

# *Microscopia alla nanoscala: il Microscopio Elettronico in Trasmissione (TEM)*



*6 Dicembre 2011*

**Stefano Frabboni**  
Dipartimento di Fisica  
Università di Modena e Reggio E.  
e CNR-INFM-S3



# Introduzione generale sul TEM

NATIONAL CENTER OF CNR-INFN

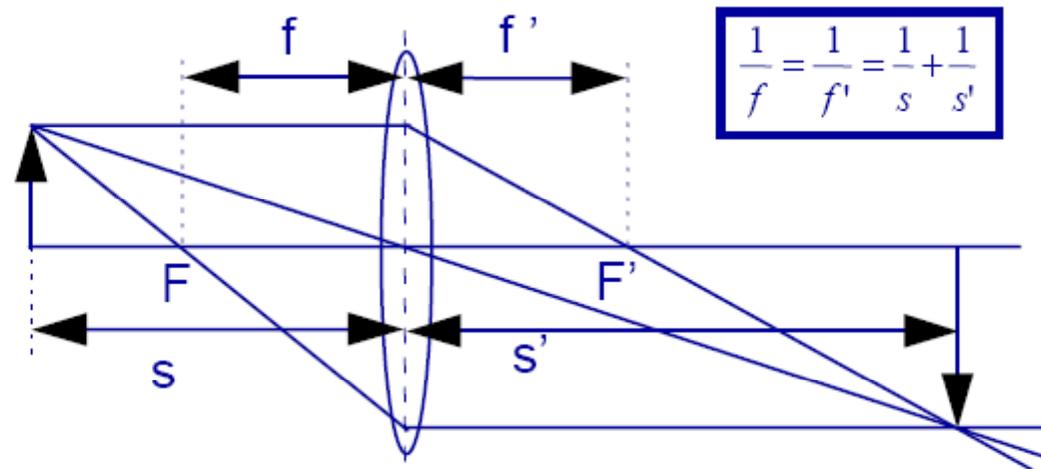


**UNIMORE**  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



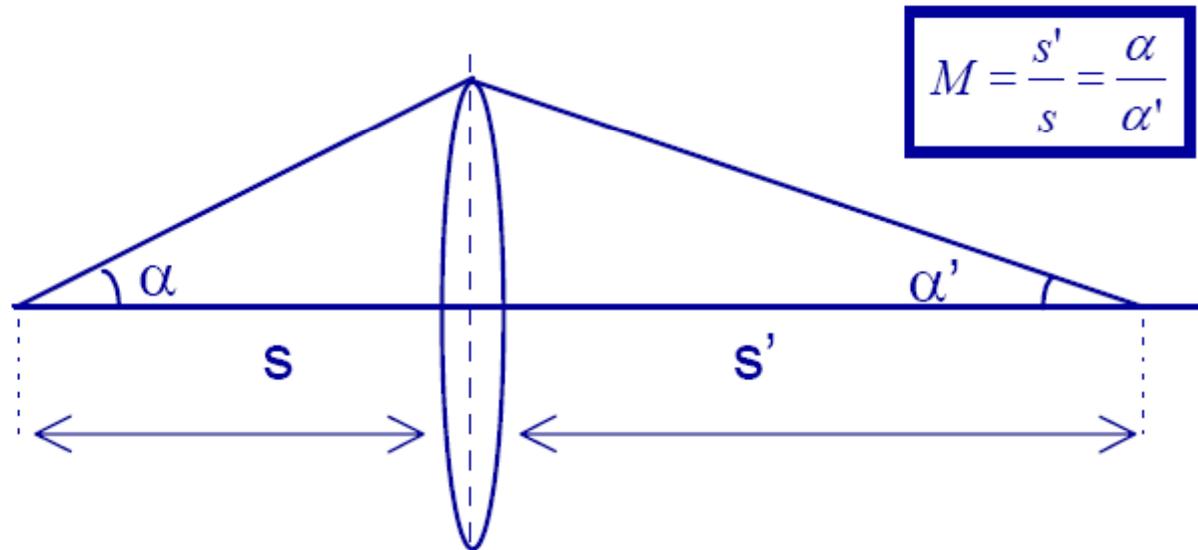
## Lente sottile

- Legge di Gauss



## Lente sottile: ingrandimenti

- Legge di Gauss



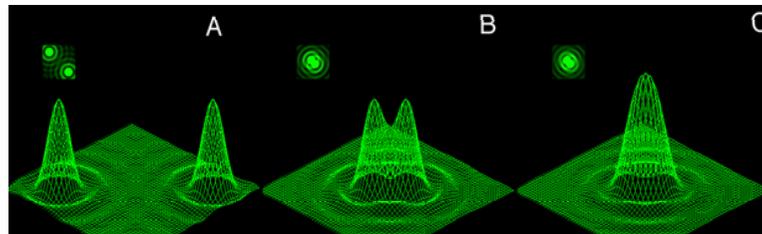
# POTERE RISOLUTIVO

Il *potere risolutivo* dell'occhio umano, ovvero la minima distanza tra due punti che ne permette una visione distinta, è circa 0,1- 0,2mm, se i due punti sono più vicini l'occhio non riesce a *risolverli* e vede quindi un'unica figura.

Un microscopio ottico moderno può raggiungere un potere risolutivo pari a 0,1- 0,2 $\mu$ m corrispondente ad un ingrandimento di circa 1000x.

Ciò che limita la risoluzione di un microscopio ottico è, in ultima analisi, la lunghezza d'onda della luce usata per illuminare il campione.

Colore	$\lambda$ ( $10^{-6}$ m)
rosso	0,780 - 0,622
arancione	0,622 - 0,597
giallo	0,597 - 0,577
verde	0,577 - 0,492
azzurro	0,492 - 0,455
violetto	0,455 - 0,380



$$d_{\min} = \frac{0.61}{n} \frac{\lambda}{\sin \alpha} \quad (2.26)$$

For a high-quality lens,  $\alpha \approx 70^\circ$ , and for  $\lambda = 450$  nm and  $n = 1.56$ ,  $d_{\min}$  reaches  $\sim 200$  nm. The term  $n \sin \alpha$  is called the numerical aperture (NA)

# Invention and Evolution of the Modern TEM

- In 1932, invented by E. Ruska *et al.*
- In 1986, Ruska received the Nobel Prize



Knoll and Ruska (1932)

the first Transmission Electron Microscope



NATIONAL CENTER OF CNR-INFM



UNIMORE  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



# Electron

De Broglie  
wavelength

$$\lambda_{non\ rel} = \frac{h}{p} = \frac{1.22}{\sqrt{E(eV)}}\ nm$$

$$\lambda_{rel} = \frac{h}{p} = \frac{h}{\left[2m_0eE\left(1 + \frac{eE}{2m_0c^2}\right)\right]^{1/2}}$$

Fundamental constants

<b>e</b>	<b>-1.602 x10<sup>-19</sup>C</b>
<b>m<sub>0</sub></b>	<b>9.109 x10<sup>-31</sup>kg</b>
<b>m<sub>0</sub>c<sup>2</sup></b>	<b>511 keV</b>
<b>h</b>	<b>6.626 x10<sup>-34</sup> J s</b>
	<b>4.14 x10<sup>-15</sup>eV s</b>
<b>c</b>	<b>2.998 x10<sup>8</sup>m/s</b>

<i>E</i> (kV)	$\lambda_{non\ rel}$ (pm)	$\lambda_{rel}$ (pm)	$\gamma$ m/m <sub>0</sub>	$v$ (10 <sup>8</sup> m/s)
100	3.86	3.70	1.196	1.644
200	2.73	2.51	1.391	2.086
300	2.23	1.97	1.587	2.330

## Electron Density

A typical electron beam has a current of about 10 picoamperes (1 pA = 10<sup>-12</sup> A). One ampere is 1 coulomb/sec. The electron has a charge of 1.6 x 10<sup>-19</sup> coulomb. Therefore, approximately 60 million electrons per second impinge on the specimen. However, because of their high speed, the average distance between electrons (at 200,000 km/second) would be over three meters. Most electrons transit the specimen one at a time.

