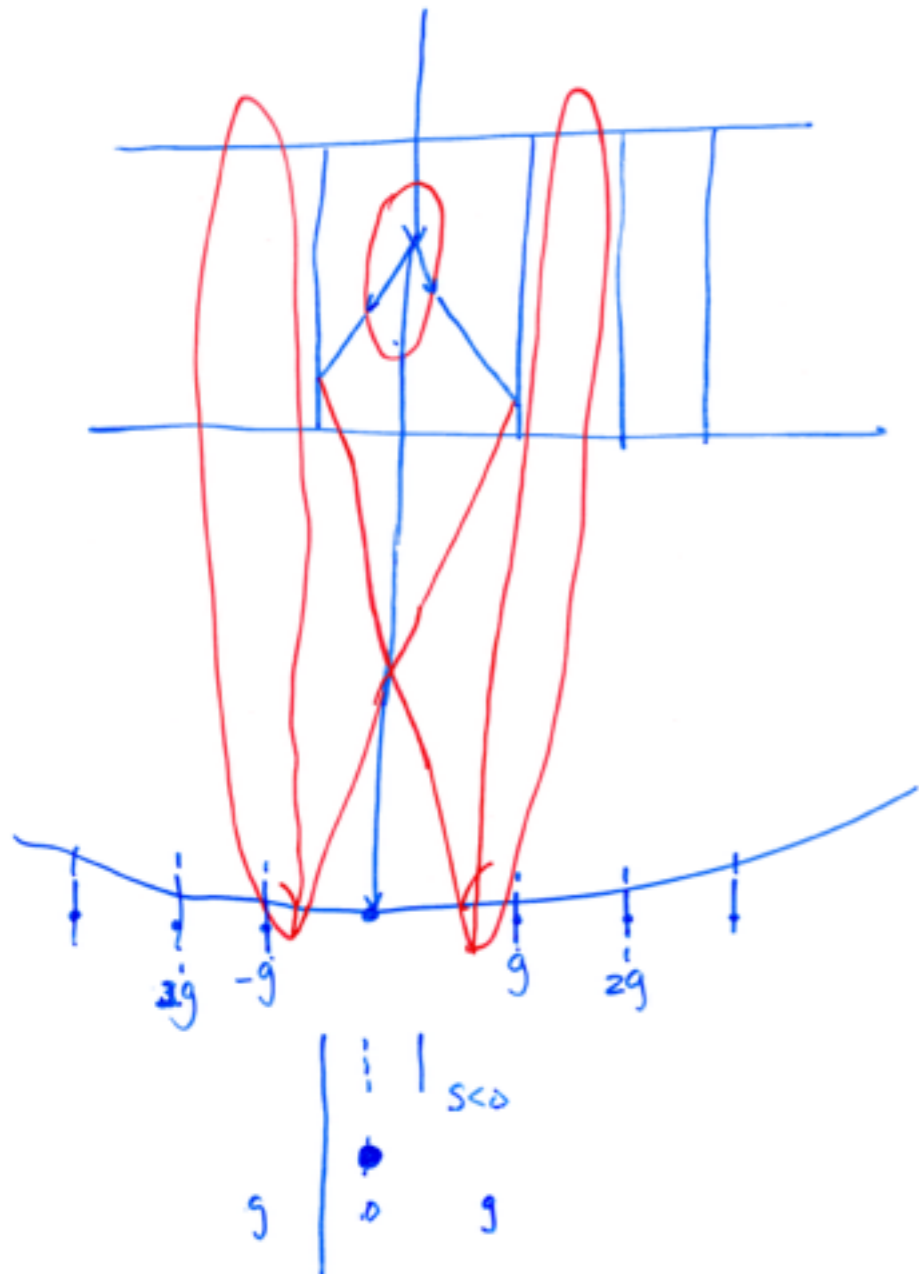
$$s = \frac{x}{Ld} = \frac{x}{d} \cdot \frac{\lambda}{Rd} \quad (19.5)$$

