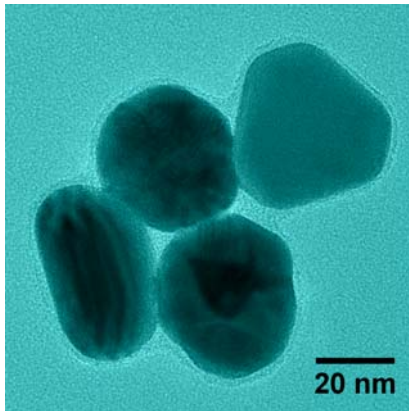


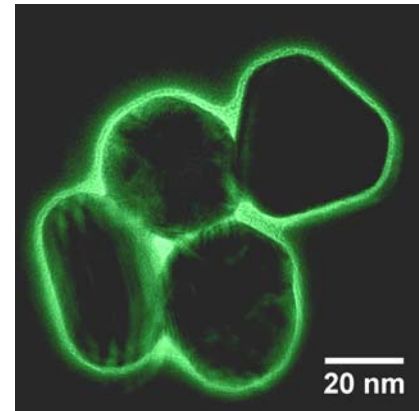
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## Elektronenmikroskopie:

Ein vielseitiges Werkzeug zur strukturellen und chemischen Analyse von Mikro- und Nanostrukturen



Dr. Armin Feldhoff



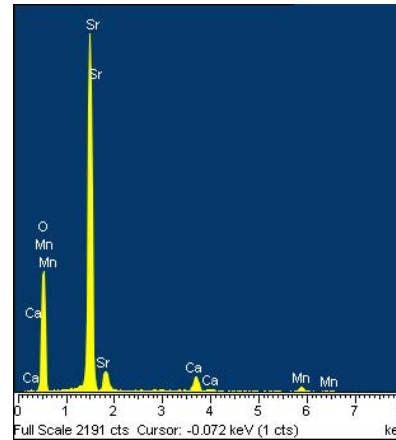
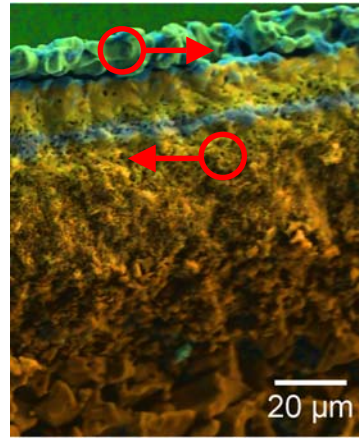
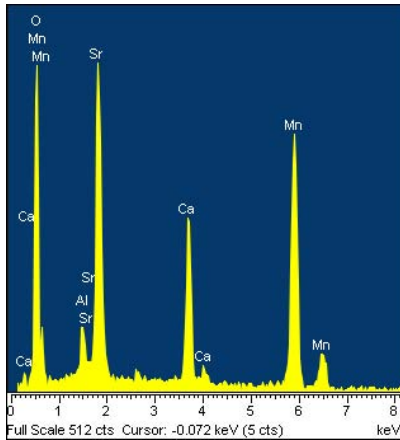
Institut für Physikalische Chemie und Elektrochemie

**Universität Hannover** 

- Sekundärelektronendetektor
- Semi-in-lens-Detektor für kleine Arbeitsabstände
- Rückstreuelektronendetektor (BSE)
- Energiedispersives Röntgenspektrometer (EDXS),  
Oxford Instruments INCA 300, Detektion ab Be (Z = 4)

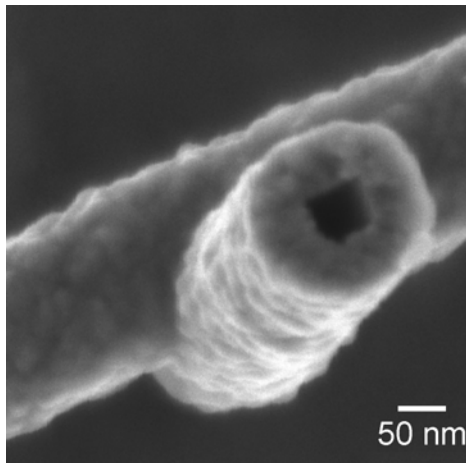
## Feldemissions- Rasterelektronenmikroskop (FE-REM)

JEOL JSM-6700F

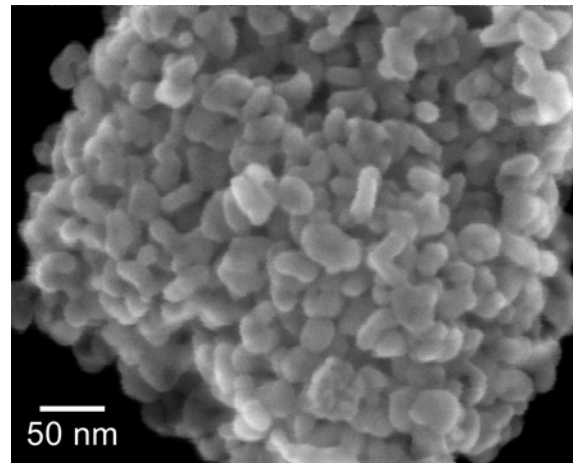


Auflösungsvermögen:  
 1,0 nm @ 15kV  
 2,2 nm @ 1kV  
 Beschleunigungsspannung:  
 0,5 - 30 kV

Bruchspiegel eines Perowskitrohrs (Membran zur Sauerstoffabtrennung)



Gekreuzte TiO<sub>2</sub>-Hohlfasern

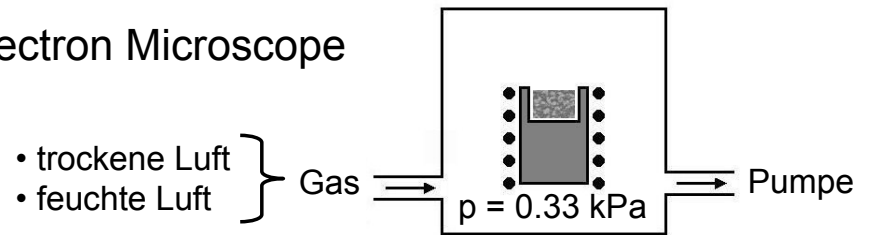


Katalysatorträger aus TiO<sub>2</sub> (Anatas)

