# Nano Malzemeler Karakterizasyon Yöntemleri

# Nanomalzemelerin Karakterizasyonu

| Teknik | Karakterizasyon Parametreleri |
|---|---|
| Dinamik ışık saçılımı (DLS)/parçacık boyut analizi | Sıvı ortam içerisinde dağıtılmış nanoparçacıkların boyutu ve boyut dağılımlarının belirlenmesi |
| Zeta potansiyel analizi | Sıvı ortam içerisinde dağıtılmış nanoparçacıkların yüzey yüklerinin belirlenmesi |
| Taramalı elektron mikroskobu (SEM) | Morfoloji ve yüzey analizi |
| Geçirimli elektron mikroskobu (TEM) | Boyut, şekil, morfoloji, iç yapı gibi analizler |
| Atomik kuvvet mikroskobu (AFM) | Şekil ve yüzey morfoloji analizi |
| Taramalı tünelleme mikroskobu (STM) | Atomik boyutta yüzey analizi ve modifikasyonu |
| Lazer taramalı eş odaklı mikroskop (LSCM) | Özellikle biyolojik malzemelerde 3boyutlu morfolojik analiz |
| Brunauer-Emmett-Teller (BET) tekniği | Yüzey alanı, gözeneklilik boyutu ve dağılımı, adsorplama kapasiteleri |
| X-Işını Kırınımı (XRD) | Kristal yapı ve faz analizi, ortalama kristalit boyutu |
| X-Işını fotoelektron spektroskopisi (XPS) | Elementel ve kimyasal bağlar hakkında bilgiler ile yüzeyden kimyasal kompozisyon analizi |
| Fourier dönüşüm infrared spektroskopisi (FTIR) | Kimyasal kompozisyon analizi |
| Diferansiyel taramalı kalorimetre (DSC) | Termal analiz ve faz dönüşüm |

# Görüntüleme Teknikleri

Elektron mikroskopları

- Taramalı elektron mikroskobu (SEM)
- Geçirimli elektron mikroskobu (TEM)

Taramalı uç mikroskopları (scanning probe microscopy)

- Atomik kuvvet mikroskobu
- Taramalı tünelleme mikroskobu

# Elektron Mikroskopları

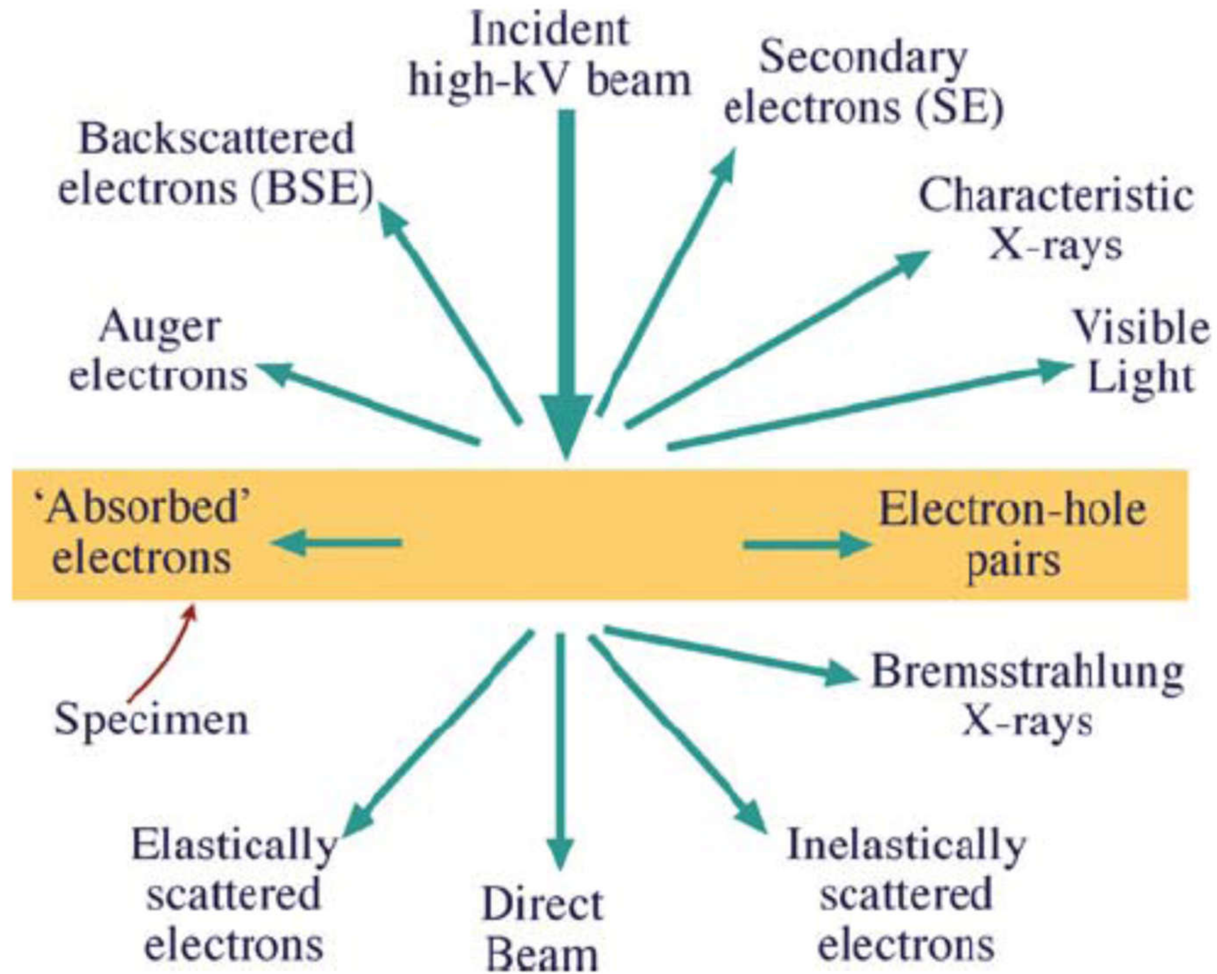
**TABLE 1.1 Fundamental Constants and Definitions**

| | |
|---|---|
| Charge (e) | (–) $1.602 \times 10^{-19}$ C |
| 1 eV | $1.602 \times 10^{-19}$ J |
| Rest mass ($m_0$) | $9.109 \times 10^{-31}$ kg |
| Rest energy ($m_0c^2$) | 511 keV |
| Kinetic energy (charge × voltage) | $1.602 \times 10^{-19}$ N m (for 1 volt potential) = J |
| Planck's constant (h) | $6.626 \times 10^{-34}$ N m s |
| 1 A | 1 C/s |
| Speed of light in vacuum (c) | $2.998 \times 10^{8}$ m/s |

**TABLE 1.2 Electron Properties as a Function of Accelerating Voltage**

| Accelerating voltage (kV) | Non-relativistic wavelength (nm) | Relativistic wavelength (nm) | Mass (× $m_0$) | Velocity (× $10^8$ m/s) |
|---|---|---|---|---|
| 100 | 0.00386 | 0.00370 | 1.196 | 1.644 |
| 120 | 0.00352 | 0.00335 | 1.235 | 1.759 |
| 200 | 0.00273 | 0.00251 | 1.391 | 2.086 |
| 300 | 0.00223 | 0.00197 | 1.587 | 2.330 |
| 400 | 0.00193 | 0.00164 | 1.783 | 2.484 |
| 1000 | 0.00122 | 0.00087 | 2.957 | 2.823 |

# Elektron Mikroskopları



# Elektron Mikroskopları

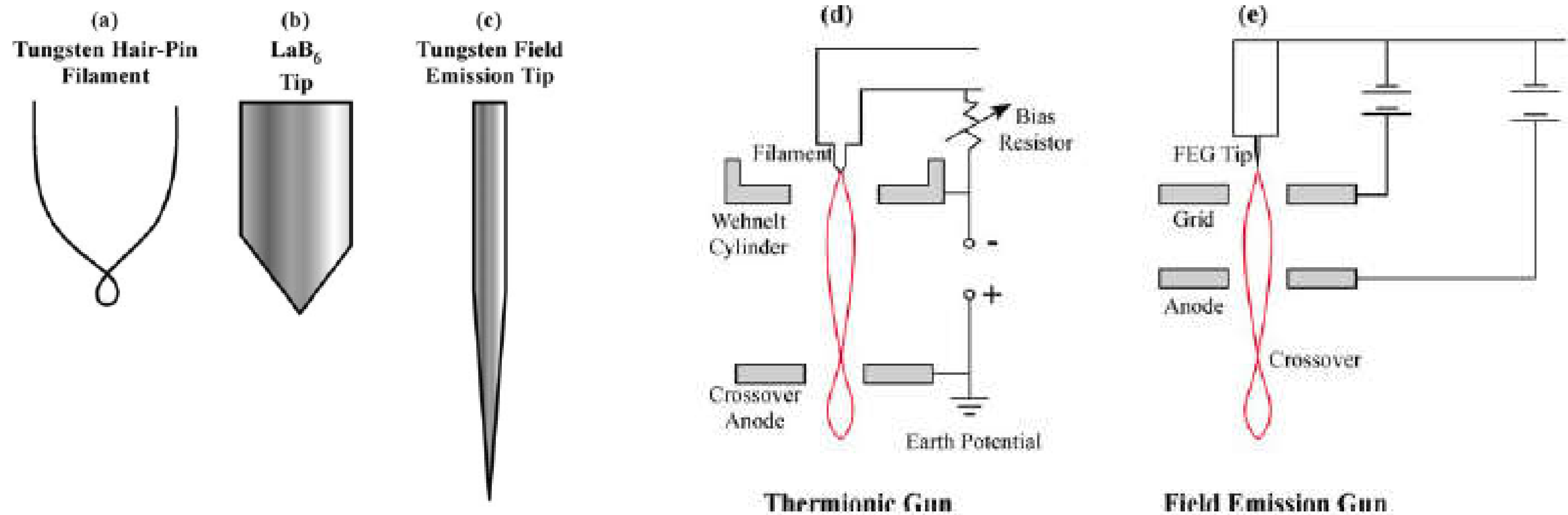


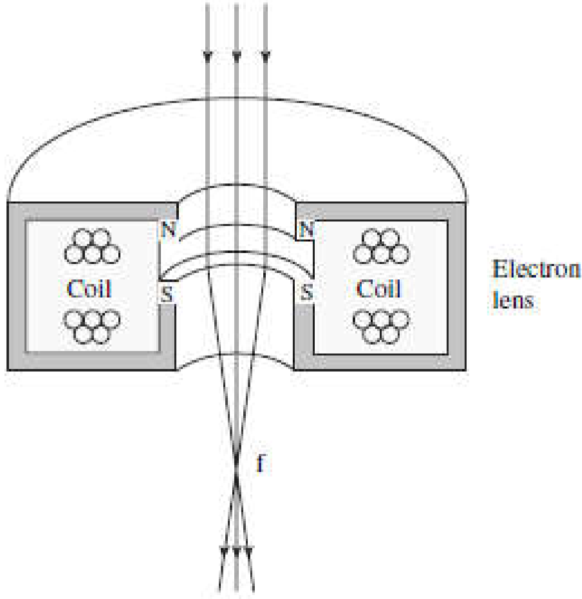

**Table 3.3** Comparison of Electron Guns

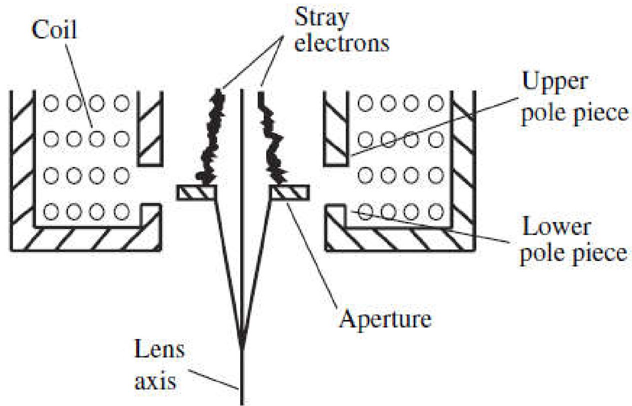
| | Tungsten filament | $LaB_6$ | Field emission Thermal | Field emission Cold |
|---|---|---|---|---|
| Operation temperature (K) | ~2800 | ~1800 | 1600 ~ 1800 | ~300 |
| Brightness[a] At 200 kV ($A\,cm^{-2}\,sr$) | $\sim 5 \times 10^5$ | $\sim 5 \times 10^6$ | $\sim 5 \times 10^8$ | $\sim 5 \times 10^8$ |
| Requirement to vacuum (Torr[b]) | $10^{-4}$ | $10^{-6}$–$10^{-7}$ | $10^{-9}$ | $10^{-9}$–$10^{-10}$ |

[a] Intensity emitted per unit cathode surface in unit solid angle.