NATIONAL CENTER OF CNR-INFN

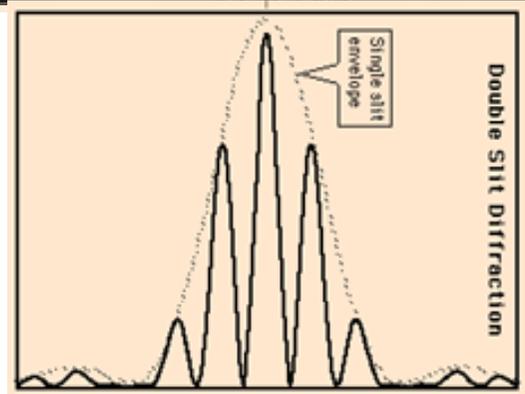
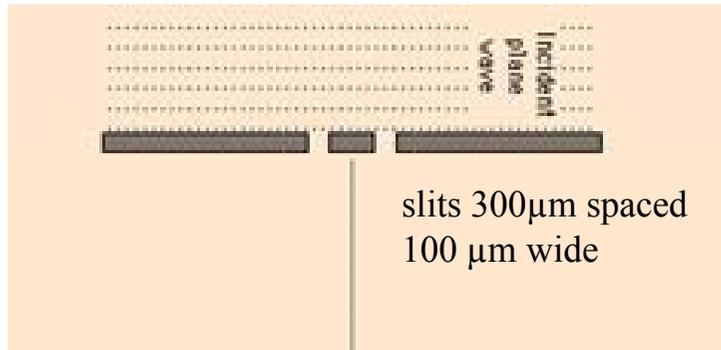


**UNIMORE**  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



# Esperimento di Young

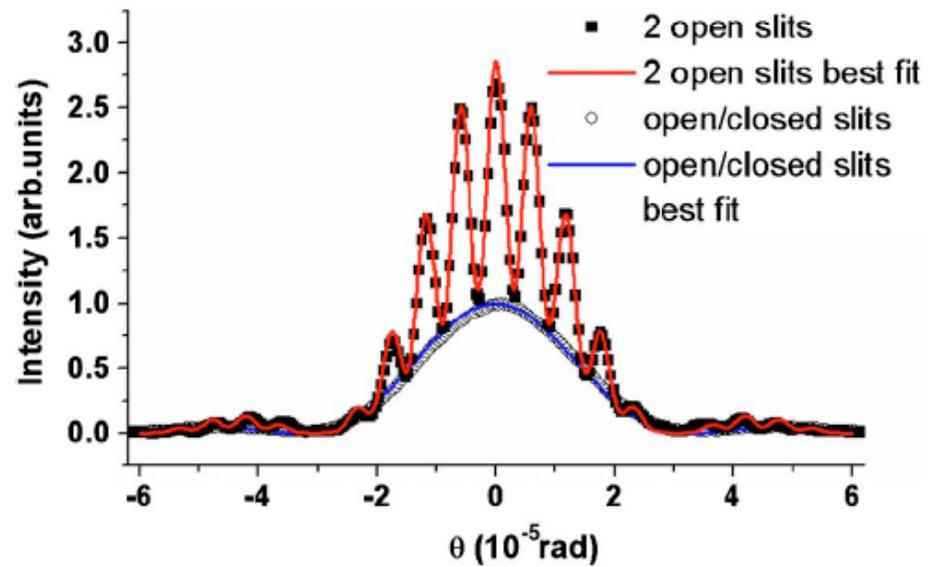
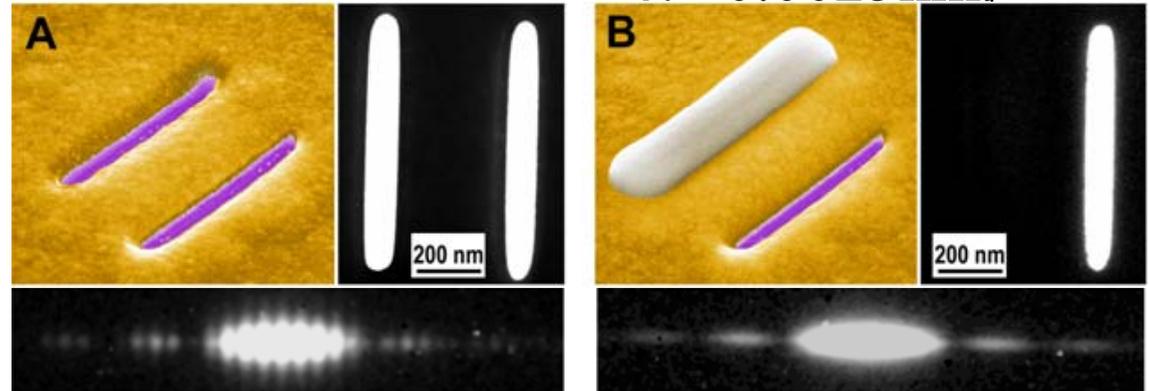
Laser He-Ne  $\lambda \sim 0.630\mu\text{m}$



$2 \times 10^{-3} \text{rad}$

FIB fabricated slits  $80\text{nm}$  wide,  $400\text{nm}$  spaced

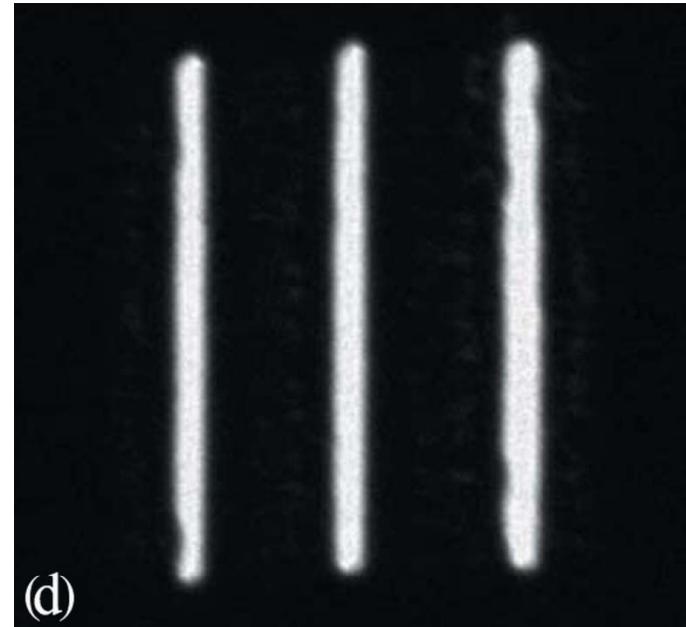
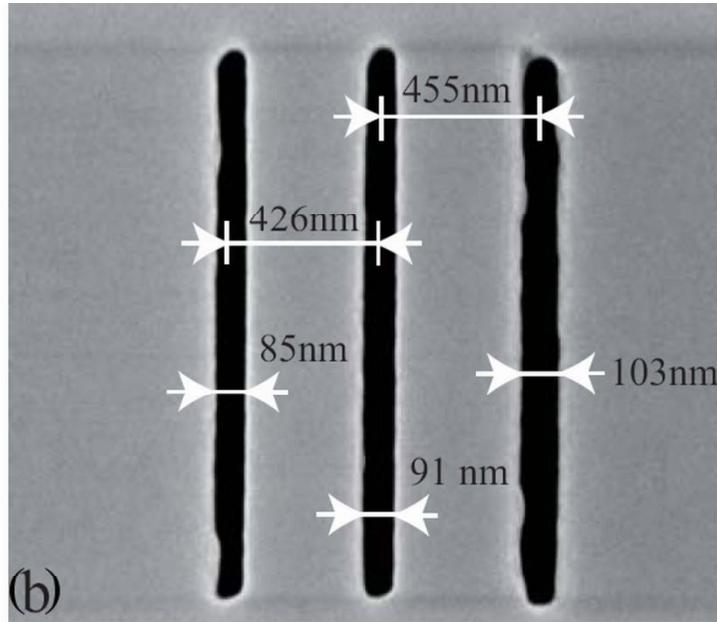
200keV electrons  
 $\lambda \sim 0.0025\text{nm}$ .