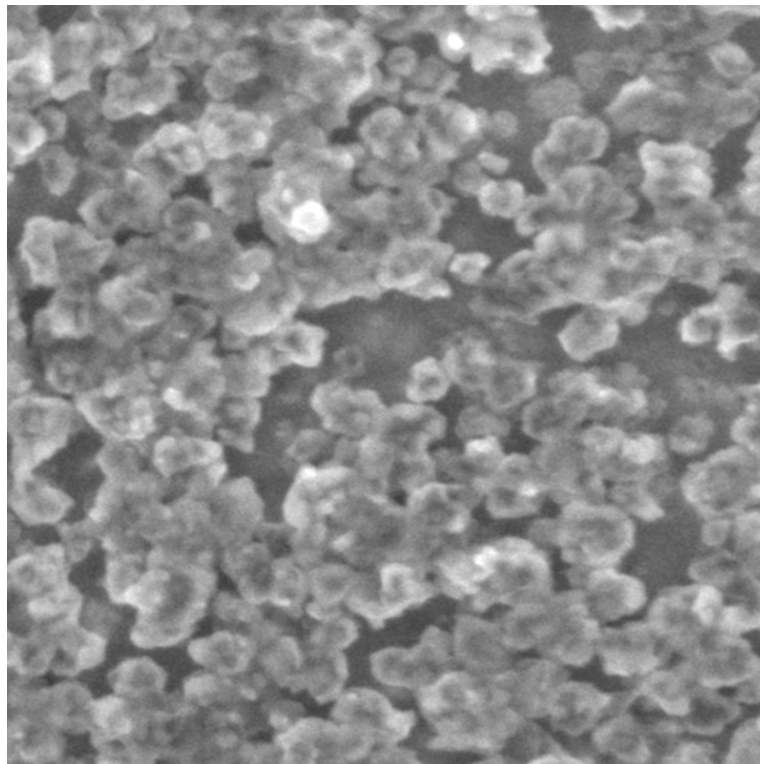


# In-situ-Beobachtung eines Oxidationsvorgangs

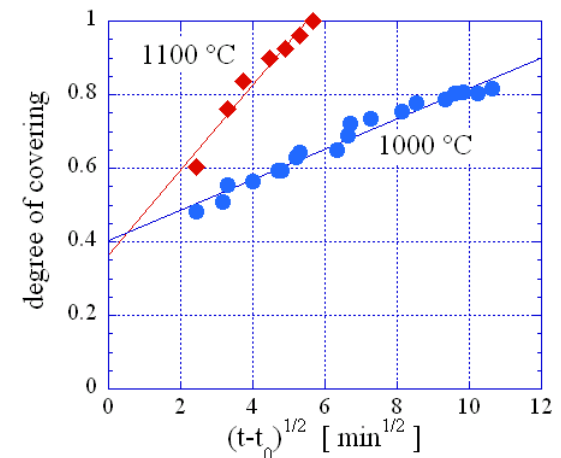
ESEM: Environmental Scanning Electron Microscope



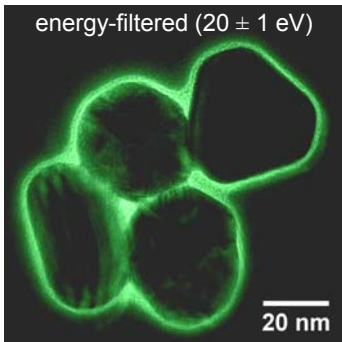
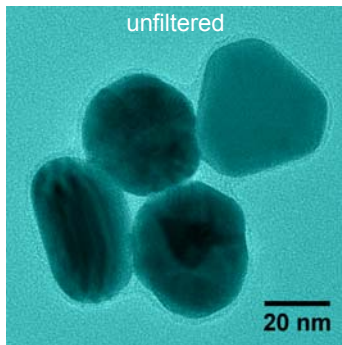
Aufsicht: 1 h Beobachtungszeit



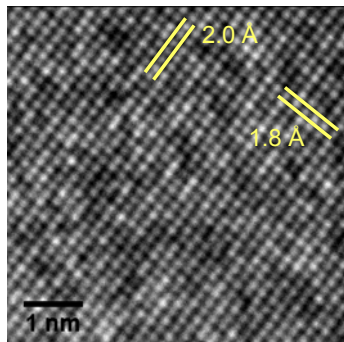
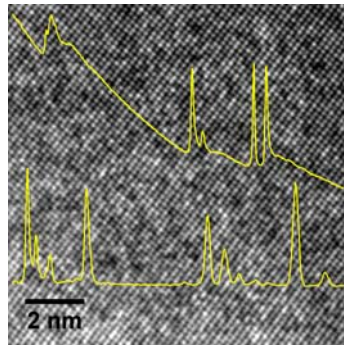
$\text{Si}_3\text{N}_4$ -TiN-Kompositkeramik bei 1100 °C:  
Kornvergrößerung und Ausbildung  
facettierter  $\text{TiO}_2$ -Kristalle (Rutil)  
begleiten das Zusammenwachsen  
einer Oberflächenschicht.



- Transmissionselektronenmikroskopie (BF, DF, HRTEM)
- Raster-Transmissionselektronenmikroskopie (STEM mit BF, HAADF)
- Elektronenbeugung (SAED, CBED, auch energiegefiltert)
- Energiegefilterte Transmissionselektronenmikroskopie (EFTEM)
- Elektronen-Energieverlust-Spektroskopie (EELS, ELNES),  
**Gatan Imaging Filter, GIF 2001, mit 1k-CCD-Kamera**
- Energiedispersive Röntgenspektroskopie (EDXS),  
**Oxford Instruments INCA 200, Detektion ab Be (Z = 4)**



Goldteilchen, oberflächenfunktionalisiert mit Proteinfilm

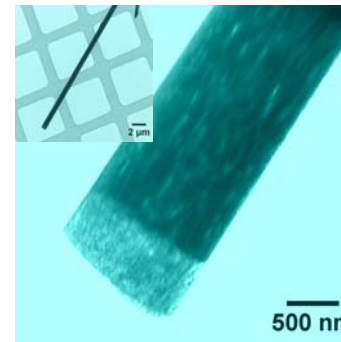
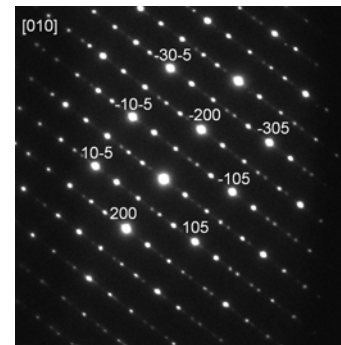


Perowskit entlang [012]  
( $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Fe}_{0.8}\text{Zn}_{0.2}\text{O}_{3-\delta}$ )

## Feldemissions-Transmissions-elektronenmikroskop (FE-TEM)

JEOL JEM-2100F-UHR

- Schottky-Feldemitter (ZrO/W(100))
- Beschleunigungsspannung: 200 kV (160 kV)
- Punktauflösung:  $\leq 0,19 \text{ nm}$  ( $C_s = 0,5 \text{ mm}$ )
- Gitterauflösung für STEM:  $\leq 0,2 \text{ nm}$
- Energieauflösung für EELS:  $\leq 0,7 \text{ eV}$



TiO<sub>2-x</sub>-Stäbchen (Anatas)  
mit planaren Defekten





# Ruska und Knoll: Die Erfindung des Elektronenmikroskops

lectures of Professor Adolf Matthias. At the end of the summer term in 1928 he told us about his plan of setting up a small group of people to develop from the Braun tube an efficient cathode-ray oscillograph for the measurement of very fast electrical processes in power stations and on open-air high-voltage transmission lines. Perhaps with the memory of my physics school lesson in the back of my head, I immediately volunteered for this task and became the youngest collaborator of the group, which was headed by Dr. Ing. Max Knoll. My first

Therefore, Hans Busch [3] at Jena calculated the electron trajectories in such an electron ray bundle and found that the magnetic field of the short coil has the same effect on the electron bundle as has the convex glass lens with a defined focal length on a light bundle. The focal length of this "magnetic electron lens" can be changed continuously by means of the coil current. Busch



Ernst Ruska (1906-1988)  
Max Knoll (1897-1969)

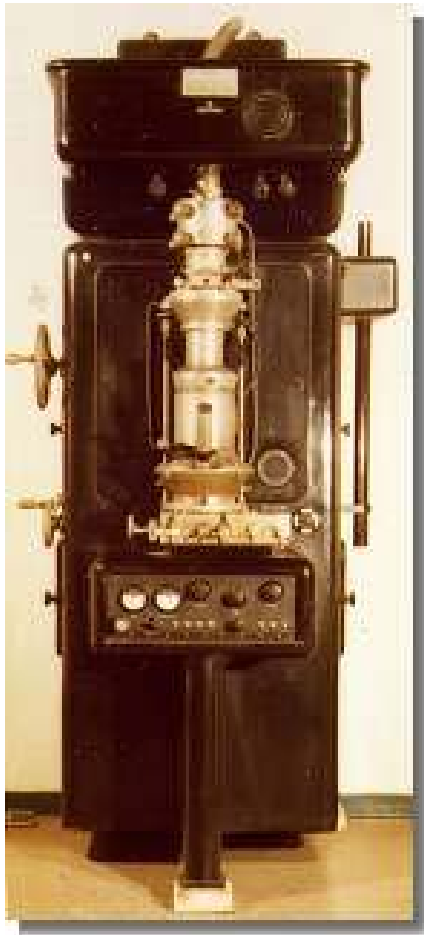


Das erste zweistufige Elektronenmikroskop,  
konstruiert von Knoll and Ruska (1931)

extremely low dimensions of the electrons. As engineers we did not know yet the thesis of the "material wave" of the French physicist de Broglie [11] that had been put forward several years earlier (1925). Even physicists only reluctantly accepted this new thesis. When I first heard of it in summer 1931, I was very much disappointed that now even at the electron microscope the resolution should be limited again by a wavelength (of the "Materiestrahlung"). I was immediately heartened, though, when with the aid of the de Broglie equation I became satisfied that these waves must be around five orders of magnitude shorter in length than light waves. Thus, there was no reason to abandon the aim of electron microscopy surpassing the resolution of light microscopy.