[b] 1 torr = 133 Pa.

# Elektron Mikroskopları



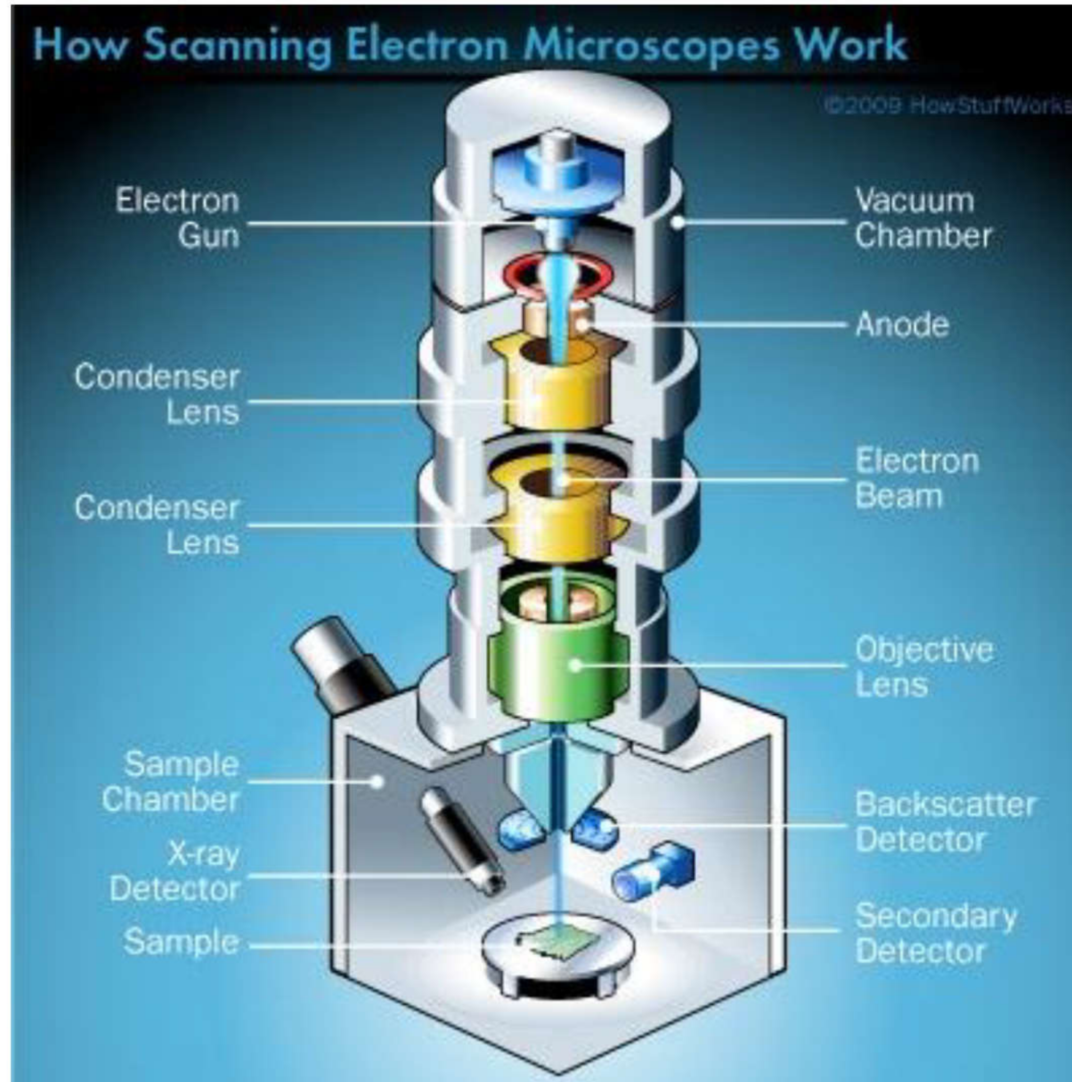




Optik mikroskoplarda cam lensler kullanılırken, elektron mikroskoplarında elektromanyetik lensler kullanılır.

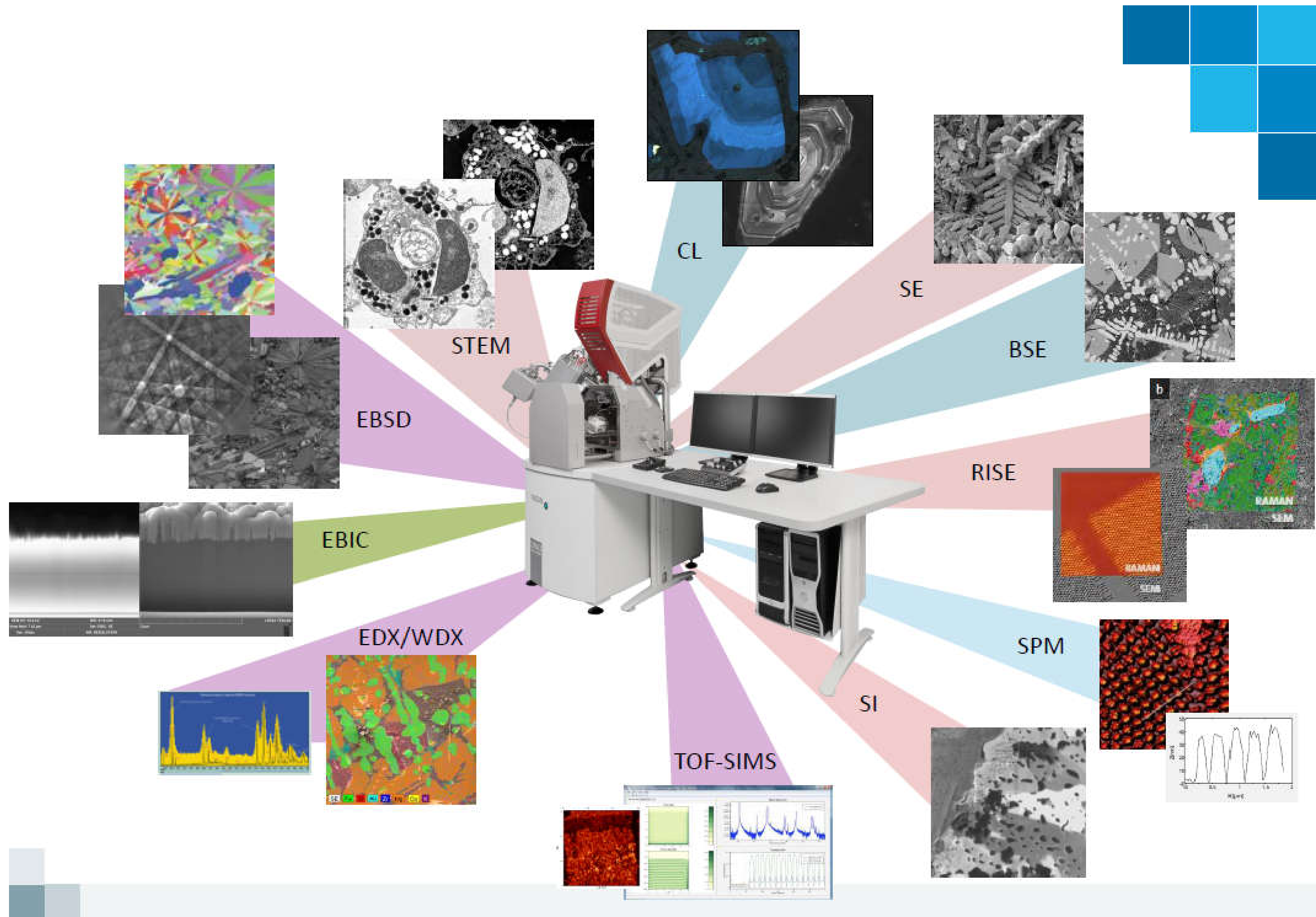
N ve S kutupları ile bir açıklıktan oluşan bu lens sisteminde oluşturulan güçlü manyetik alan ile elektronlar kontrol edilir. Manyetik alanın elektrik akımını değiştirerek kolaylıkla kontrol edilebilmesi sayesinde, bir elektromanyetik lensin büyütme gücü lensten geçen akımı değiştirerek kontrol edilebilir. Cam lenslerde böyle bir özgürlük yok.

Elektron mikroskoplarında ‘aperture’ lensin içerisine yerleştirilmiş dairesel bir metal bloğudur. Bu açıdan optik mikroskopa benzerlik gösterir.

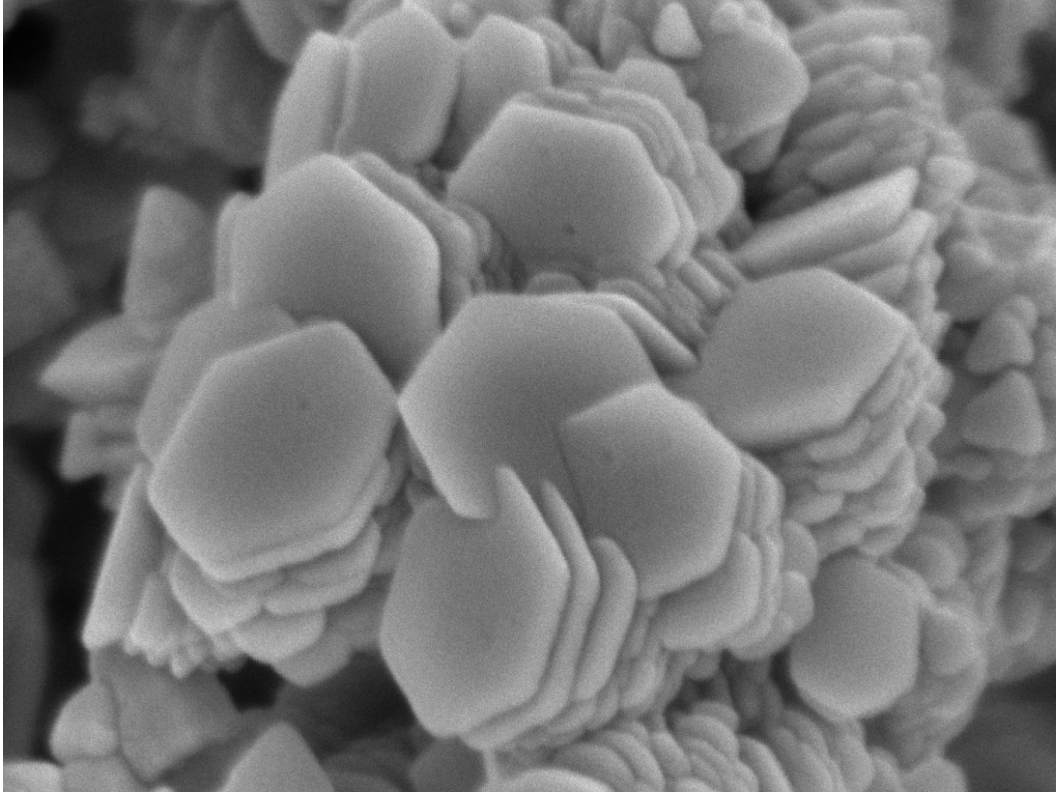
# Taramalı Elektron Mikroskopları (SEM)