NATIONAL CENTER OF CNR-INFN

# 3 fenditure:

Immagine SEM e TEM



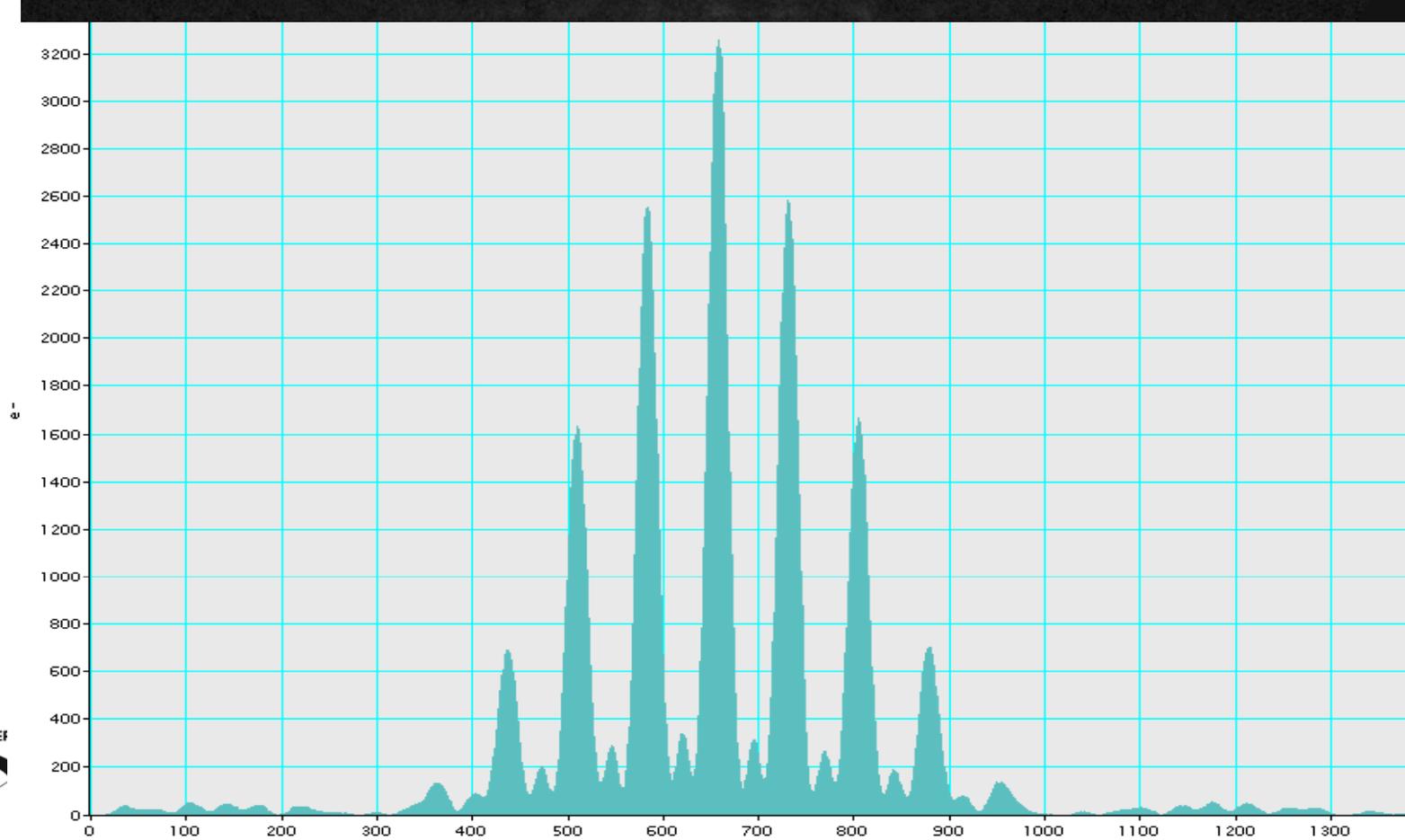
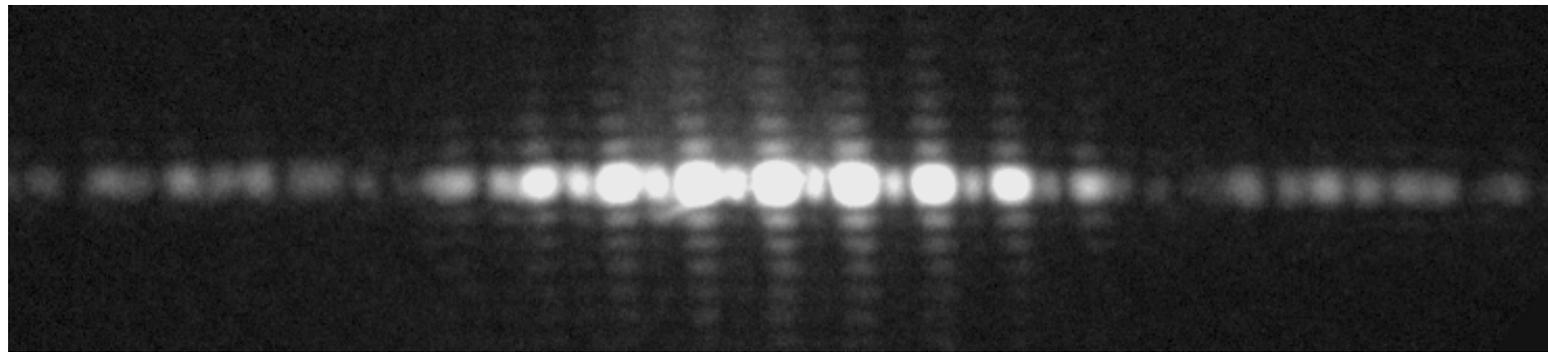
NATIONAL CENTER OF CNR-INFM