1939: das erste seriengefertigte TEM, Fa. Siemens & Halske AG, Berlin

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*Dr.-Ing.*  
Ernst Ruska  
(1906-1988)



*Dr.-Ing.*  
Bodo von Borries  
(1905-1956)



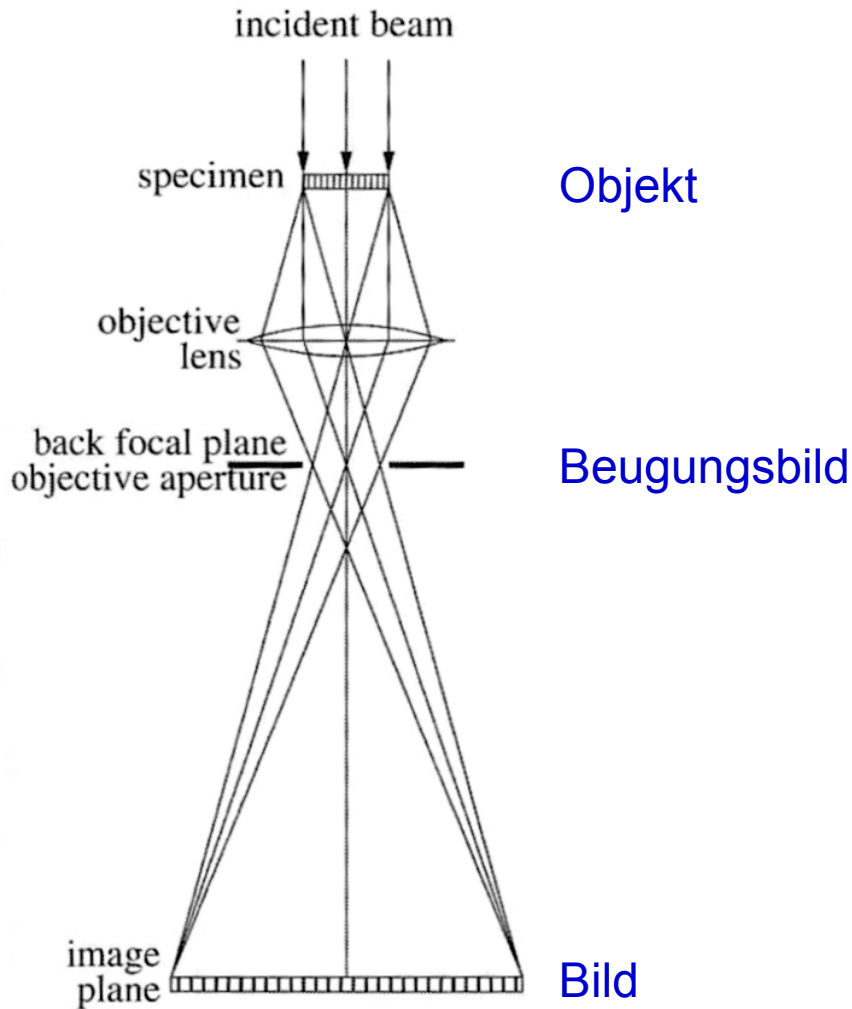
*Dr. med.*  
Helmut Ruska  
(1908-1973)

„Die Sichtbarmachung  
der Viren“  
(1940)

erreichte 30.000-fache Vergrößerung

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# Abbildung mit einem Durchstrahlungsmikroskop



Realraum  
= Ortsraum



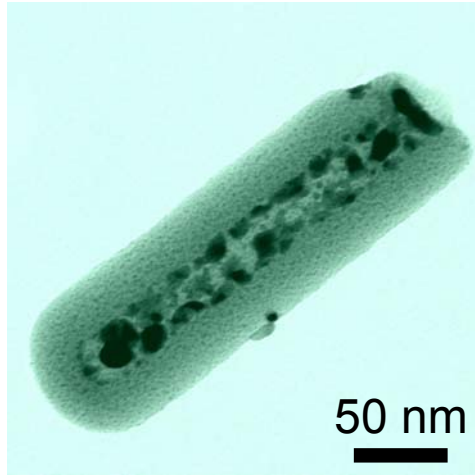
reziproker Raum  
= Impulsraum



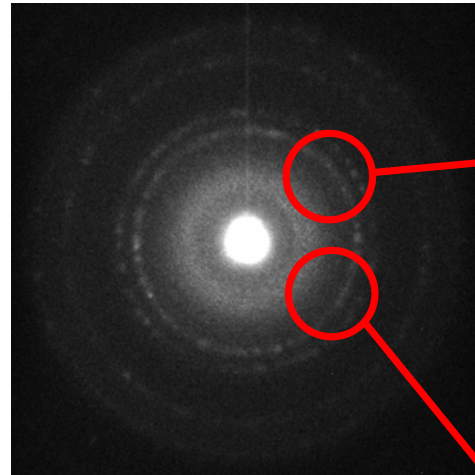
Realraum  
= Ortsraum

# SiO<sub>2</sub>-Nanoröhrchen, gefüllt mit Platin

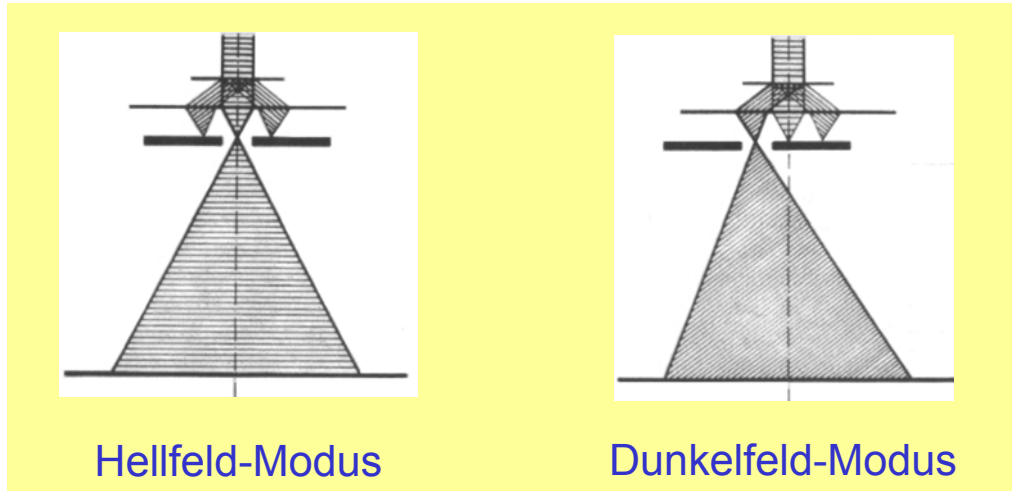
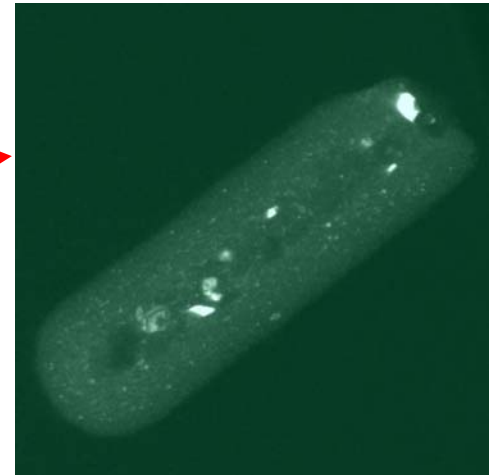
Hellfeld (BF)



