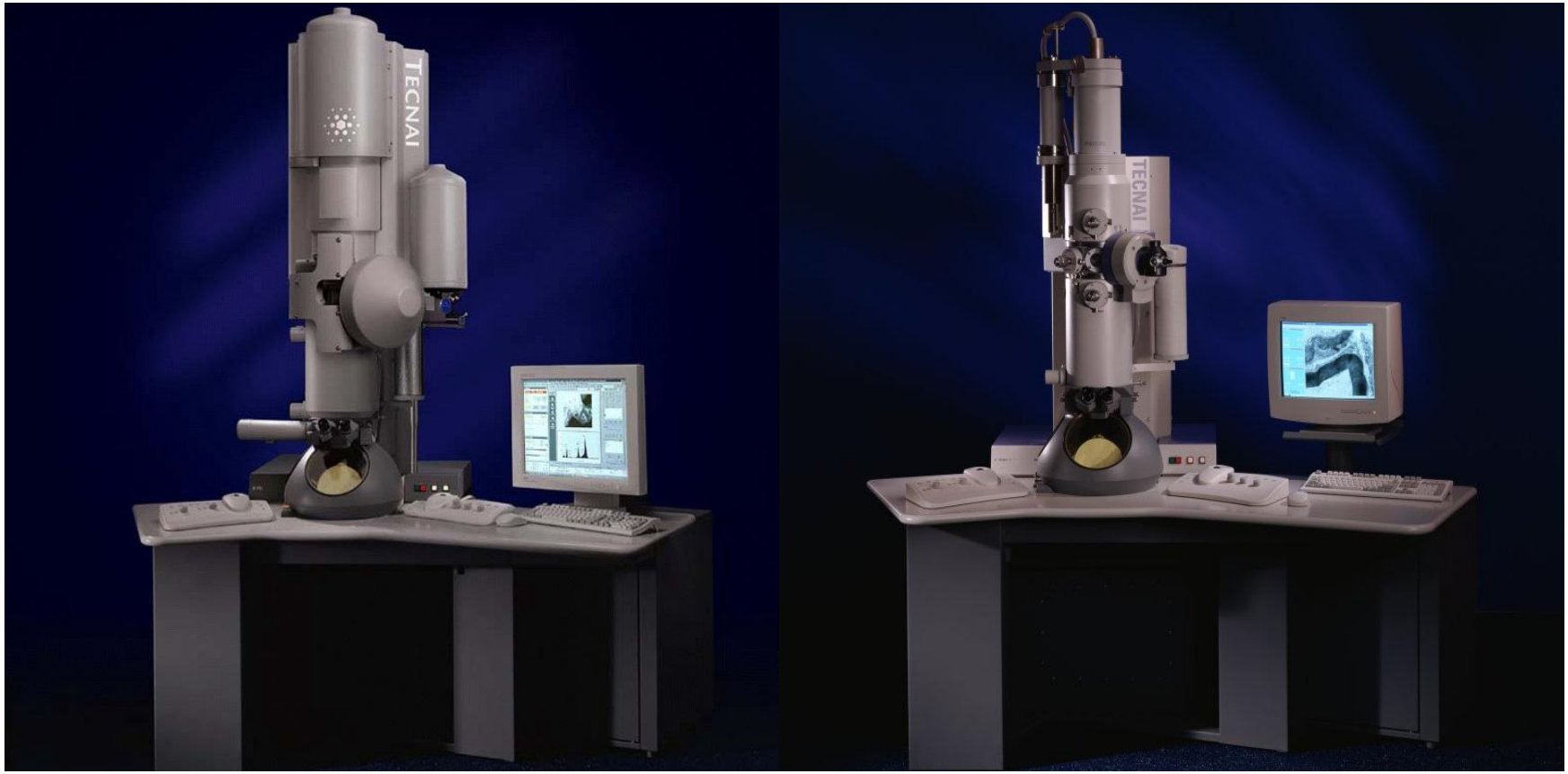
TEM（透射电镜） 操作简介

镇江乐华电子科技有限公司

王义林 总经理



Tacnai Basic Operation

电子显微镜历史

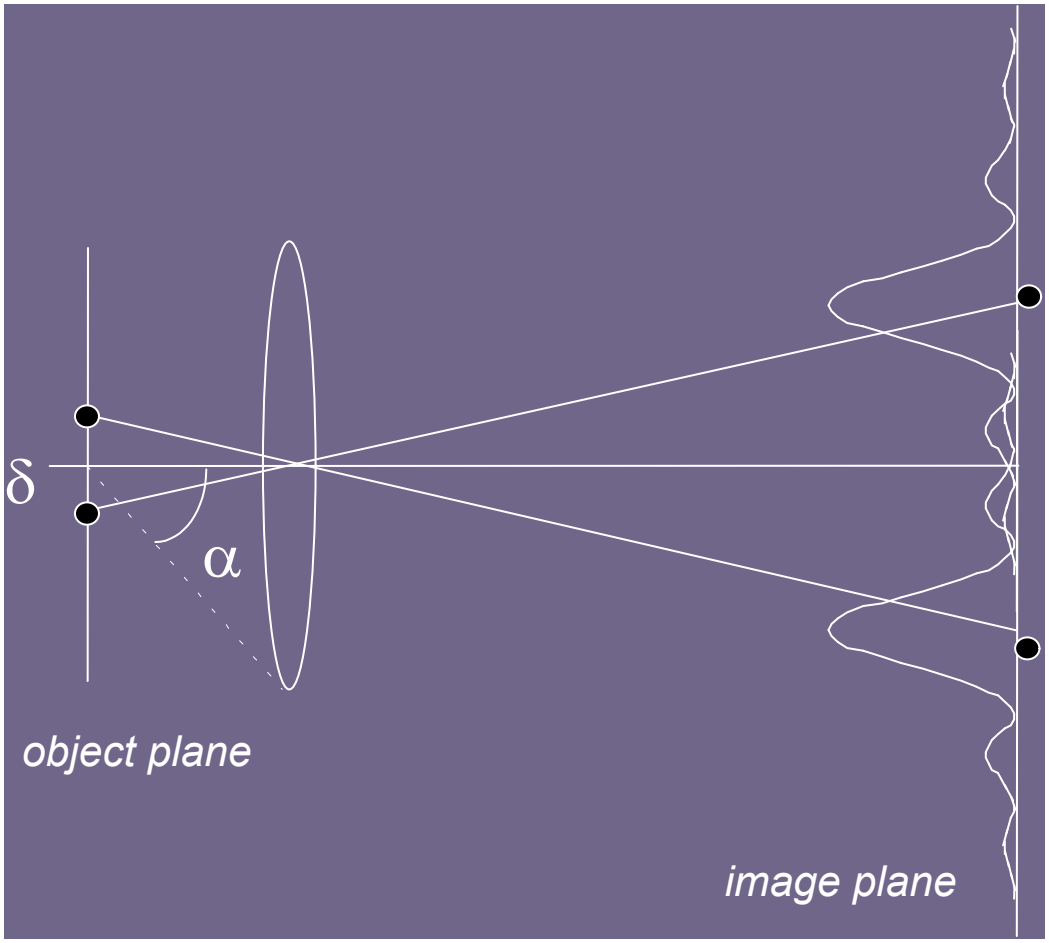
1897: *Thompson* describes the existence of negatively charged **particles** (electrons)

1925: *De Broglie* theorised that electrons have **wave**-like characteristics, addressing the wave/particle duality

1927: *Thompson and Reid* demonstrated the **wave** nature of electrons by diffraction experiments

1931: *Ruska* et al. build the first electron microscope (Nobel Prize in 1986)

透射电镜成像原理 / 分辨率



Rayleigh Criterion:

$$\delta = \frac{0.61\lambda}{n \sin \alpha}$$

光学显微镜:
δ= 200 nm

电子显微镜(TF30):
δ= 0.2 nm

为何用电子?

不同高压下的电子波长

U	Relativistic
100kV	λ=3.7 pm
120kV	λ=3.4 pm
200kV	λ=2.5 pm
300kV	λ=2.0 pm

显微镜 / 分辨率

电子波长

λ~0.002 nm

电子显微镜分辨率

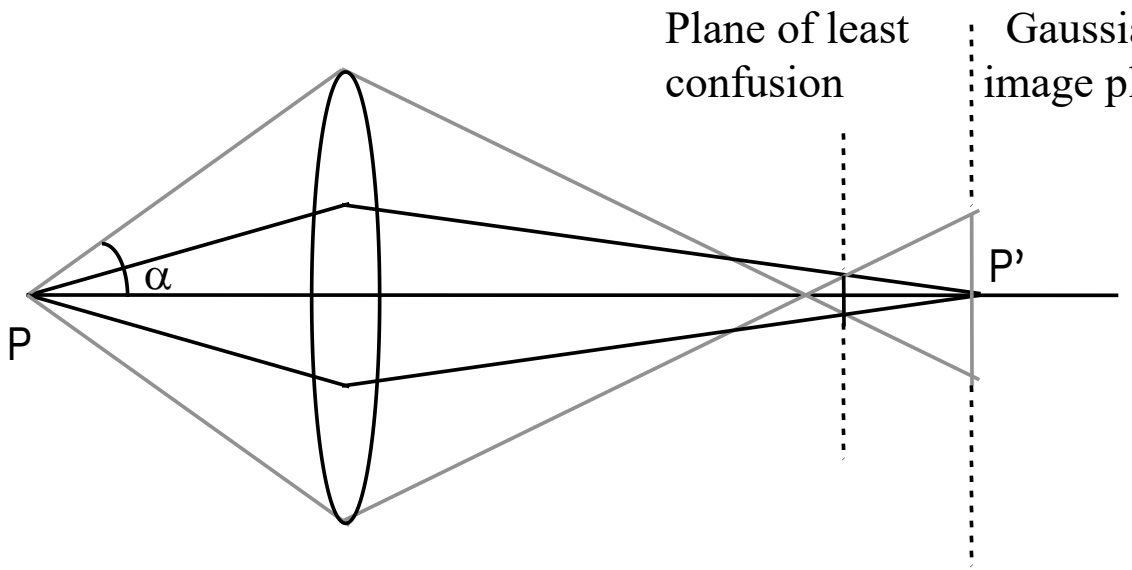
δ~0.1 nm

为什么?

- Lens aberrations (*e.g.* C_s, C_c): can be corrected by multipole lenses or focus-series reconstruction
- Instabilities (*e.g.* HT, electronic, mechanical / acoustic, thermal)

电磁透镜: 球差

Lens imperfections lead to different focal lengths in centre (weaker field) and at edges (stronger field) of lens



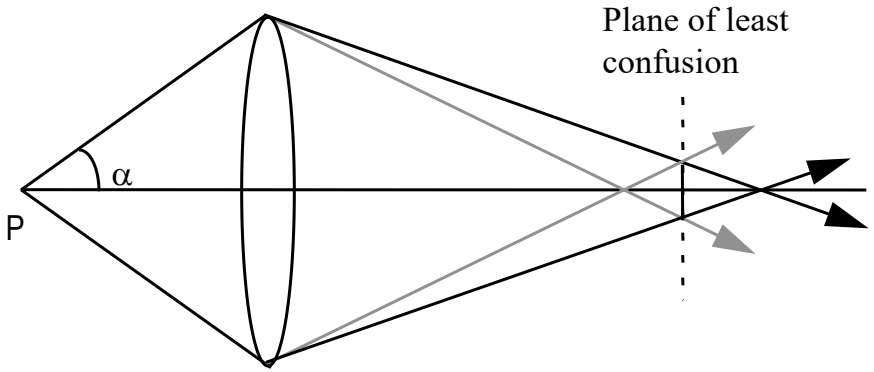
$$\delta_s = C_s \alpha^3$$

Outer zones have stronger strengths

球差电镜, 赛默飞公司Spectra， 日本电子Arm200F

电磁透镜: 色差

Blurring due to energy spread in electron beam and lens current fluctuations

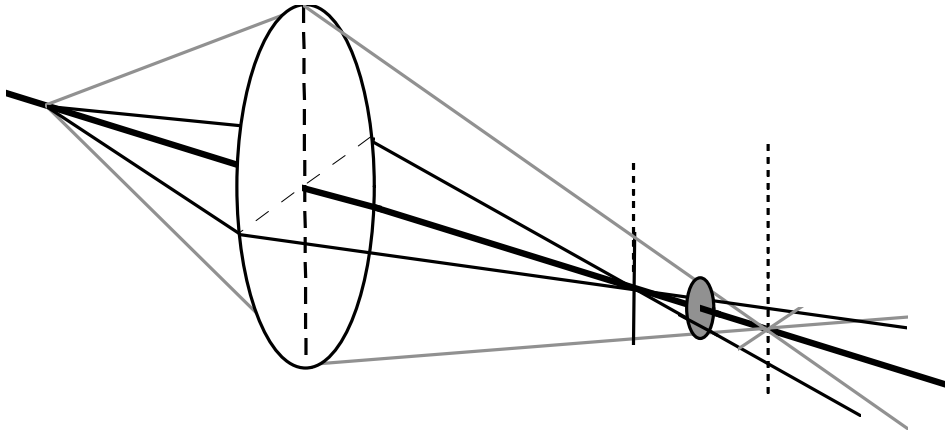


$$\delta_c = C_c \alpha \left(\frac{\Delta E}{E} + \frac{2\Delta I}{I} \right)$$

最新透射电镜发展方向: 色差校正器 Cc corrector

电磁透镜: 像散

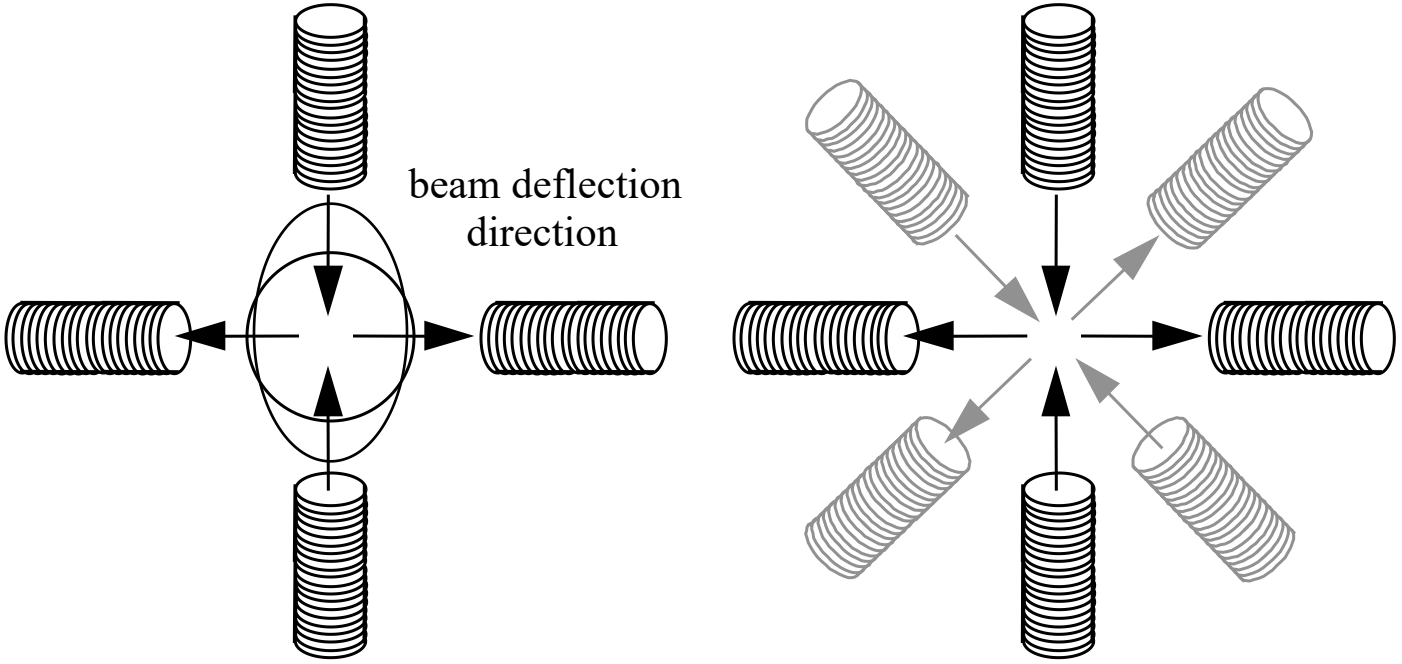
Lens defect caused by magnetic field asymmetry



can be corrected using stigmators!

电磁透镜系统: 消像散器

工作原理：



TEM Modes

透射电镜模式

Bright Field Imaging	明场成像
Dark Field Imaging	暗场成像
Diffraction	电子衍射
STEM	扫描透射
EDX	X射线能谱
EFTEM	能量过滤透射
Tomography	体层摄影术

TEM明场像

Standard imaging methods

Imaging modes:

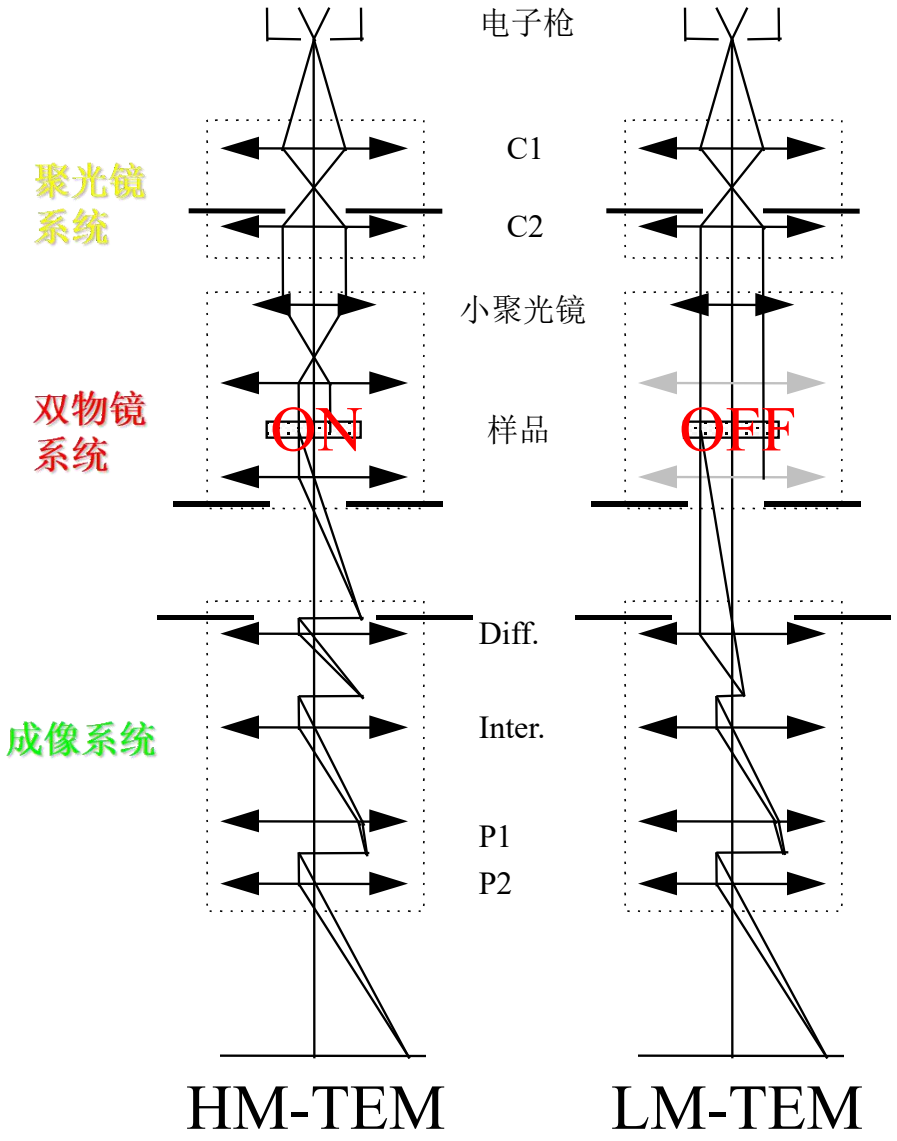
- LM (objective nearly off)
- HM (objective on)