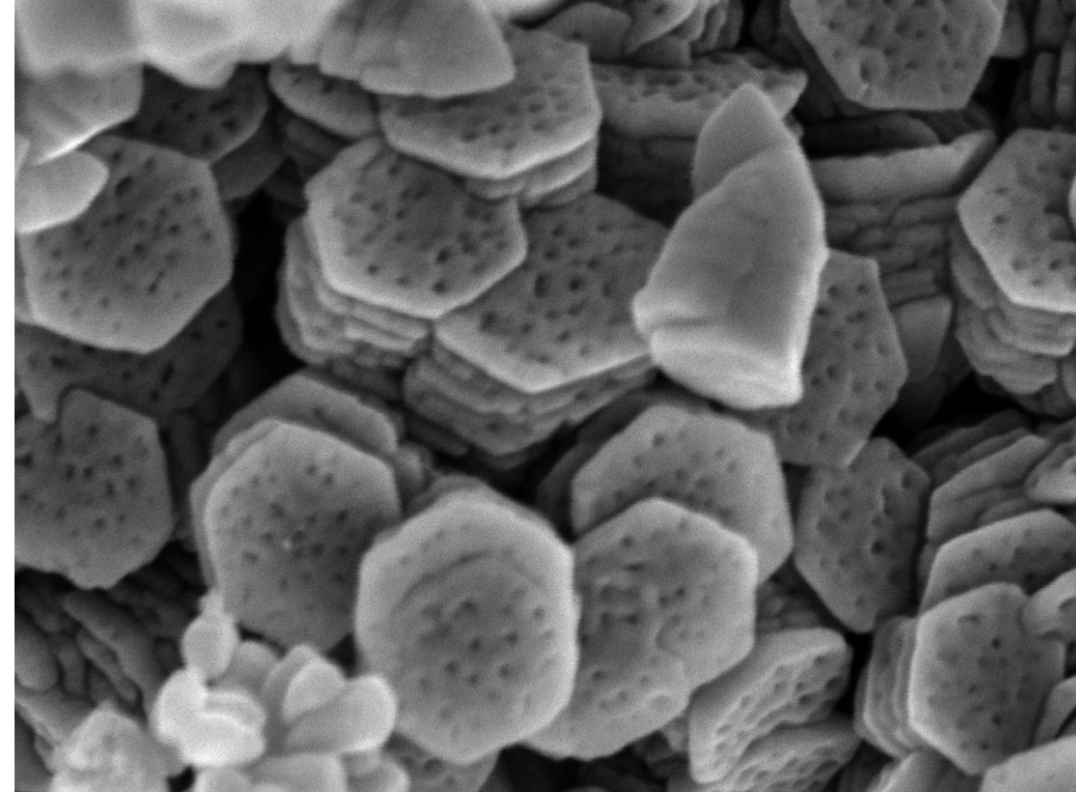


# Taramalı Elektron Mikroskopları (SEM)

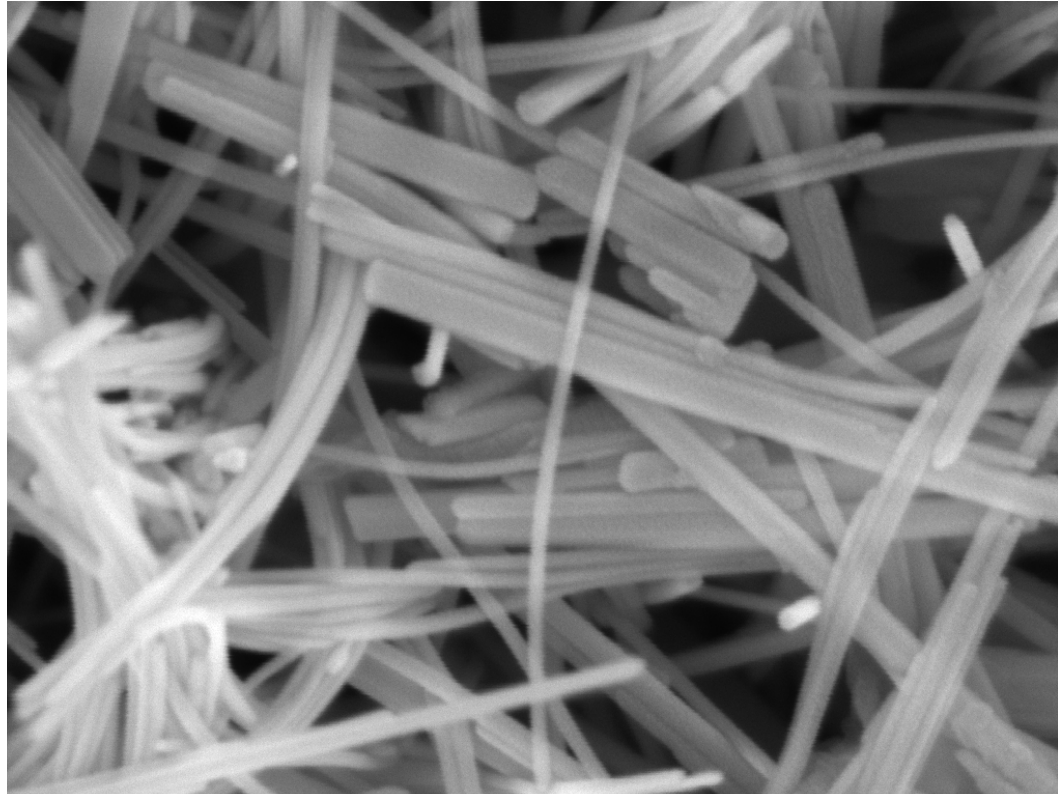


# Taramalı Elektron Mikroskopları (SEM)

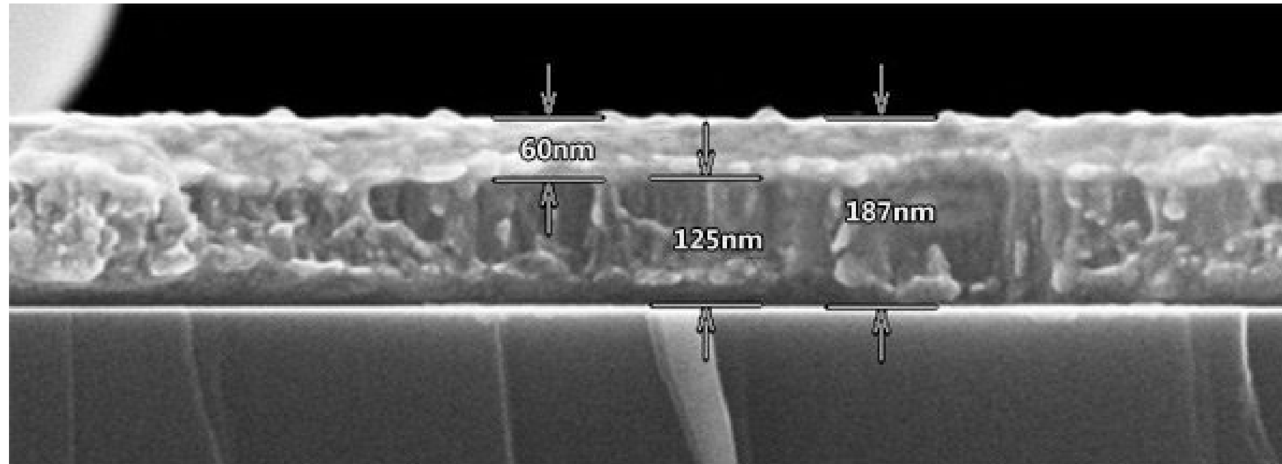


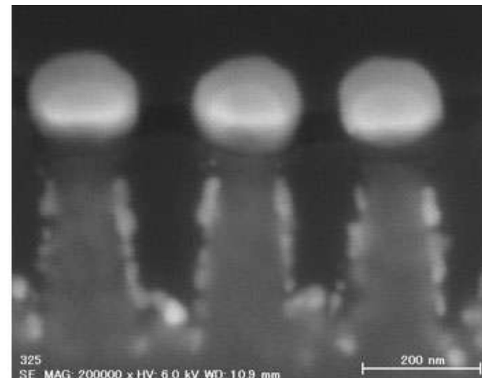



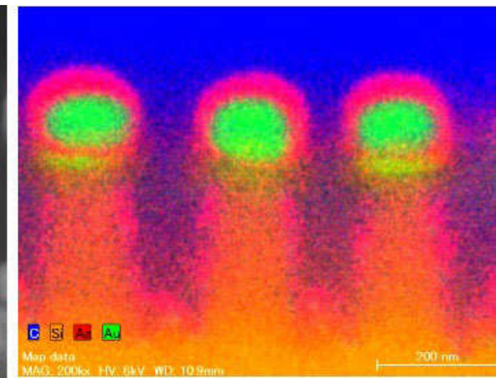
# Taramalı Elektron Mikroskopları (SEM)

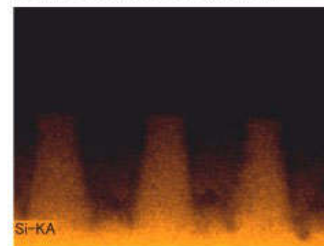


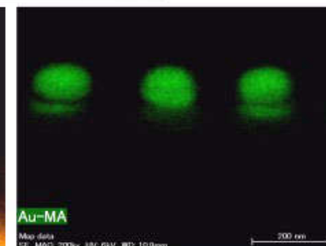
# Taramalı Elektron Mikroskopları (SEM)