Feinbereichsbeugung (SAED)

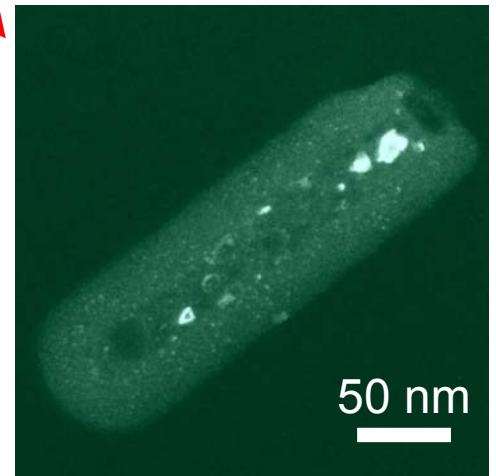


Dunkelfeld (DF)



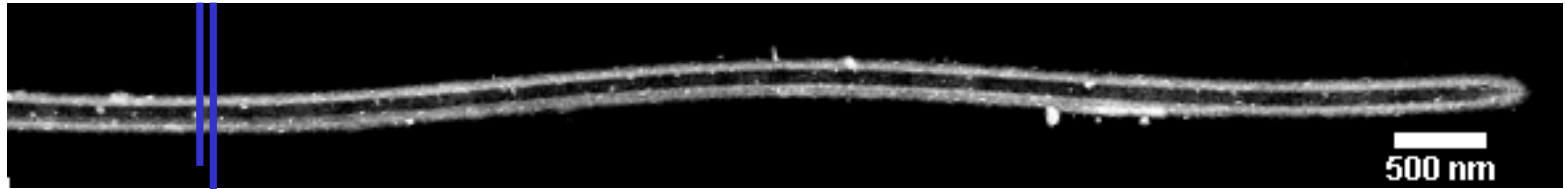
Hellfeld-Modus

Dunkelfeld-Modus

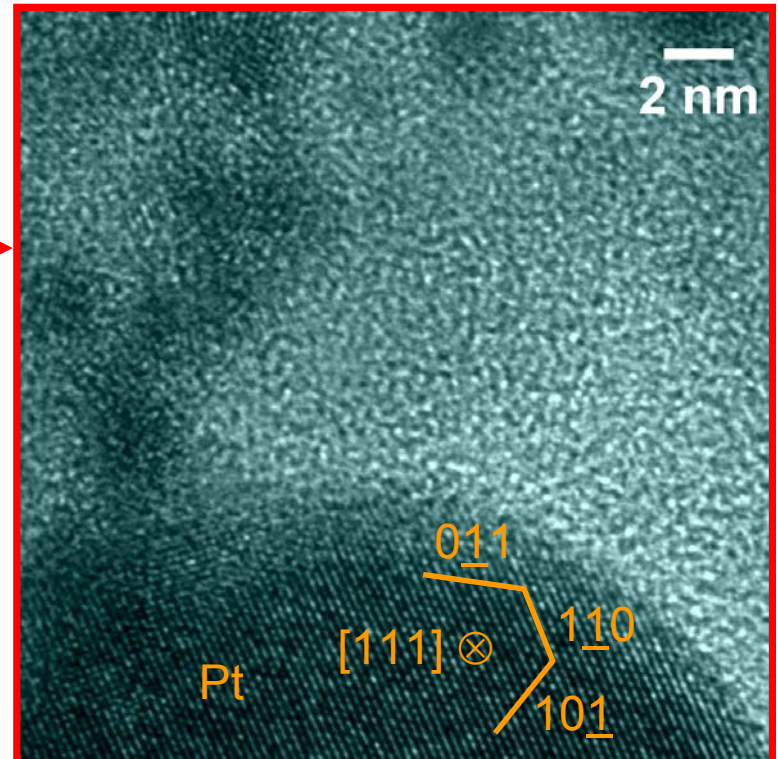
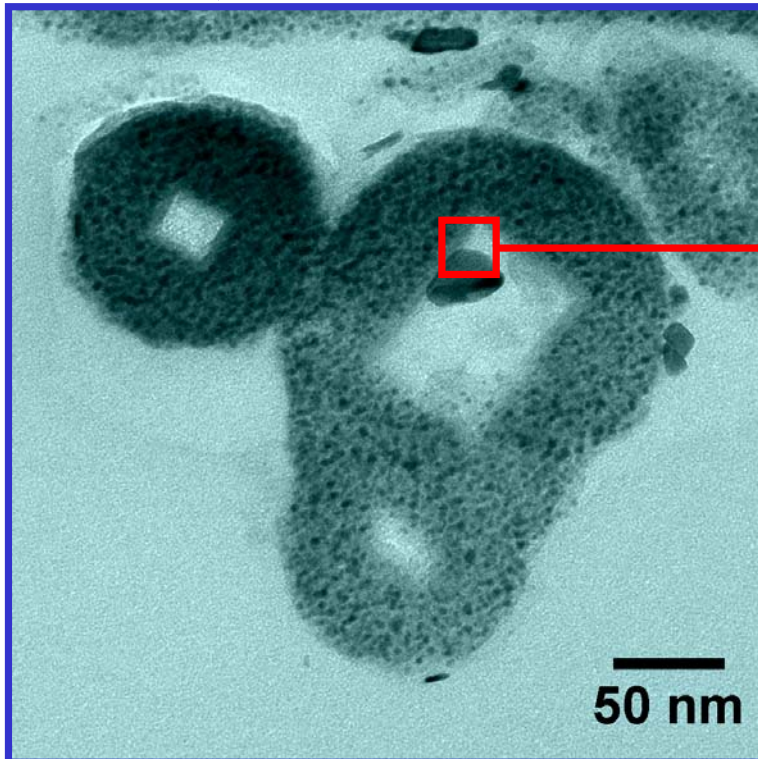




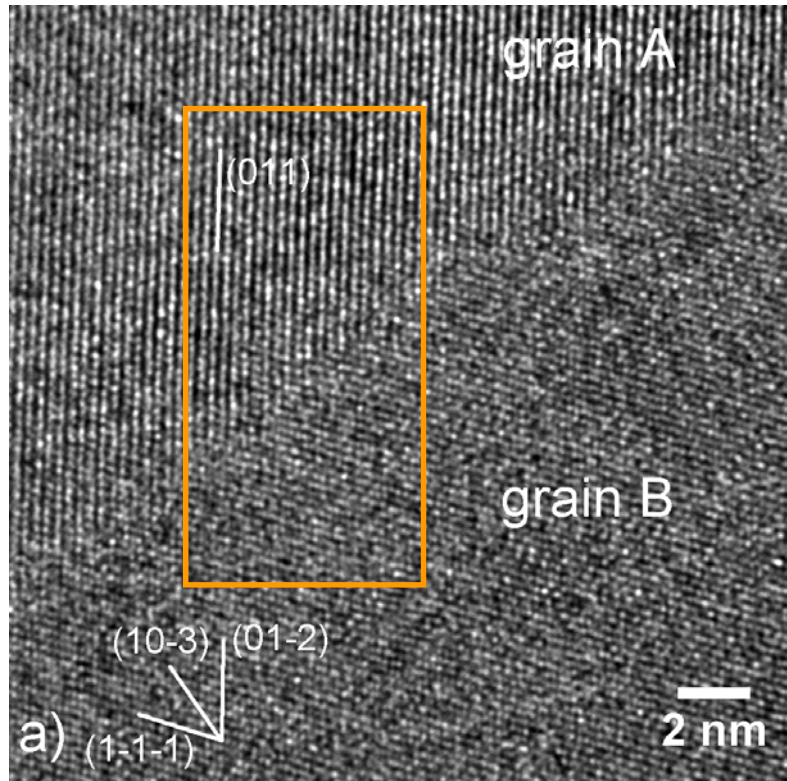
# SiO<sub>2</sub>-TiO<sub>2</sub>-Nanoröhrchen, gefüllt mit Platin