标准成像方法

成像模式:

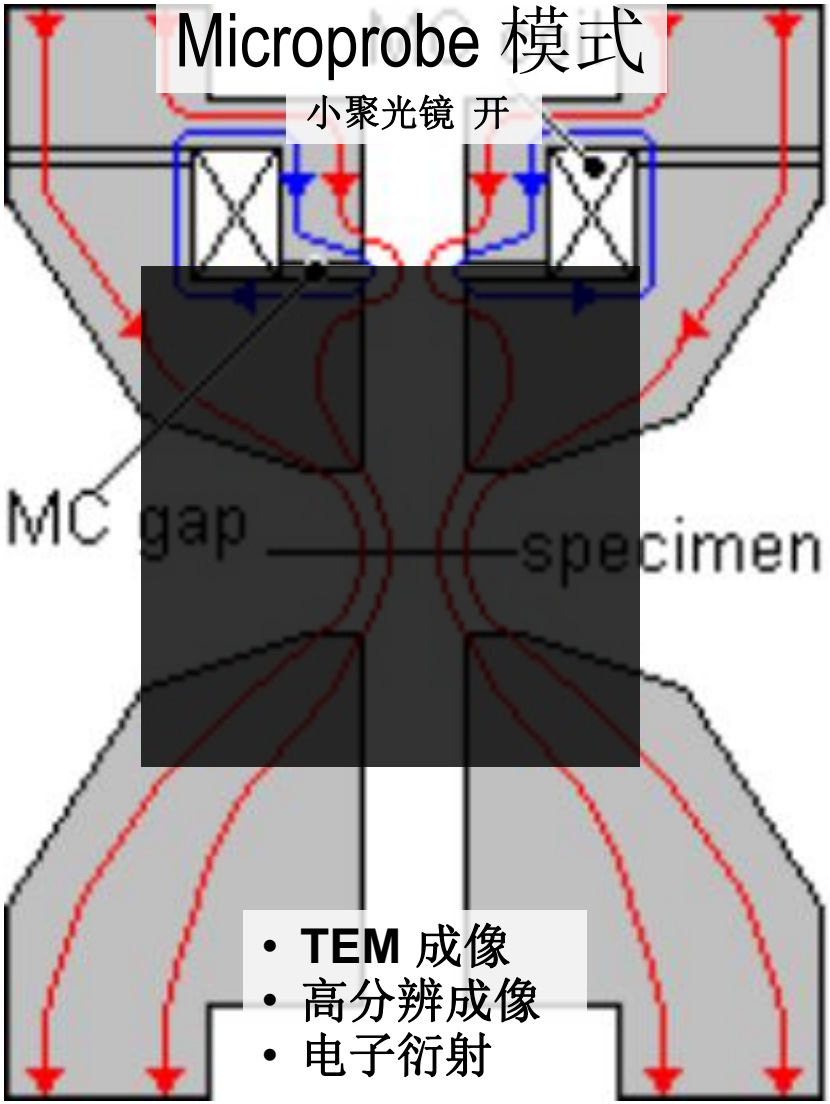
- 低倍率模式（物镜关闭）
- 高倍率模式（物镜通电）

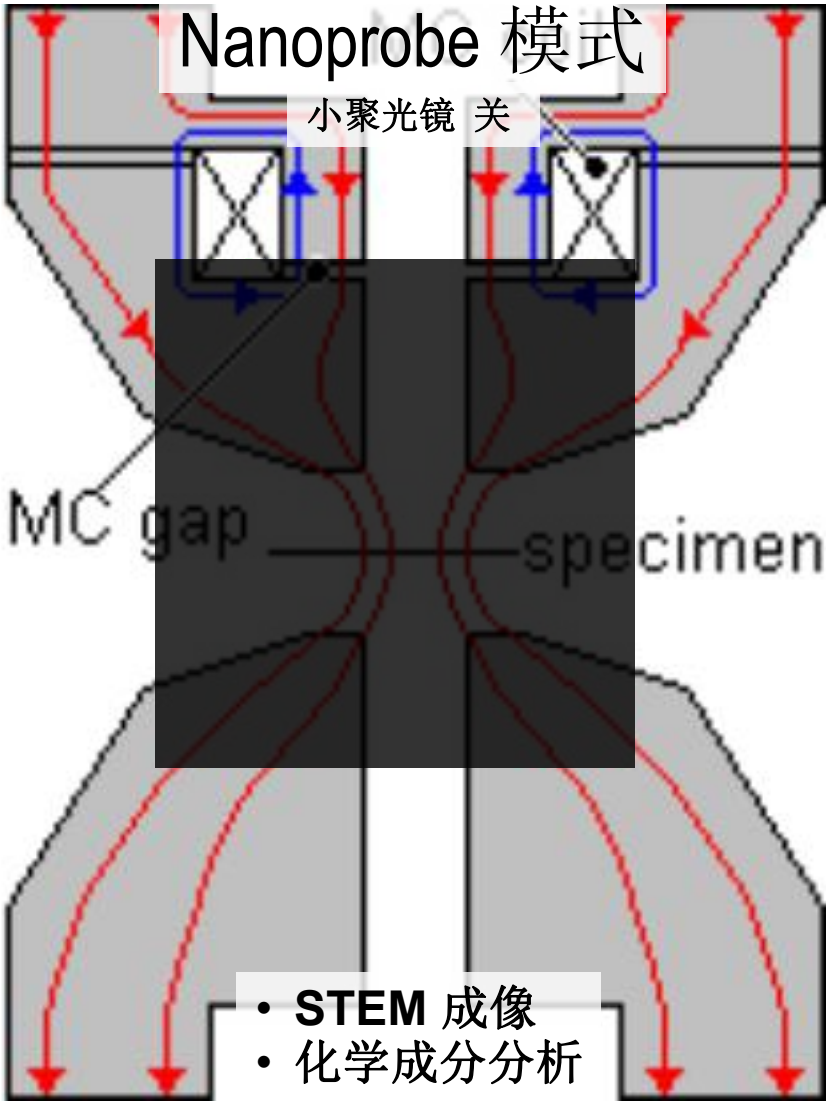
TEM明场像: LM低倍率 / HM高倍率



成像系统的不同聚焦模式

TEM明场像: 双物镜


Microprobe 模式
小聚光镜 开
MC gap
specimen
• TEM 成像
• 高分辨成像
• 电子衍射

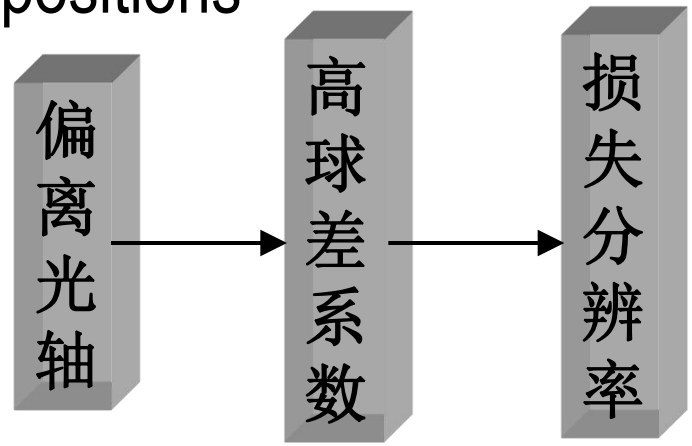

Nanoprobe 模式
小聚光镜 关
MC gap
specimen
• STEM 成像
• 化学成分分析

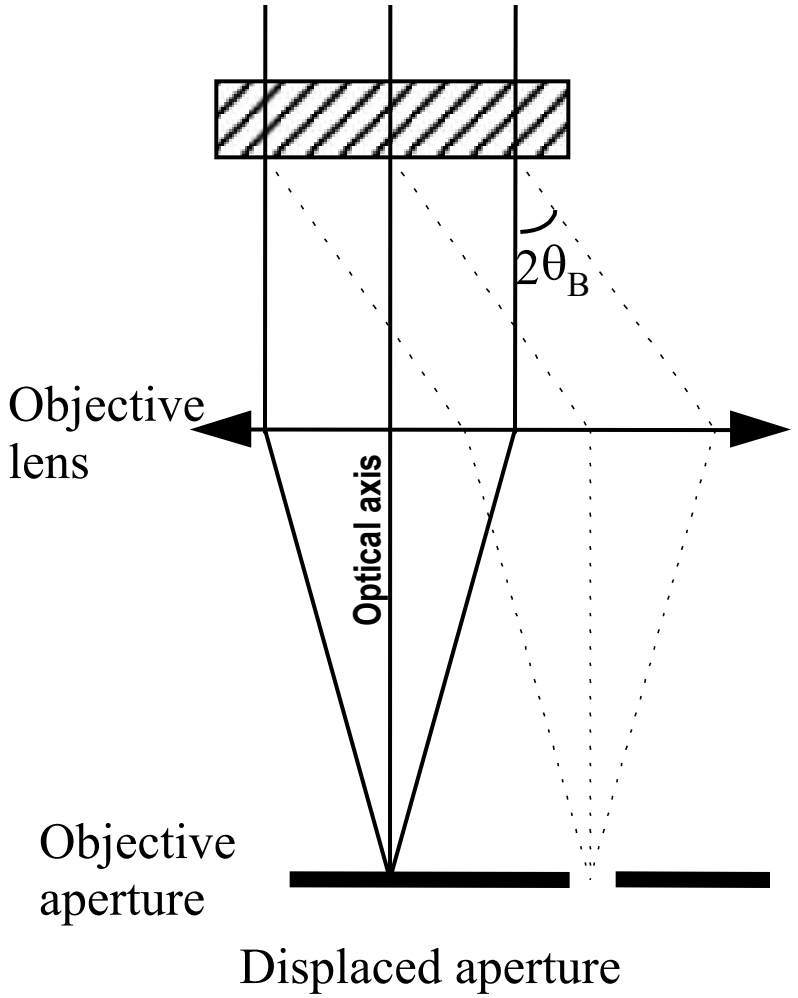
TEM暗场像: 离轴成像

Off-axis imaging

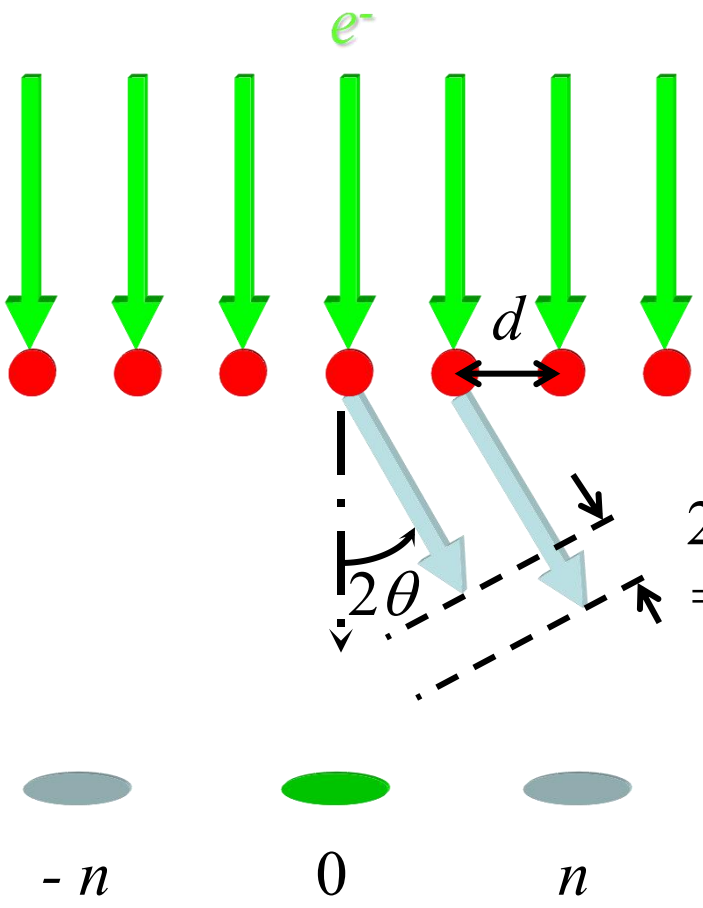
离轴成像

Loss of resolution due to higher C_s at off-axis positions





电子衍射: 原理



样品置于物平面
进行衍射

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

=> *constructive interference*

衍射斑点形成
于物镜后焦面

电子衍射: 成像模式

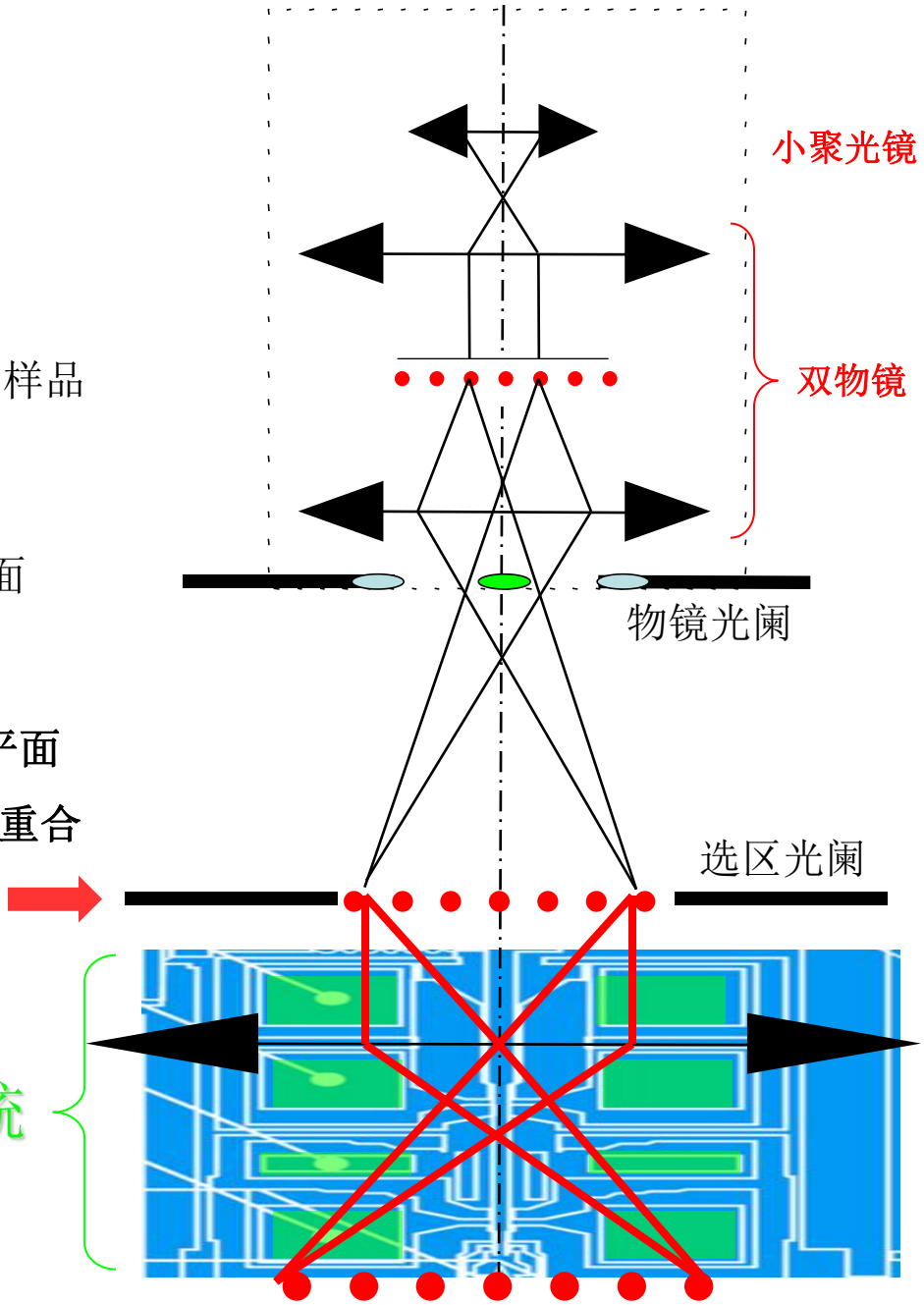
成像系统的**物平面**

成像 模式:

- 一级中间镜
  相平面

衍射 模式:

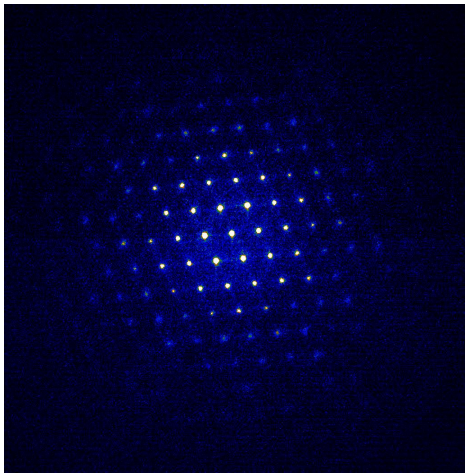
- 物镜后焦面



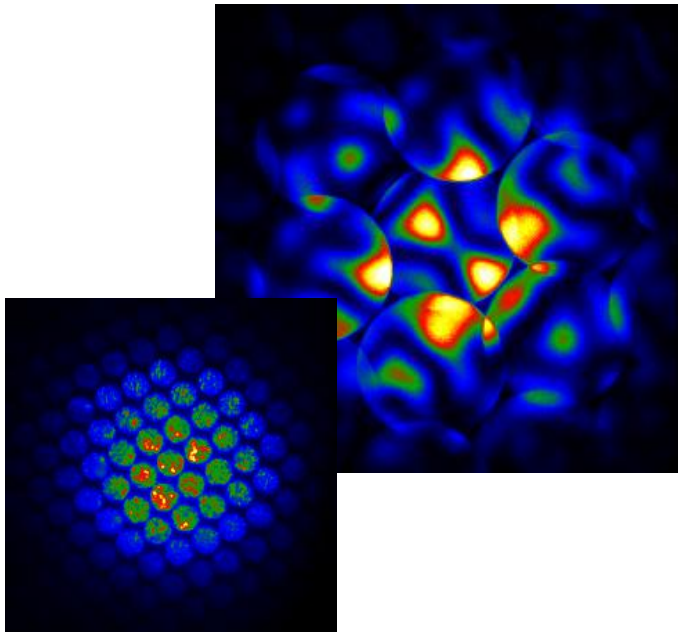
电子衍射: NBD and CBED

束斑定位于样品感兴趣区域产生相应的电子衍射

- **纳米束衍射 (NBD)**
  - 最小 5 nm (10 or 20 um C2 光阑)
  - **平行电子束**
- **会聚束衍射 (CBED)**
  - 最小 0.3 nm
  - **会聚束**