$$s = \frac{x \lambda}{R d^2} = \frac{x}{R} \lambda g^2 \quad (19.6)$$

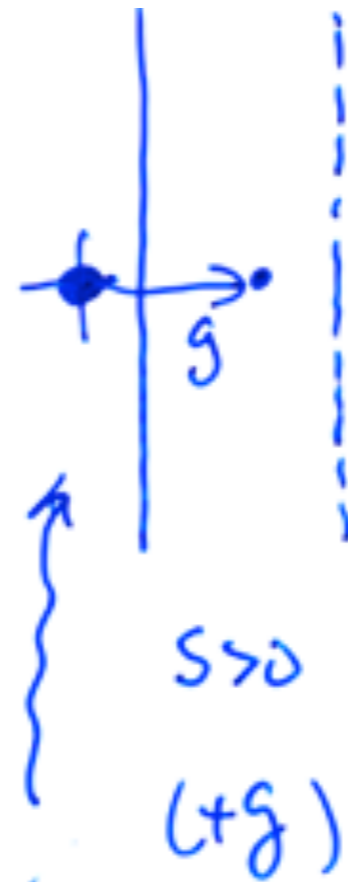
在晶軸時

- 光束平行原子平面，所以的

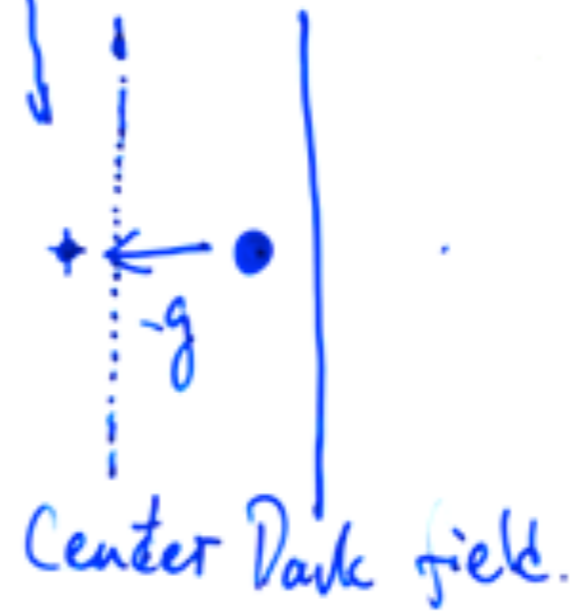
$$\bar{g}, s < 0$$



- Kikuchi lines在 0 及 g 之中央，且亮度相等
 og 及 $o(-g)$ 之平分線



光軸

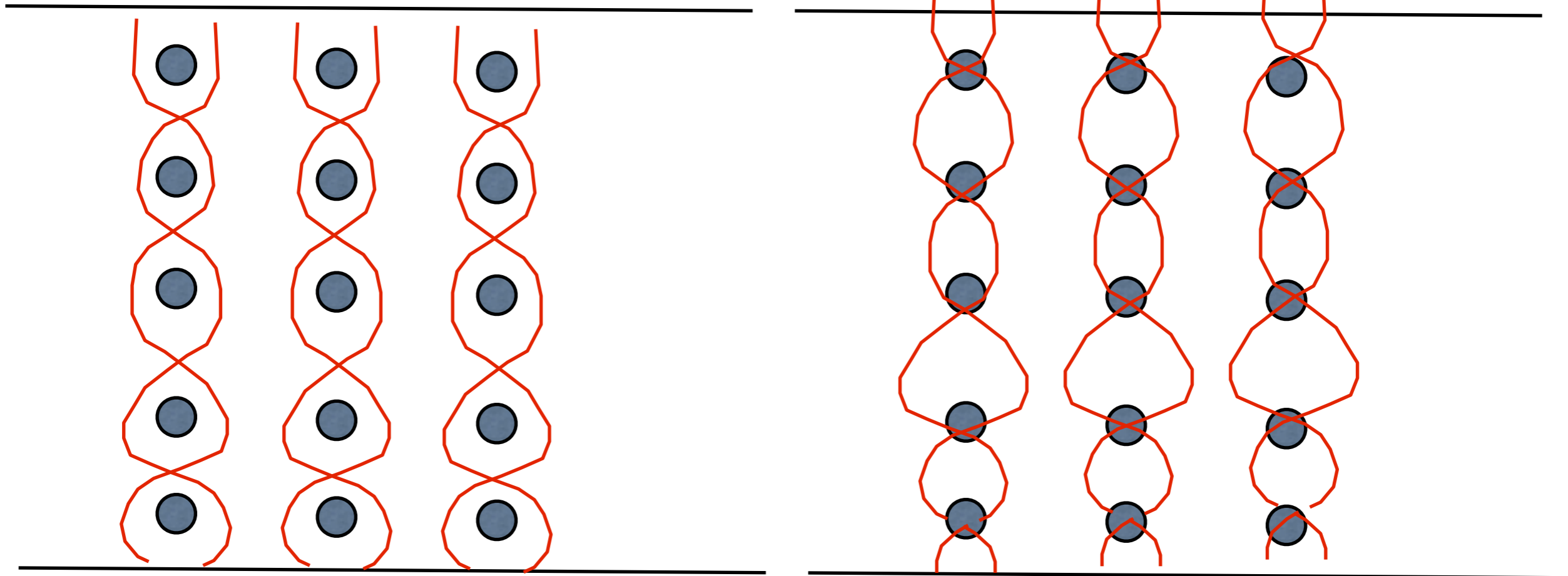


- off-axis dark field 有像差.