↓ Ultramikrotom-Dünnschnitt

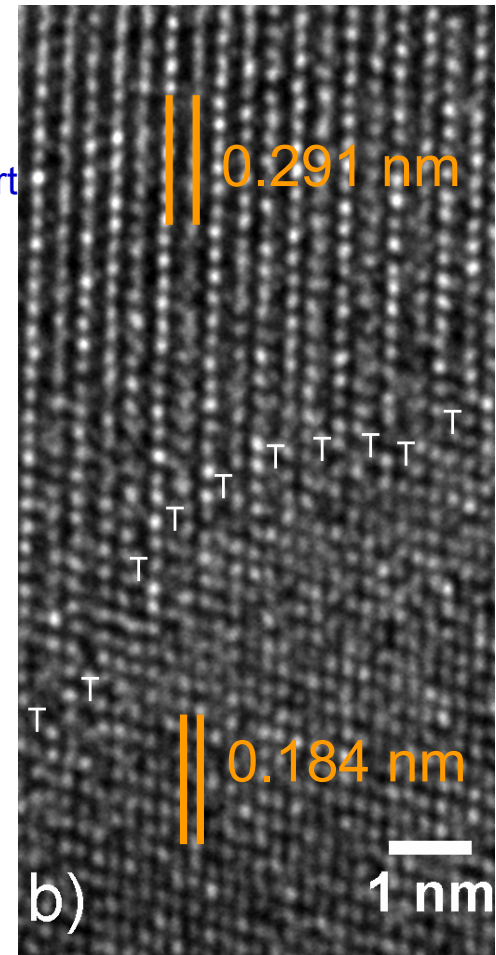


# Korngrenze in einer $(\text{Ba}_{0.5}\text{Sr}_{0.5})(\text{Zn}_{0.2}\text{Fe}_{0.8})\text{O}_{3-\delta}$ -Keramik



Ladungstransport  
über innere  
Grenzflächen:

ionische  
+  
elektronische  
Leitung

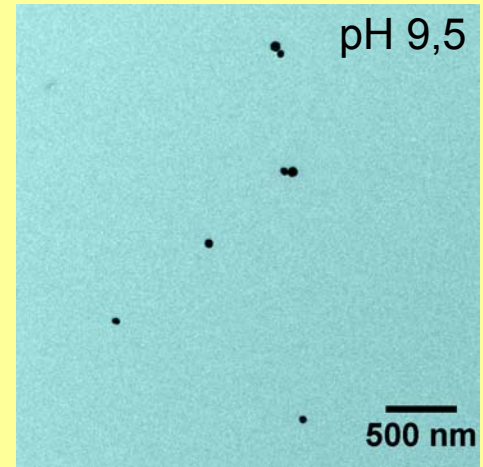
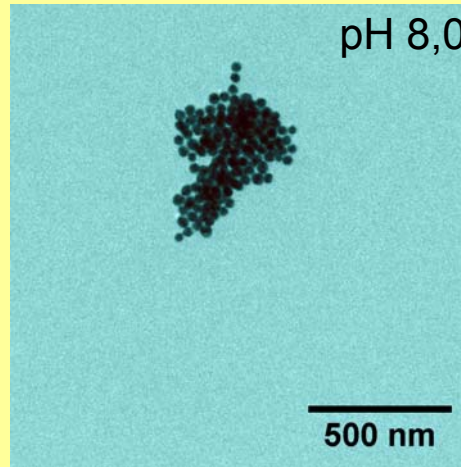
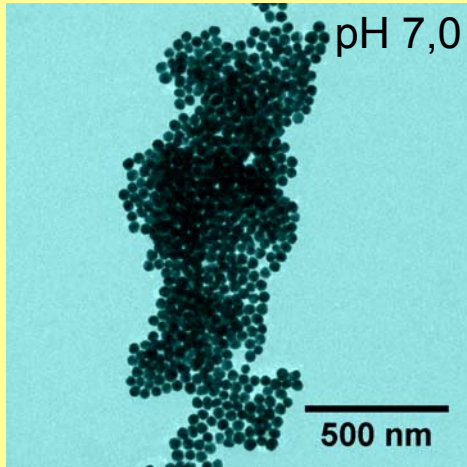


Gitterfehlpassung  $f$  wird durch Einbau von Anpassungsversetzungen ausgeglichen.

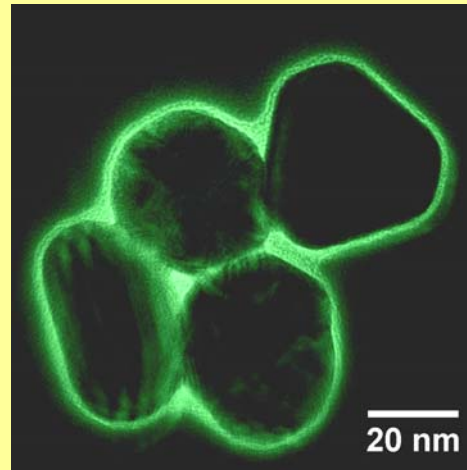
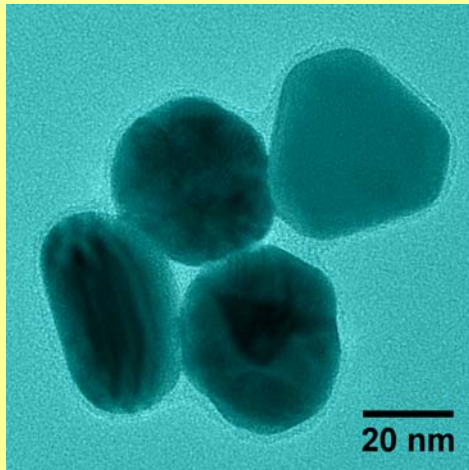
$$f = \frac{d_{011} - d_{01-2}}{\frac{1}{2} \cdot (d_{011} + d_{01-2})} = \frac{0,291 \text{ nm} - 0,184 \text{ nm}}{\frac{1}{2} \cdot (0,291 \text{ nm} + 0,184 \text{ nm})} = \frac{1,07}{2,38} = 45 \%$$