Points



Discs

电子束—样品的相互作用

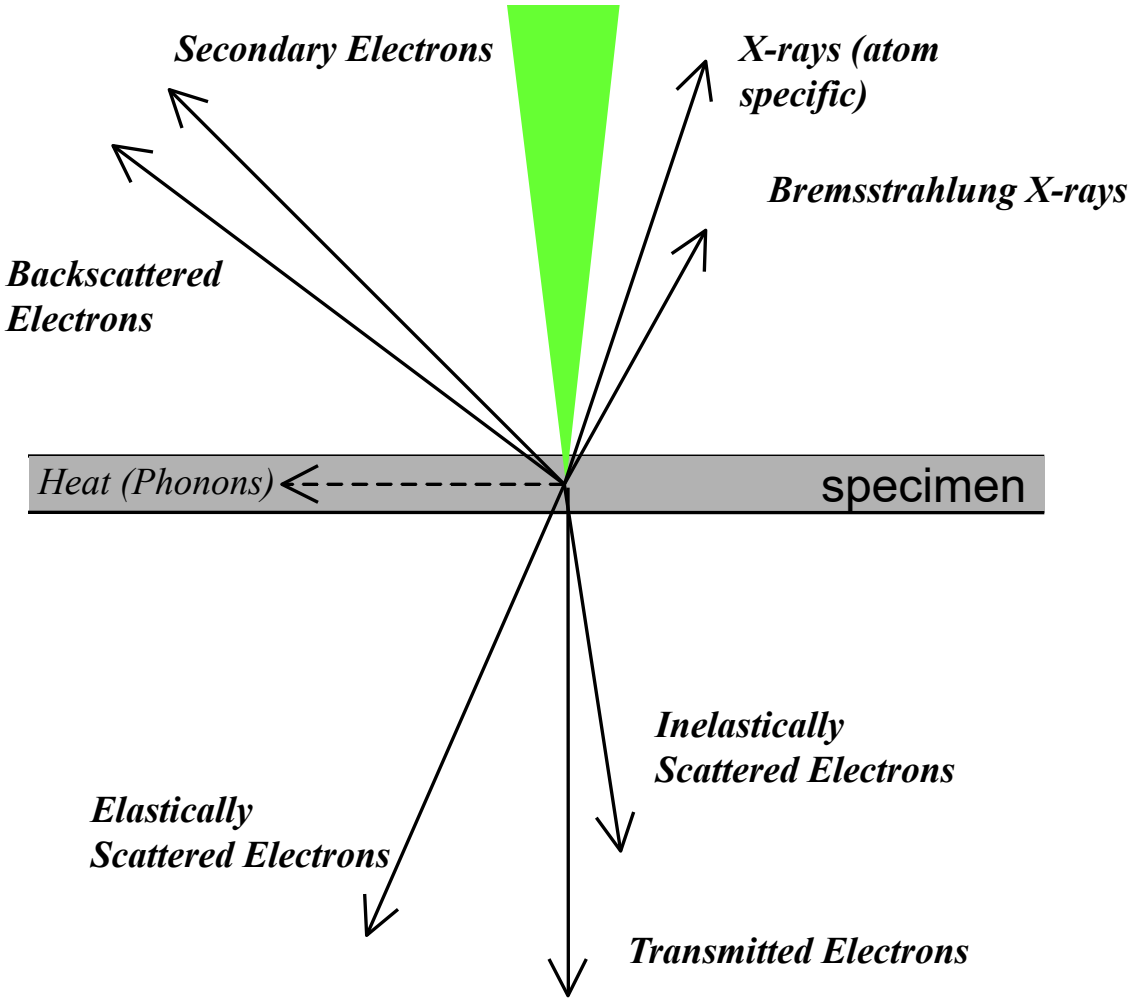
弹性散射

- 具有方向性
- 散射角正比于Z^2/E

非弹性散射

- 具有方向性, 能量发生改变
- 作用体积E^2/Z

电子束—样品的相互作用


Secondary Electrons
X-rays (atom specific)
Bremsstrahlung X-rays
Backscattered Electrons
Heat (Phonons)
specimen
Inelastically Scattered Electrons
Elastically Scattered Electrons
Transmitted Electrons

STEM （扫描透射电镜）与 TEM（透射电镜）相比的优势

图像更容易解释

- 高衬度及信噪比
- 非相干性: 没有相位衬度,不会因欠焦量大小改变图像衬度

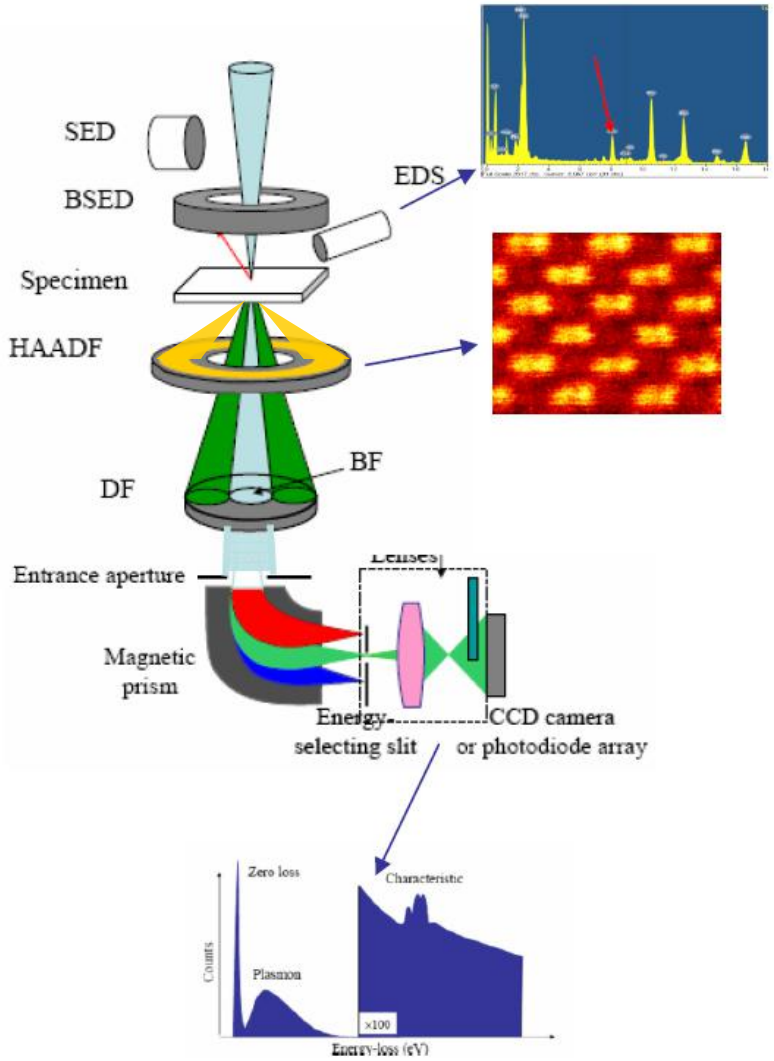
信号强度的变化

- 与样品厚度、密度成线性变化
- 与原子序数成正比 $Z^{4/3}$

不受色差影响

Low Dose模式下可以精准控制照射区域

可同时采集图像和化学成分信息



STEM 扫描透射模式

聚焦电子束在样品表面进行扫描，在每个扫描点产生信号；信号被探头采集的同时，屏幕上生成相应位置的STEM图像

- 可进行图像成分分析 (elemental mapping)：每一个像素点的图像信号与EDX信号同时采集

STEM 扫描透射模式

Different detectors are used to collect electrons transmitted at different angle range, thus giving different contrasts:

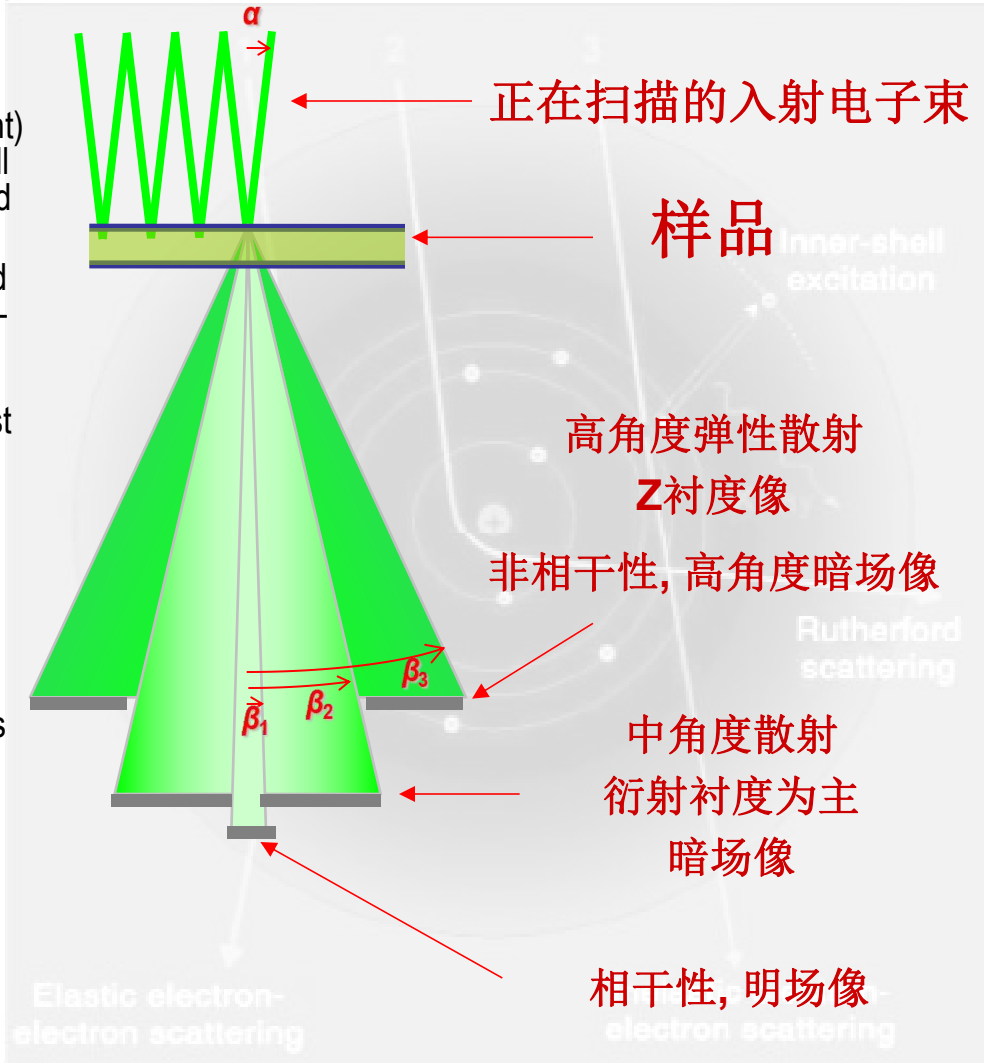
- **Bright-field detector:** collects (partially coherent) electrons not scattered or scattered to very small angle; the contrast will be similar to that obtained in CTEM
- **Dark-field detector:** collects electrons scattered at intermediate angle, equivalent to conical dark-field in CTEM with spread illumination, when the illumination is tilted with respect to the optical axis of the objective lens, and the image contrast formed by electrons diffracted or scattered to intermediate angles
- **HAADF detector** (single-electron counting): collects (incoherent) electrons scattered at high-angle (typically above 50 mrad); the collected intensity is proportional to ~ $\rho\, t\, Z^{4/3}$ thus produces Z-contrast images

The collection angle β of the HAADF detector depends on the camera length (CL) used;

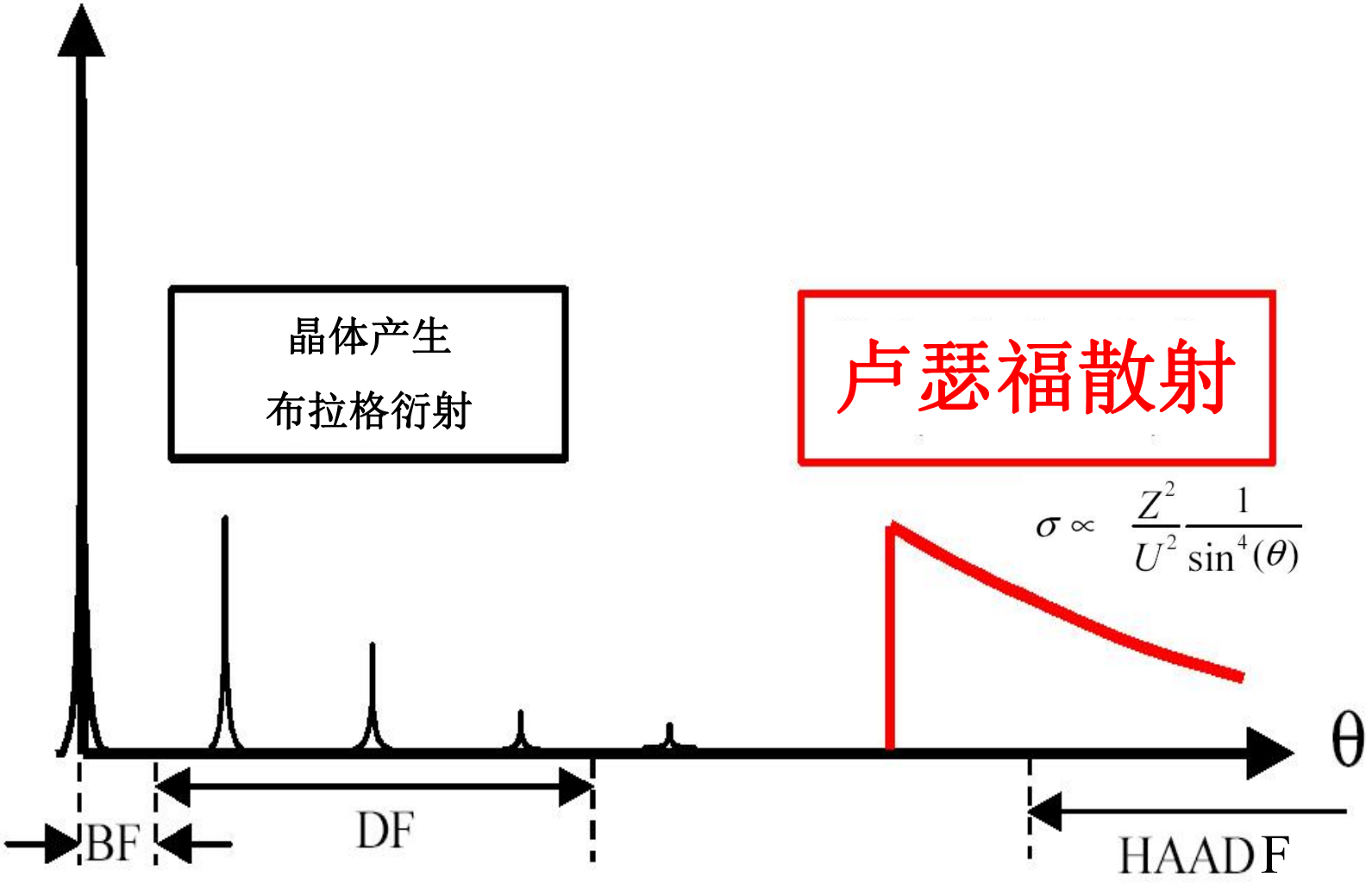
E.g. on the FEI Tecnai F20:

β_2 [rad] = 2 / (0.276 * CL) = 72mrad @CL=100mm

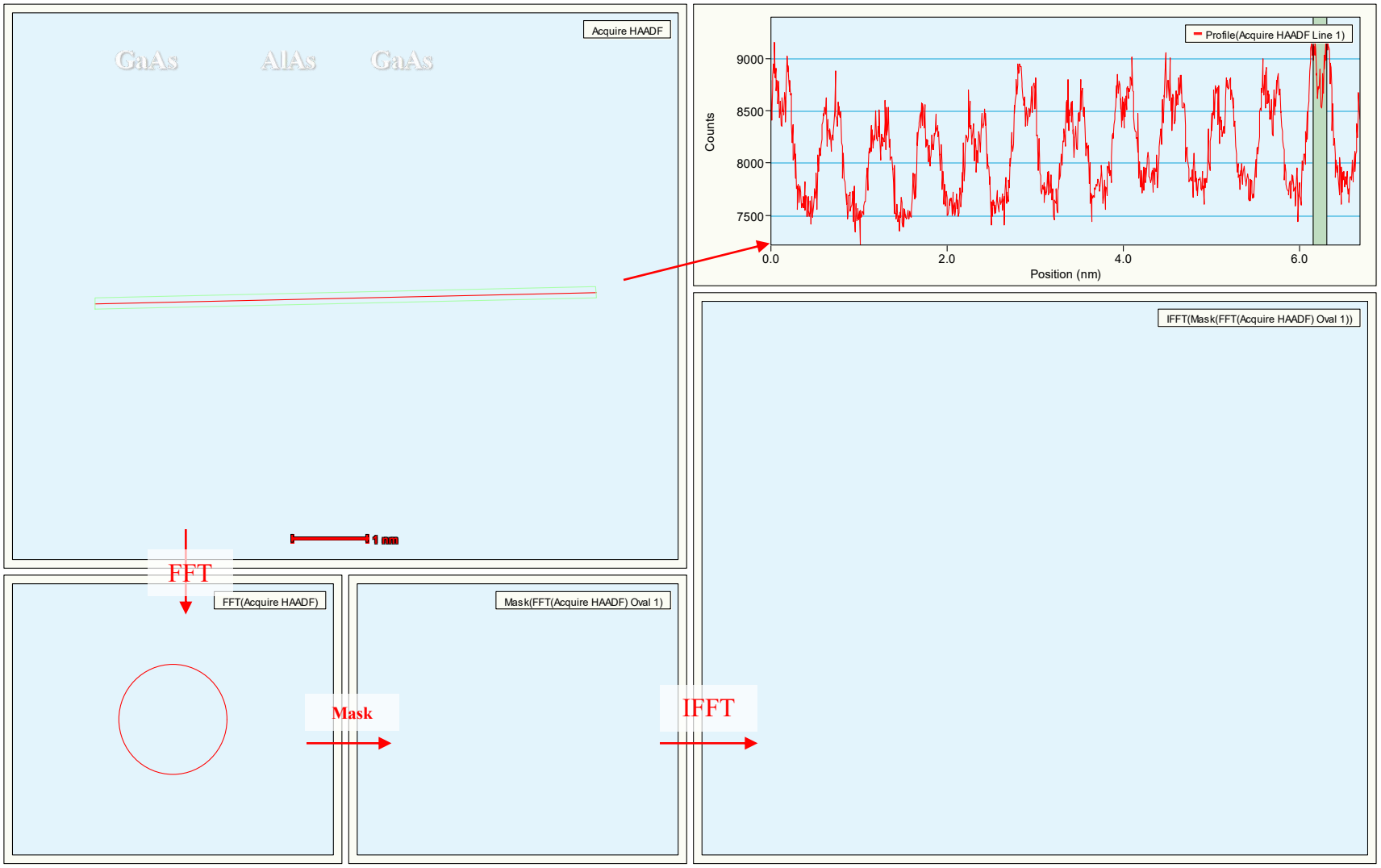
β_3 [rad] = 10 / (0.276 * CL); βmax ≤ 227 mrad



STEM 明场像 / 暗场像 / 高角度暗场像

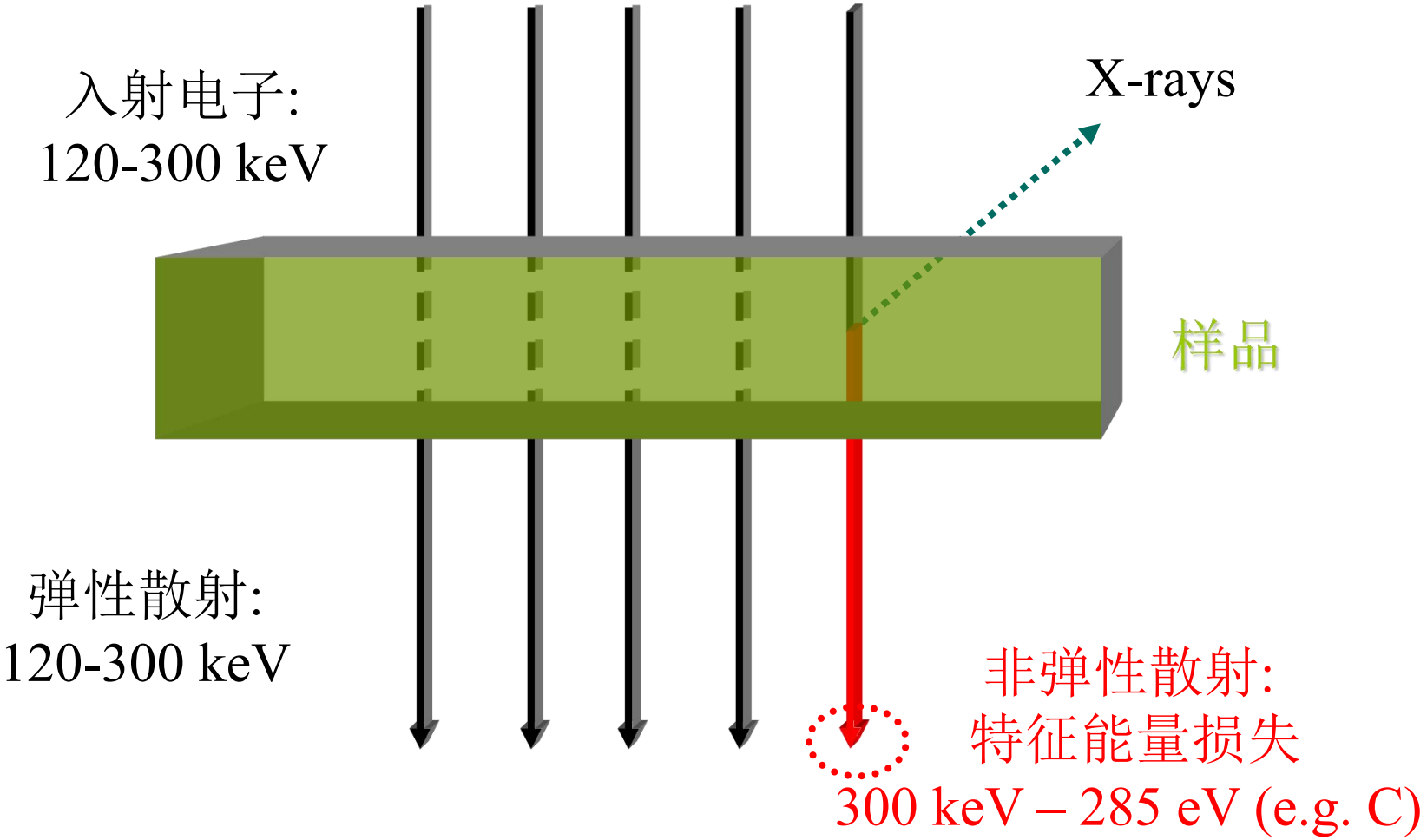


高角环形暗场像 (HAADF)