**UNIMORE**  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



# 3 fenditure: Fraunhofer e massimi secondari



NATIONAL CENTER



NANOSTRUCTURES AND  
BIOSYSTEMS AT SURFACES

UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



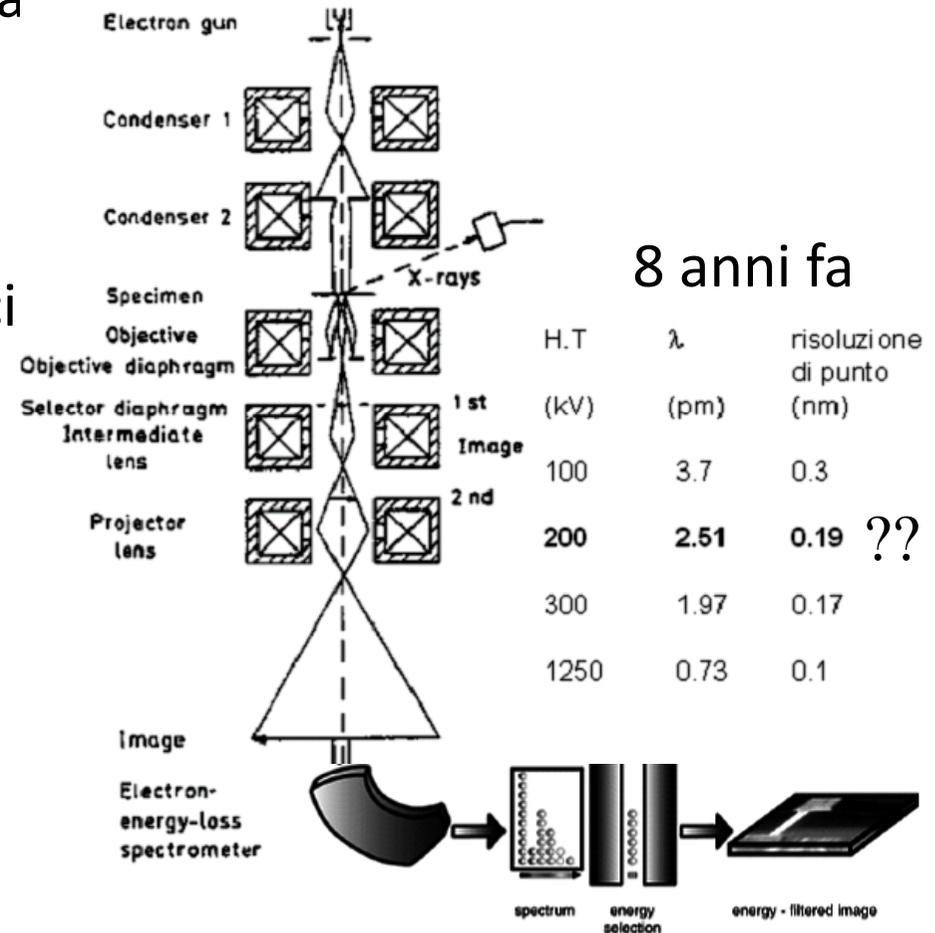


NATIONAL CENTER OF CNR-IR



# Hardware:

- Impianto da vuoto
- Sorgente elettronica
  - Termoionica
  - Field Emission
  - Schottky
- Deflettori magnetici
- Diaframmi
  - Condensatore
  - Obiettivo
  - Campo
- Lenti magnetiche
  - Condensatrici
  - Obiettivo
  - Intermedia
  - Proiettore
- Spettrometri per elettroni o raggi X
- Rivelatori (CCD)

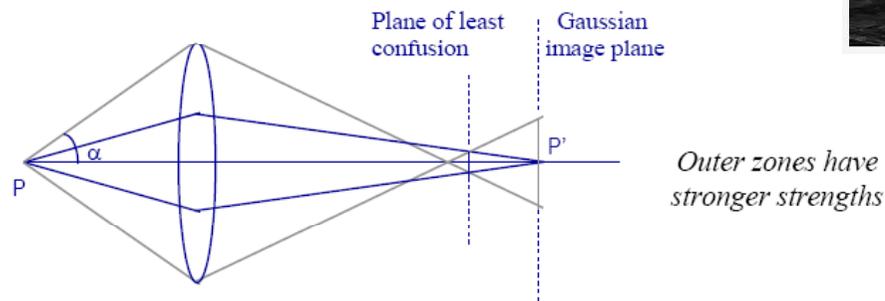


8 anni fa

# Lenses

## Spherical Aberration

- Lens imperfections lead to different focal lengths in centre and at edges of lens



$$\rho_s = C_s \alpha^3$$

$$C_s = 1 \text{ mm (tipico)}$$

$$\rho_s = 0.1 \text{ nm} \implies \alpha \sim 5 \text{ mrad} \sim \theta_{\text{Bragg}} (100 \text{ keV})$$

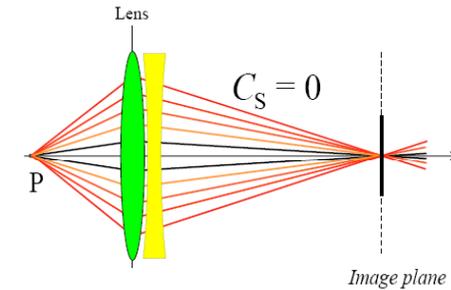
NATIONAL CENTER OF CNR-INFN



UNIMORE  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



La nuova era...



**TEAM**

Transmission Electron  
Aberration-Corrected  
Microscope

2009, march

**Moving atoms in graphene**  
Working with TEAM 0.5, researchers have made a movie that shows in real-time carbon atoms repositioning themselves around the edge of a hole that was punched into a graphene sheet. Viewers can observe how chemical bonds break and form as the suddenly volatile atoms are driven to find a stable configuration.

**Beating the Bohr radius**  
Germanium atoms are arranged in rows of dumbbell pairs aligned end-to-end. Ordinarily, the dumbbells are too small to be resolved with STEM. But, as the figure shows, the LBNL microscope could resolve the 47-pm separation between two paired atoms.

**Test driving TEAM 0.5**  
The development of an ultrastable electron microscope with aberration-correcting optics and a monochromated high-brightness source has significantly improved instrument resolution and contrast. The instrument's new capabilities were exploited to detect a buried grain boundary and observe the dynamic arrangements of single atoms and atom pairs with sub-angstrom resolution.

NATIONAL CENTER OF CNR-INFM



<http://ncem.lbl.gov/TEAM-project/>

**UNIMORE**  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



# Thermoionic emission

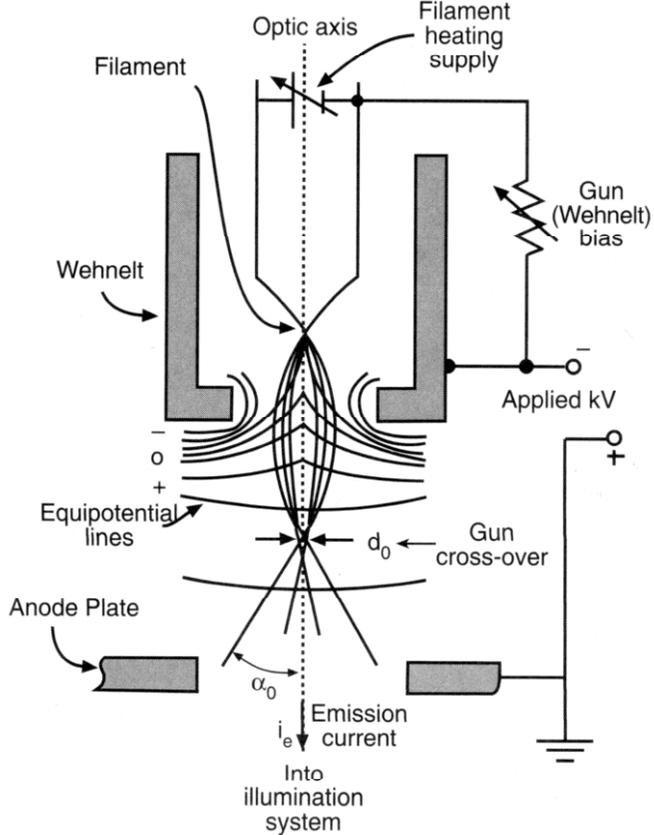
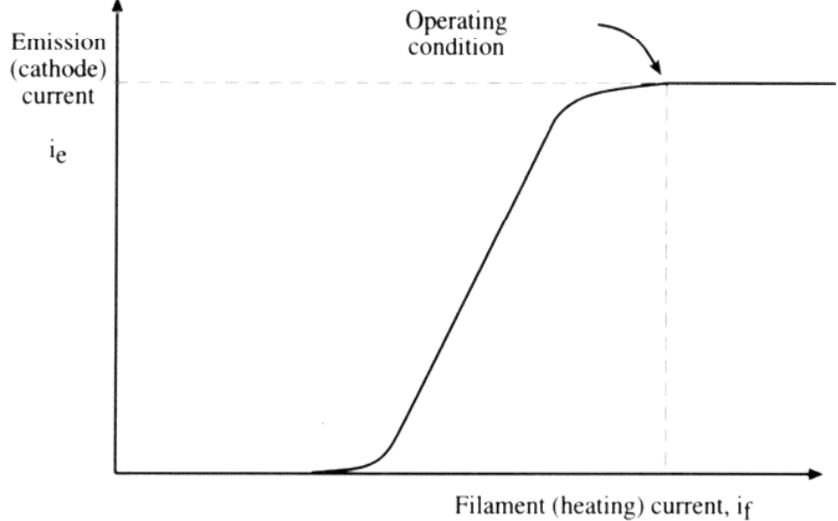
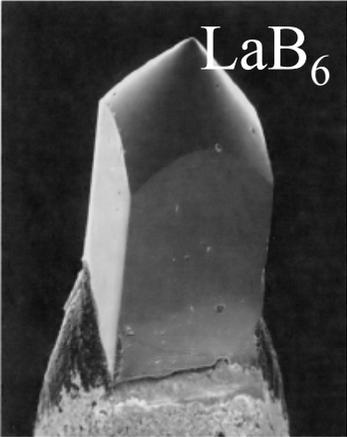


