**LABORATORIUM KIMIA FISIKA**
**Jurusan Kimia - FMIPA**
**Universitas Gadjah Mada (UGM)**

# KIMIA ZAT PADAT
## Pengamatan Kerusakan pada Kristal

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## Pengamatan dislokasi

***Teknik mikroskop elektron seperti*** Transmission Electron Microscopy (TEM) dapat digunakan untuk pengamatan dislokasi yang terjadi pada mikrostruktur bahan kristalin.

Secara virtual dapat dibayangkan bahwa semua bahan kristalin akan memiliki kerusakan dislokasi yang terjadi selama proses pemadatan, pada waktu deformasi plastik, dan sebagai akibat penekanan termal pada proses pendinginan bahan.

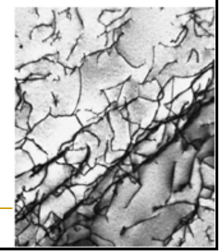

**Figure 4.6** A transmission electron micrograph of a titanium alloy in which the dark lines are dislocations. 51,450×.

Dislokasi dari atom-atom yang tidak tersusun baik terlihat dalam warna gelap.



## MICROSCOPIC TECHNIQUES

Mbanyak kerusakan yang secara struktural teramati dengan bantuan mikroskop.
Terdapat dua jenis mikroskop yakni optik dan elektron. Masing-masing dikoneksikan dengans fasilitas fotografi untuk merekam secara visual.
Perkembangan teknik dilakukan dengan menggunakan model transmisi dan reflektif.

Teknik mikroskop:
- **Microscopi optik**
- **Microscopi Elektron :**
  - transmission electron microscope (TEM)
  - scanning electron microscope (SEM)
  - Scanning probe microscope (SPM)



## Skala ukuran kerusakan

## Kisaran kemampuan pengamatan

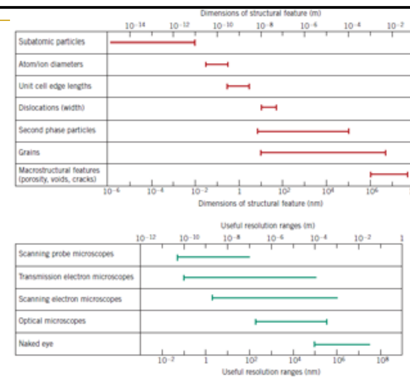


**Figure 4.15** (*a*) Bar-chart showing size ranges for several structural features found in materials. (*b*) Bar-chart showing the useful resolution ranges for four microscopic techniques discussed in this chapter, in addition to the naked eye.



## Mikroskop optik

Dengan mikroskop optik, cahaya mikroskop digunakan untuk mengamati mikrostruktur sampel.
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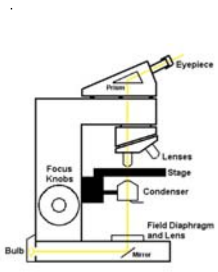




## Mikroskop optik

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Contoh:

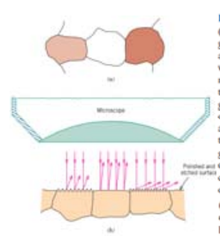


**Figure 4.13**
(*a*) Polished and etched grains as they might appear when viewed with an optical microscope. (*b*) Section taken through these grains showing how the etching characteristics and resulting surface texture vary from grain to grain because of differences in crystallographic orientation.
(*c*) Photomicrograph of a polycrystalline brass specimen. 60×.

## Mikroskop optik

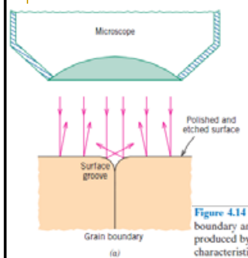
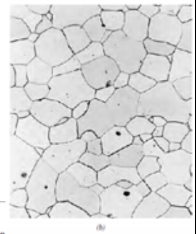


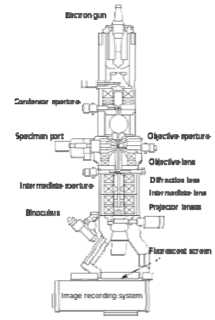

**Figure 4.14** (*a*) Section of a grain boundary and its surface groove produced by etching; the light reflection characteristics in the vicinity of the groove are also shown. (*b*) Photomicrograph of the surface of a polished and etched polycrystalline specimen of an iron-chromium alloy in which the grain boundaries appear dark. 100×.