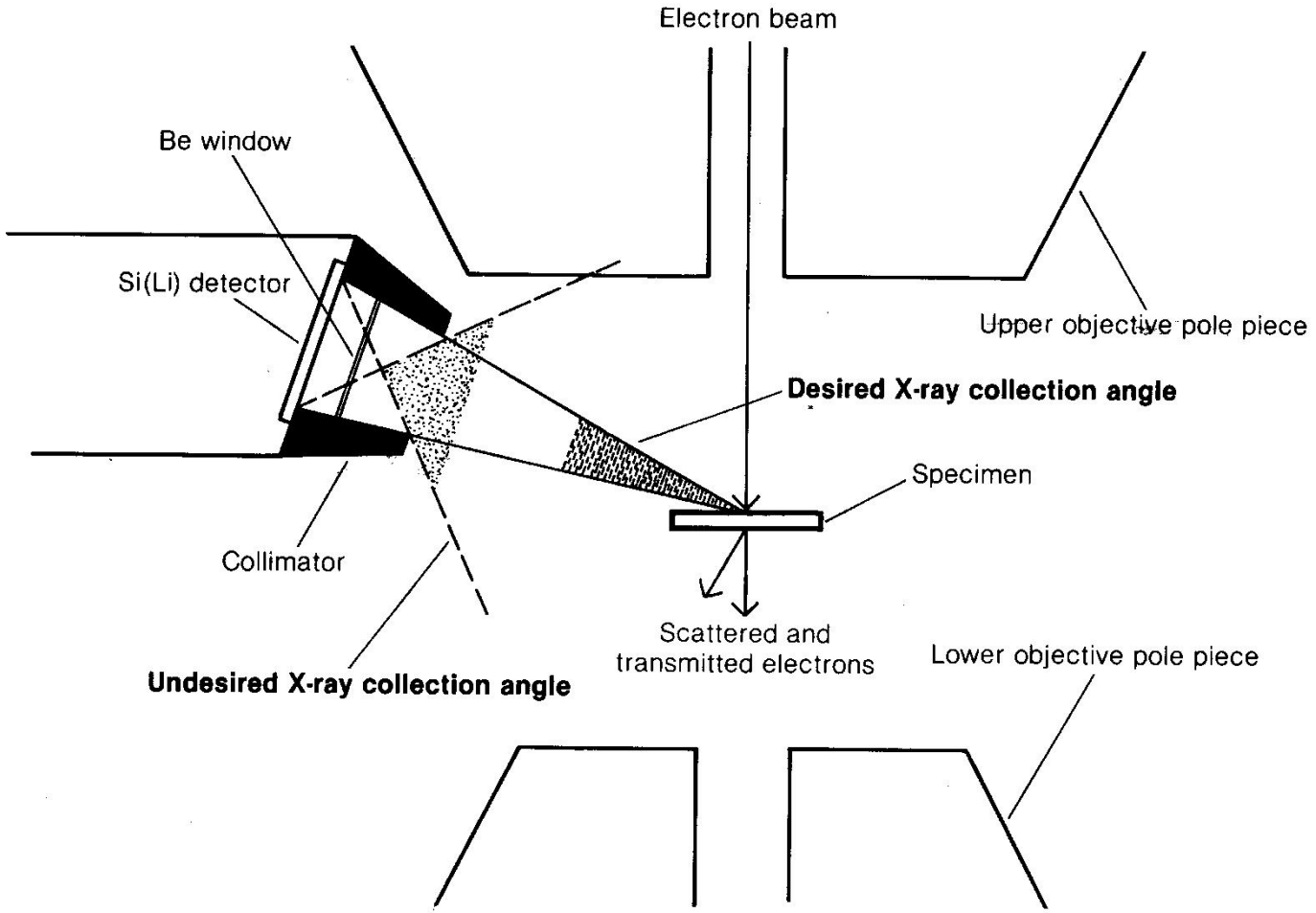


束斑尺寸 < 0.140 nm

EDX / EELS（能谱/能量损失谱）：非弹性散射



EDX（能谱）


Electron beam
Be window
Si(Li) detector
Upper objective pole piece
Desired X-ray collection angle
Specimen
Collimator
Scattered and
transmitted electrons
Undesired X-ray collection angle
Lower objective pole piece

EDX

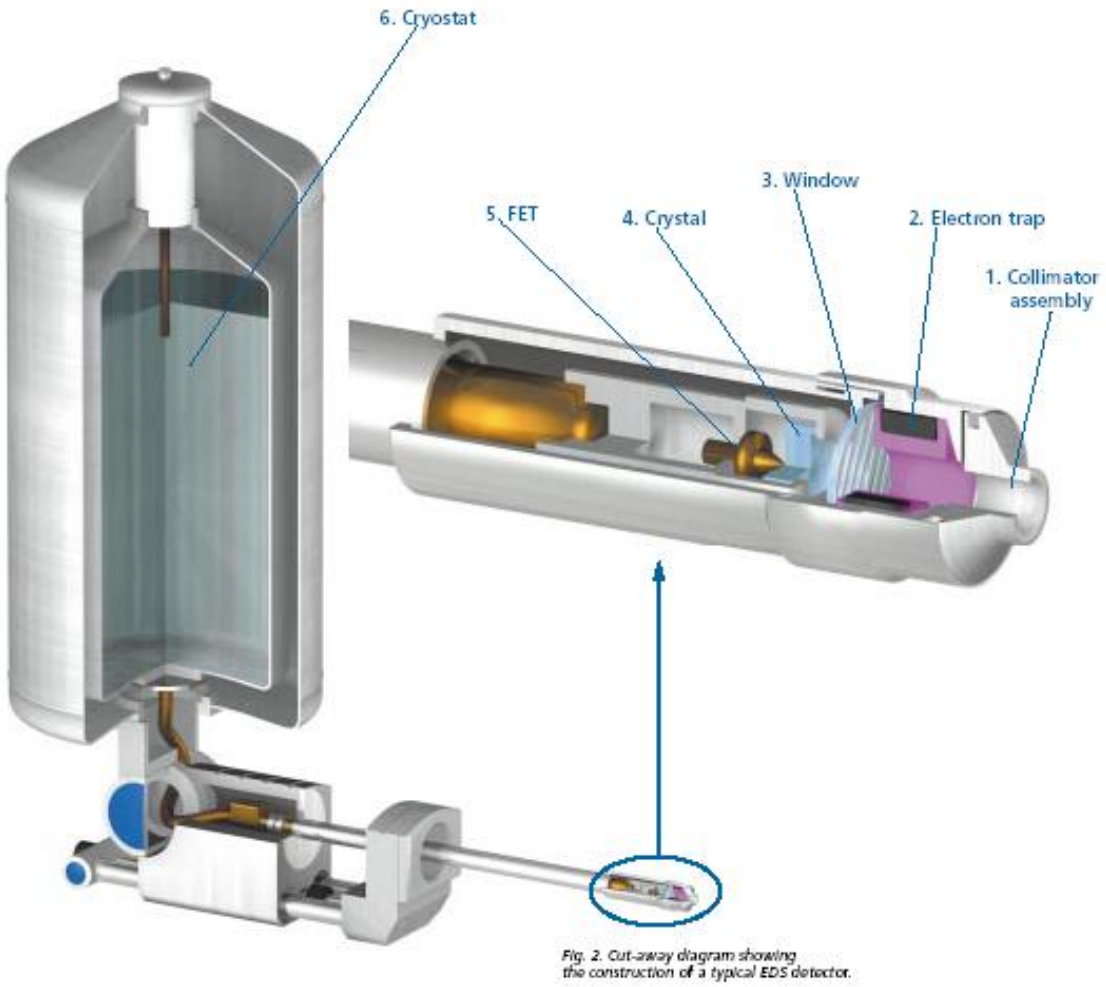
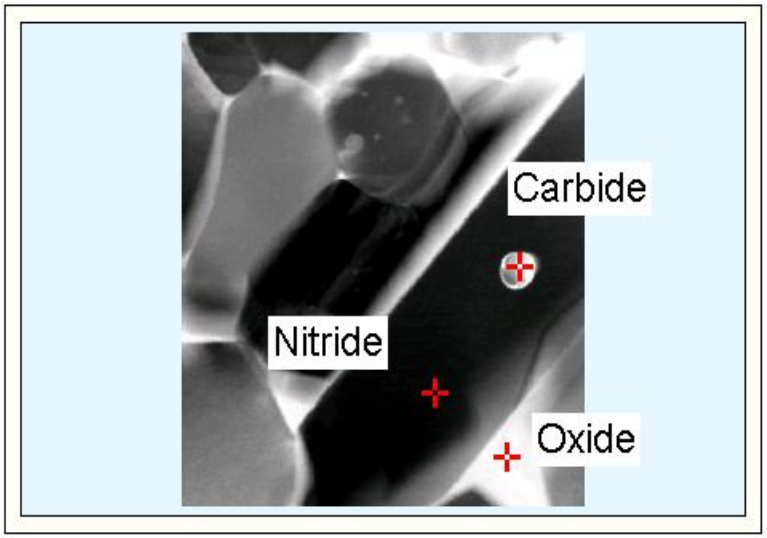
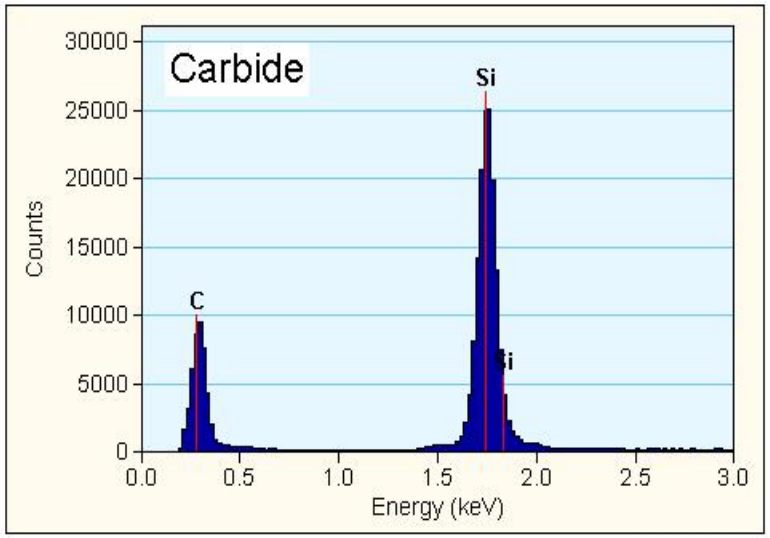


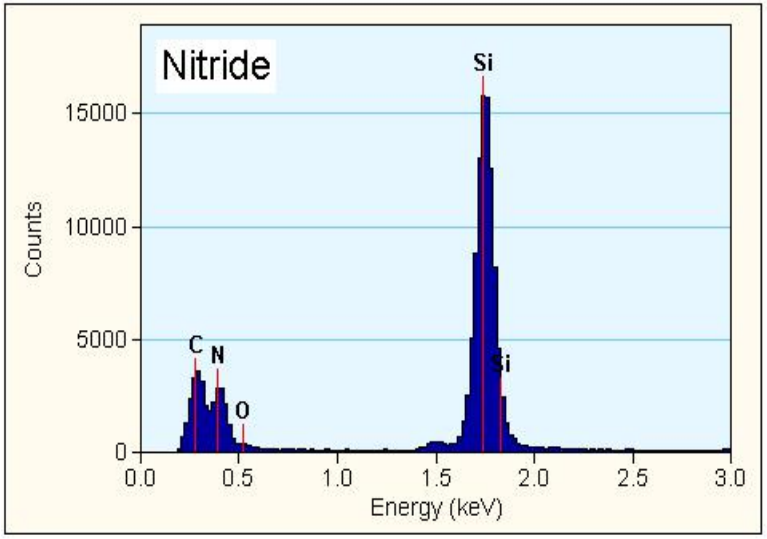
Fig. 2. Cut-away diagram showing the construction of a typical EDS detector.

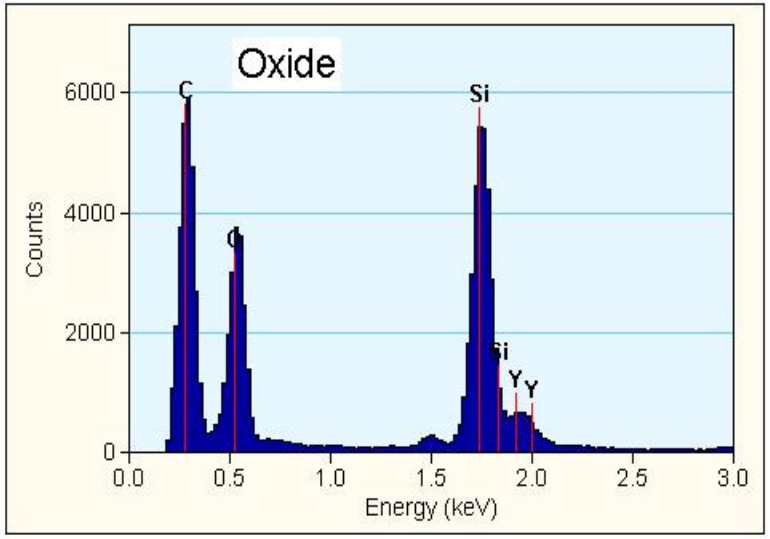
Schematic courtesy of Oxford Instruments

EDX

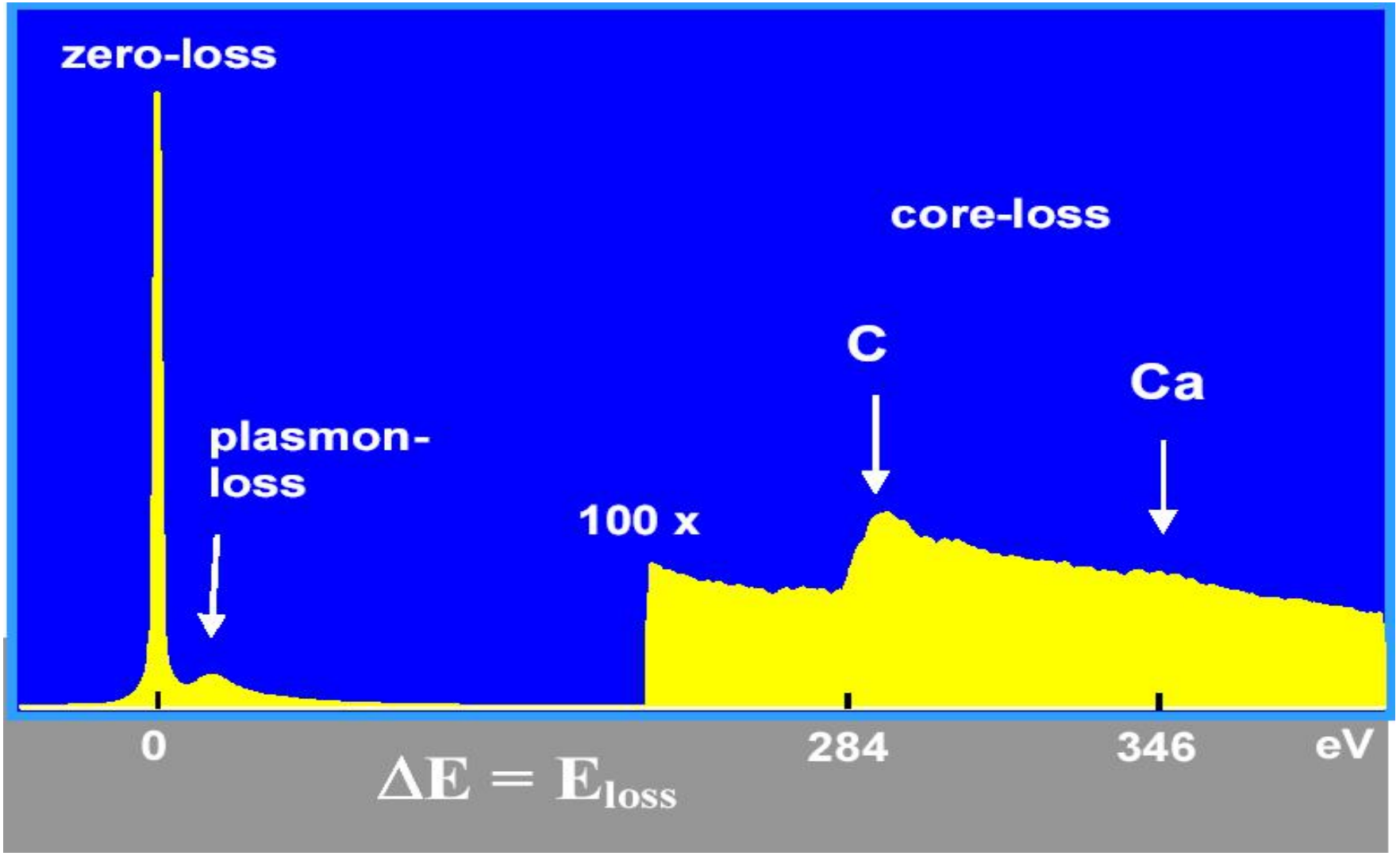

Carbide
Nitride
Oxide


Carbide
Si
C
Si
Counts
Energy (keV)

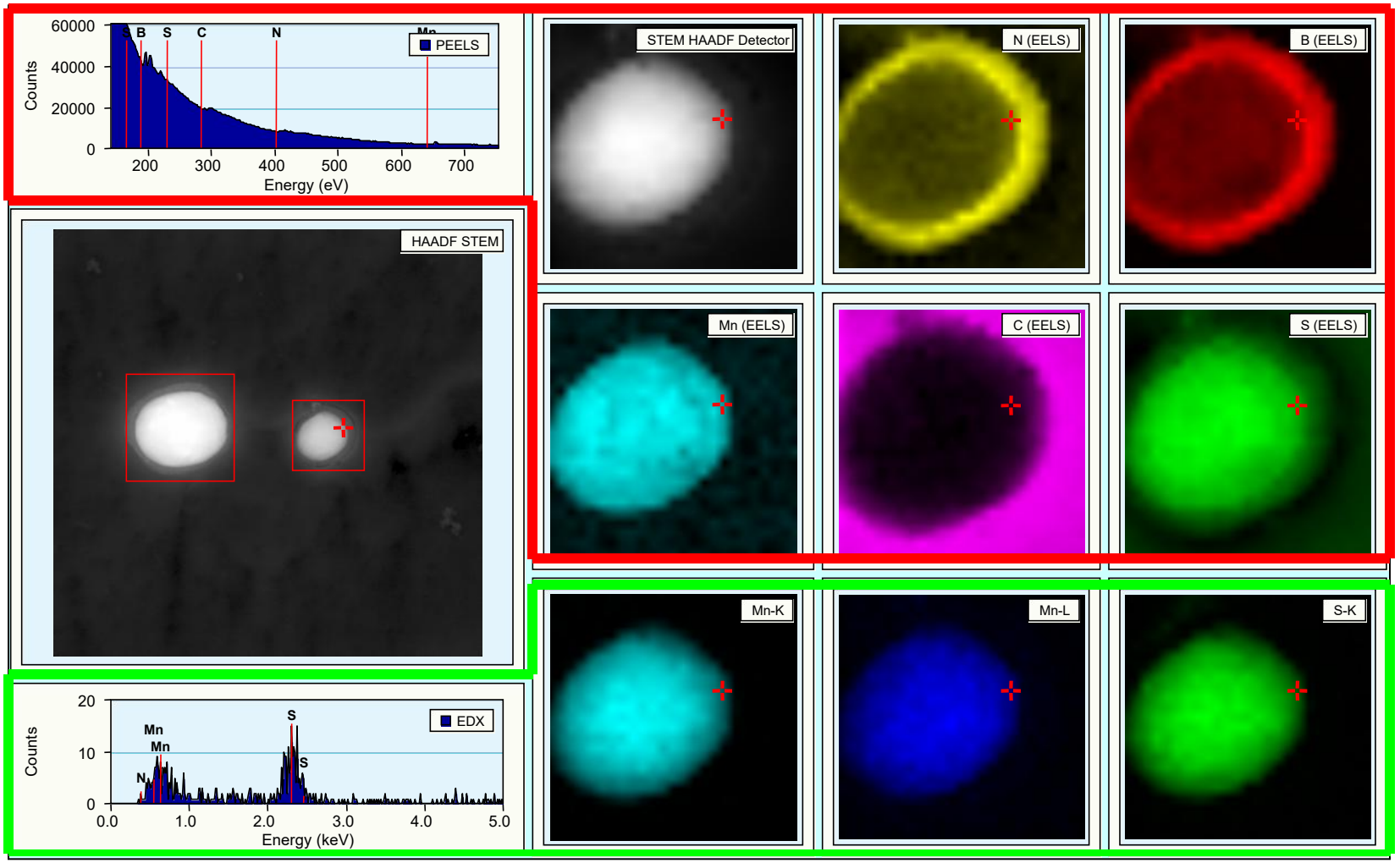

Nitride
Si
C
N
O
Si
Counts
Energy (keV)


Oxide
C
Si
O
Si
Y
Y
Counts
Energy (keV)

EELS（能量损失谱）


zero-loss
plasmon-loss
core-loss
C
Ca
100 x
0
284
346
eV
$\Delta E = E_{loss}$

HAADF-STEM in combination with EELS/EDX



EDX + EELS: elemental maps. Mn-K, Mn-L and S-K refer to EDX maps, all other maps being computed from the EELS spectrum. As illustrated here, PEELS and EDX are complementary techniques to detect both light and heavy elements.