Figura 4 - Caratteristica del cannone elettronico



from Williams, Carter "Transmission electron microscopy"

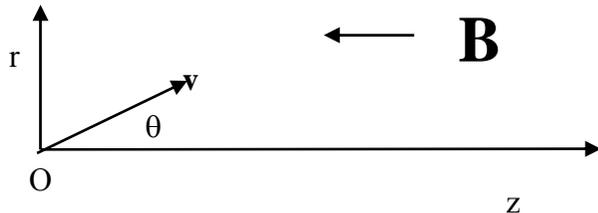


# Magnetic deflection (focusing)

$$\mathbf{F} = q \mathbf{v} \times \mathbf{B}$$

$$\mathbf{v} \perp \mathbf{B} \Rightarrow r = \frac{mv}{qB} = \frac{v}{\omega_c}$$

$\mathbf{v} \parallel \mathbf{B} \Rightarrow$  no deflection



Hp ::

$$\mathbf{v} = v_r \hat{r} + v_z \hat{z} \quad ; \quad \mathbf{B} = -B_z \hat{z}$$

$$v_z = v \cos \theta$$

$$\rho = \frac{m v_x}{qB} = \frac{m v}{qB} \sin \theta$$

Helical trajectories :

$$L_z = v_z T = v_z 2\pi / \omega_c$$

$$L_z = \frac{2\pi m v}{qB} \cos \theta = \frac{2\pi m v}{qB} \left( 1 - \frac{1}{2} \theta^2 + \dots \right) = L_0 \left( 1 - \frac{1}{2} \theta^2 + \dots \right)$$

$$\Delta z = \frac{L_0}{2} \theta^2 \Rightarrow \rho = \Delta z \tan \theta \approx \frac{L_0}{2} \theta^3 \approx C_s \theta^3$$

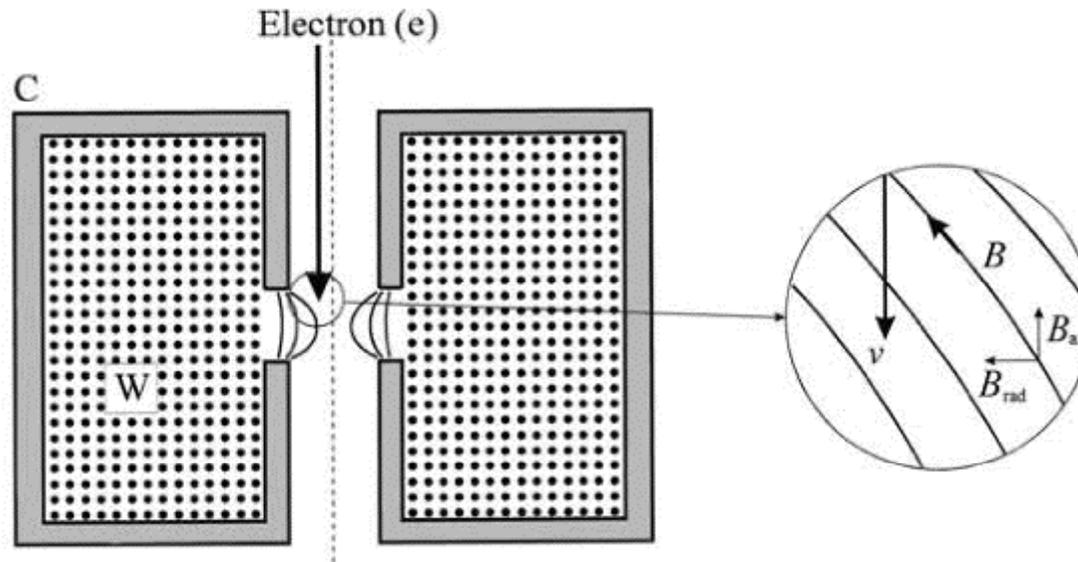
Spherical  
aberration

NATIONAL CENTER OF CNR-INFN



UNIMORE  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



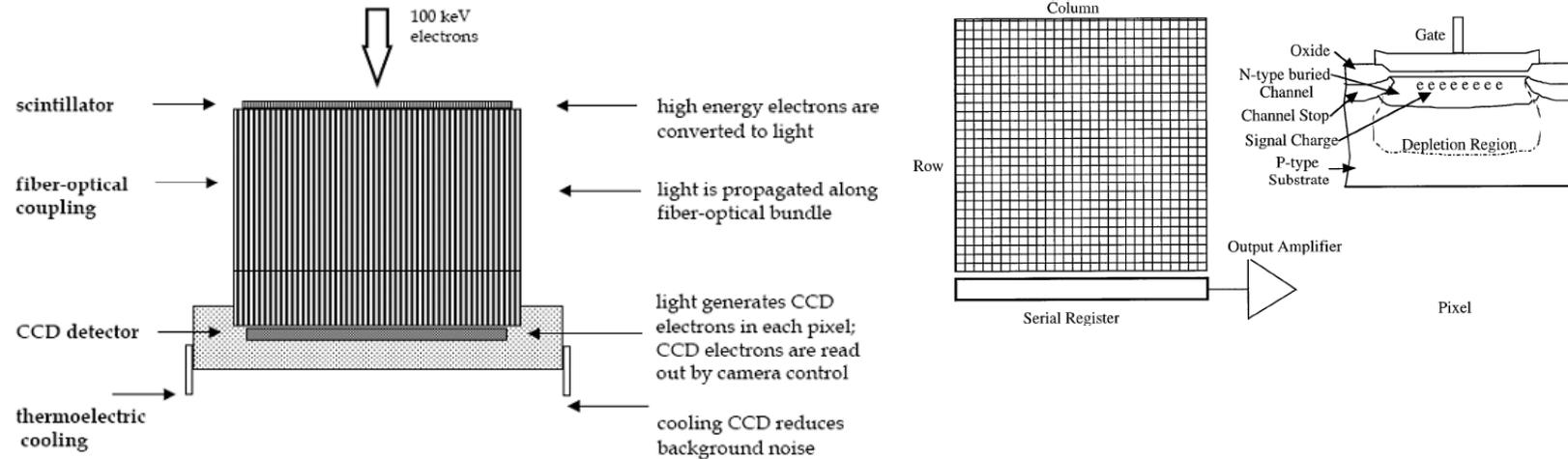


A typical electromagnetic lens is designed to provide a magnetic field almost parallel to the direction of travel of the electrons. An electron entering the lens (Figure 2.5) experiences a magnetic field  $\mathbf{B}$  which can conveniently be resolved into components  $B_{ax}$  along the axis of the microscope and  $B_{rad}$  in a radial direction. Initially the electron is unaffected by  $B_{ax}$ , which is parallel to its direction of travel, but experiences a small force of magnitude  $B_{rad} ev$  from

the small radial component. This force causes the electron to travel in a helix along the lens. As soon as it starts to spiral it has a component of velocity  $v_{circum}$  perpendicular to the plane of the paper and therefore experiences a force of magnitude  $B_{ax} ev_{circum}$  in a radial direction. Thus the helical path follows a tighter and tighter radius and the effect is that a parallel beam of electrons entering the lens is caused to converge to a point exactly as light is focused by a glass lens. If the magnetic field only extends over a short distance along the axis, then the lens behaves as a 'thin lens' and all the geometrical expressions quoted in Chapter 1 apply.