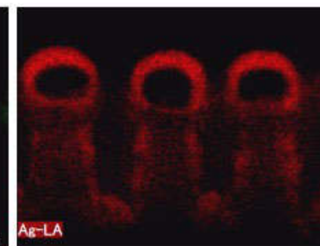




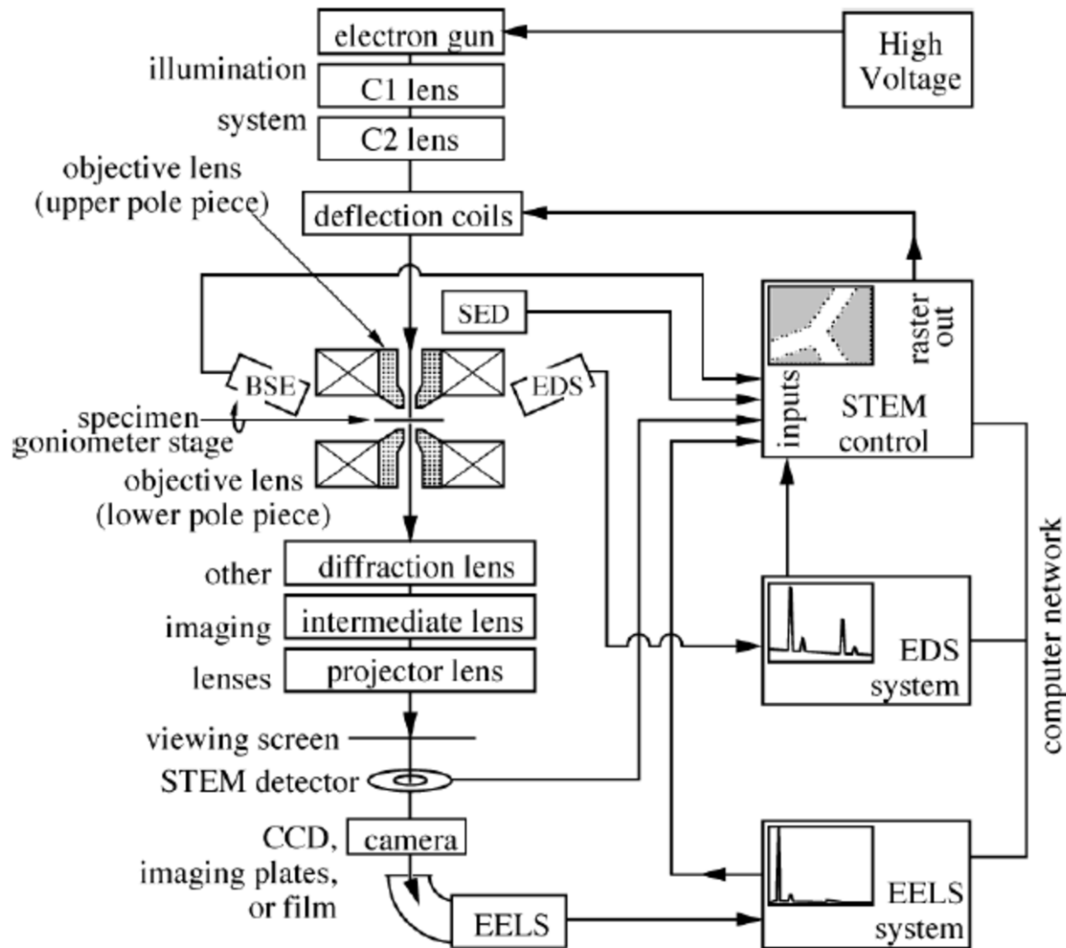



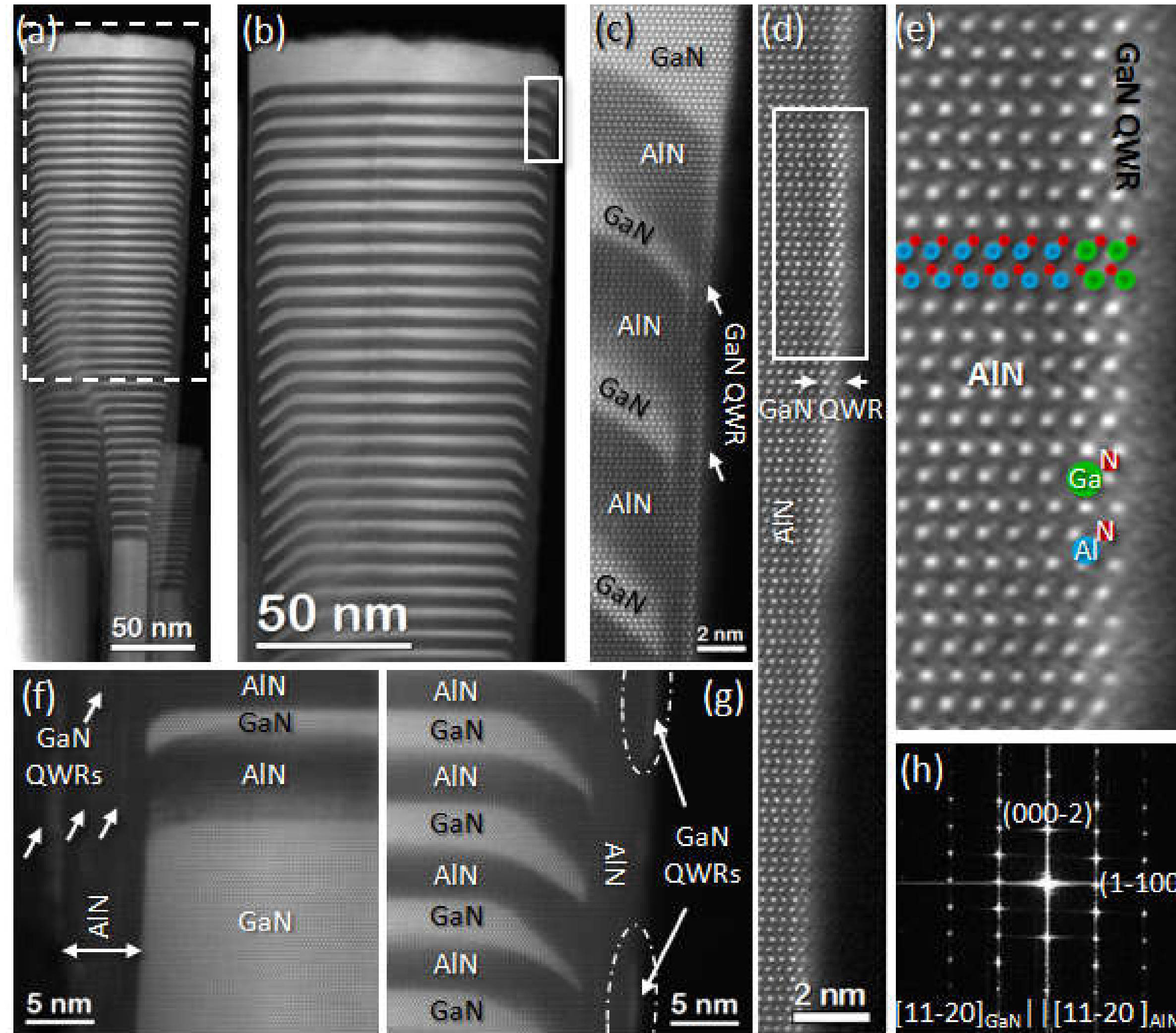



# Geçirimli Elektron Mikroskopları (TEM)



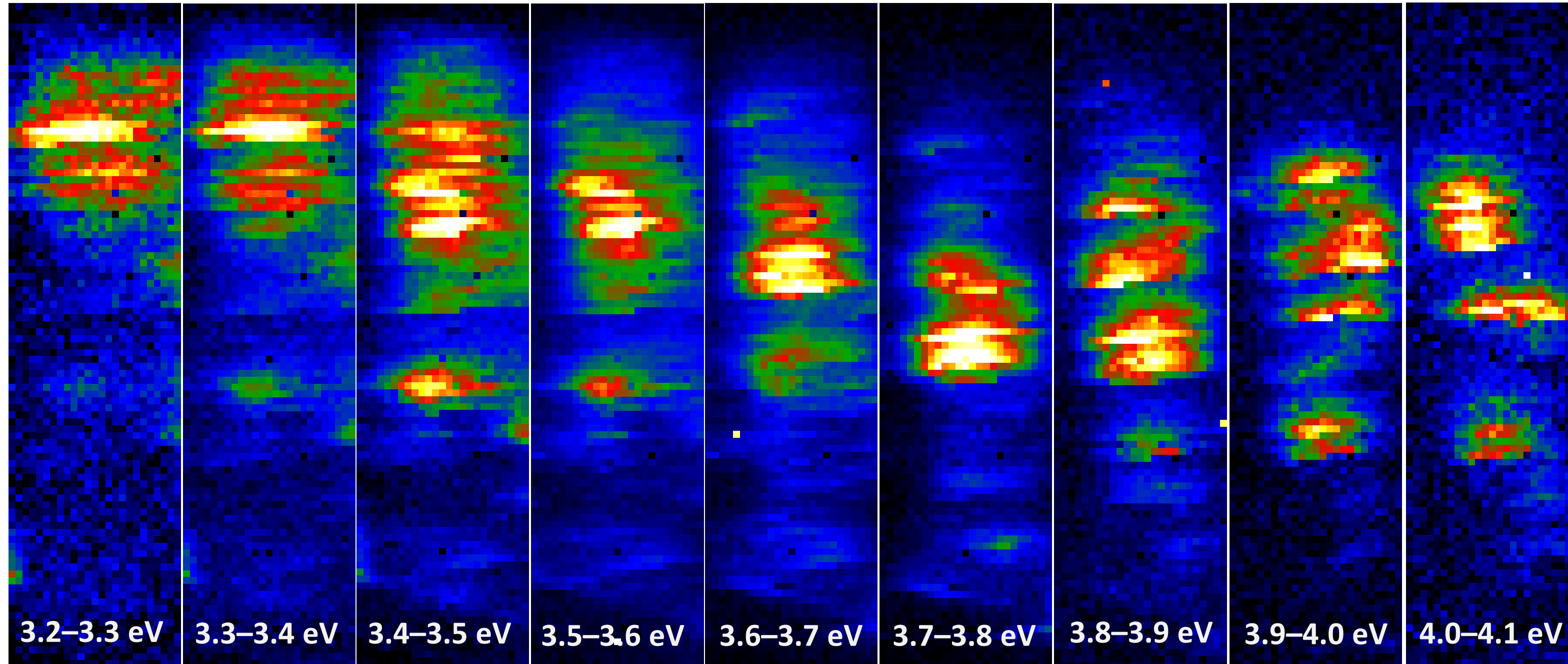
# Geçirimli Elektron Mikroskopları (TEM)



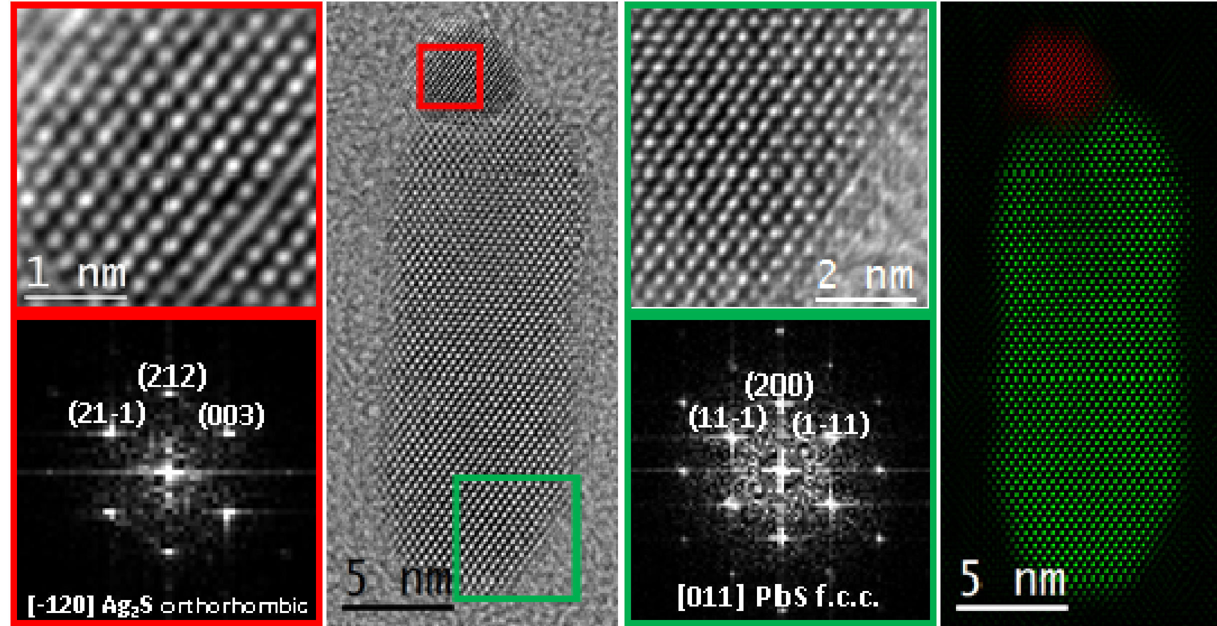

AlN-GaN Heteroyapıların Optik Özellikleri

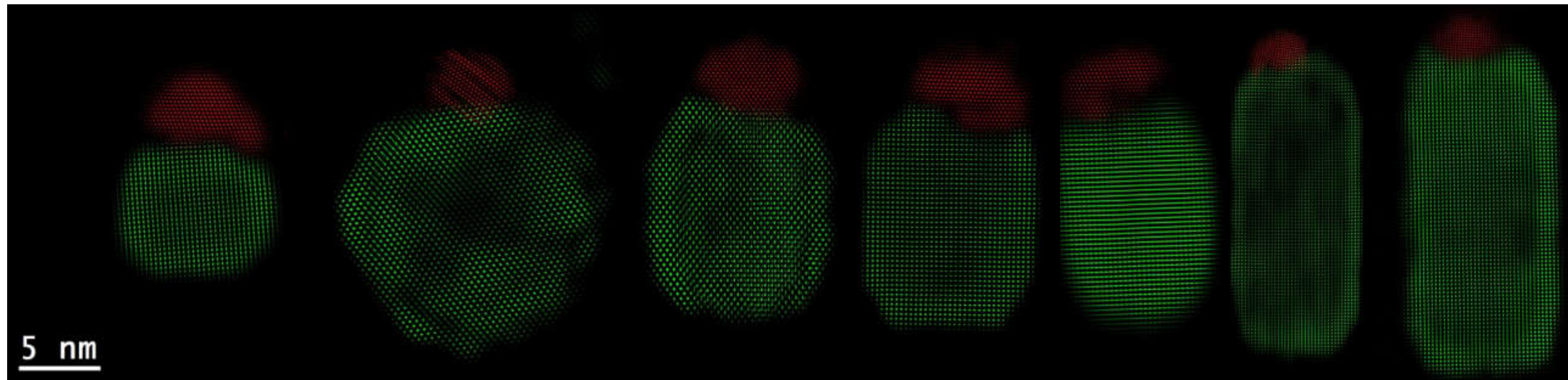
# Geçirimli Elektron Mikroskopları (TEM)