Bloch Waves

Bloch wave 1

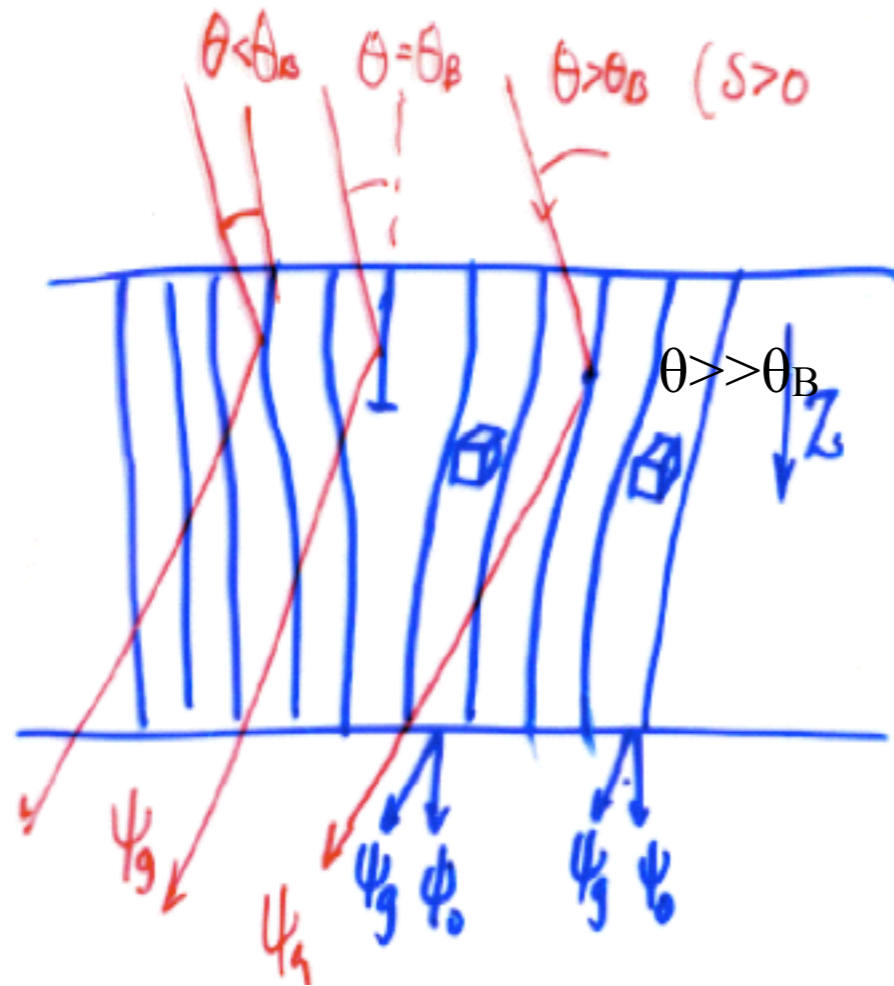
Bloch wave 2



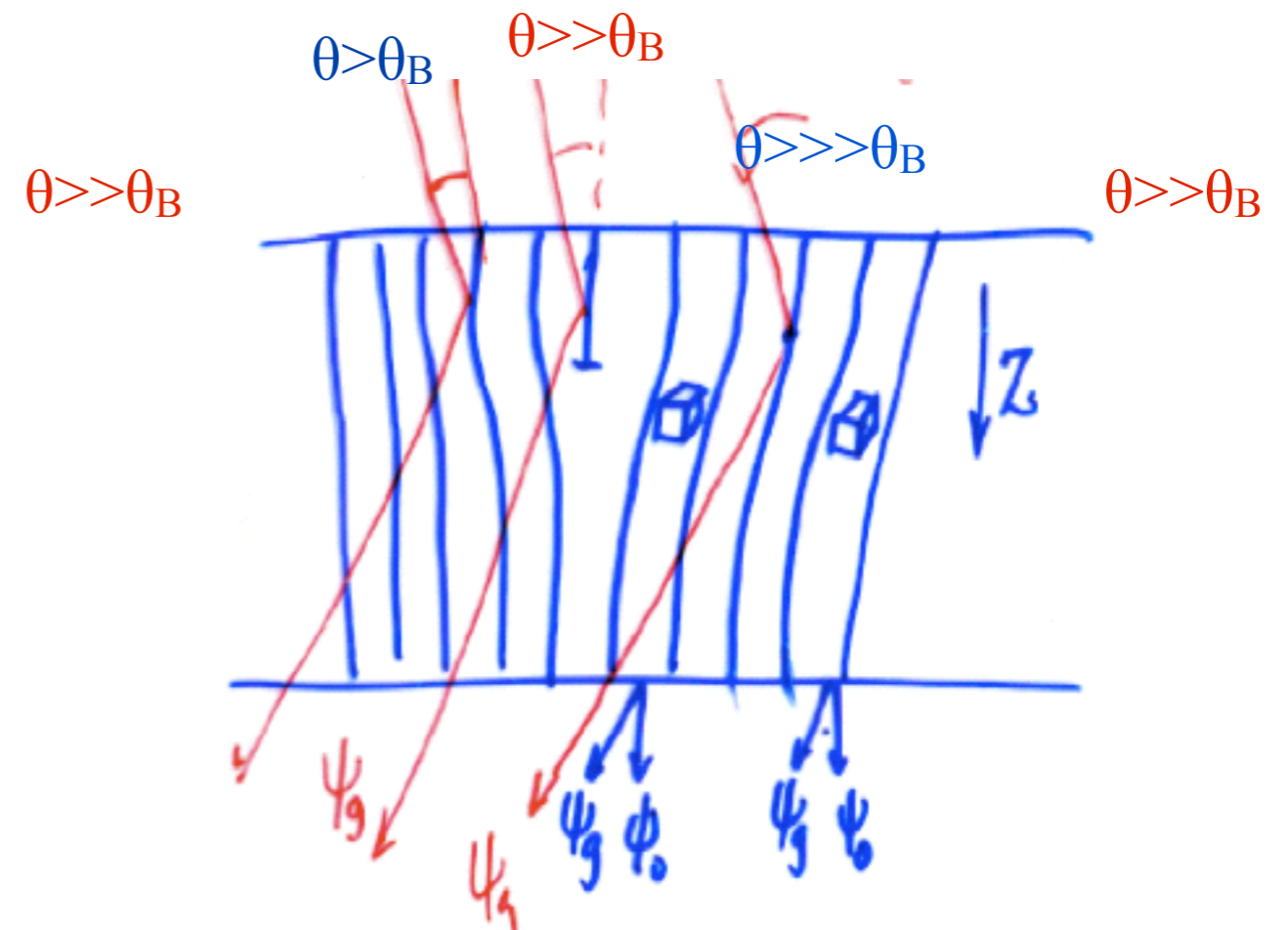