## TRANSMISSION ELECTRON MICROSCOPE (TEM)

- Gambar yang dihasilkan dari TEM terbentuk dari pancaran elekttron yang melewati sampel.
- Detail mikrostruktur dapat diamati melalui pengaturan kekontrasan perbedaan elektron yang dipancarkan dan yang didifraksikan.

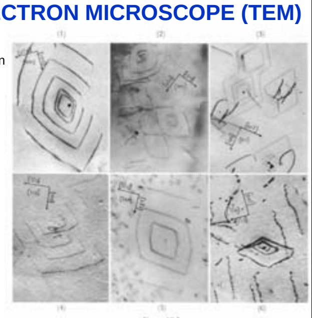





## TRANSMISSION ELECTRON MICROSCOPE (TEM)

- Mengingat padatan mengabsorpsi elektron, maka sampel harus disiapkan dalam bentuk lapis tipis. Pancara elektron yang ditransmisikan dikenakan pada lapisan fluoresen atau lapisan film sehingga dapat memunculkan gambar.
- Perbesaran dapat diperoleh sampai 1,000,000 X yang memungkinkan untuk pengamatan dislokasi.

Mikrograf dari TEM untuk pengamatan dislokasi pada logam.

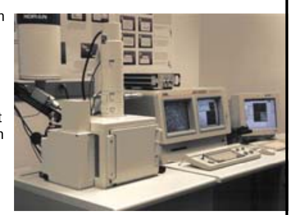


Contoh dislokasi loop pada logam.

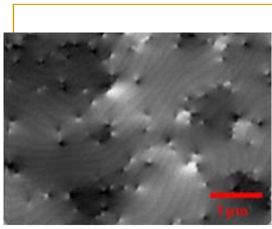


## SCANNING ELECTRON MICROSCOPE (SEM)

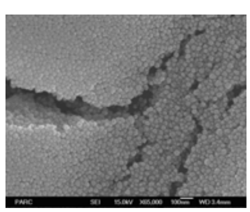
- Permukaan sampel dikenai pancaran elektron dan hasil yang dipantulkan dikumulkan untuk diolah menjadi gambar pada tabung sinar katoda. Hasil gambar berupa tampilan permukaan sampel.
- Permukaan sampel haruslah bersifat konduktif, biasanya sampel yang non konduktif akan diselaput dengan lapisan logam.
- Perbesaran dapat berlangsung dari 10- 50.000 kali
- Instrumentasi dapat dilengkapi untuk keperluan analisis kualitatif dan semikuantitatif.







**SEM tentang dislokasi lapis tipis nitrida**



SEM tentang lapis tipis tinta berbasis perak



## Perbedaan TEM dan SEM

**Comparison of TEM and SEM**

RJL 11/14/98 rev. 11/22/05

definitions: e- = electron
GFPC = gas filled proportional counter
PMT = photomultiplier tube
SCD = semiconductor detector (Si or Ge)

| | SEM | TEM |
|---|---|---|
| Invented: | Zworykin et al., 1942 | Ruska, 1933 |
| Commercially Available: | Cambridge Instr., 1965 | Vickers, 1936 |
| Design: | 2 CRTs, with synchronized e-beams scanning raster patterns. | 1 CRT, raster scan not essential. |
| Electron column: (CRT #1) | Electron gun, 2 lenses 1 aperture, sample and movable stage, various detectors. See below. | Electron gun, 4 lenses, 2 apertures, sample & movable stage. Half the lenses & apertures are above & half below the sample. |
| Sample Chamber: | Large. Allows for sample tilt & rotation. May also allow electrical connections and mechanical test apparatus. | Small. Allows for sample tilt and rotation. |
| Typical Voltage & Resolution: | 1 to 50 kV 30 Ang. or more, depends on sample. Great depth of field. | 50 to 300 kV, even a million volts! 10 Ang. or more. atomic planes visible |

| | | |
|---|---|---|
| Typical Voltage & Resolution: | 1 to 50 kV<br>30 Ang. or more, depends on sample. Great depth of field. | 50 to 300 kV, even a million volts!<br>10 Ang. or more. atomic planes visible |
| Display: | CRT #2 provides a TV-like display. Display brightness is determined by detector output, adjusted for brightness & contrast. | A fluorescent screen inside the electron column at the bottom.<br>... or an area detector |
| Photography: | Take photo of the CRT display or capture image digitally for analysis. | Load film cartridge inside the e-column under the fluorescent screen. Flip up screen to expose the film. |
| Standard Detector: | Secondary e- (<50 eV) uses scintillator & PMT. Gives good topographical contrast. | Just the fluorescent screen and photographic film. |