**CL map with a pixel size of 3 nm** 120 keV, Cryo



# Geçirimli Elektron Mikroskopları (TEM)




Termoelektrik uygulamalar için PbS – $Ag_2S$ hibrit nanoyapıları

# Geçirimli Elektron Mikroskopları (TEM)

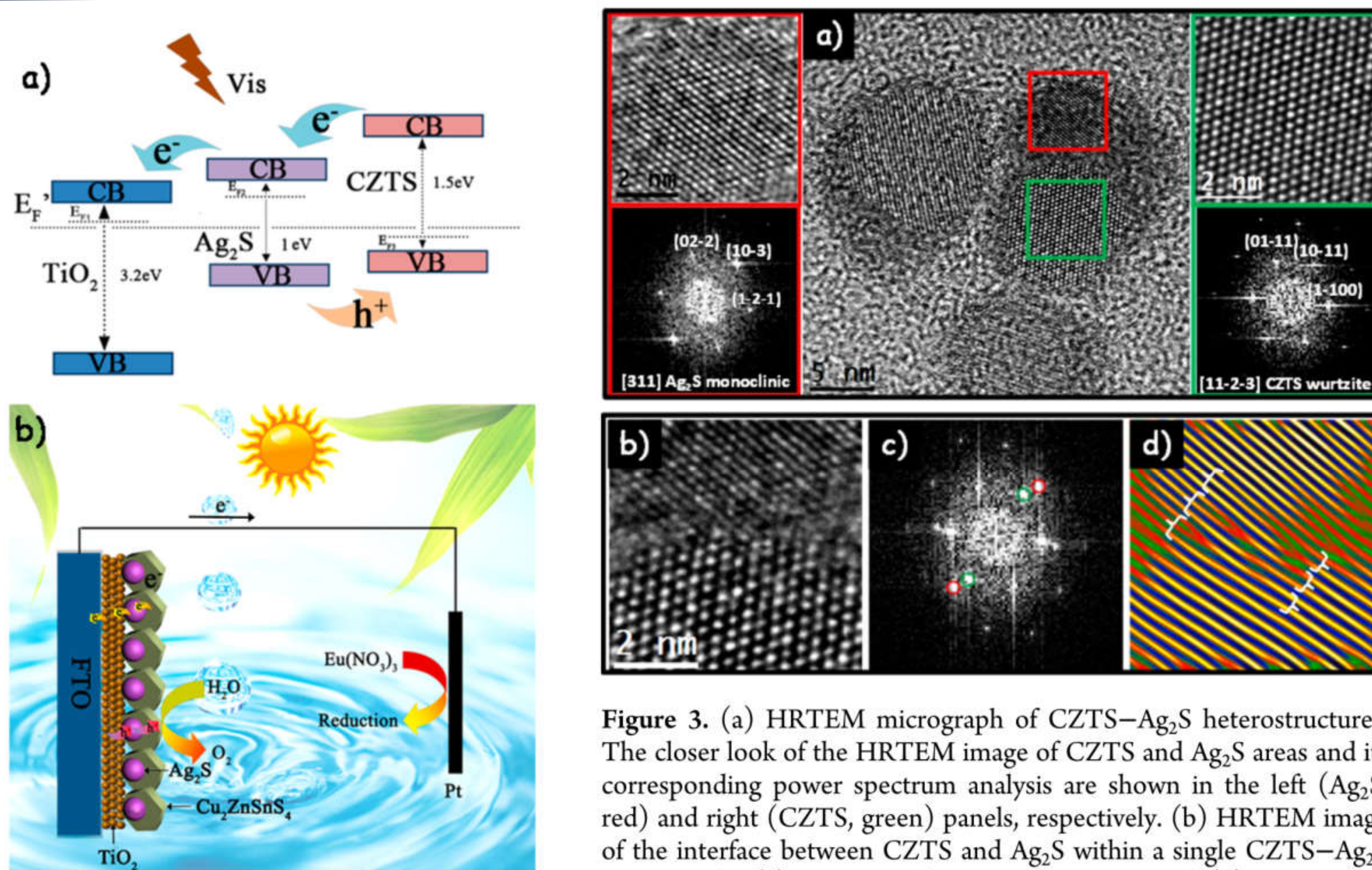



**Figure 5.** (a) Schematic of the energy band diagram of heterostructures and the possible model of electron transfer. (b) Schematic diagram of water splitting reaction in the photo-electrochemical cell.

**Figure 3.** (a) HRTEM micrograph of CZTS−$Ag_2S$ heterostructures. The closer look of the HRTEM image of CZTS and $Ag_2S$ areas and its corresponding power spectrum analysis are shown in the left ($Ag_2S$, red) and right (CZTS, green) panels, respectively. (b) HRTEM image of the interface between CZTS and $Ag_2S$ within a single CZTS−$Ag_2S$ nanoparticle. (c) Corresponding power spectrum and (d) inverse FFT of the circled diffraction spots showing the epitaxial relation.

DOI: 10.1021/acs.langmuir.5b02490
*Langmuir* 2015, 31, 10555−10561

# Geçirimli Elektron Mikroskopları (TEM)

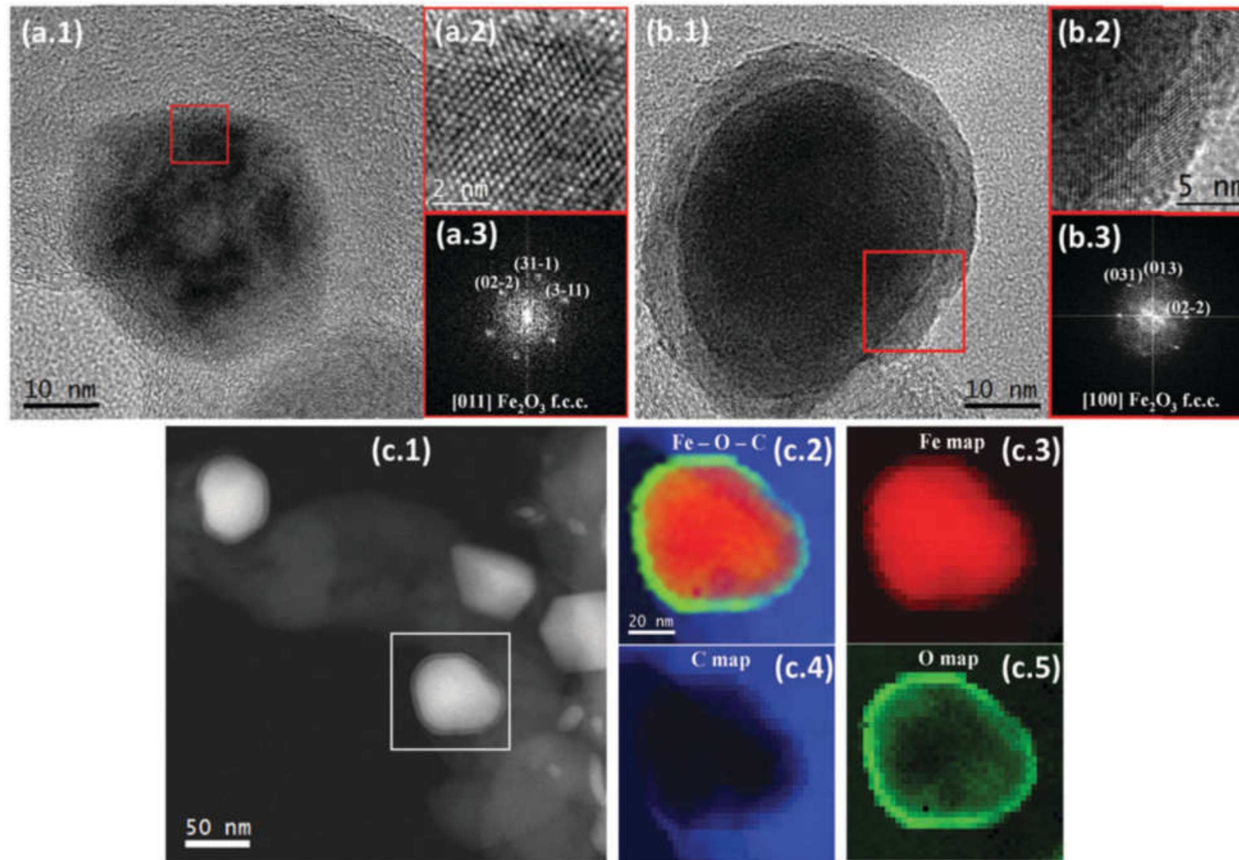
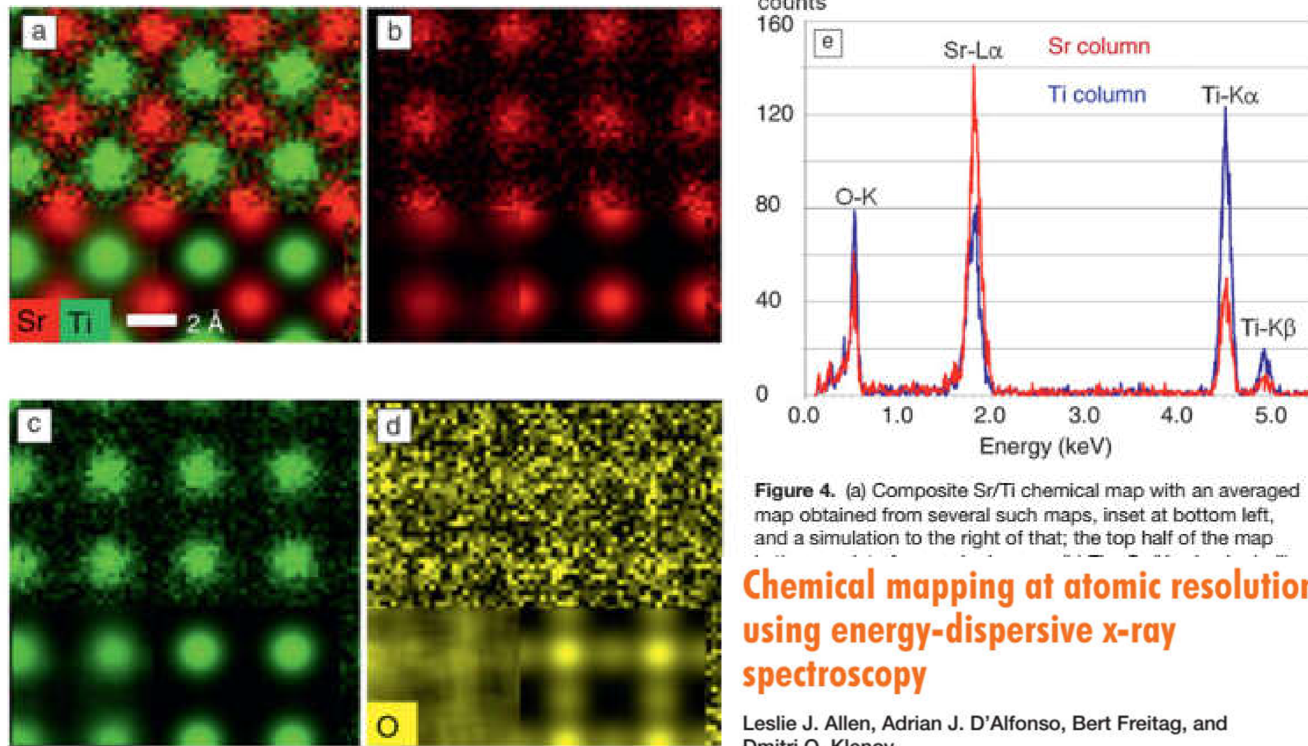
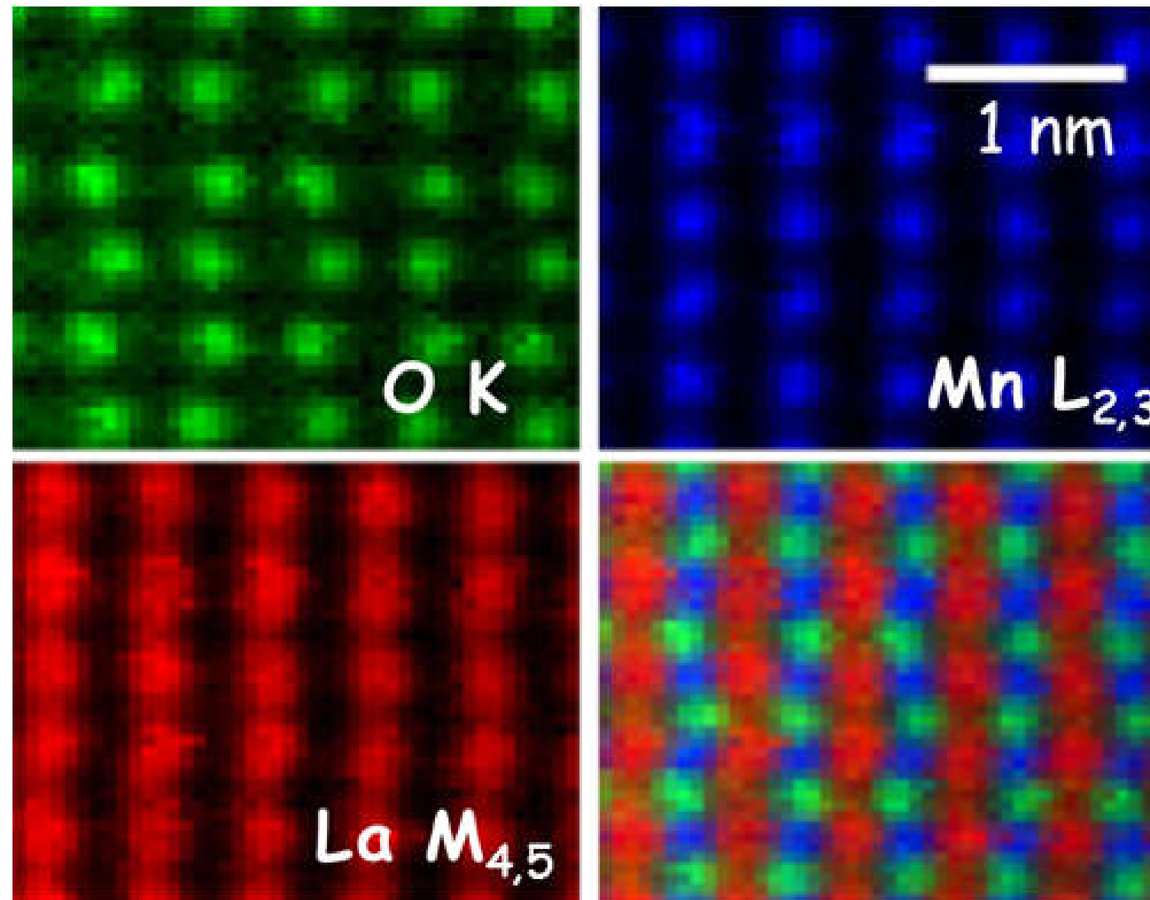



**Fig. 2** (a) and (b) HRTEM micrographs of $FeO_x$(650)/C along with the power spectra (FFTs) of the selected areas. (c) HAADF STEM of $FeO_x$(650)/C and EELS composition maps of the indicated area. Individual Fe (c.3 in red), C (c.4 in blue) and O (c.5 in green) maps along with (c.2) Fe–O–C.