# Detector: CCD camera

Fiber-optical coupling:



Linear Dyn range	<b>10<sup>4</sup> counts</b>
Number of pixels	<b>2<sup>10</sup>x2<sup>10</sup>-2<sup>11</sup>x2<sup>11</sup></b>
Pixel size	<b>25x25micron<sup>2</sup></b>
PSF $h(i,j)$ (from MTF)	<b>2-5 pixels</b>
DQE [SNR <sub>out</sub> /SNR <sub>in</sub> ] <sup>2</sup>	<b>~0.8</b> (100-1000)e/pix
Gain ( $g$ )= $\langle I \rangle / \langle N_e \rangle$ :	<b>2</b>

$$I^{raw}(i, j) = g \cdot h(i, j) \otimes I_0(i, j) + B(i, j)$$

$$I(i, j) = \frac{I^{raw}(i, j) - I^{dark}(i, j)}{I^{gain}(i, j) - I^{darkref}(i, j)}$$

NATIONAL CENTER OF CNR-IRFM



# Electron-specimen interactions

CTEM

HREM

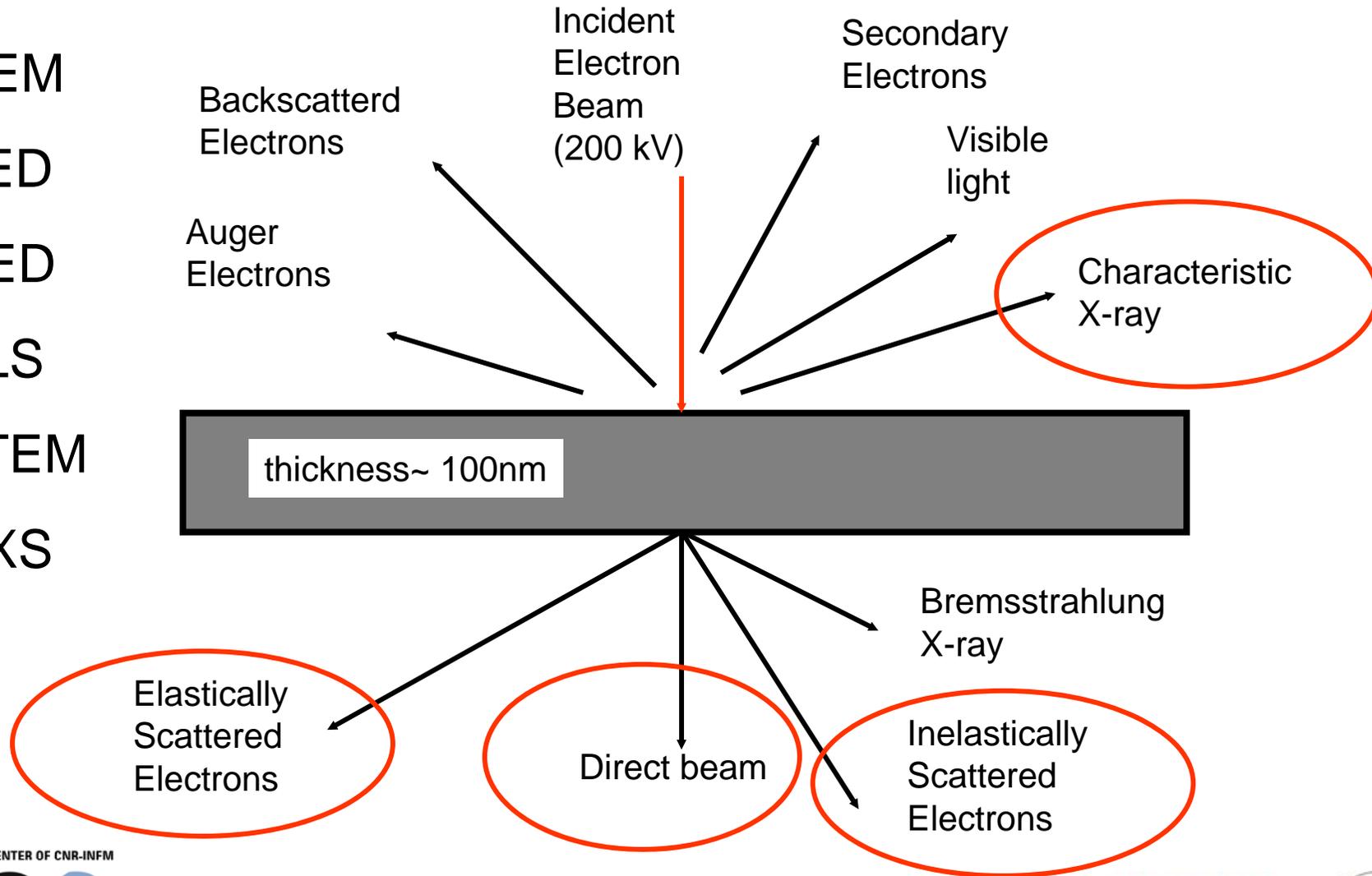
SAED

CBED

EELS

EFTEM

EDXS



NATIONAL CENTER OF CNR-IFM



PERCHE' ASSOTTIGLIARE IL CAMPIONE?

NATIONAL CENTER OF CNR-INFN



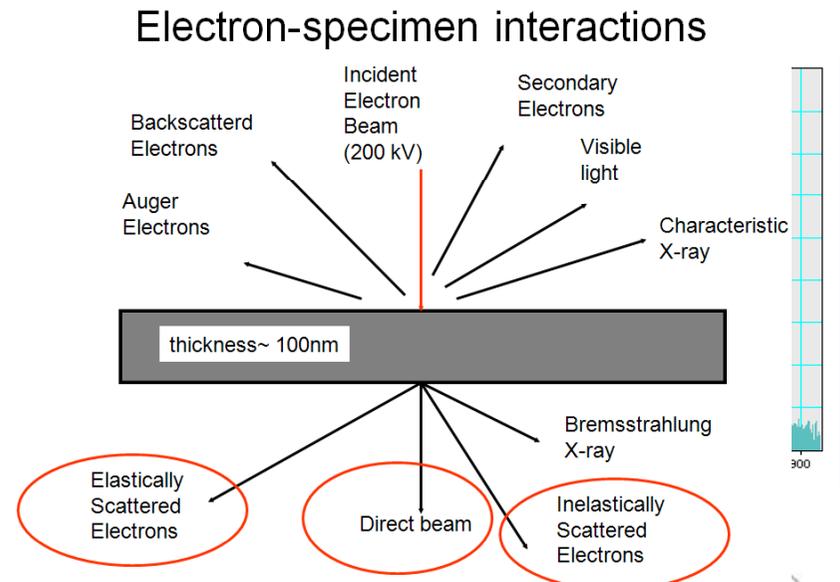
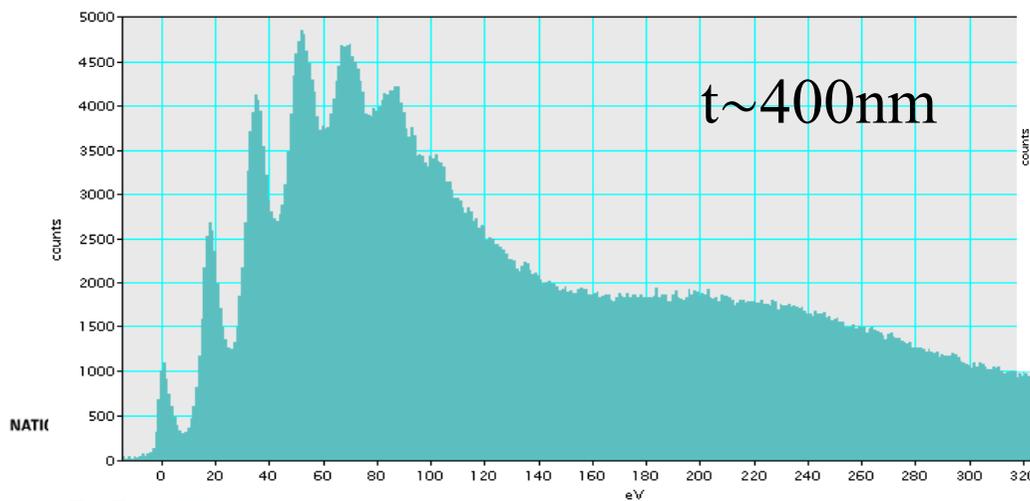
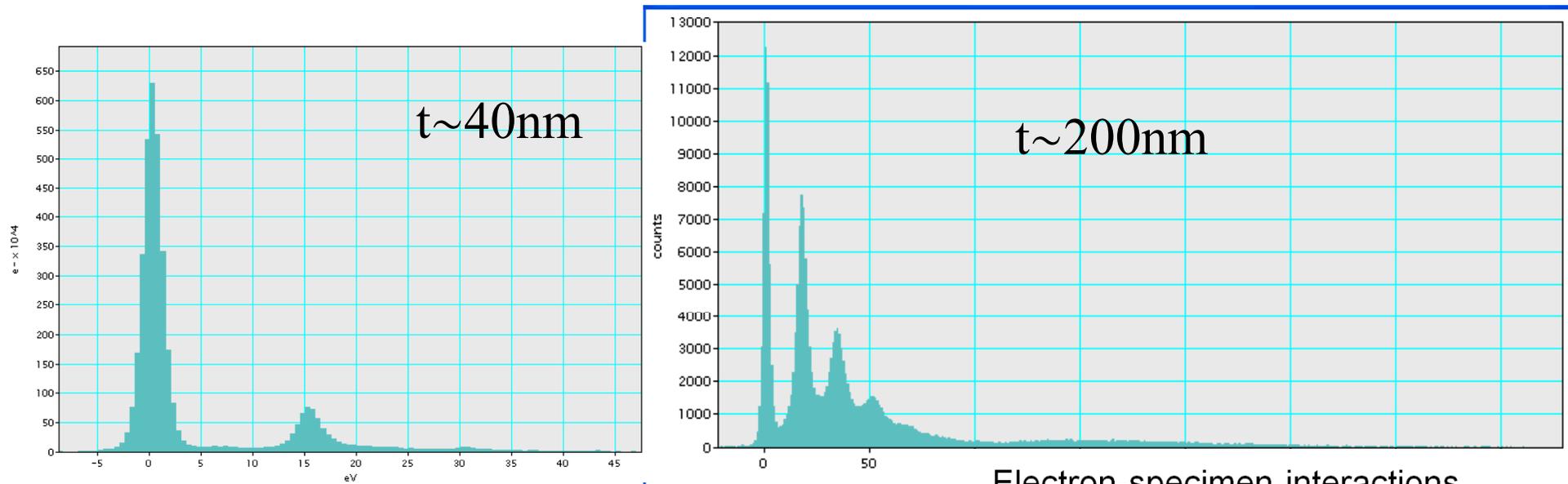
NANOSTRUCTURES AND  
BIOSYSTEMS AT SURFACES