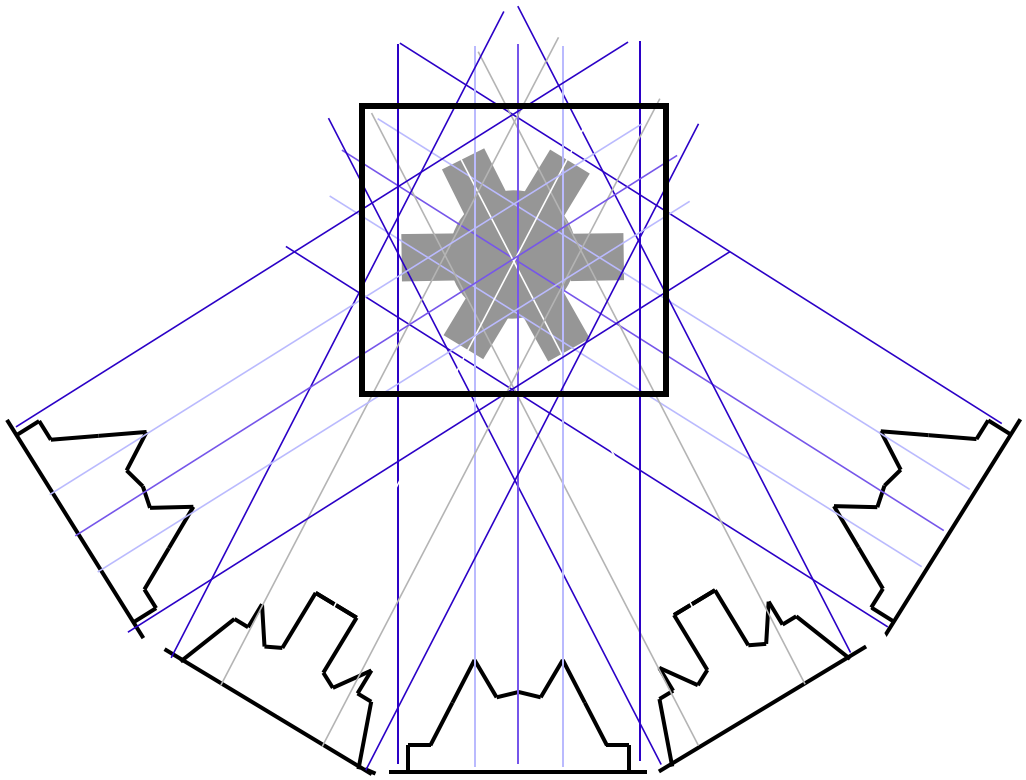
Electron Tomography（三维重构）

Basic Idea

采集不同方向的投影像

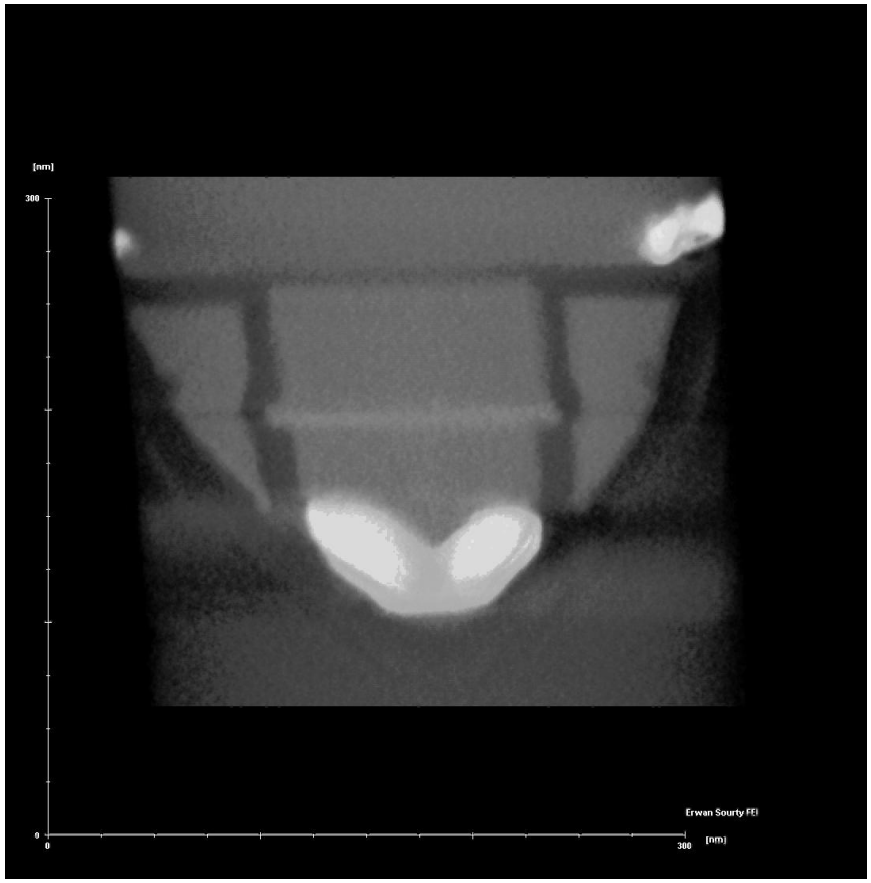
投影像进行合轴

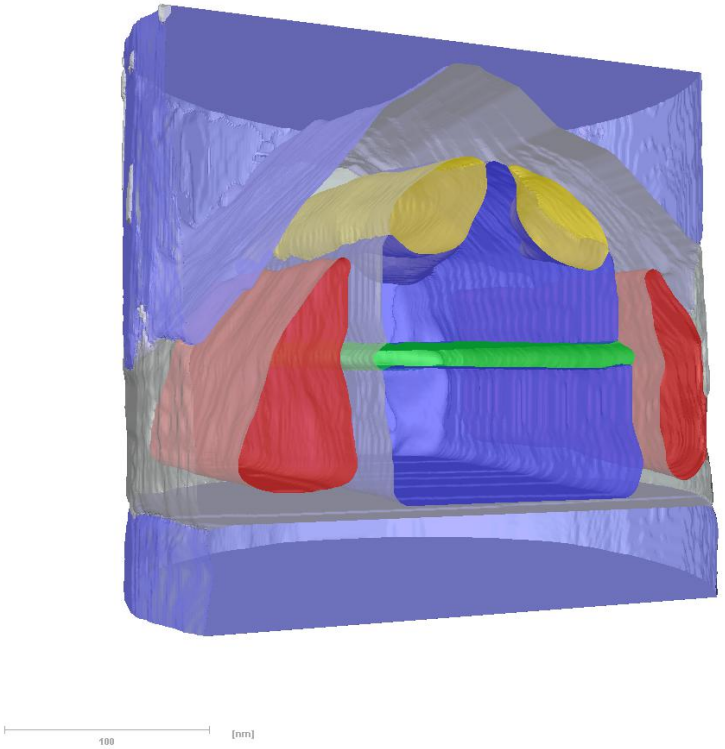
逆运算进行重构



Resolution ~ π D / N

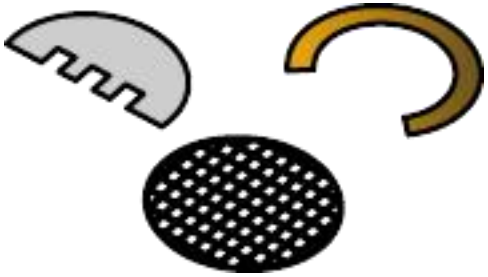
Electron Tomography
with Xplore3D™





3D-analysis of semiconductor structures by electron tomography
Microelectronic Engineering, *Volume 84, Issue 11*, *November 2007*, *Pages 2707-2713*
H. Bender, O. Richard, A. Kalio and E. Sourty

TEM样品要求



小

- 3 mm 直径

薄

- 一般 < 500 nm*

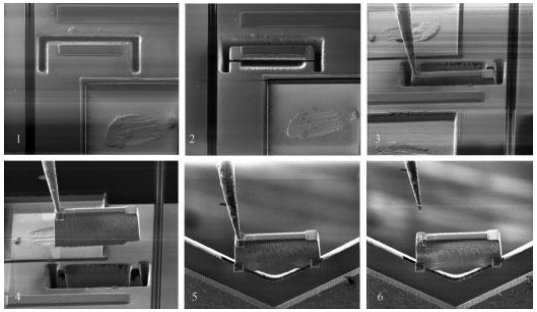
生物样品

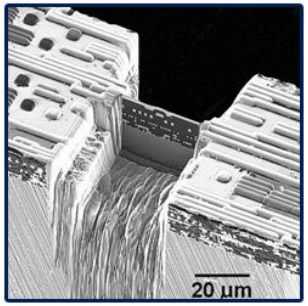
- 在碳膜上沉积样品组织
- 冷冻制样(病毒)

材料样品

- 粉末分散于溶液中，沉积于碳膜上
- 离子减薄，电子束在孔洞边缘透射成像
- 树脂包埋＋超薄切片

- **FIB lift-out or H-bar =>**





** 1 nm = one-millionth of a millimetre*

= ca. 5x size of single atom

TEM基本组成部分（以TF30为例）

电镜镜筒

电源柜

TEM控制柜

STEM控制柜

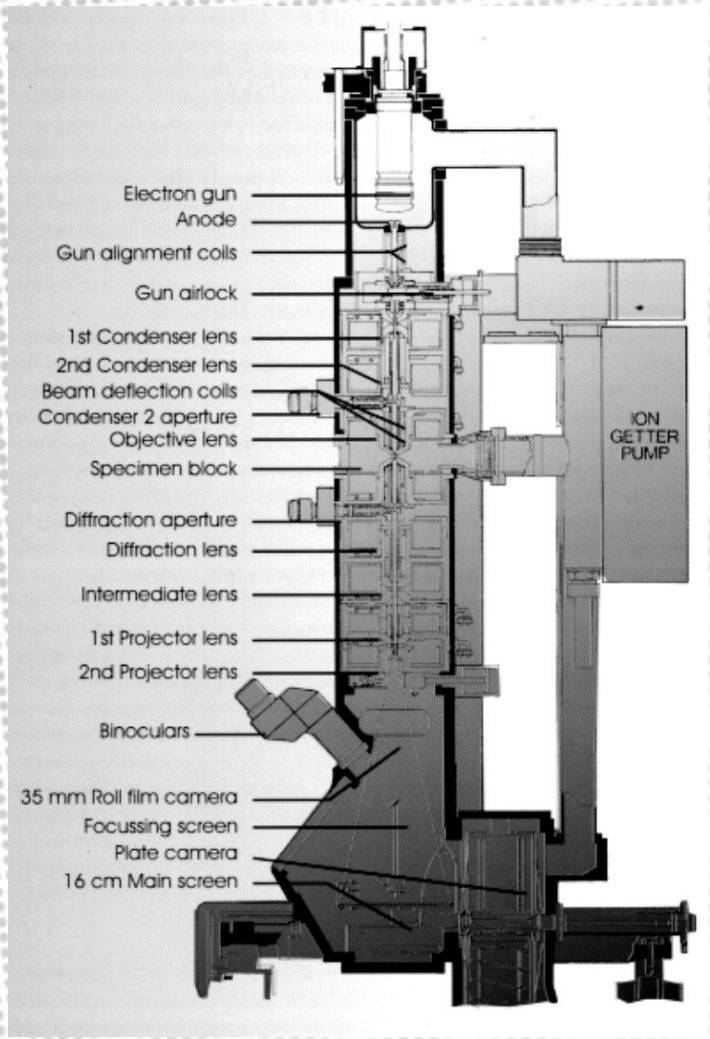
高压发生器HT TANK

电镜计算机

左右操作面板

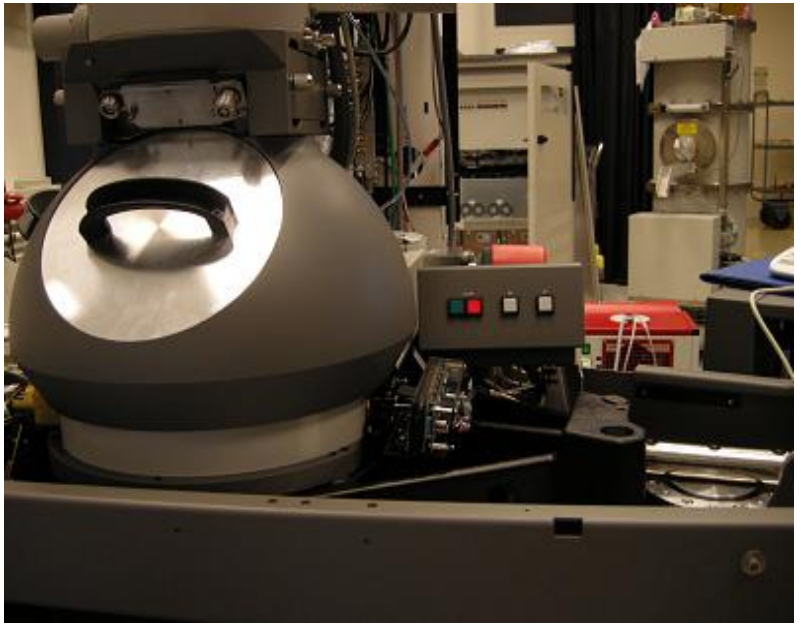
能谱探头

TEM基本结构

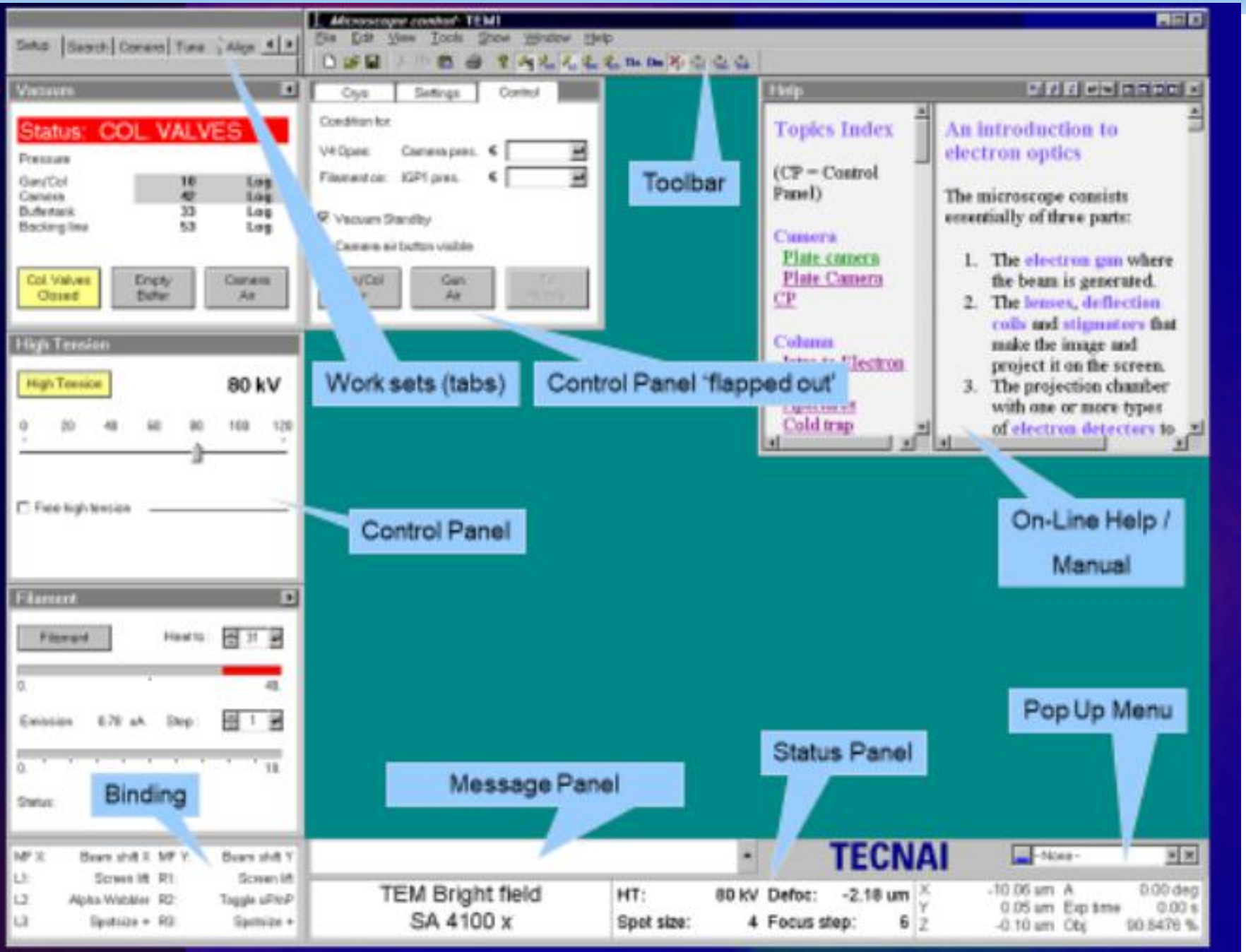

Electron gun
Anode
Gun alignment coils
Gun airlock
1st Condenser lens
2nd Condenser lens
Beam deflection coils
Condenser 2 aperture
Objective lens
Specimen block
Diffraction aperture
Diffraction lens
Intermediate lens
1st Projector lens
2nd Projector lens
Binoculars
35 mm Roll film camera
Focussing screen
Plate camera
16 cm Main screen
ION GETTER PUMP