# Geçirimli Elektron Mikroskopları (TEM)

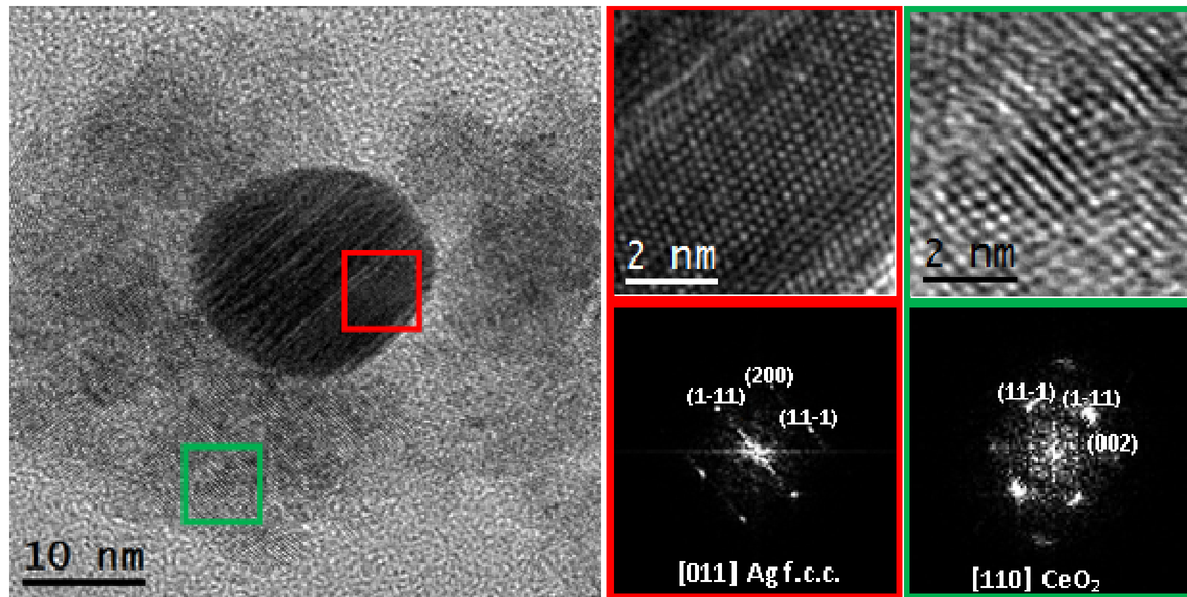


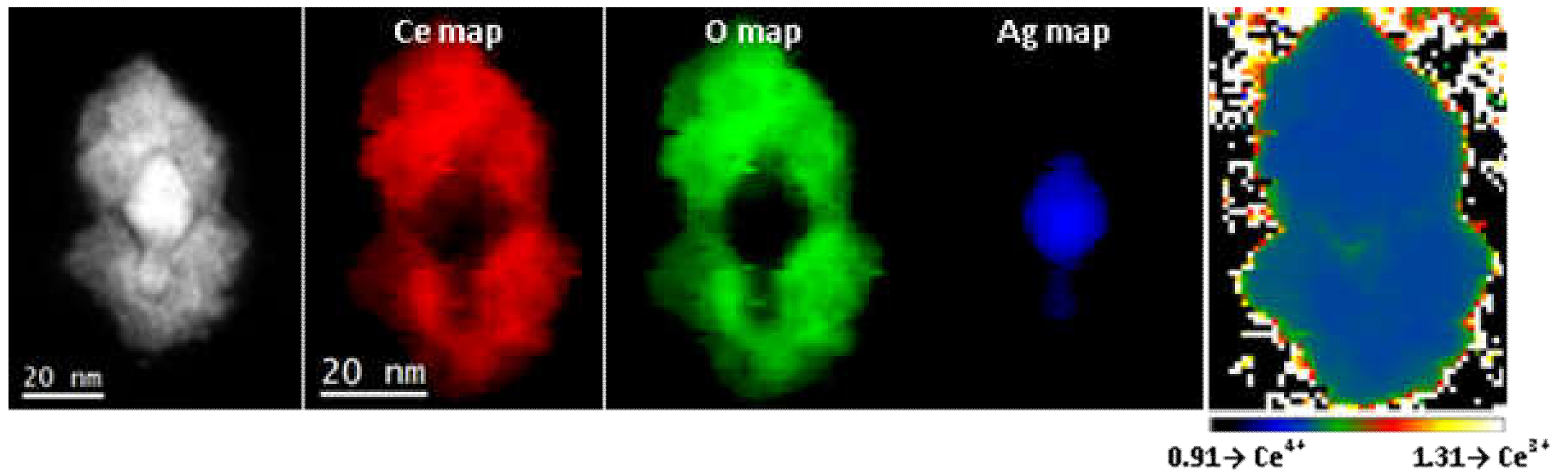
# Geçirimli Elektron Mikroskopları (TEM)



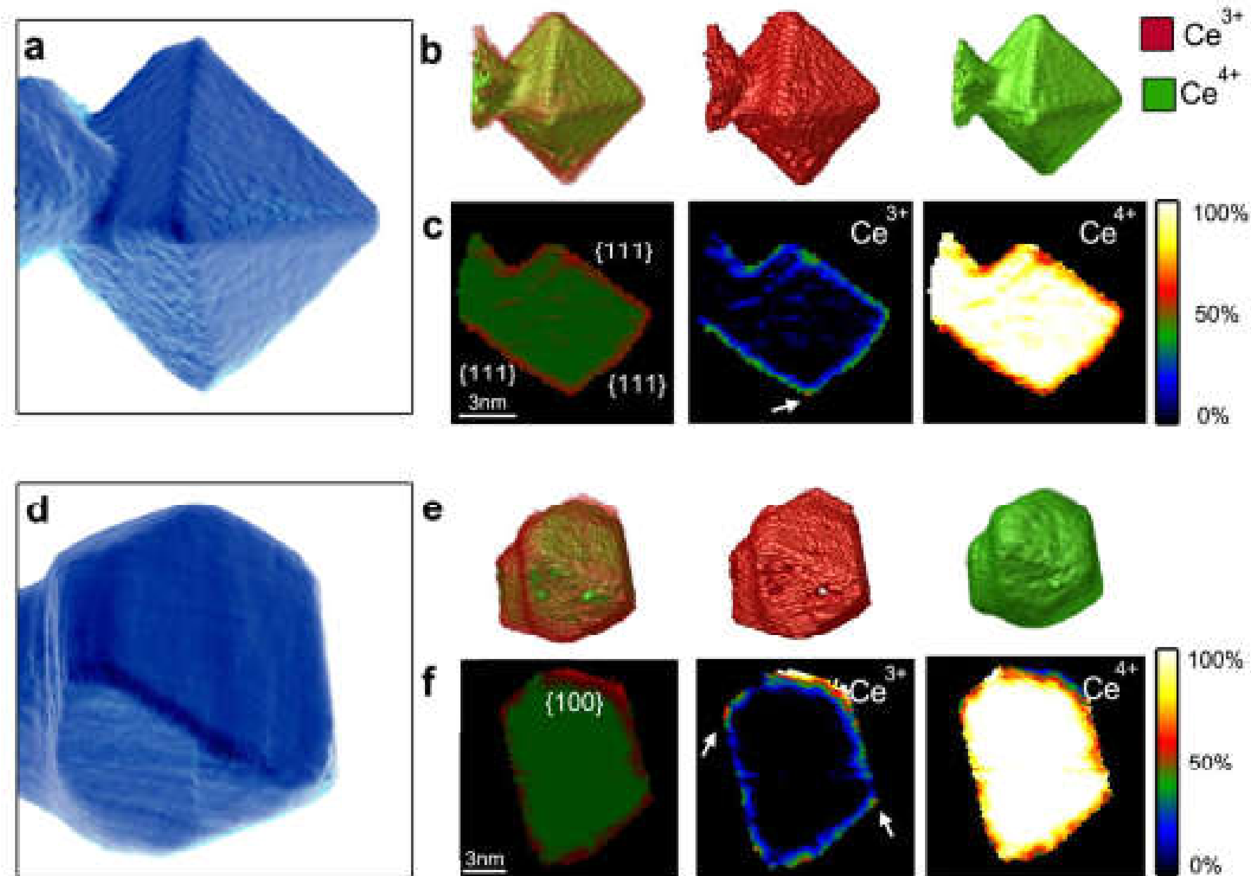

Spectroscopic images from a Nion UltraSTEM operating at 60kV that not only differentiate the elements in $LaMnO_3$, but also show clearly the octahedral rotations of the O sublattice. Data recorded by M. Varela and processed using Principle Component Analysis.

# Geçirimli Elektron Mikroskopları (TEM)



# Geçirimli Elektron Mikroskopları (TEM)




**Three Dimensional Valency Mapping in Ceria Nanocrystals**

Bart Goris, Stuart Turner, Sara Bals, and Gustaaf Van Tendeloo

# Taramalı Tünelleme Mikroskobu (STM)

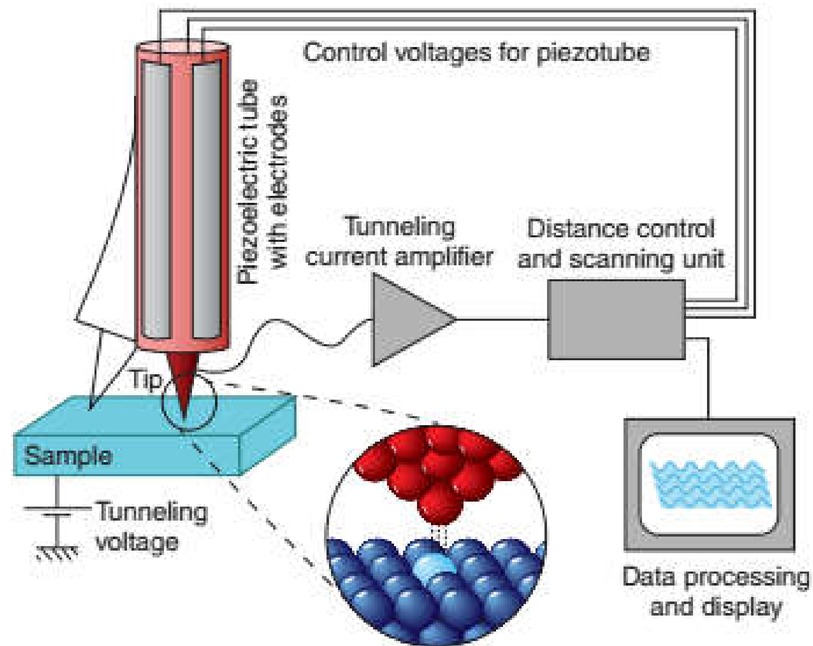



**Figure 3.13** Essential components of an STM with a close-up view of the tunneling effect between tip of the probe and the surface atoms. http://en.wikipedia.org/wiki/Scanning_tunneling_microscope.