# Goldkolloide, oberflächen-funktionalisiert mit Maus-Gen (IgC)

Ermittlung des optimalen pH-Werts für die Konjugation Antikörper + Gold



ungefiltert

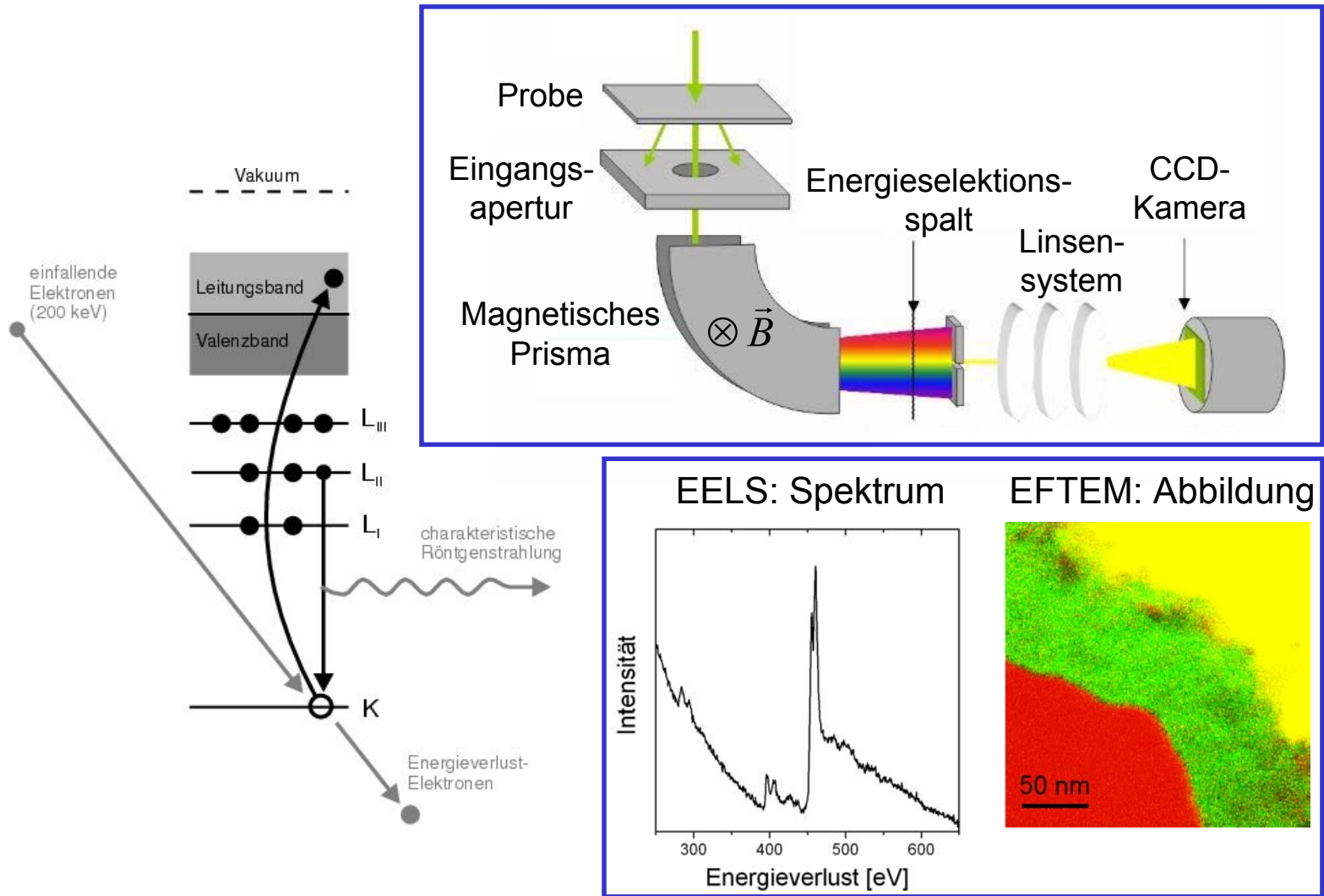


Energie-gefilterte  
Abbildung  
( $20 \pm 1$  eV):

Mit Elektronen,  
die inelastisch an  
Proteinmolekülen  
gestreut wurden.



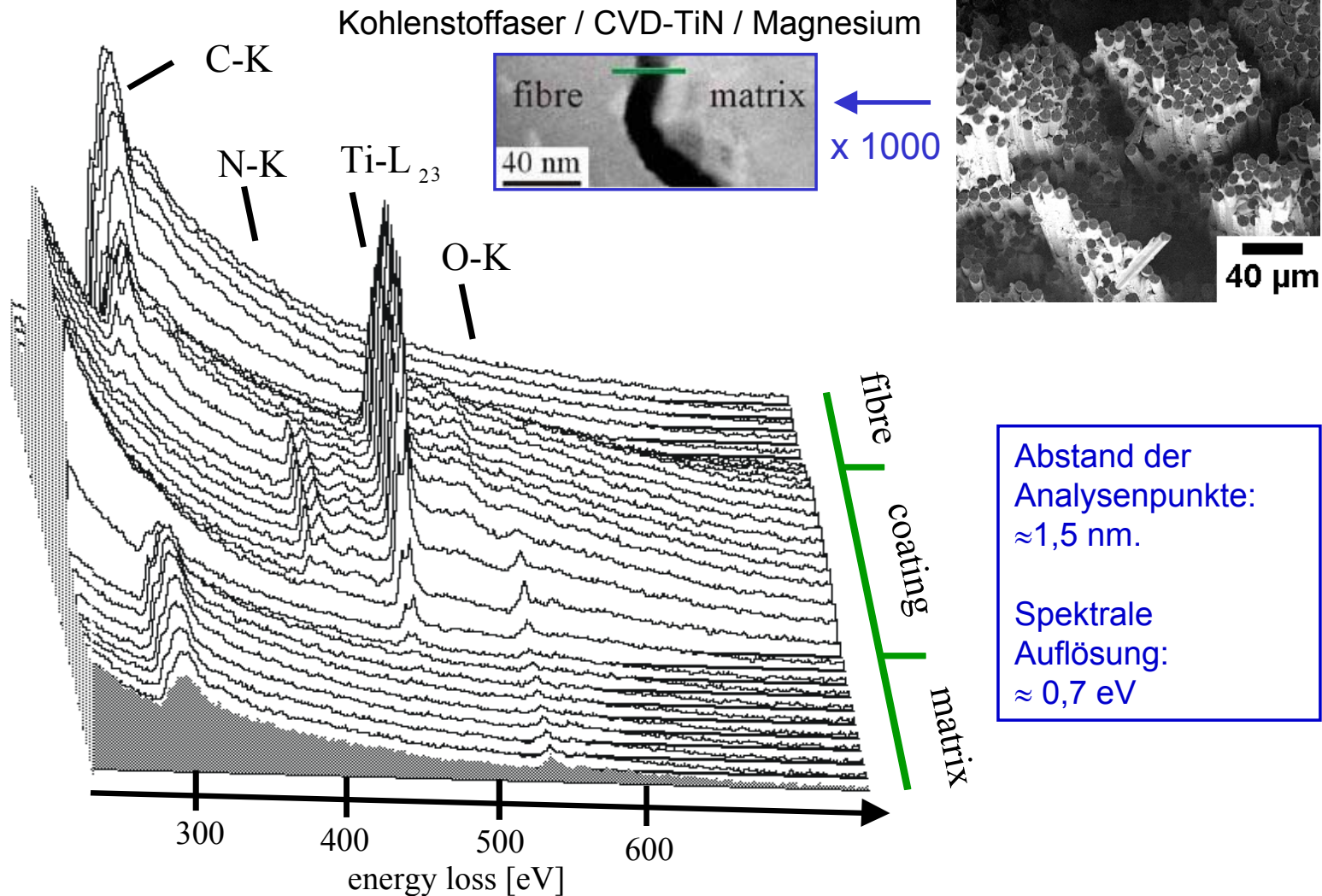
# Welche Energie haben die transmittierten Elektronen?

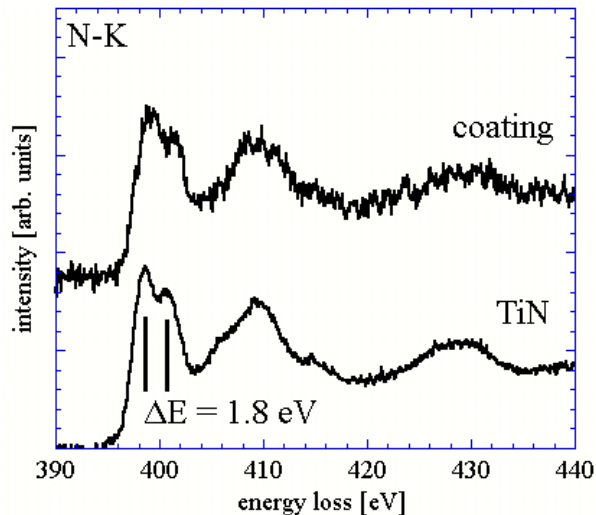
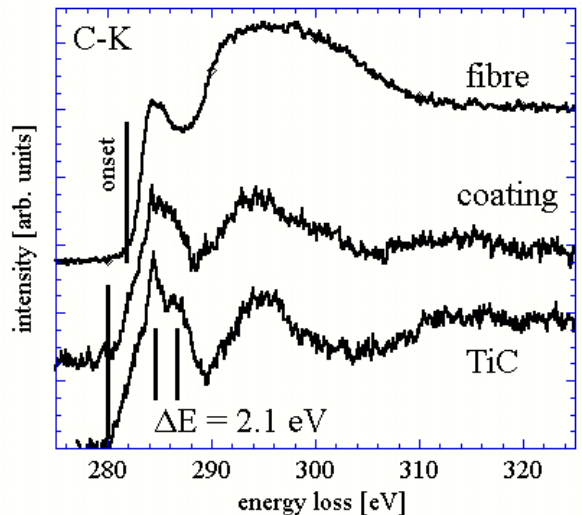