电镜开机过程

1. 开电源柜主开关，在右面的所有开关朝上，24V电源灯亮
2. 开计算机和显示器，等待计算机完全启动
3. 开电镜主机电源：在SOOP处，按一下 [ON] 键 ，电源柜的所有电源灯都亮，表明电源主机启动.
4. TEM控制柜的CCB板开始与计算机通讯连接，当所有灯都灭了，通讯完成
5. 用正确的用户名和密码登录计算机
6，运行Tecnai UI



电镜操作界面: TEMUI



左右操作面

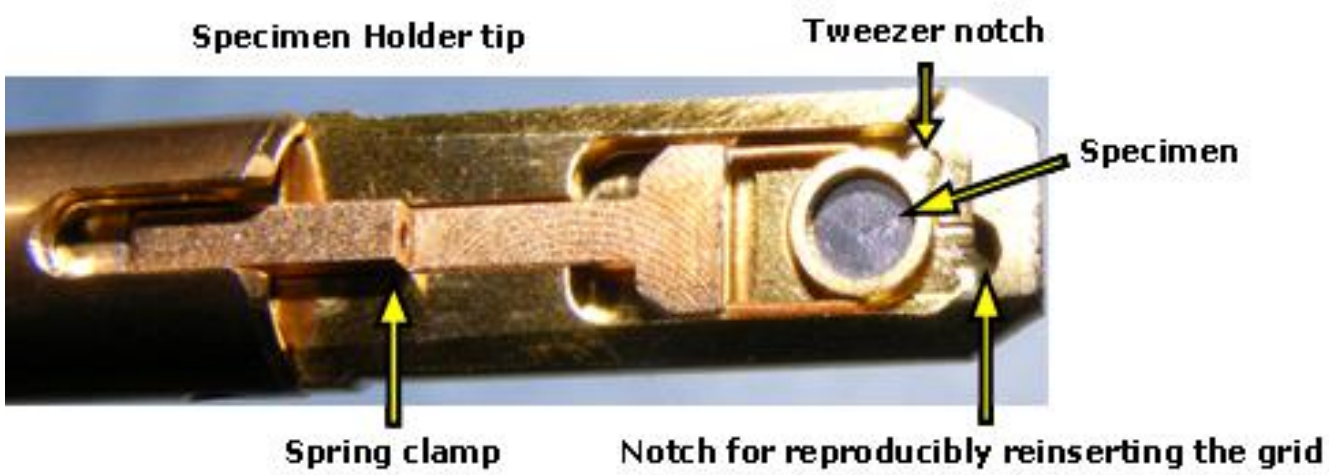
左操作面板

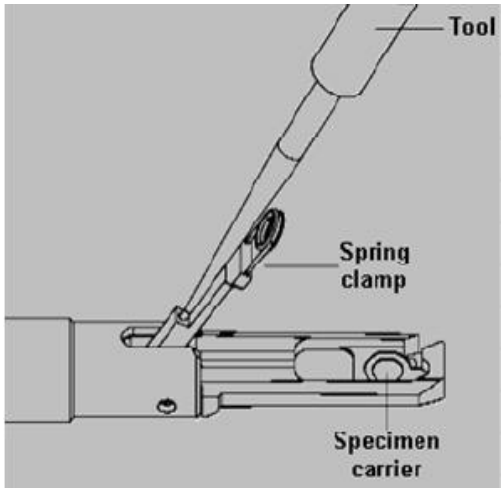
Exposure	曝光按钮，现在不用
Stigmator	消像散按钮
α tilt	样品台a旋转
β tilt	样品台β旋转
Track ball （ Beam shift ）	移动电子束
MF+/-	改变移动速度
Multi function X	根据选择功能改变
Intensity	调节电子束大小
Fine/Coarse	改变调节电子束快慢
L1	个性化键根据客户习惯设定
L2	个性化键根据客户习惯设定
L3	个性化键根据客户习惯设定

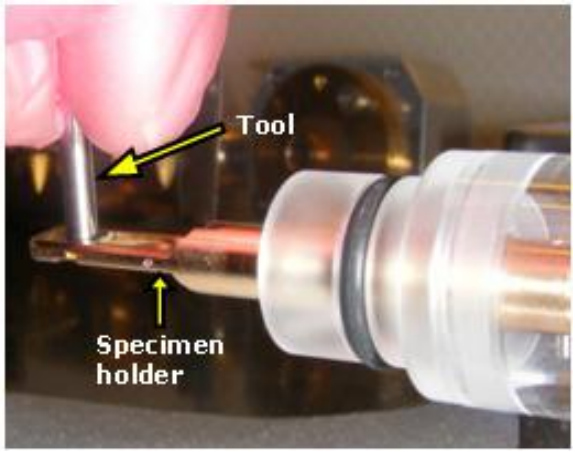
右操作面板

R1	个性化键根据客户习惯设定
R2	个性化键根据客户习惯设定
R3	个性化键根据客户习惯设定
Trackball / Joystick	X,Y方向移动样品
Z-axis	Z方向移动样品
Eucentric focus	物镜电流设定共心高度
Wobbler	电子束摇摆
Diffraction	切换到衍射模式
Dark Field	切换到暗场模式
Focus Step	设定聚焦快慢
Focus	聚焦样品
Magnification	设定放大倍数
Multifunction Y	根据选择功能改变

安装样品(单倾杆/ 低背景双倾杆)



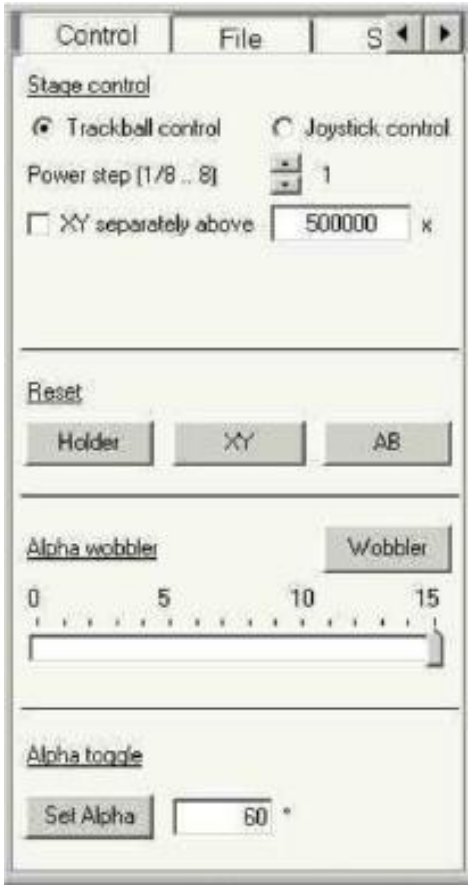




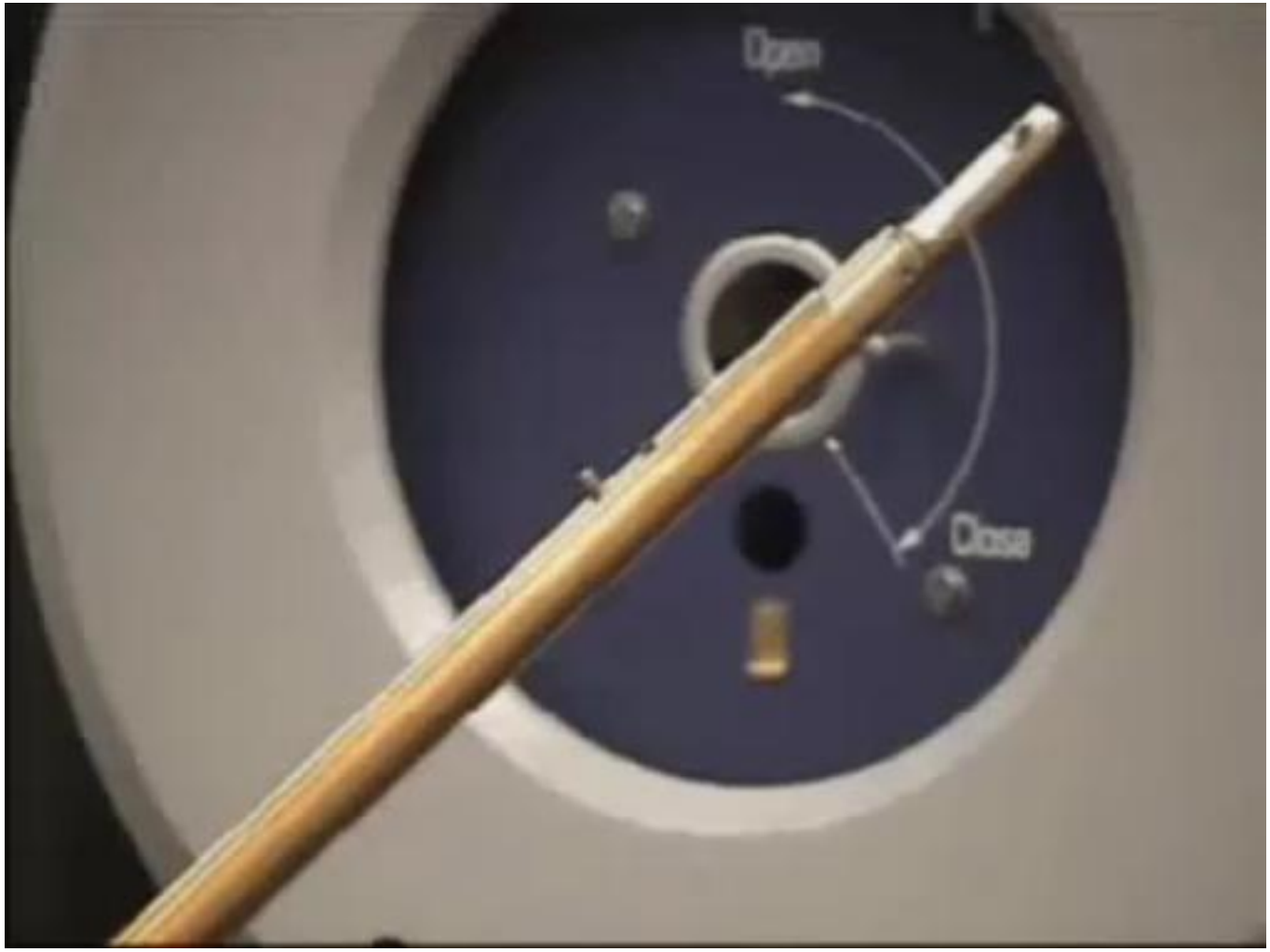
插样品杆前准备工作

- 1，加上液氨

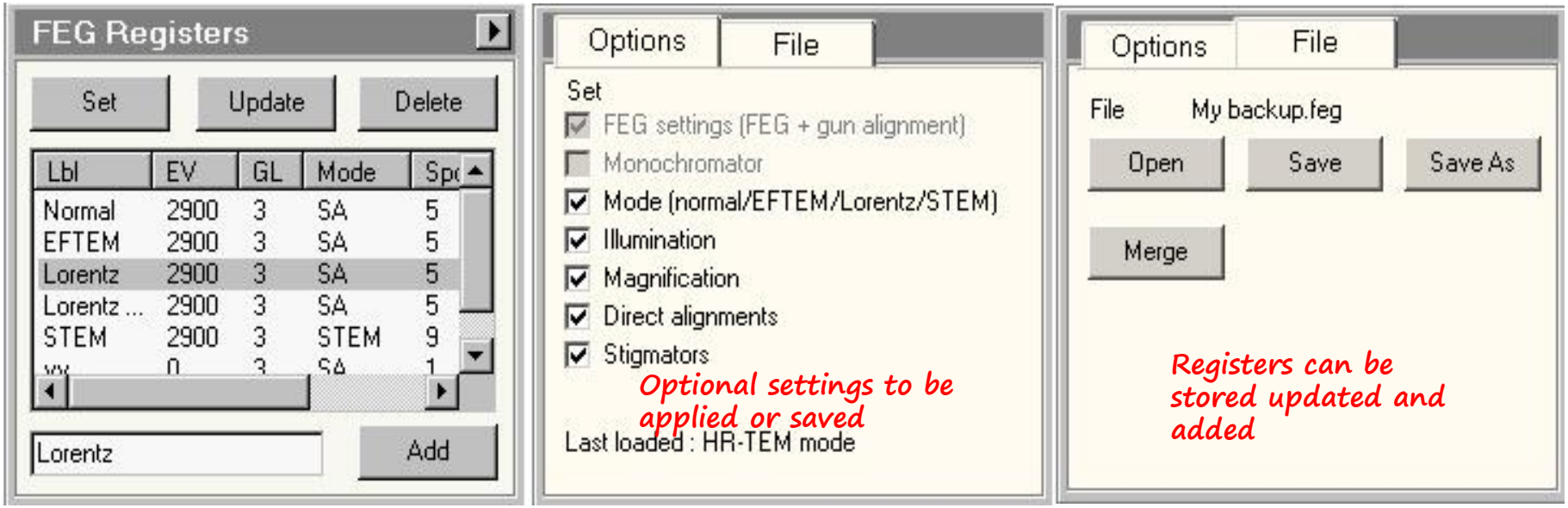
- 2，检查样品台X/Y/Z/a是否为0，
- 如果不是，在Stage的Control窗口中，
- 点Reset Holder



插入样品杆



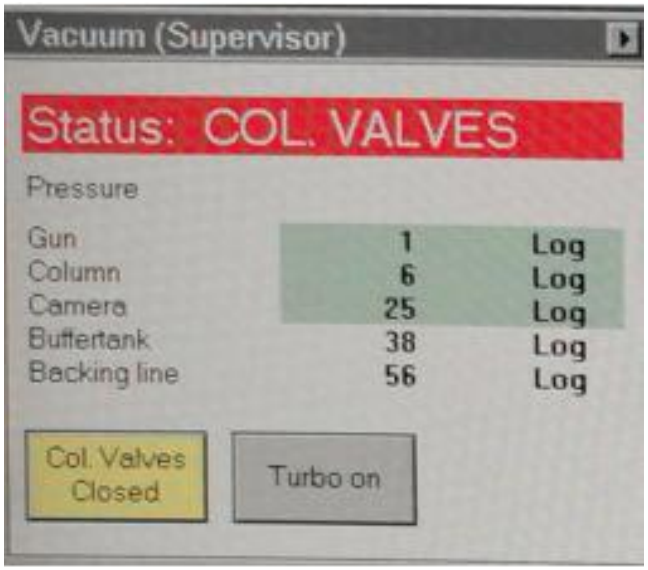
场发射电镜（TF30）FEG register 菜单调用一个Gun的合轴文件



FEG registers

选择将使用的文件，按Set键.

打开镜筒阀(V7& V4)



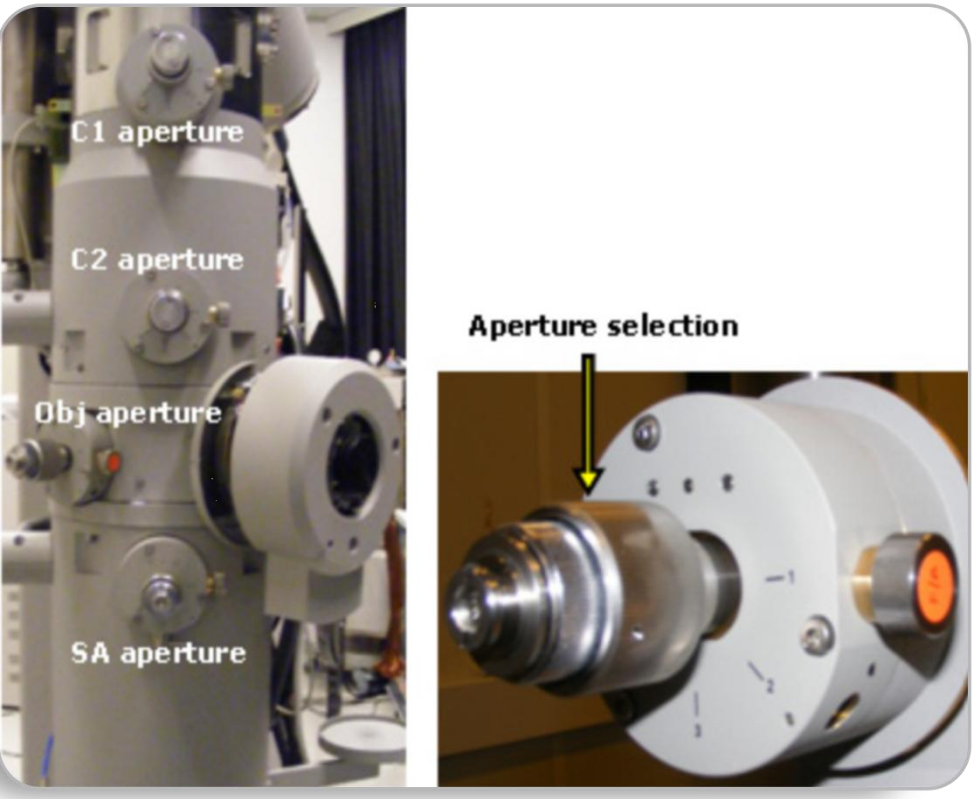
1. 在电镜使用界面，选择[Vacuum] 窗口
2. 检查镜筒真空（非常重要！！！在镜筒真空不达到要求值，打开镜筒，会损坏灯丝）

Instrument Column vacuum level before opening valves

LaB6 or Tungsten	< 35 log
FEG (XFEG and SFEG)	< 20 log

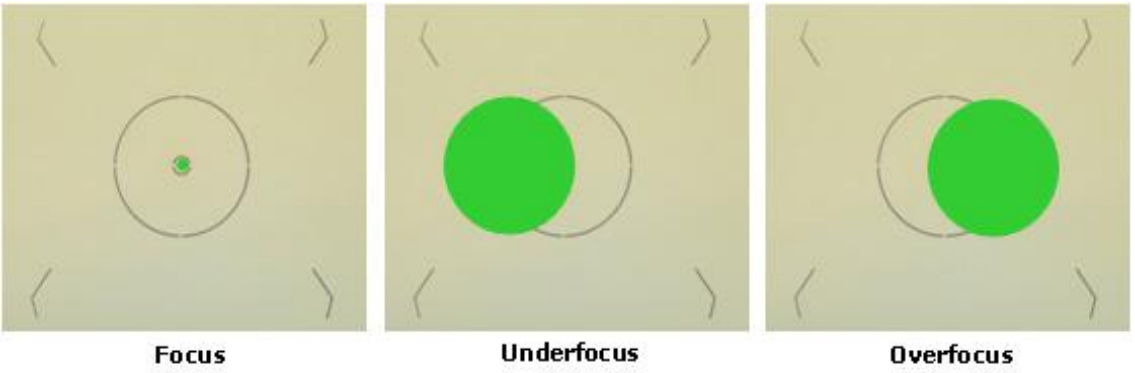
3. 用鼠标点一下[Col. Valves Closed]，打开镜筒阀 ，在[Vacuum]窗口. 在镜筒阀打开状态，按钮颜色变成灰色