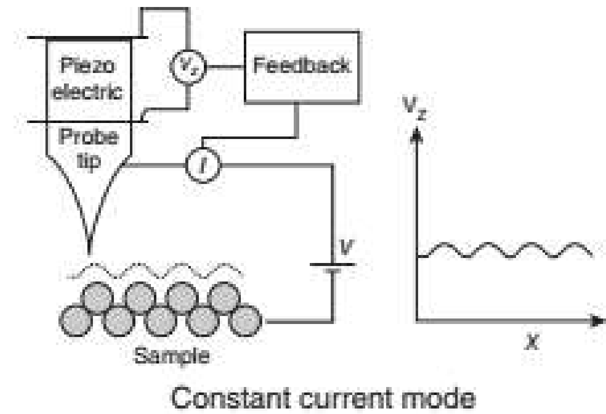
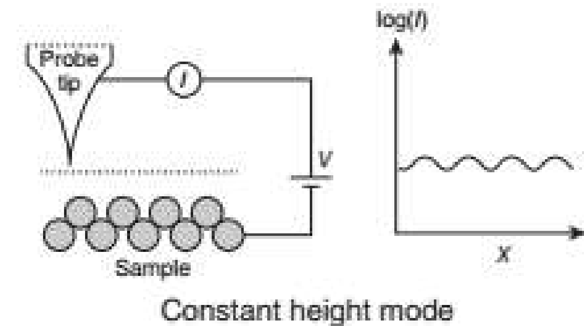



**Figure 3.14** (a) Constant current and (b)




) constant height imaging modes of an STM.

# Taramalı Tünelleme Mikroskobu (STM)

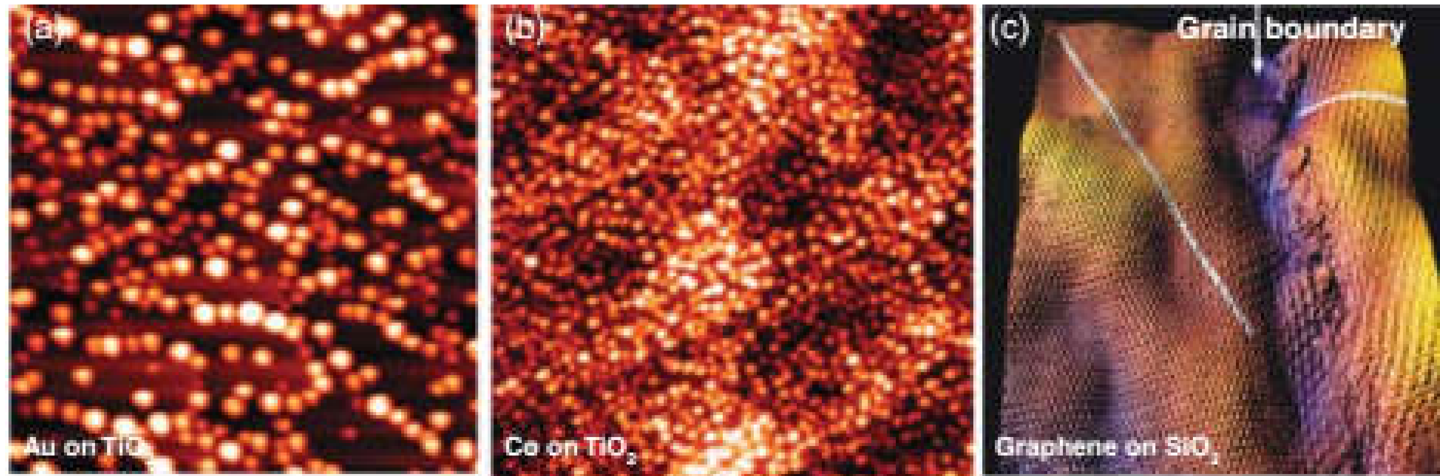
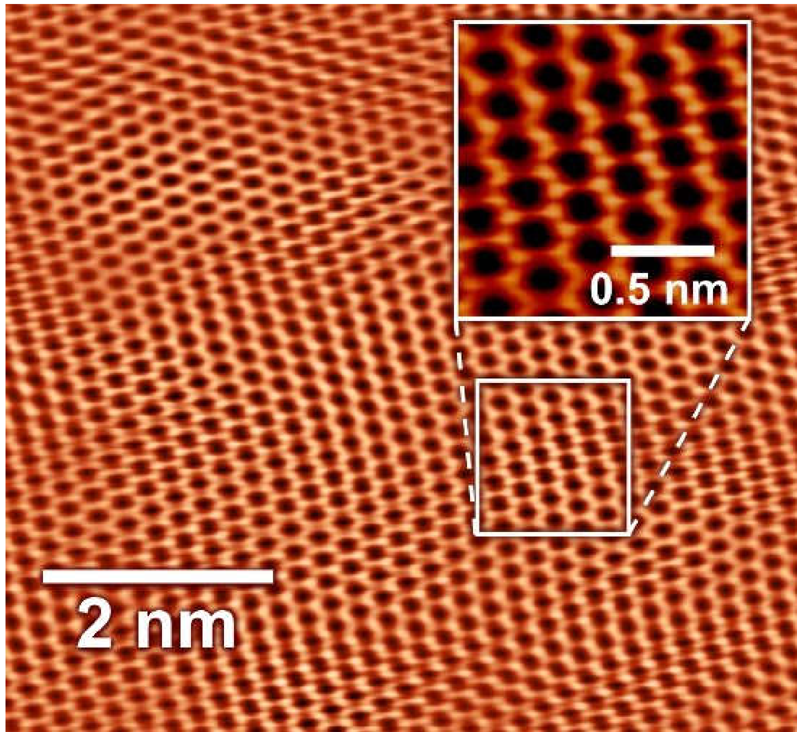
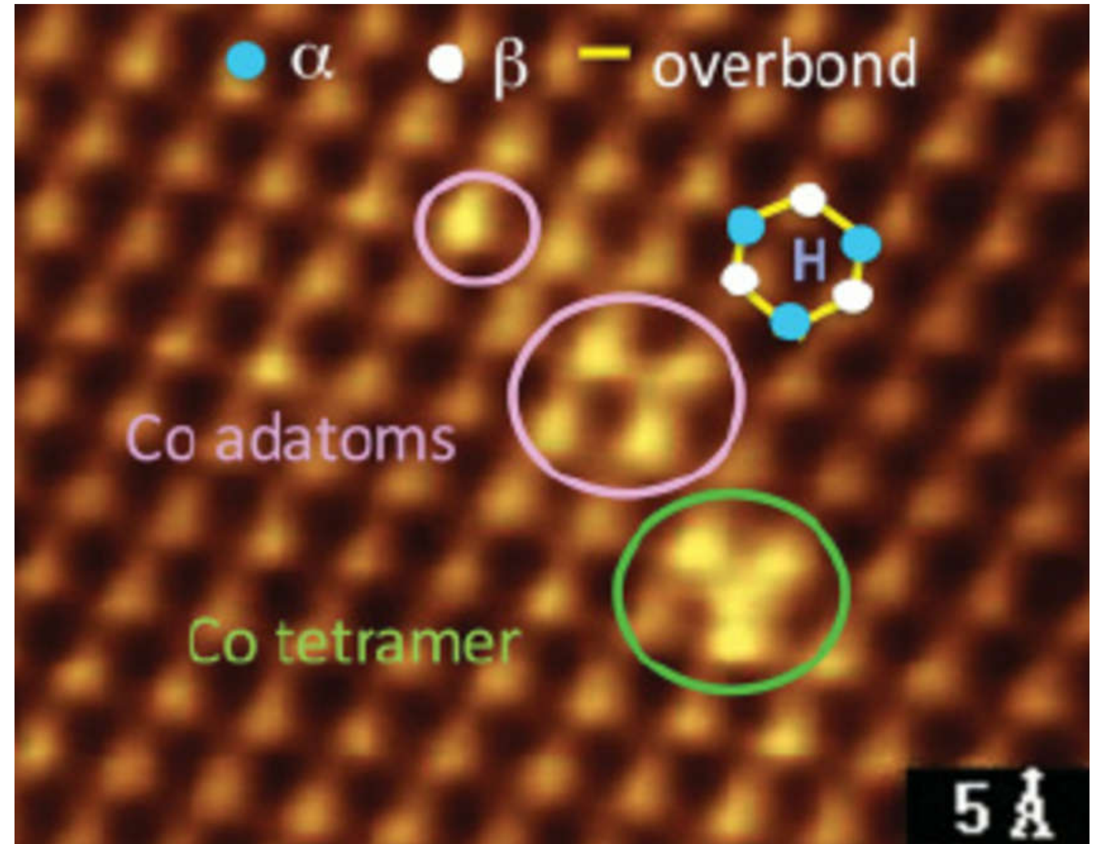



**Figure 3.15** STM images of (a) nano-gold on $TiO_2$. Galhenage et al. [17b], © 2013. With permission of ACS; (b) nano-cobalt on $TiO_2$ surface Galhenage et al. [17b], © 2013. With permission of ACS; (c) graphene on $SiO_2$ showing grain boundary. Koepke et al. [19], © 2013. With permission of ACS. (*See color plate section for the color representation of this figure.*)

# Taramalı Tünelleme Mikroskobu (STM)




Grafit

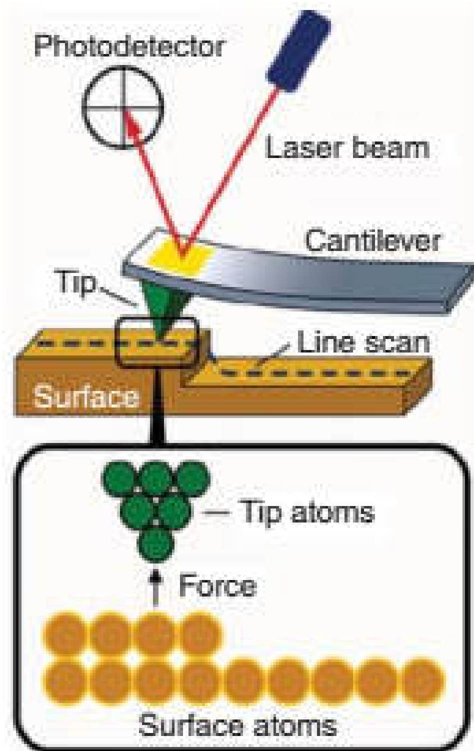


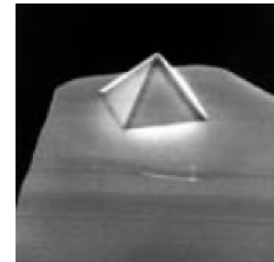

PHYSICAL REVIEW B **84**, 054420 (2011)

**Growth mechanism and interface magnetic properties of Co nanostructures on graphite**

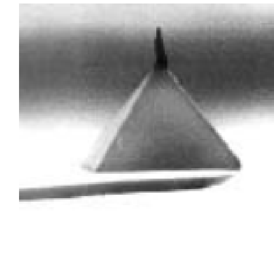
P. K. J. Wong, M. P. de Jong,* L. Leonardus, M. H. Siekman, and W. G. van der Wiel

