HRTEM Aplicada a Ciência dos Materiais

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Conteúdo

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  - 3 – Canhões de Elétrons
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  - 5 – Correção de Aberração Esférica (Cs)
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Referências Bibliográficas

**TEM:**

Análise de TEM

- **Electron Gun (FEG)**
  - Brightness, Coherence, \( \lambda \) (Resolution)
- **Thin Specimen**
  - Preparation Techniques / Sample Holder
- **Objective Lens**
  - Low C\(_o\), C\(_c\) (Resolution)
- **Column**
  - Clean High Vacuum, Controlled Atmosphere
- **Images**
  - BF, DF, HR
  - Resolution to 1Å
- **Diffraction**
  - Structure Spatial 10Å
- **Spectroscopy**
  - (EDS)
  - Z = 6 to 45, 0.1%
  - Spatial Res. 100Å (EELS)
  - All Elements Bonding, Electronic States
  - Spatial Resolution 1Å
- **Magnetic Imaging**
  - (Holography)
  - Magnetic Domains Imaging
  - Spatial Res. 50Å
Canhões de Elétrons

<table>
<thead>
<tr>
<th>Thermal emission</th>
<th>Field emission</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brightness (A/cm²sr) at 200kV</td>
<td>W</td>
</tr>
<tr>
<td>~5x10⁸</td>
<td>~5x10⁸</td>
</tr>
<tr>
<td>Electron Source Size</td>
<td>50µm</td>
</tr>
<tr>
<td>Energy Width (eV)</td>
<td>2.3</td>
</tr>
<tr>
<td>Operating Condition</td>
<td>Vacuum (Pa)</td>
</tr>
<tr>
<td>Temperature (K)</td>
<td>2800</td>
</tr>
<tr>
<td>Emission</td>
<td>Current (µA)</td>
</tr>
<tr>
<td>Short term stability</td>
<td>1%</td>
</tr>
<tr>
<td>Long term stability</td>
<td>1%/hr</td>
</tr>
<tr>
<td>Maintenance</td>
<td>Start-up takes time</td>
</tr>
<tr>
<td>Price &amp; Operation</td>
<td>Low &amp; simple</td>
</tr>
<tr>
<td>Lifetime</td>
<td>3 months</td>
</tr>
<tr>
<td>UIC instruments</td>
<td>JEM-100CX</td>
</tr>
</tbody>
</table>

TEMs Modernos

FEI TECNAI G2

Canhões de Elétrons

| Operating Conditions | Vacuum (Pa) | 10⁻¹ | 10⁻¹ | 10⁻¹ | 10⁻¹ |
| Temperature (K) | 2800 | 1800 | 1800 | 1600 | 300 |
| Emission | Current (µA) | ~100 | ~100 | ~100 | 20-100 |
| Short term stability | 1% | 1% | 1% | 7% | 5% |
| Long term stability | 1%/hr | 3%/hr | 1%/hr | 6%/hr | 20% |
| Maintenance | Start-up takes time | Build up necessary after change | Flash every few hours |
| Price & Operation | Low & simple | Low & simple | High & easy | High & easy | High & complicated ? |
| Lifetime | 3 months | 1 year | >4 years (UIC 8 years +) | ? | ? | 1 year |
| UIC instruments | JEM-100CX | JEM-3010 | JEM-2010F | NA | NA | HB601 |
TEMs Alta Voltagem

Osaka University, Osaka, Japan

Publications
- Observation of the X-ray Diffraction from a Nanometer-Sized Dislocation Loop, K Arakawa, K Otsu, M Hatanaka, E Kuramoto, M Ishikawa and H Mori, Synops 31B (2005) p1056

See also: http://www.uhvem.osaka-u.ac.jp/pub-2007.html

URL
www.uhvem.osaka-u.ac.jp/index-j.html

<table>
<thead>
<tr>
<th>Manufacturer and Model:</th>
<th>Hitachi H-3000</th>
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</thead>
<tbody>
<tr>
<td>Electron Accelerating-Voltage:</td>
<td>0.5 to 3.5 MV</td>
</tr>
<tr>
<td>Magnification:</td>
<td>0.5 to 1000 kx</td>
</tr>
<tr>
<td>Resolution:</td>
<td>0.14 nm (5 nm STEM)</td>
</tr>
<tr>
<td>Angle between Electron and Ion Beams:</td>
<td>Film, CCD and harpicon camera</td>
</tr>
<tr>
<td>Recording Facilities:</td>
<td></td>
</tr>
<tr>
<td>Additional TEM-Analysis:</td>
<td></td>
</tr>
</tbody>
</table>