UNIMORE

UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



Effetto dello spessore del campione sulla distribuzione energetica del fascio trasmesso: spettri EELS in trasmissione in funzione dello spessore del campione.



# Specimen preparation

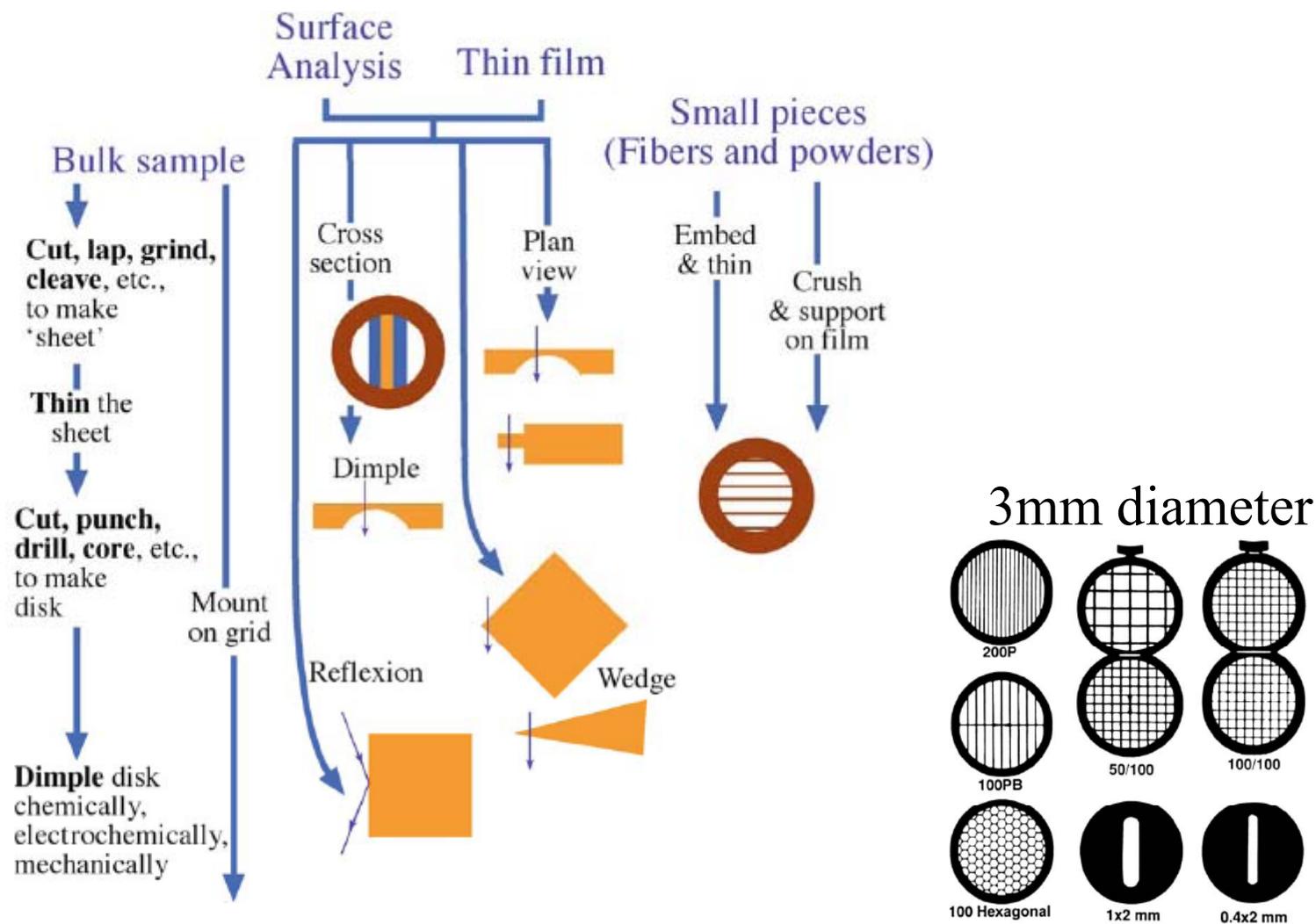


FIGURE 10.26. Summary flow chart for specimen preparation.

NATIONAL CENTER OF CNR-IRFM



from Williams, Carter "Transmission electron microscopy"

**UNIMORE**  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



# Analisi strutturali e composizionali con AEM/HREM

## Determinazione del tipo di stato condensato (Diffrazioni & Immagini)

- amorfo
- policristallo
- monocristallo

## Riconoscimento di una struttura nota

- Composizione chimica (EDX and EELS)
- Dimensioni e simmetria cella unitaria da confrontare con data-base di strutture note (D&I)
- Funzione radiale negli amorfici (D, EXELFS)

## Caratterizzazione di modifiche a strutture note analisi difetti cristallografici

- Campi di deformazione  $\Rightarrow$  strain ( D & I )
- Misure di disordine statico (D)
- Studio difetti cristallografici (D,I) (HREM+Image Simulation)
- Mappe elementali (EDXS, EELS)

## Determinazione di una nuova struttura

- Composizione chimica (EDX, EELS)
- Dimensioni e simmetria cella unitaria (D & I)
- Posizioni atomiche nella cella unitaria (D & I)
- Studio del legame chimico (D & ELNES)