# Atomik Kuvvet Mikroskobu (AFM)

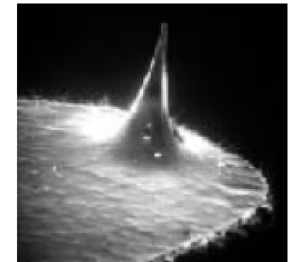


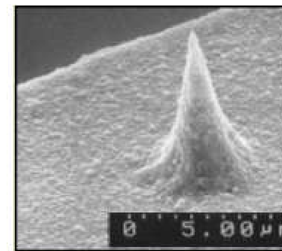
Normal Tip



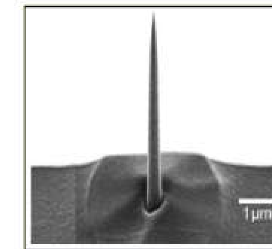
Supertip



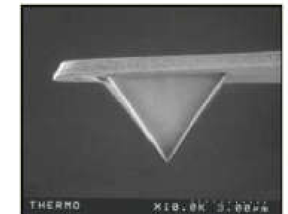
Ultralever




Diamond-coated tip




FIB-sharpened tip



Gold-coated $Si_3N_4$ tip

**Figure 3.16** Schematic representation of AFM, Courtesy of Keysight Technologies.

# Atomik Kuvvet Mikroskobu (AFM)

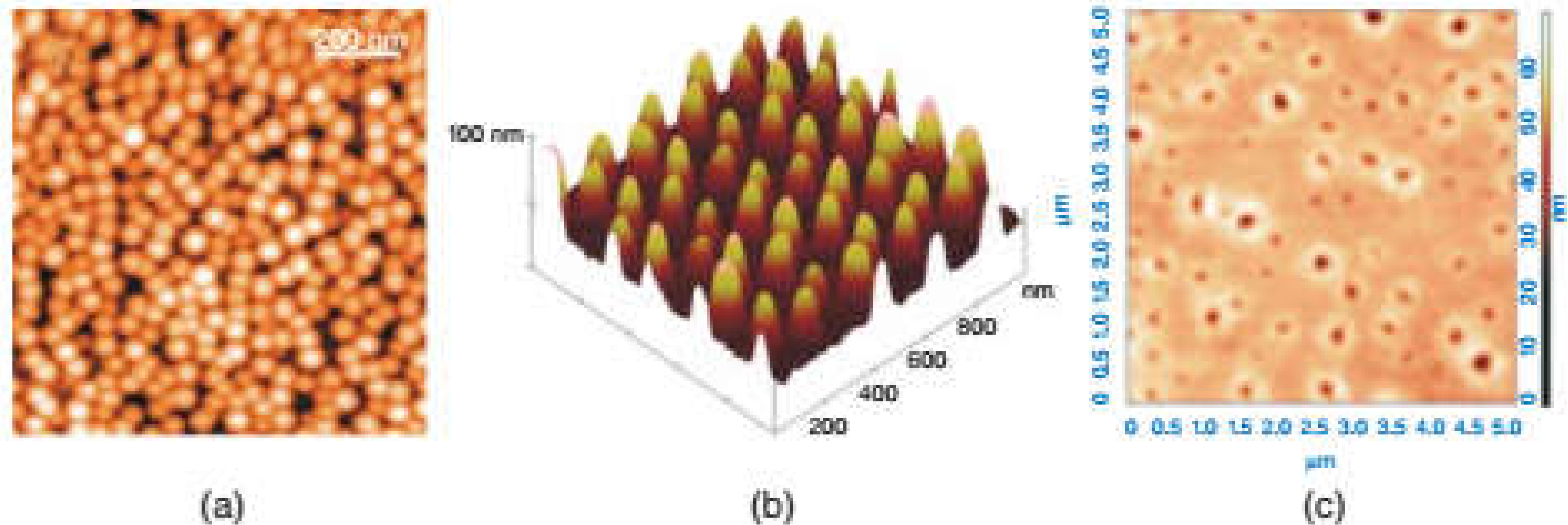
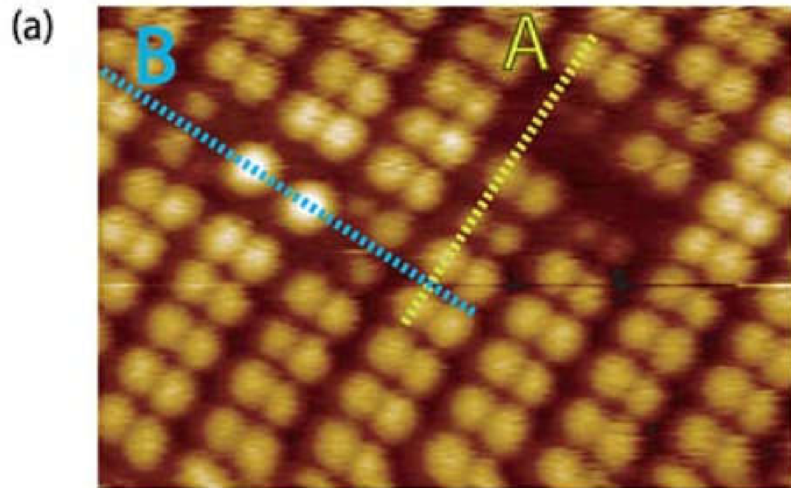
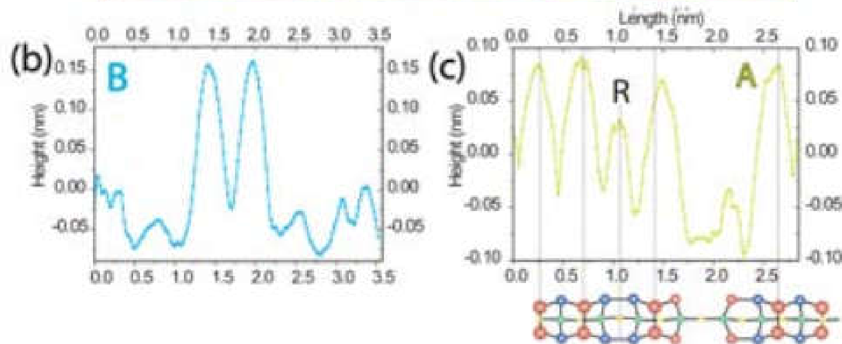



**Figure 3.17** AFM images showing (a) InAs/GaAs QDs grown on a Ge/Si substrate. Chen et al. [21a], © 2015. With permission of licensee MDPI, Basel, Switzerland; (b) Pt nanoparticles coated with Si nanopillars. Li et al. [21b], © 2015. With permission of NPG; (c) cholesterol–B, SA conjugate. Gehlot et al. [22], © 2013. With permission of NISCAIR, CSIR, New Delhi. (*See color plate section for the color representation of this figure.*)

# Atomik Kuvvet Mikroskobu (AFM)

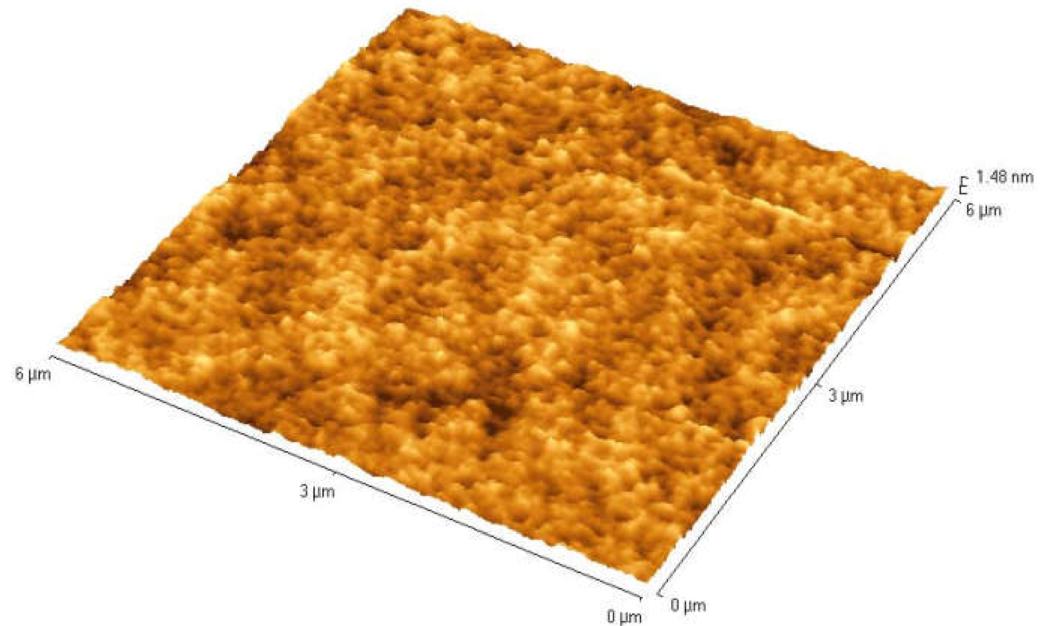



**Si(100) surface taken at 77 K.**

**Scanned with non-contact mode.**

Clean glass surface: roughness ~ 0.8 nm



# Spektroskopi Teknikleri

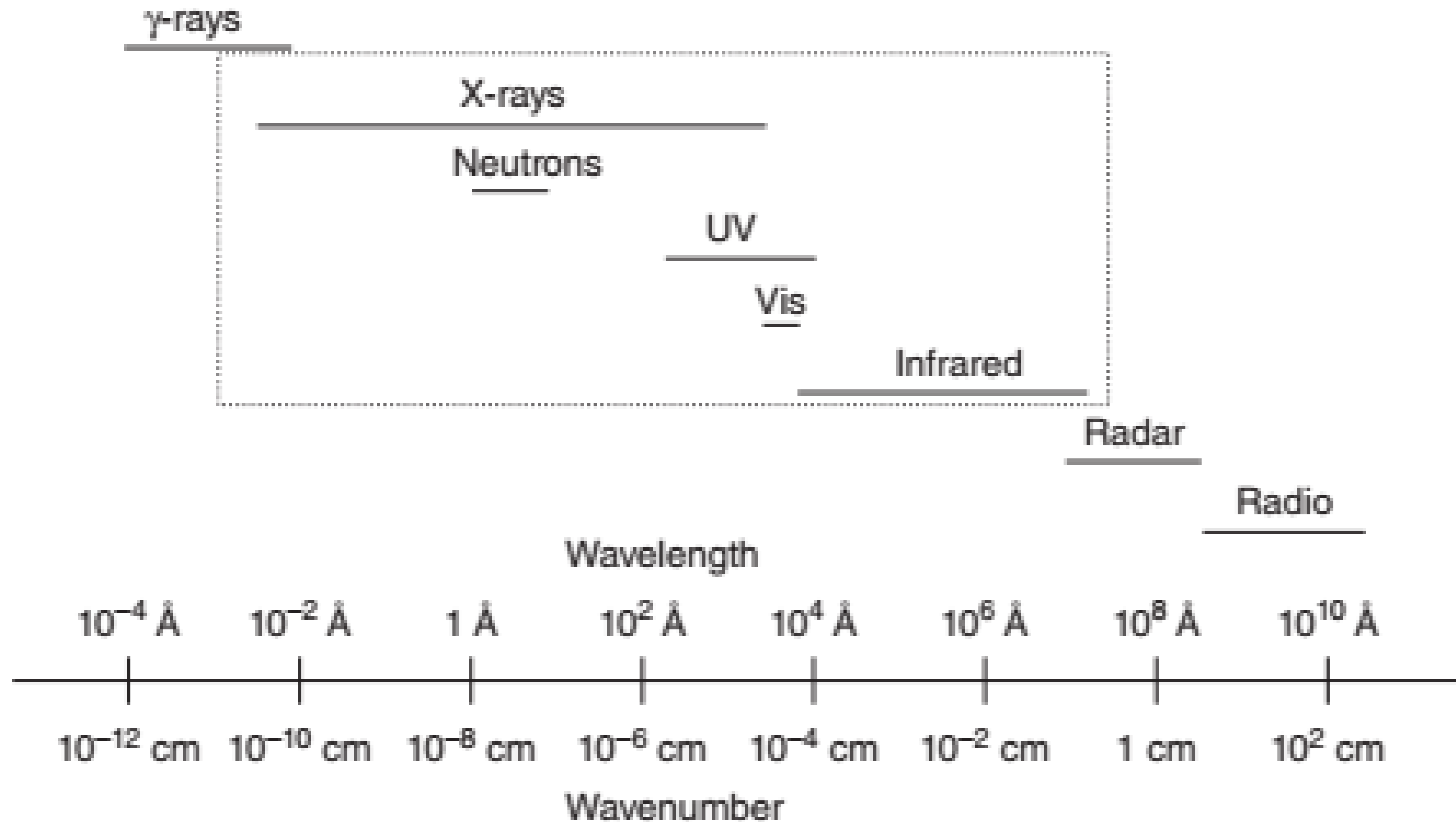



**Figure 3.19** Spectrum of various electromagnetic radiations.

# Saçılım Teknikleri

**TABLE 3.7 Radiations Used in Scattering-Based Characterization Techniques**

| Scattering Techniques | Type of Radiation (Wavelength, $\lambda$) | Sample Thickness | Limitations |
|---|---|---|---|
| SAXS, WAXS | X-rays (0.1–5 Å) | <1 mm | Absorption |
| SANS, WANS | Neutrons (1–15 Å) | 1–2 mm | Low fluxes |
| LEED | Electrons (0.1 Å) | 100 μm | Low penetration |
| SLS, DLS | Laser light (1 μm) | 1–5 mm | Scattering from dust |

**SAXS: Düşük açılı X-Işını saçılımı, WAXS: Geniş açılı X-ışını saçılımı**
**SANS: Düşük açılı nötron saçılımı, LEED: Düşük enerji elektron kırınımı, DLS: Dinamik ışık saçılımı**