选择 C2 aperture

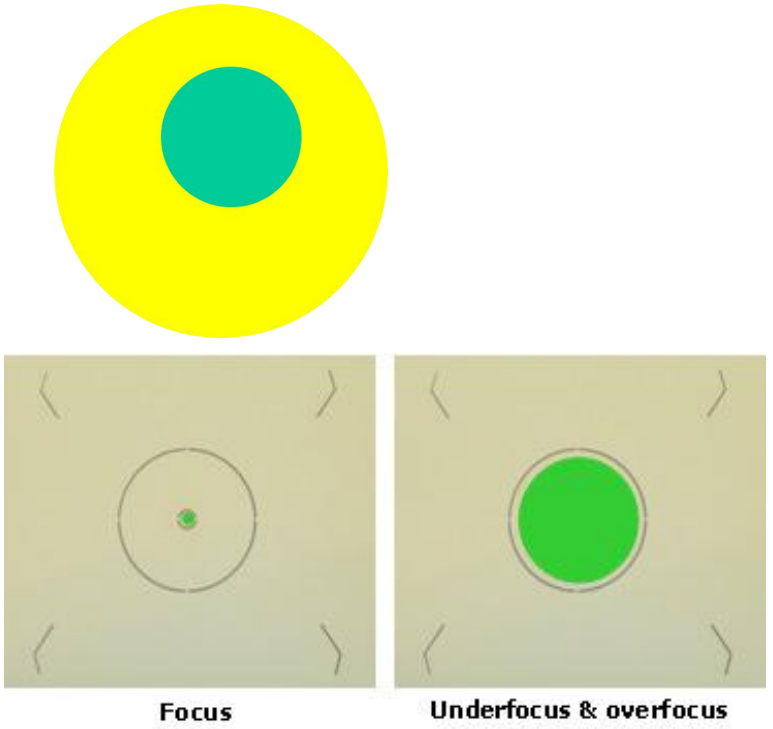


C2 光阑 150um 100um 70um 50um 对应位置4 3 2 1

对中 C2 Aperture



Misaligned Aperture

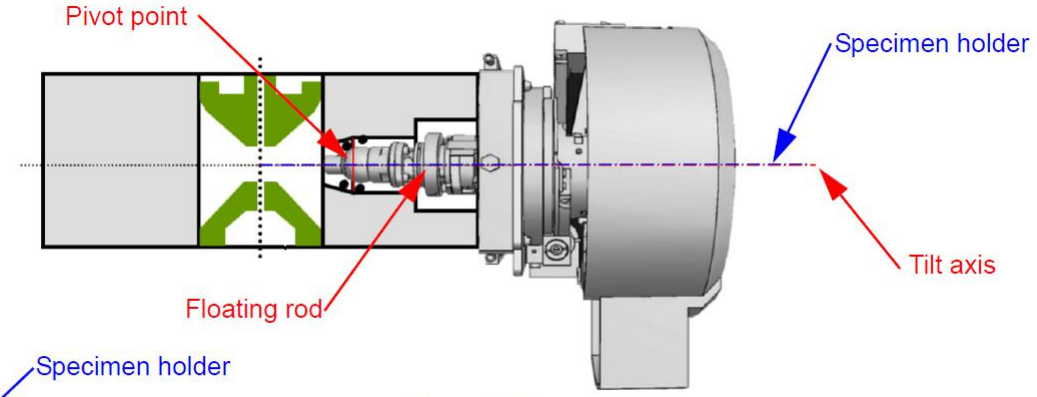


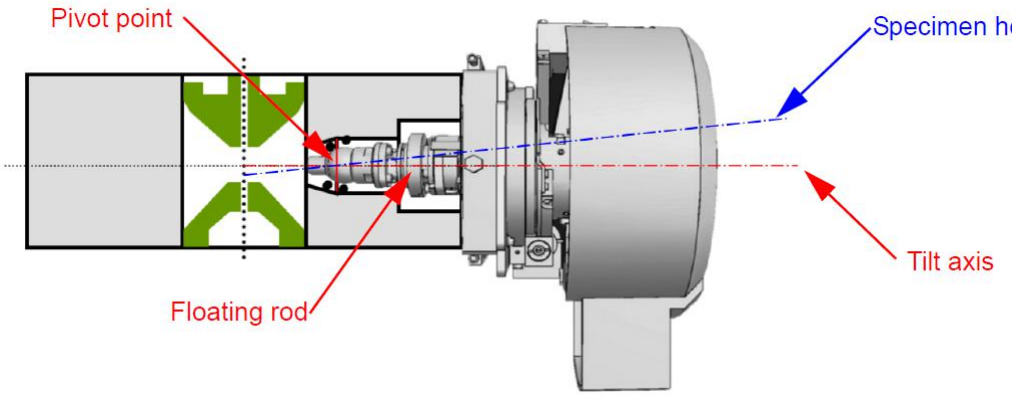
Aligned Aperture

1. 移开物镜（OBJ）和选区光阑（SA）.
2. 选择spotsize 3
3. 选13500x或者以上
4. 聚焦 beam， 用左面板[Intensity] 旋钮
5. 用左面板的轨迹球，将光移到荧光屏中心
6. 顺时针选择[Intensity] 旋钮，过焦散开光束
7. 如果光束不对中，用聚光镜光阑的2个调节旋钮将光束对中荧光屏
8. 重复第4-7步，直到过焦散开光束，光束中心与荧光屏中心重合

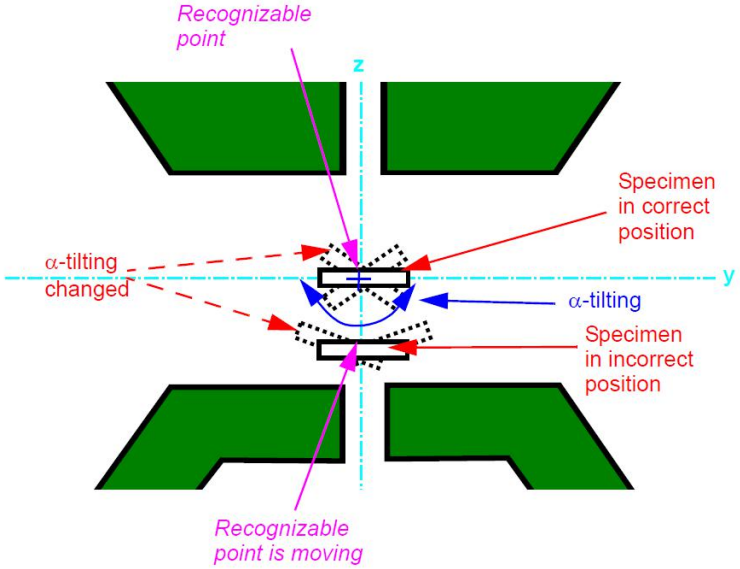
调节样品高度到共心高度(Eucentric Height)

方法一、在10000X-20000X，在Compustage菜单里面 左右转到样品台 (Wobbler)，调节Z轴高度，在荧光屏中心的样品移动最小



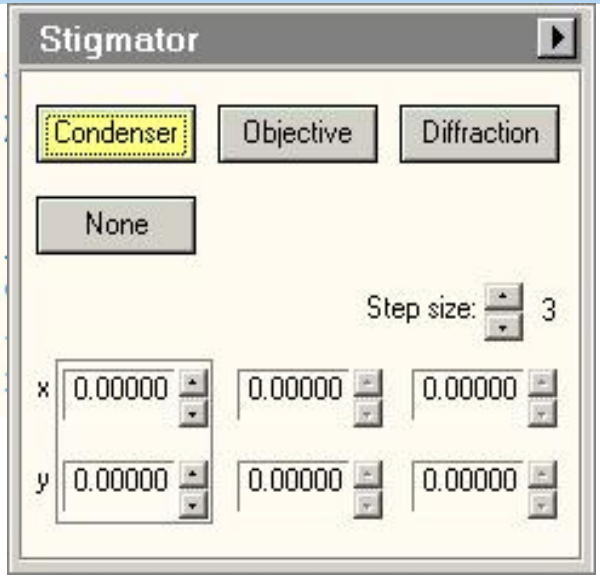


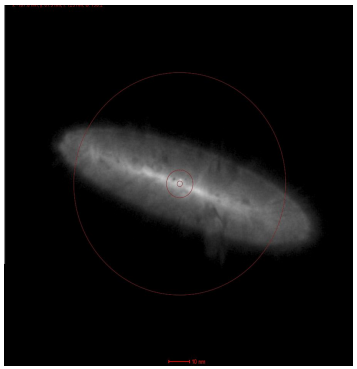
方法二、按一下Eucentric focus，调节Z轴高度，在荧光屏中心的样品的衬度最小



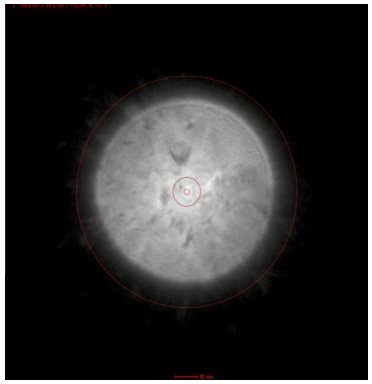
校正光镜C2像散

MF Y: Cond stig Y
MF X: Cond stig X
L1:
L2:
L3: Spotsize -
R2: Toggle µP/nP
R3: Spotsize +
R1: Screen lift





Uncorrected

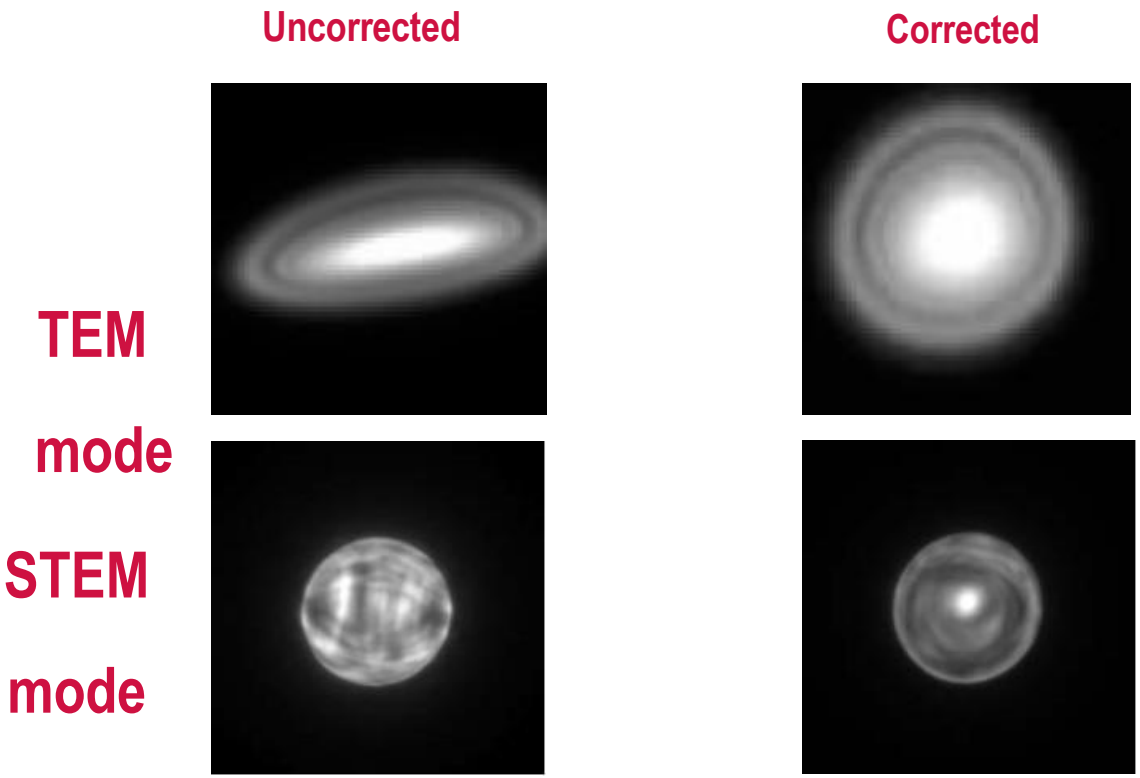


Corrected

1，Lab6/W灯丝电镜，放大倍数在10Kx左右

2，FEG电镜，放大倍数在100KX及以上

校正聚光镜像散(Condenser Astigmatism)

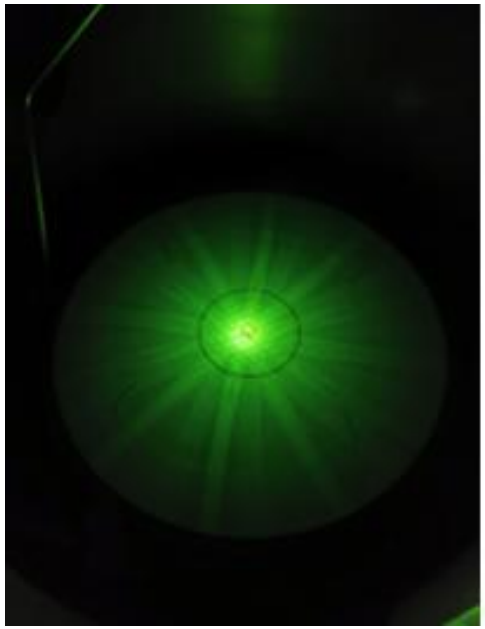


调正样品晶带轴

1，放大倍数在SA范围

2，切换到衍射模式

3，观察衍射花样，如果衍射花样不是沿中心衍射斑对称，需要通过样品台α、β旋转，调正晶带轴

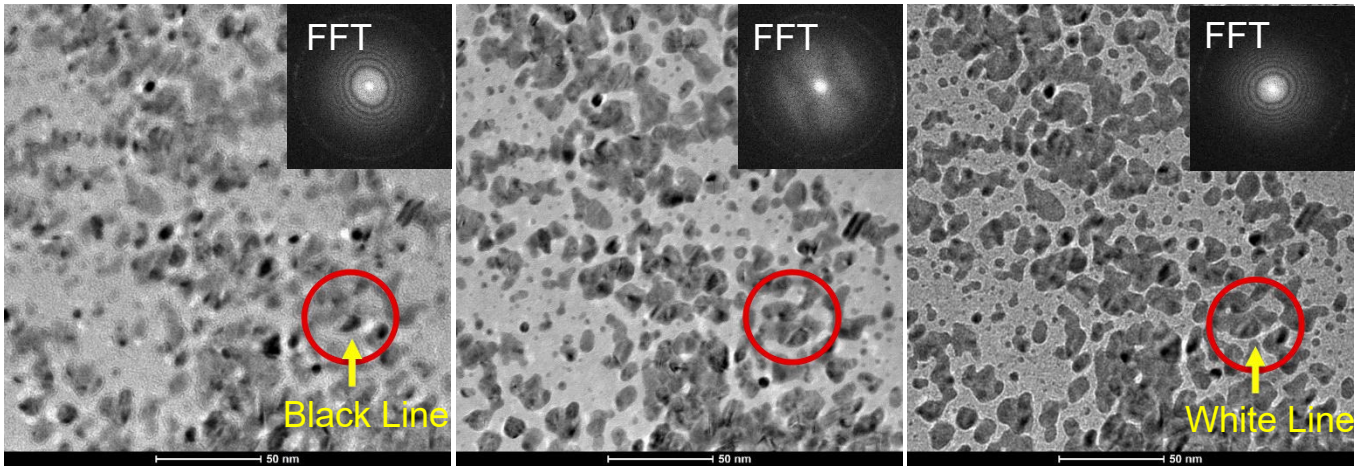


聚焦Focus

1， focus step有5档，1-3档微调，4-5档粗调
2，大小2个圆圈，旋转大的圆圈设定step，旋转小的圆圈改变焦距，顺时针方向旋转是过焦，逆时针方向旋转是欠焦

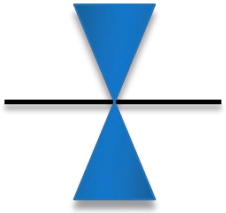
聚焦样品

Objective Astigmatism

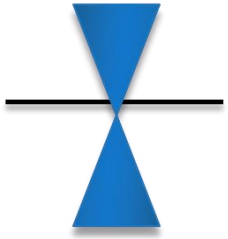


Over focus

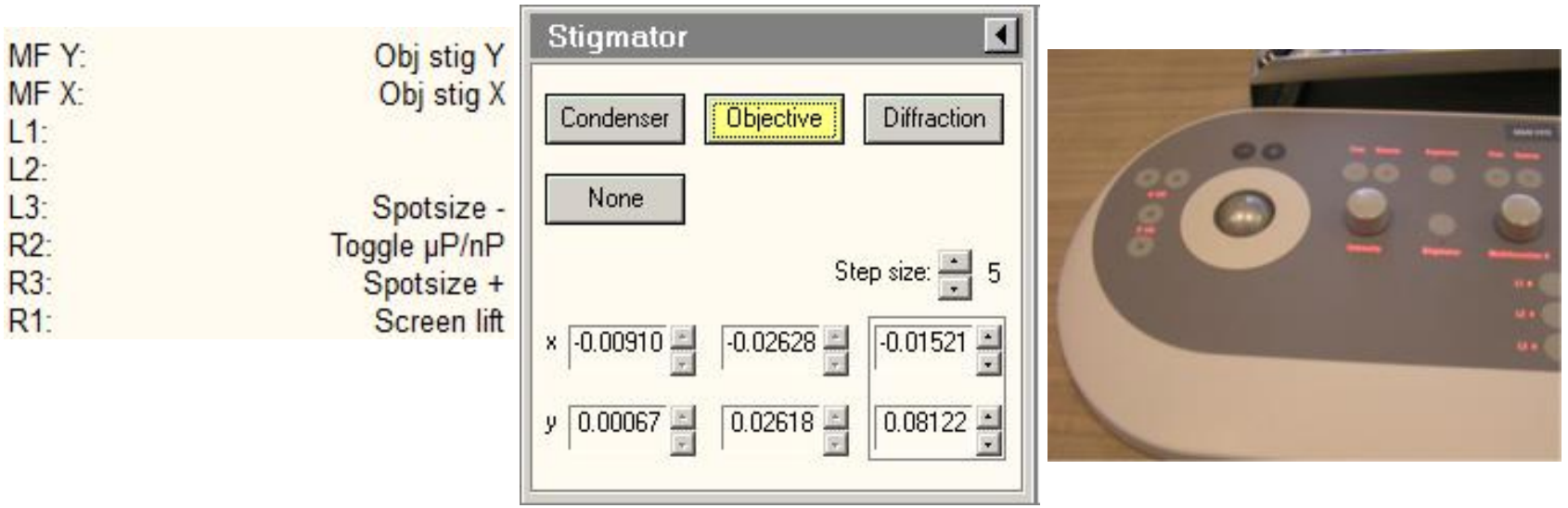
On focus



Under focus



校正物镜像散Objective Stigmator

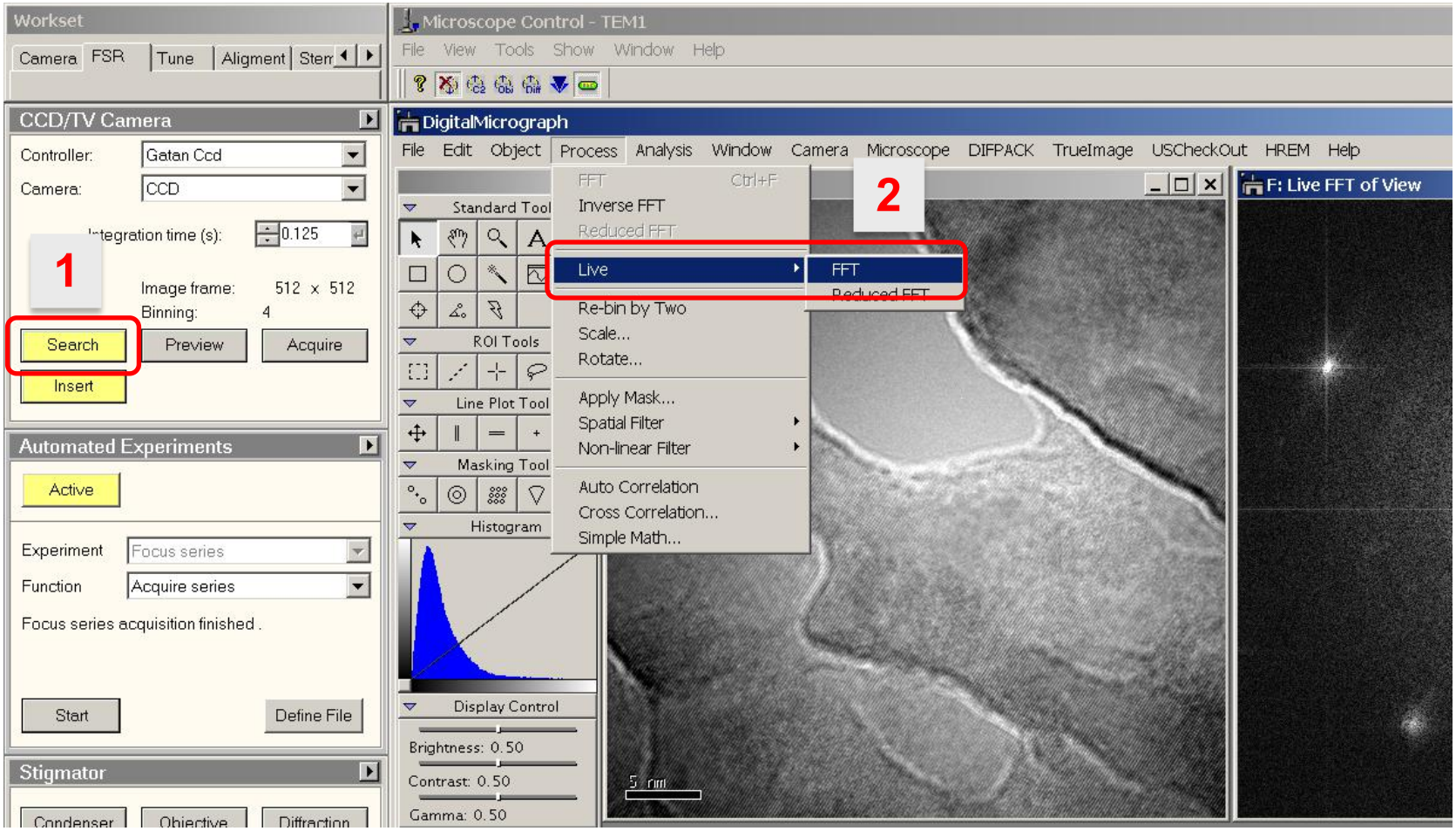


1， 放大倍数在100Kx及以上

2，选择一个无定形的样品区域，如碳膜

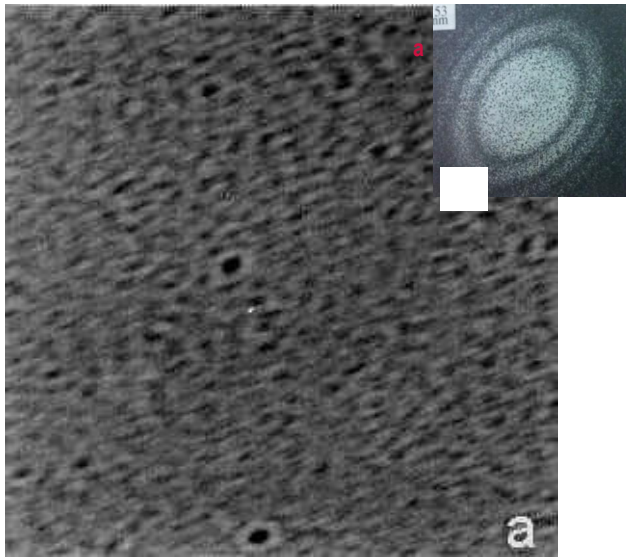
3，在左操作面板上按Stigmator按键，或者在control 菜单里面选择Sigmator，点击Objective