TEMs Modernos

Titan™ G2 60-300
Deep sub-Ångström performance with the largest high tension range in STEM imaging and analysis optimized for a wide range of materials

Dynamic in situ exploration of nanomaterials at the atomic scale with variable gas pressures and temperatures
TEMs Modernos

Titan<sup>3</sup>™ G2 60-300
Ultimate performance and high imaging and analysis in C<sub>3</sub>-corre

The Titan<sup>3</sup>™ G2 60-300™ is the most powerful high res electron microscope (S)TEM with the largest accelerati for 2D and 3D material characterization and chemical a The novel, environmentally isolated platform design all in S/TEM imaging and chemical analysis by combining image C<sub>3</sub>-correctors), a monochromator, and a novel, u gun (X-PEG) in one instrument.

The option to combine two correctors enables studies i probe and in TEM mode with a parallel beam with 70 p specimen area in the same microscope. Hence the ben can be obtained in a double-corrected S/TEM platform. environmental enclosure, the system can transfer infor
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HITACHI
HF-3300 300 kV FE-TEM

HITACHI
HD-2700 Cs-Corrected FE-STEM

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JEM-ARM200F JEOL
Atomic Resolution Analytical Microscope
HAADF–STEM image of Opx recorded along the [001] direction. (Center) Fourier transform (FT) of the image. (Right) Radial distribution of the signal in the FT. (a) Raw image. (b) Filtered image from (a) by using the Wiener filter. (c) Low pass filtered image from (b) by cutting the signal >8 nm$^{-1}$ in the FT.

Filtered image of the pyroxene along the c-axis with a boundary between Opx (upper half) and augite lamella (lower half). The inset at the upper left is the image from the same specimen taken by CTEM with a point resolution of 2.0 Å (the contrast is reversed from the original image to compare with the HAADF–STEM image). Partial enlargements of the image to indicate the corresponding cation sites are shown at the right side. (b) Analysis of the contrast at the octahedral slabs in Opx (upper) and augite (lower). See text for the detail. The crystal structures corresponding to the contrasts are also shown in the right.
HRTEM - Exemplos


Application to semiconductor interfaces, (a) SiO2/Si, (b) surface of GaAs.

Direct observation of oxygen atoms in a MgO crystal of 1 nm thick near a hole.

Fig. 5: ADF-STEM image of a stacking fault of a SiGe alloy.

Fig. 6: World first Cs-corrected ADF-STEM observation of an antimony (Sb) dopant moving in silicon. R & L show positions in the dumbbell images.

HRTEM - Exemplos

Experimental ABF/HAADF STEM and HR-TEM images
LaB₆ [100] specimen

Comparison between the different imaging techniques (HR-TEM/HR-STEM) on one material.
HRTEM - Exemplos

HR-TEM on gold nanoparticles on carbon film
TITAN image corrected with 80, 200, 300kV

- Cs-corrected images on one instrument at the same area with different acceleration voltages
- At heavy elements 300kV shows the clearest image of the surface
- At 80kV the carbon contrast is maximized and a step in thickness in the center of the particle becomes visible
Titan G2 performance exemplified in results:
Best S/TEM resolution, analytics, and material science

Best HR-S/TEM performance in the market (70pm)

Phase of the exit wave
Exit wave
Reconstruction
After processing
20 HRTEM
2k x 2k images
Nb, O, Ba columns and Na/Ba columns can be identified
In Phase image of Electron Exit wave
**Atomic-Column STEM-EELS**

La$_{1.2}$Sr$_{1.8}$Mn$_2$O$_7$


La$_{0.7}$Sr$_{0.3}$MnO$_3$/SrTiO$_3$


La$_{0.5}$Sr$_{0.5}$MnO$_3$


Bi$_{0.5}$Sr$_{0.5}$MnO$_3$


CaMnO$_3$