$S > 0$ Bloch wave 1 dominates

$S < 0$ Bloch wave 2 dominates

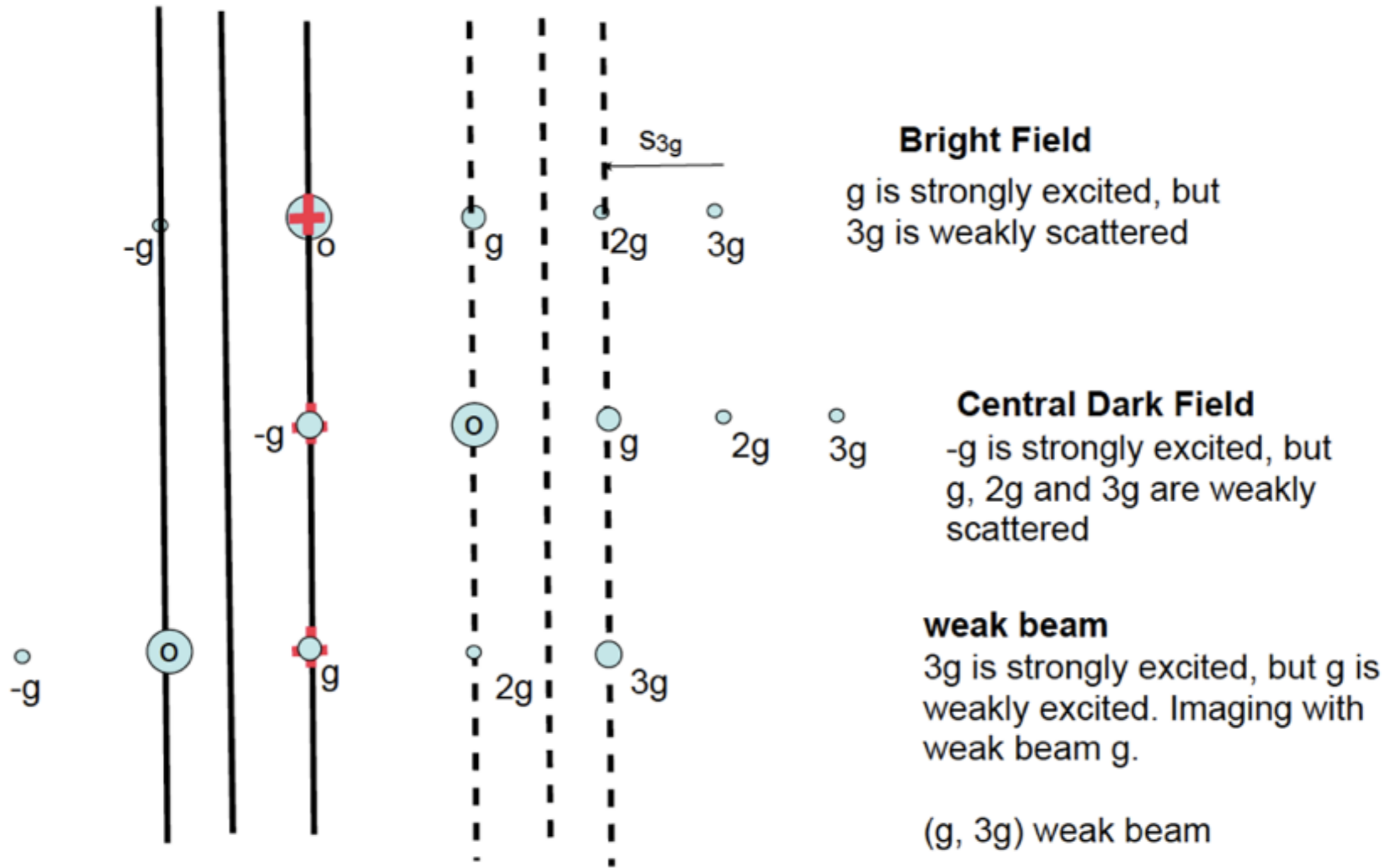
Regular Two Beam