1. Choose SA or Mh magnification range
2. Lift screen and start CCD
3. In TIA Or DM, select Process>Live>FFT

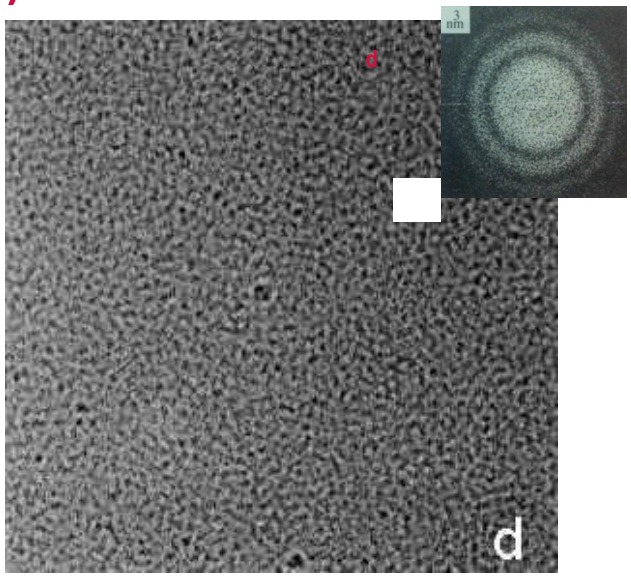


校正物镜像散(Objective Astigmatism)

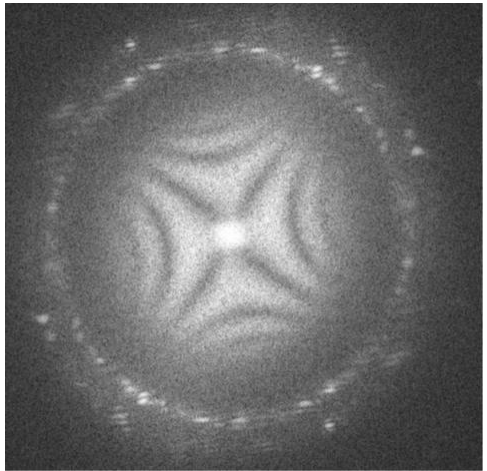
Astigmatism can be corrected using stigmator deflection coils (on carbon)

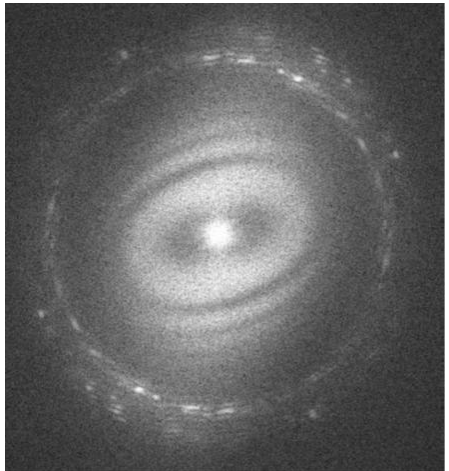


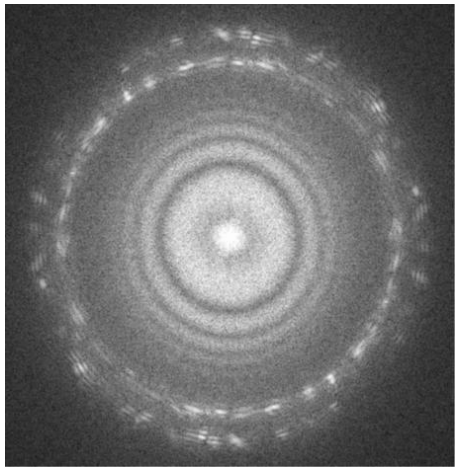
Overfocus / uncorrected



Overfocus / corrected





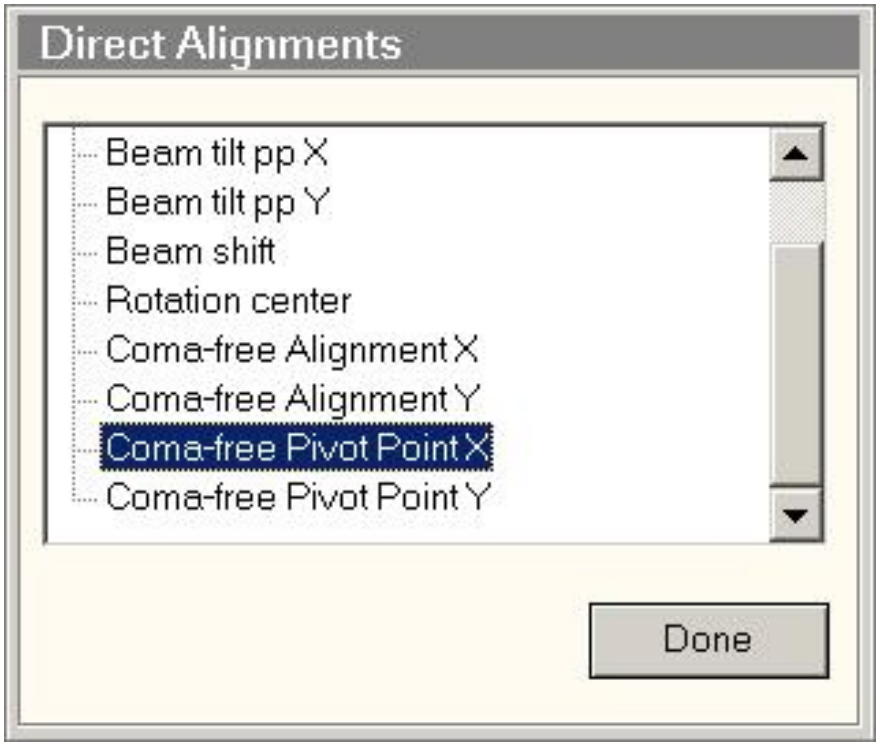


Direct Alignment

Direct access to specific alignments

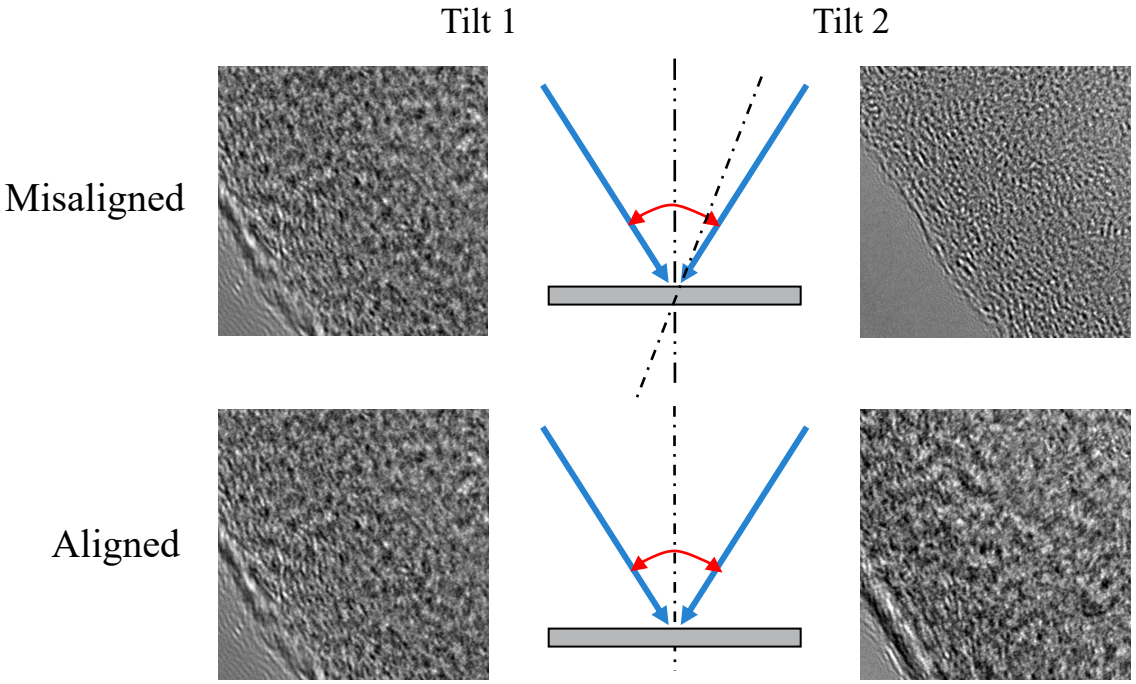
Quick, efficient and straight forward

Different set at different operational mode



Coma-free alignment

- Rotation centre fine alignment
- Two amorphous carbon images (Tilt 1 & Tilt 2) are similar in contrast



Coma-free alignment

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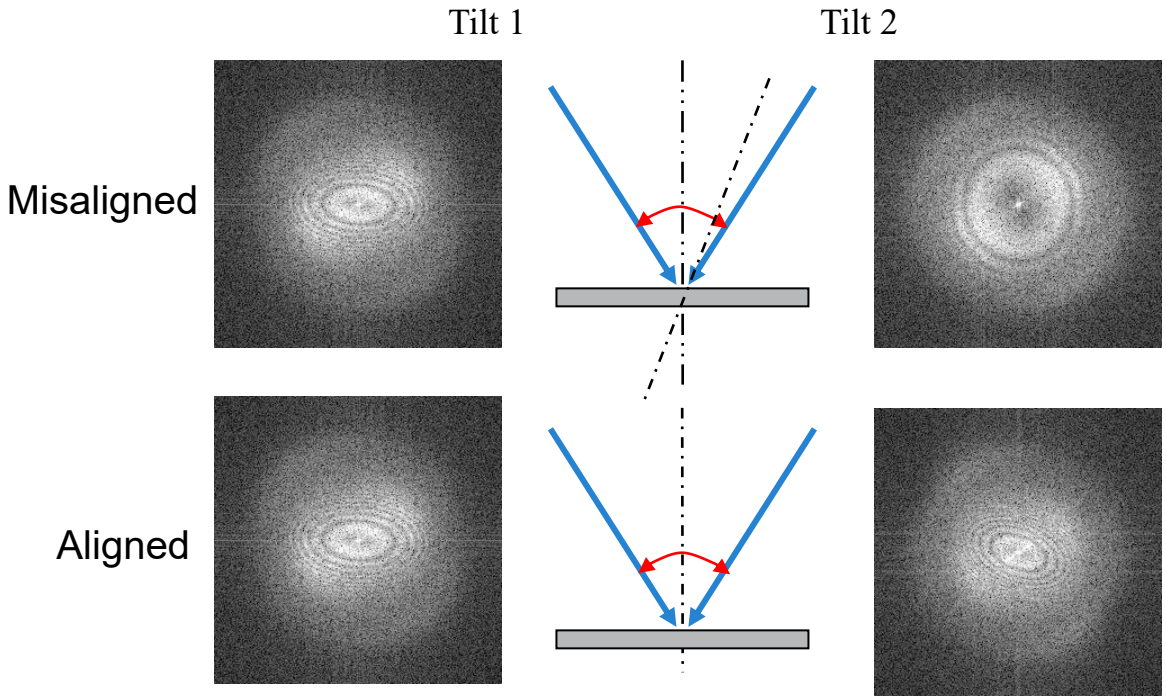
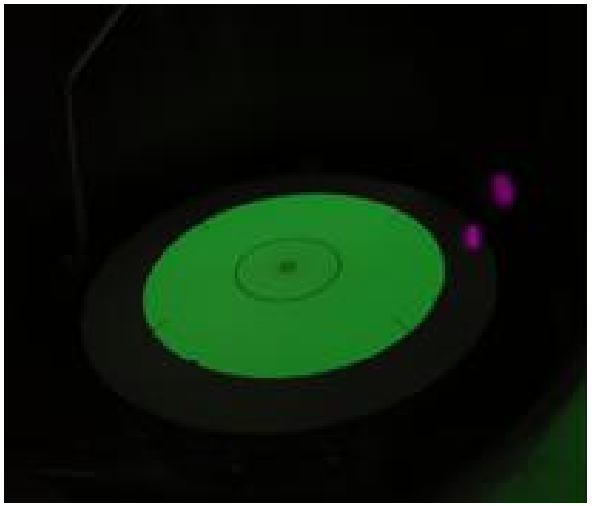
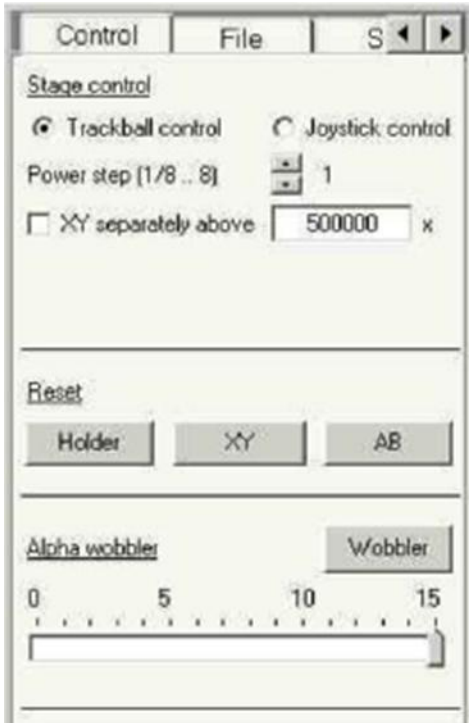
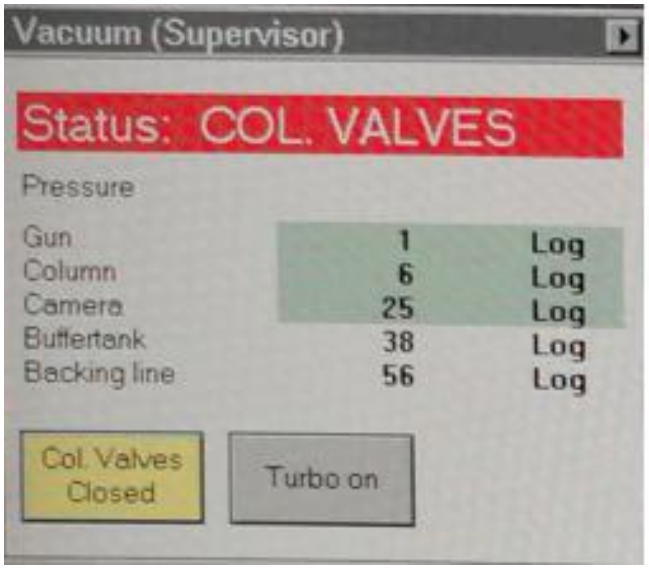


工作结束时准备工作

- 1，将放大倍数设定到M，比如：2250X，
- 将电子束对中，并散开接近荧光屏满屏
- 2，在Stage的Control窗口中，
- 点Reset Holder，将X/Y/Z/ α /β归零
- 3，关闭镜筒阀（V7 &V4）





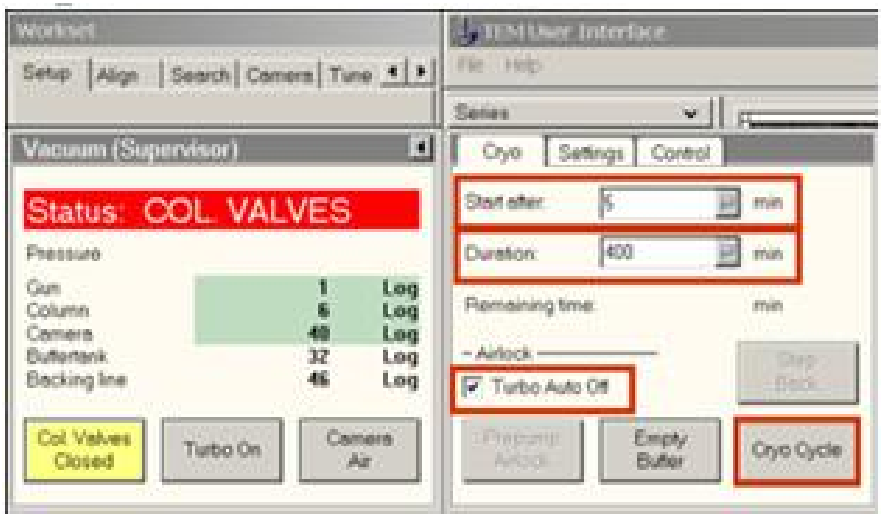


取出样品杆

cryo cycle

Start a cryo cycle

- 1、在[Vacuum] control 窗口，设定好[Start after] and [Duration]时间. 一般开始时间5-10分钟，结束时间大于240分钟。
- 2、点击[Cryo Cycle] 按钮，变黄，开始工作。



Typical FEG setups

Application	C1	C2	GL	Spotsize
TEM	-	70/100	3	1-3
EFTEM	-	150	3	1-3
EDX	(30)	70	3	4-6 (nanoP)
STEM	-	70	3	6 (nanoP)
HR-STEM	-	70	3	9-10 (nanoP)
Holography	-	70	3	3

注意事项

1，插入样品杆和拔出样品杆，必须样品台位置归零，X/Y/Z/ α /β值接近0，避免样品杆与物镜极靴相碰，损坏极靴和样品杆

2，打开镜筒阀（V7 &V4）必须检查Column真空值小于20 Log，避免损伤灯丝

3，衍射时，用Beam Stop挡住中心衍射点，同时，衍射斑尽量弱，避免损伤荧光屏和CCD的闪烁体

4，样品杆安装样品，仔细检查样品是否压紧？双倾杆螺丝是否水平进入样品座？必须将样品杆旋转180°，轻轻敲击样品杆后部，检查样品是否安装好，避免样品掉入镜筒

- Thank You