EELS: Electron Energy-Loss Spectroscopy, EFTEM: Energy-Filtered TEM

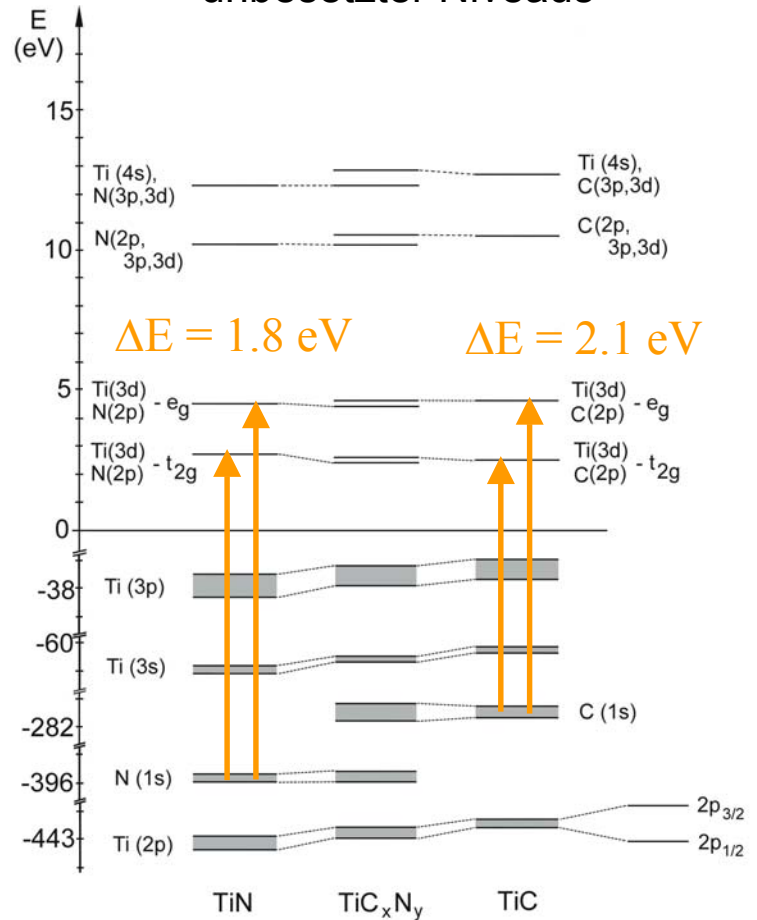


# EELS: Linienprofil über eine Grenzschicht

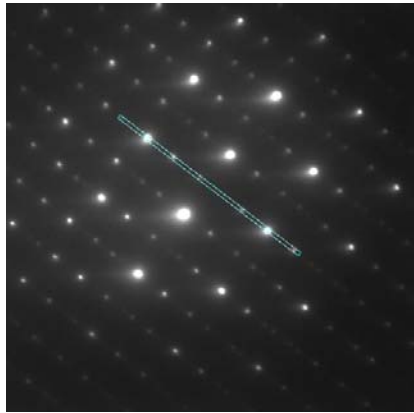




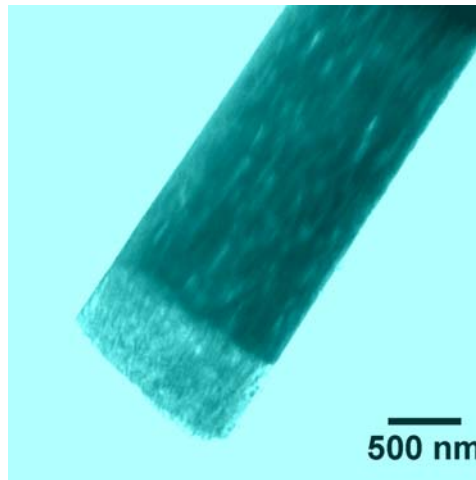
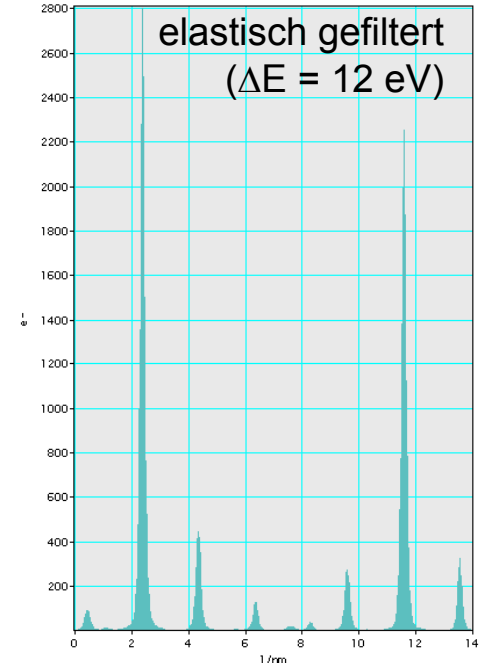
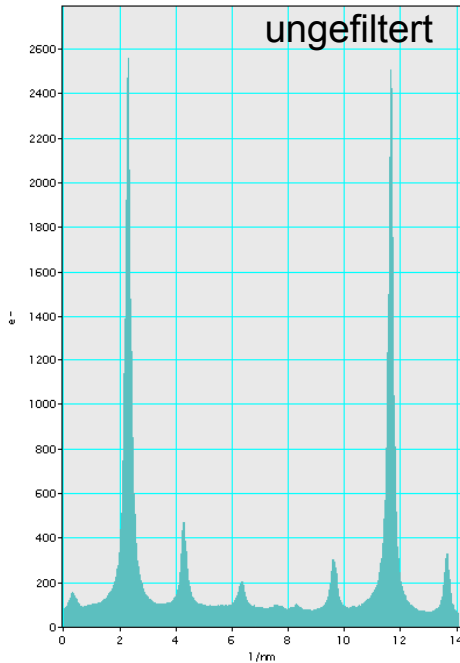
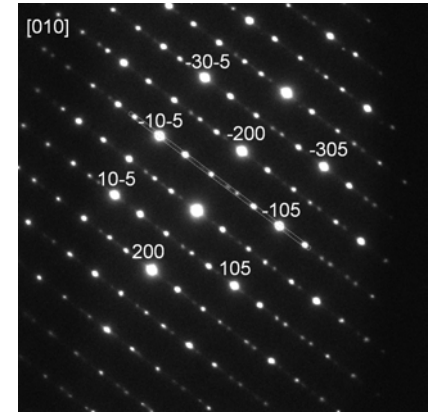
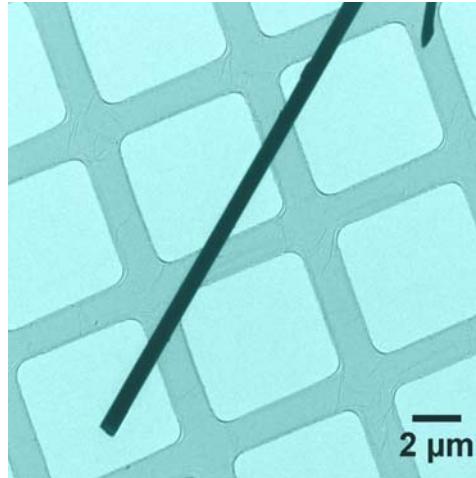
## Energien besetzter und unbesetzter Niveaus