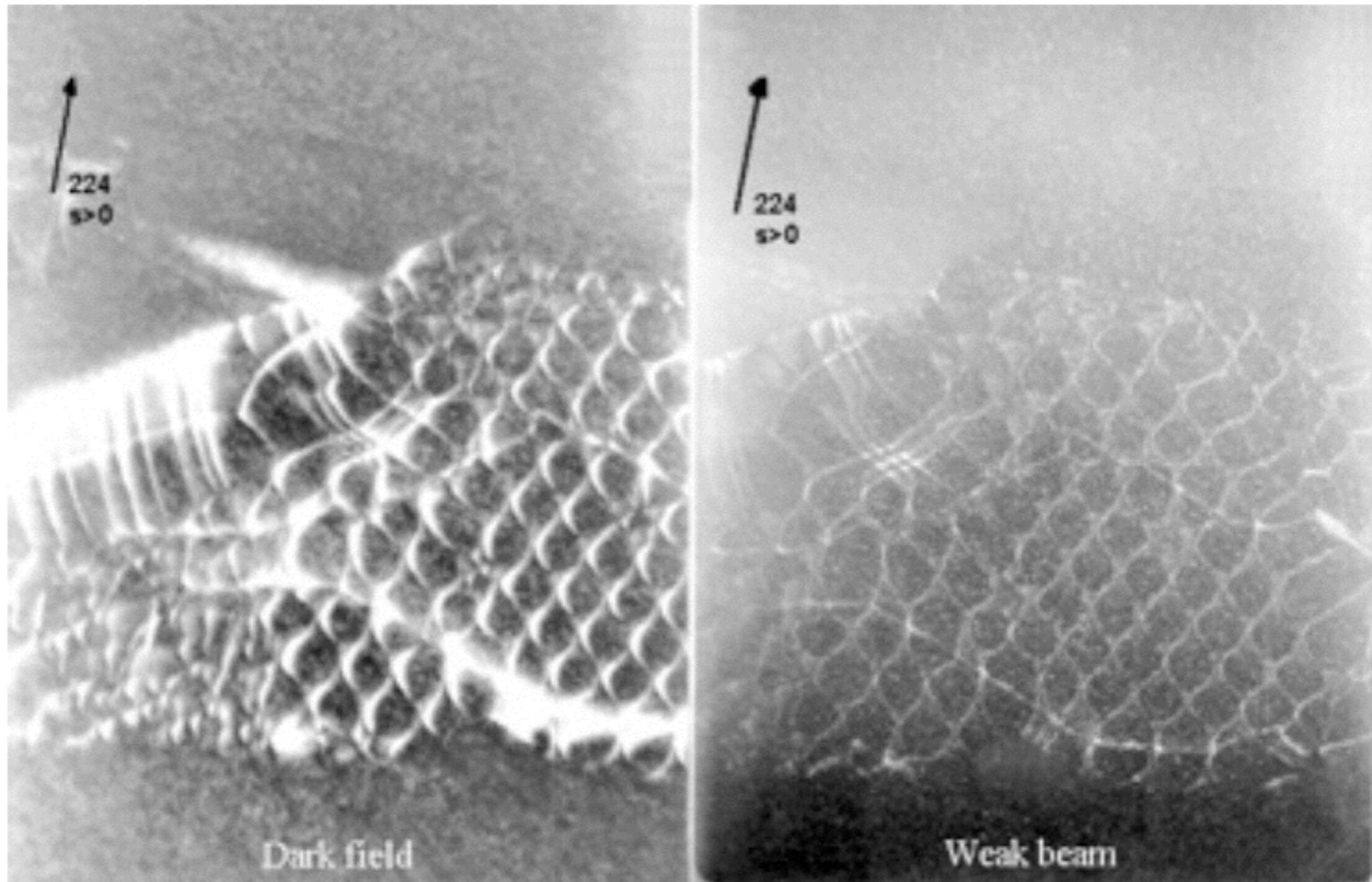
Weak Beam

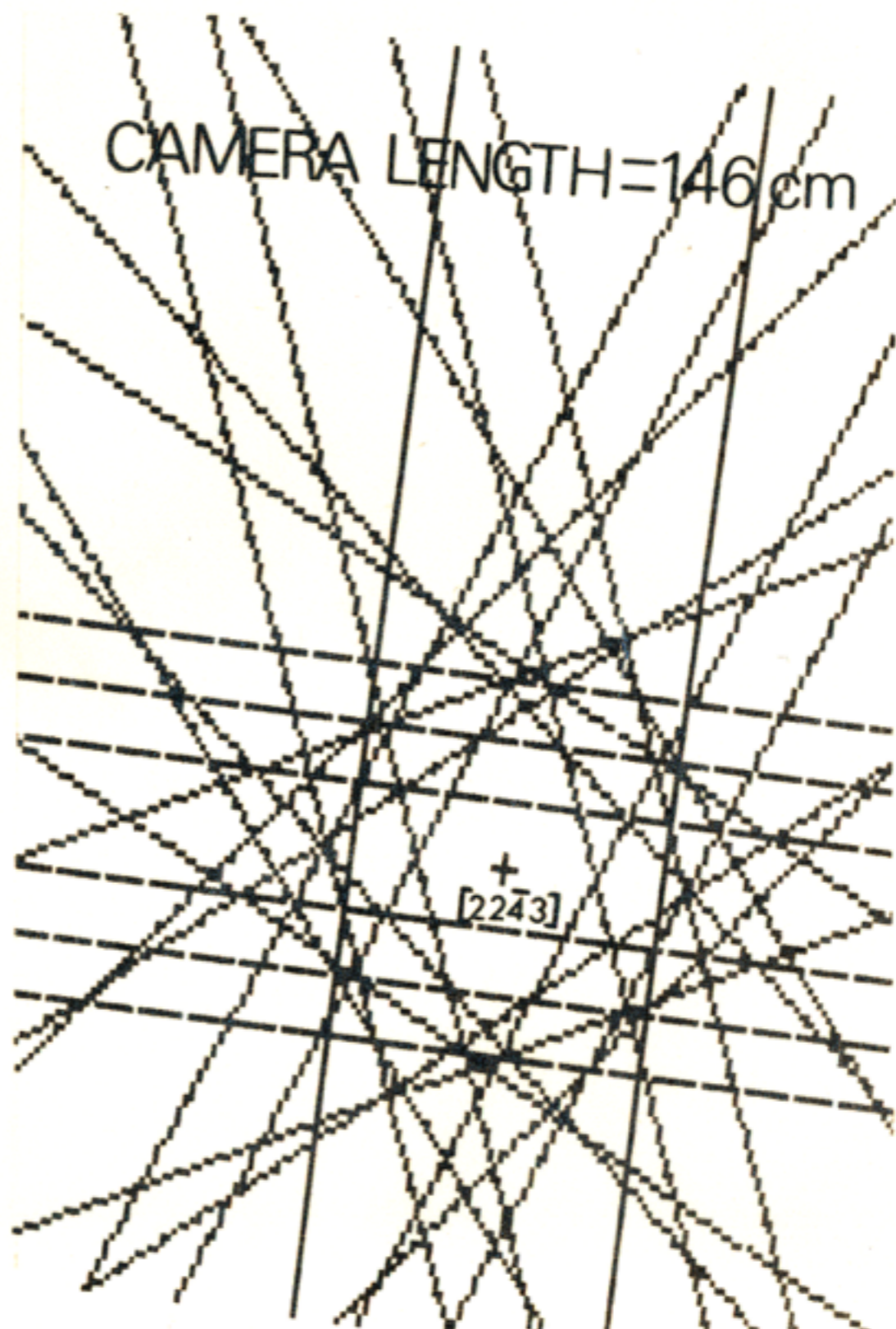
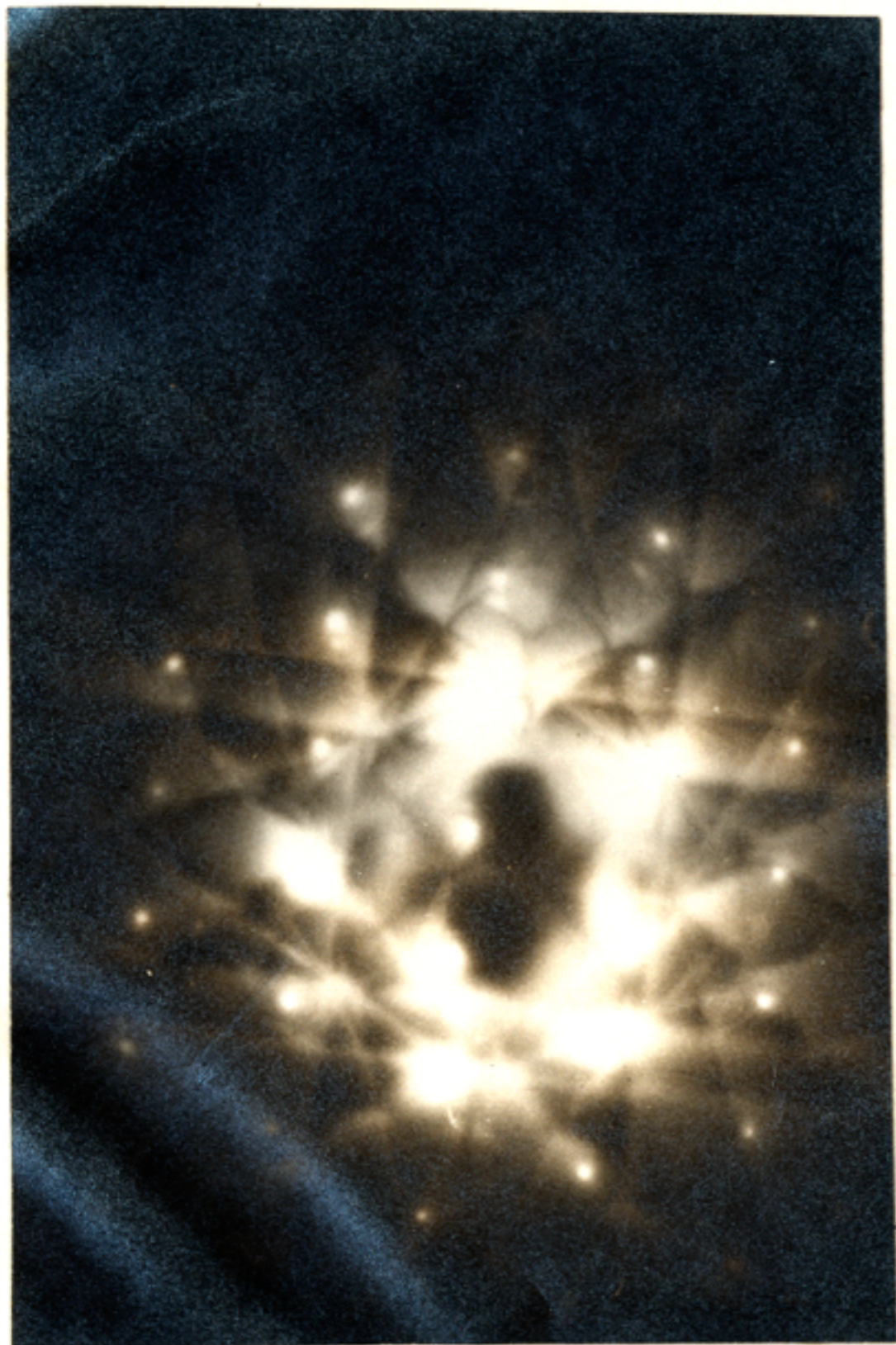


Dark Field and Weak Beam



Dark field and Weak beam





Kikuchi (菊池) 圖的應用

- 因菊池圖比繞射圖形對晶向的改變更敏感，因此菊池圖提供更準確的晶向訊息
- 菊池圖 \sim better than 0.1度
- 繞射圖 \sim better than 10度
- 決定繞射條件 (偏離Bragg's條件之遠近)
 - $s > 0$ 明場像之條件 亮度及對比較差
 - $s \ll 5 \times 10^{-2}$ 弱光束之條件
- 傾轉晶體分析晶體缺陷 $g \cdot R = 0$
- $L\lambda = dx$ 校準相機常數
- 分析晶體缺陷 (位錯, 晶界, 疊差, 位錯還之線
(trace Analysis)