NATIONAL CENTER OF CNR-IFM

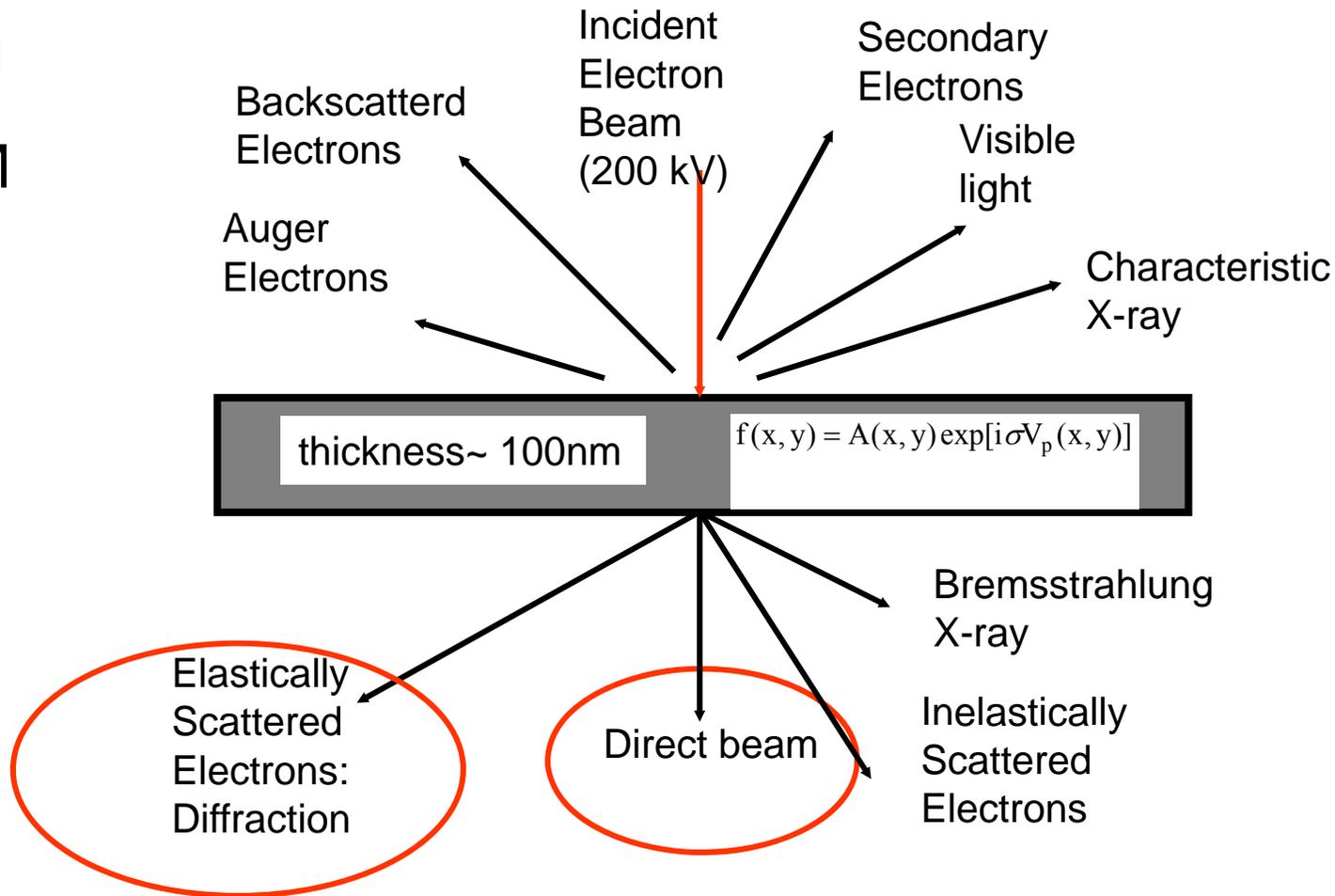


UNIMORE  
UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA



# Elastic electron-specimen interactions

CTEM  
HREM  
SAED  
CBED

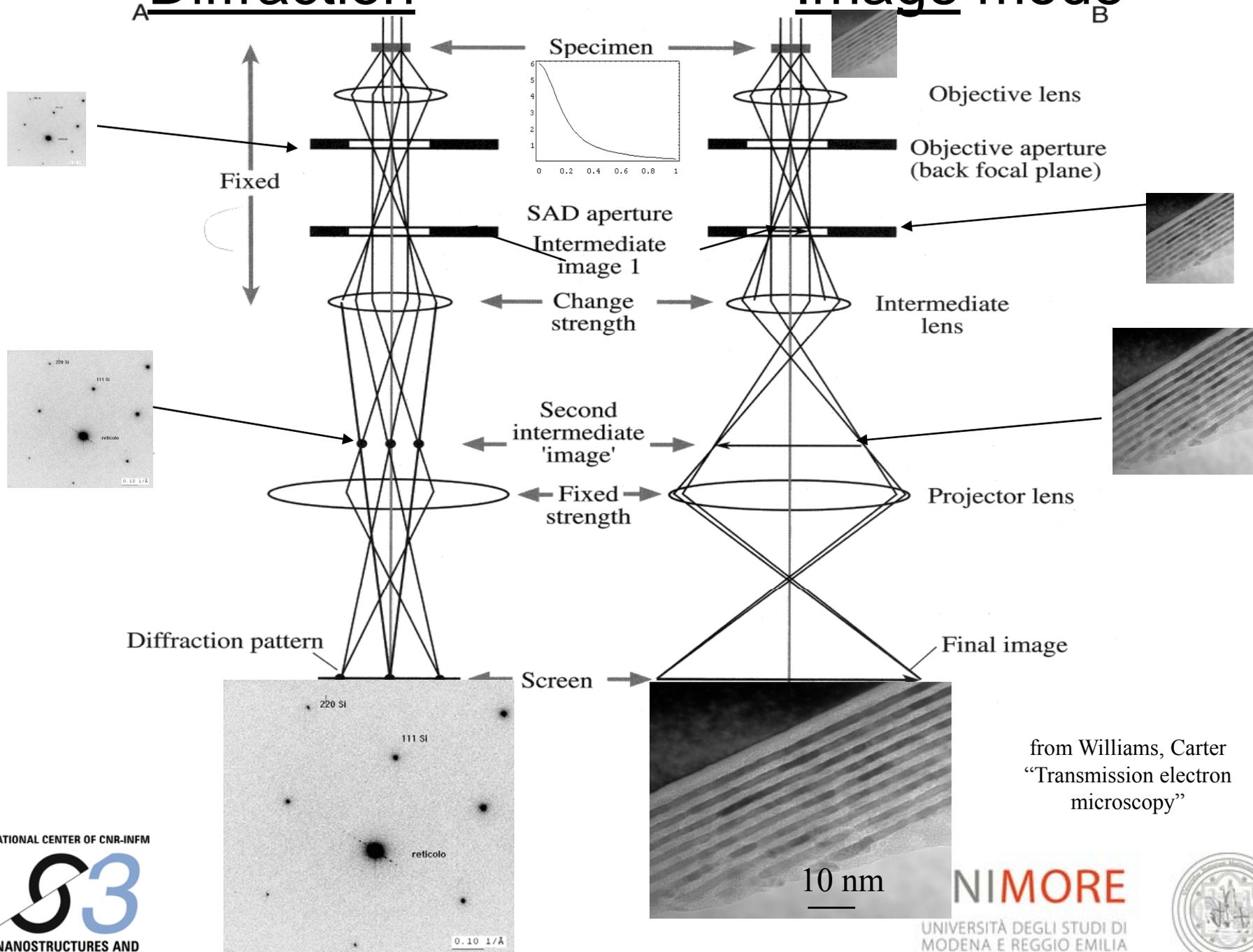


$$f(x, y) = A(x, y) \exp[i\sigma V_p(x, y)]$$

$$\approx (1 - a(x, y))(1 + i\sigma V_p(x, y)) \approx (1 - a(x, y) + i\sigma V_p(x, y))$$

# A Diffraction

# B Image mode



from Williams, Carter  
"Transmission electron  
microscopy"

NATIONAL CENTER OF CNR-INFN



**NIMORE**

UNIVERSITÀ DEGLI STUDI DI  
MODENA E REGGIO EMILIA