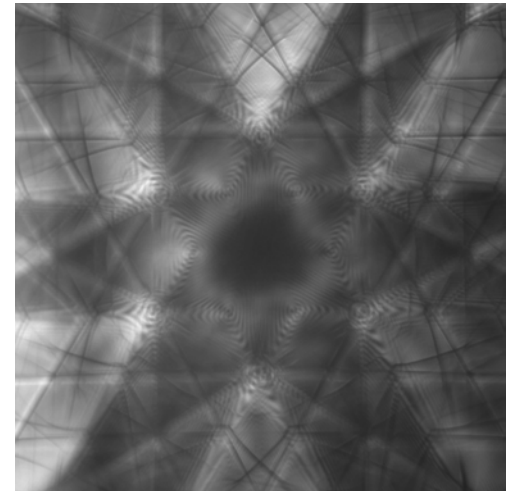
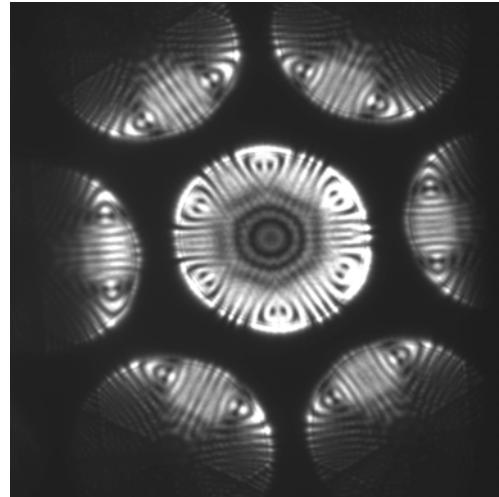
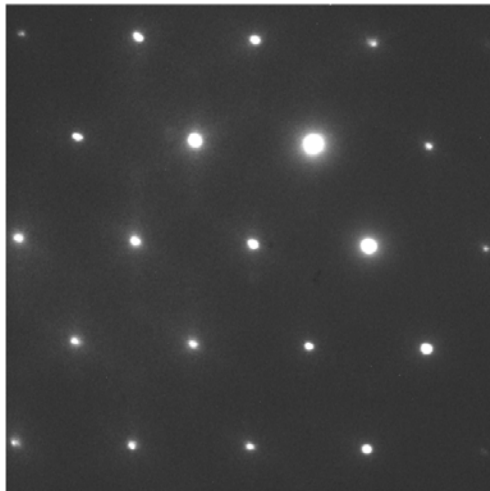
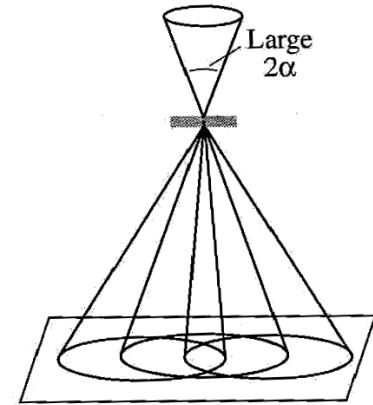
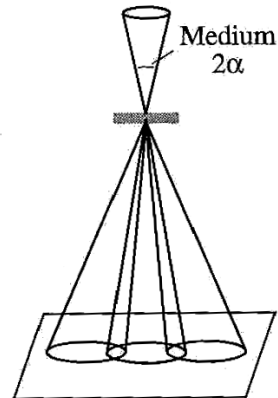
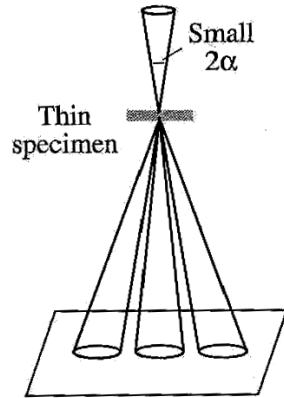
# Energie-gefilterte Elektronenbeugung



$\text{TiO}_{2-x}$ -Nanostabchen  
(substochiometrischer Anatas)



# Konvergente Elektronenbeugung (CBED)



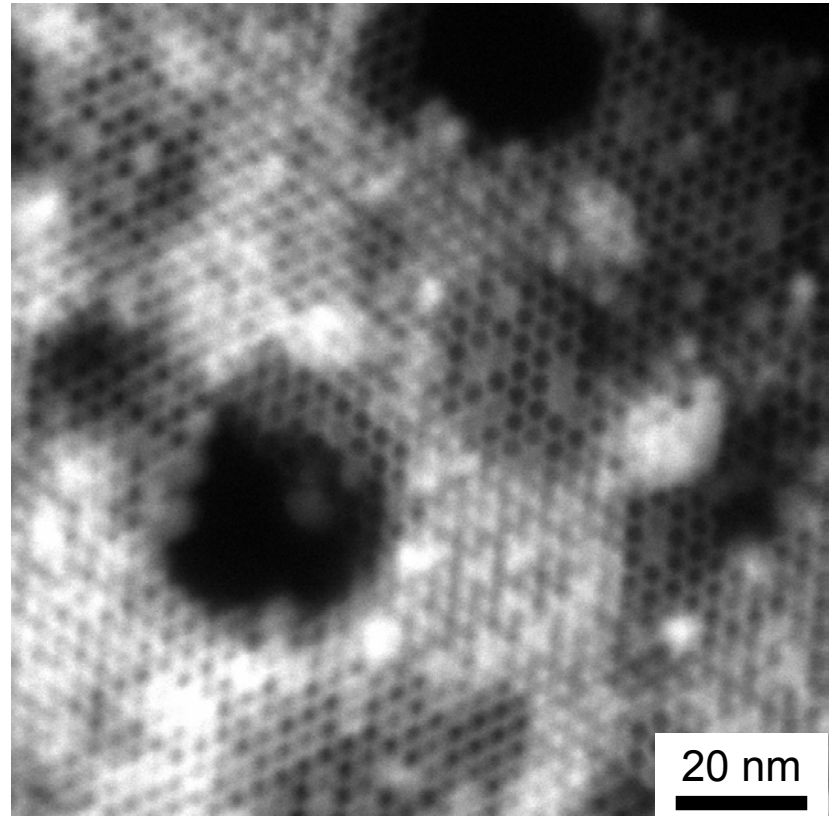
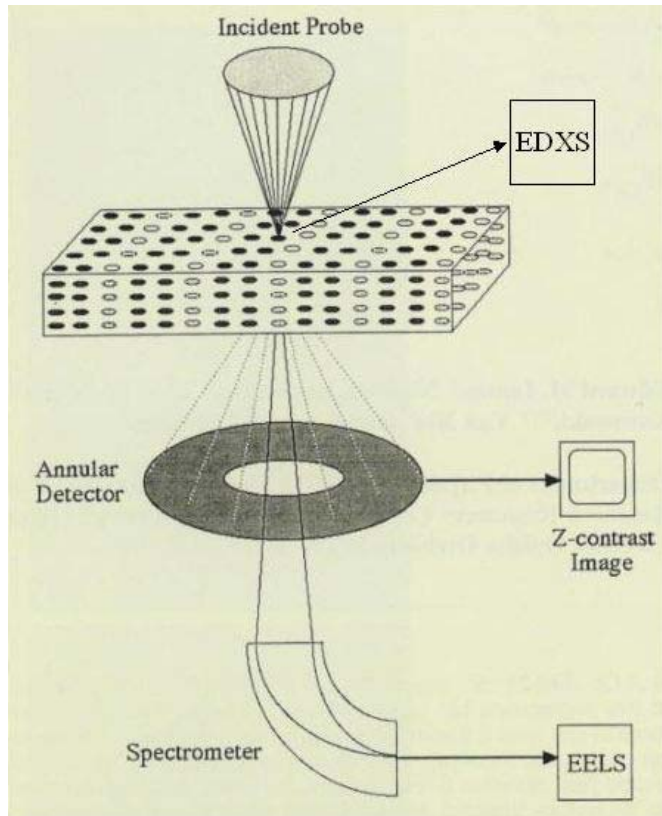
Beispiel: Silizium entlang [111]



# Raster-Transmissionselektronenmikroskopie (STEM)

## Z-Kontrast-Abbildung

$$Z(\text{O}) = 8, \quad Z(\text{Si}) = 14, \quad Z(\text{Sn}) = 50$$



SiO<sub>2</sub>- Molekularsieb MCM-41,  
Kanäle teilweise gefüllt mit SnO<sub>2</sub>

# Analytische Elektronenmikroskopie:

Eine leistungsstarke Methodenkombination zur strukturellen und chemischen Charakterisierung fester Stoffe bis hinab zur atomaren Skala.