| | | |
|---|---|---|
| Optional Detectors: | Backscattered e- (same energy as incident beam) uses SCD or scintillator & PMT. Gives good compositional contrast. | Electron energy loss spectrometer detects lighter elements using quadrupole magnetic detector in the transmitted beam. |
| | Energy dispersive X-ray uses SCD, detects heavy elements. | Energy dispersive X-ray. |
| | Wavelength dispersive x-ray uses crystal diffractometer with GFPC. Detects lower concentrations, lighter elements and avoids peak convolution. Works slow. | Secondary e- detector, plus raster scan capability. |
| | Photoemission (a.k.a. cathodoluminescence) uses a mirror & PMT. Good for non- or semi-conductors. | |
| | Specimen current to ground = beam - secondary - backscatter. | |
| | Voltage contrast uses a slightly modified secondary e- detector to image regions of varying potential. Ideal for IC chips. Strobe the beam off & on to "freeze" periodic signals. | |
| | Electron beam induced current, flows between two contacts to the sample, not to ground. Good for semiconductors. | |
| | Thermal wave uses a piezoelectric microphone to detect acoustic noise generated in sample by pulsing (blanking) the e- beam. Good for imaging features which conduct heat poorly. | |



| | | |
|---|---|---|
| Sample form: | Almost any clean solid. Big, thick samples are OK. | Foil or powders < 1000 Ang. thick. or surface replicas. |
| Sample prep: | First clean off dirt & grease. Insulators must be coated with a conducting layer ~100 Ang thick. Sputter or evaporate metal or C. Sample prep is usually simple. | Use ion mill, focused ion beam, electropolishing, jet polishing, dimpling, etc. Sample prep is usually a lot of work and may irreversibly change the material. |
| Most useful for: | Fracture, wear or corrosion surfaces, powders, polished & etched microstructures, IC chips, chemical segregation. | Selected area e- diffraction, imaging of dislocations, tiny precipitates, grain boundaries and other defect structures in solids. |

Both SEM and TEM are useful in biology and geology, as well as in materials science.



## SCANNING PROBE MICROSCOPY (SPM)

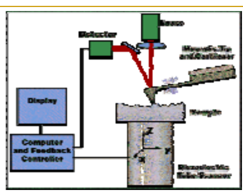
SPM berbeda dari dua teknik mikroskop sebelumnya karena tidak menggunakan cahaya atau elektron untuk membentuk gambarnya. Mikroskop ini membangkitkan peta topografis pada skala atomik untuk merepresentasikan permukaan sampel yang diuji.

Beberapa fitur SPM:

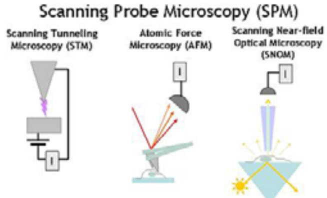
- Pemeriksaan pada skala nanometer sangat dimungkinkan karena perbesaran setinggi yang mungkin dengan hasil resolusi yang jauh lebih baik dibandingkan dengan teknik mikroskopis lainnya.
- Gambar dapat diolah menjadi tiga dimensi untuk memberikan informasi topografi tentang fitur yang menarik.
- Beberapa SPMs dapat dioperasikan di berbagai lingkungan (misalnya, vakum, udara, cair), dengan demikian spesimen tertentu dapat diperiksa dalam lingkungan yang paling sesuai.
- Scanning probe mikroskop menggunakan probe kecil dengan ujung yang sangat tajam yang dibawa ke dalam jarak sangat dekat (yaitu, untuk dalam pada urutan nanometer) dari permukaan spesimen.



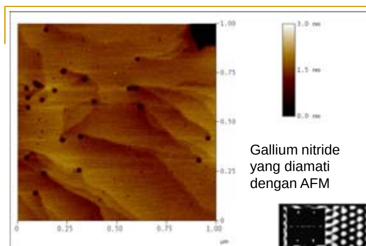
## SPM



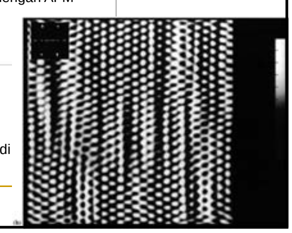

Tipe lain





Gallium nitride yang diamati dengan AFM



Gambar molekular dari lapisan cadmium arachidate di atas mika