We combine two complementary areas of analytical microscopy—electron microscopy and proximal-probe techniques—and use a variety of state-of-the-art imaging and analytical tools. We also design and build custom instrumentation and develop novel techniques that provide unique capabilities. In our work, we collaborate with you to solve materials- and device-related R&D problems. This sheet summarizes the uses and features of four major tools: transmission electron microscopy, scanning electron microscopy, the dual-beam focused-ion-beam workstation, and scanning probe microscopy.

**TRANSMISSION ELECTRON MICROSCOPY**

In transmission electron microscopy (TEM), a thin sample, less than 200 nm thick, is bombarded by a highly focused beam of single-energy electrons. The beam has enough energy for the electrons to be transmitted through the sample, and the transmitted electron signal is greatly magnified by a series of electromagnetic lenses. The magnified transmitted signal may be observed in several ways—by electron diffraction, diffraction-contrast imaging, or phase-contrast imaging. Transmission electron diffraction (TED) patterns help to determine the crystallographic structure of the material. Diffraction-contrast and high-resolution TEM images yield information about the chemistry and microstructure of the material and its defects. Phase-contrast imaging or high-resolution TEM imaging gives information about the microstructure of the material and its defects at an atomic resolution.

With scanning transmission electron microscopy (STEM), the electron beam is raster-scanned across the material, which produces a variety of electron and X-ray signals used for compositional and electronic analysis. The transmitted electrons at high scattering angle can be collected to form high-resolution, chemically sensitive Z-contrast images.

**Applications**

- **Crystallography.** Electron diffraction enables one to determine the crystallographic structure of materials on a fine scale.
- **Microstructure.** Diffraction-contrast and high-resolution TEM yields information on the composition, microstructure, and atomic structure of materials and defects. High-resolution STEM Z-contrast imaging provides directly interpretable, chemically sensitive images of the structure of materials, defects, and interfaces in samples with atomic resolution.
- **Composition.** Energy-dispersive X-ray spectroscopy (EDS) and electron energy-loss spectroscopy (EELS) provide quantitative and qualitative compositional analysis of materials to sub-nm spatial resolution for almost any element. STEM EDS and EELS enable elemental mapping to be performed with nm-scale resolution. EELS also provides information on the electronic properties of materials. A Gatan image filter (GIF) enables elemental mapping to be performed by energy-filtered TEM with high spatial resolution.
- **Cross-sectional analysis.** Investigates the structure, composition, and perfection of multilayer films and interfaces.

**Features**

- **Field-emission gun STEM.** Enables structural, compositional, and electronic cross-sectional analysis. Investigates the structure, composition, and perfection of multilayer films and interfaces.

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**MAJOR INSTRUMENTATION FOR TRANSMISSION ELECTRON MICROSCOPY**

<table>
<thead>
<tr>
<th>System</th>
<th>Analytical Technique</th>
<th>Typical Applications</th>
<th>Lateral Resolution</th>
<th>Special Features</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phillips CM30</td>
<td>Transmission electron microscopy</td>
<td>Structural and compositional analysis and lattice imaging</td>
<td>0.23 nm</td>
<td>High-resolution, EDS</td>
</tr>
<tr>
<td>FEI F20 (UT)</td>
<td>Field-emission scanning transmission electron microscopy</td>
<td>Structural, electronic, and compositional analysis; elemental mapping; lattice imaging</td>
<td>0.19 nm for HRTEM; 0.14 nm for Z-contrast STEM</td>
<td>High-resolution, Z-contrast, EDS, EELS, Energy filtering, Field-emission electron source</td>
</tr>
<tr>
<td>FEI NOVA 200</td>
<td>Field-emission scanning electron (SEM) and ion (SIM) microscopy</td>
<td>Preparation of TEM and SEM samples, Fabrication of nanostructures</td>
<td>1.1 nm @ 15 kV for SEM; 7 nm for SIM</td>
<td>THERMO EDS; FIB etch and deposition; High-resolution SEM</td>
</tr>
</tbody>
</table>
analysis with sub-nm spatial resolution.

- **STEM high-angle annular dark-field detector.** Enables high-resolution (~0.14 nm) STEM Z-contrast microscopy, providing directly interpretable, chemically sensitive atomic resolution images.

- **Gatan image filter (GIF).** Enables EELS spectroscopy and elemental mapping to be performed for a wide range of elements and energy-filtered electron diffraction and imaging.

- **Energy dispersive X-ray spectroscopy (EDS) systems.** Enables compositional analysis and elemental mapping with high spatial resolution.

- **Low-energy low-temperature ion milling system.** Reduces damage during sample preparation.

- **Dual-beam focused-ion-beam (FIB) workstation.** Enables site-specific preparation of TEM samples.

### SCANNING ELECTRON MICROSCOPY

In scanning electron microscopy (SEM), highly energetic (0.1–40 keV) electrons are focused and scanned over the surface being observed, causing multiple interactions: emission of secondary and backscattered electrons, photons, X-rays, excitation of phonons, and diffraction under specific conditions. Because the electron beam is scanned in the X-Y plane, an image for each of these different processes can be obtained when using a suitable detector. Detection of secondary electrons—a standard practice in SEM—provides the topography of the surface being observed with a resolution on the order of 1–2 nm and magnification from 10x to 500,000x. In addition, we can map information on composition, phase, and other multiple properties (e.g., electrical, optical, thermal) with excellent resolution by adding appropriate detectors. The SEM is perhaps the most versatile instrument in materials science because—beyond being a stand-alone instrument—it represents an integrated platform for a wide variety of characterization techniques.

#### Applications

- **Topography.** Secondary electrons, with energies from 0 to 50 eV, provide an image of the surface being observed with very high resolution and depth of field at very high scanning speeds. Best resolution is obtained with in-lens detectors in microscopes with field-emission sources.

- **Composition.** Backscattered electron emission is sensitive to composition and is used to form compositionally sensitive images. Moreover, using characteristic X-rays, maps of different elements can be obtained with micrometer resolution.

- **Electron-beam-induced current (EBIC).** In the presence of an electrostatic field (p-n junction), electron-hole (e-h) pairs excited by the electron beam can be collected, and the resulting current is amplified with a picocommunity and used to form an EBIC image. EBIC provides information about the location of electron-beam-induced current (EBIC).

#### MAJOR INSTRUMENTATION FOR SCANNING ELECTRON MICROSCOPY

<table>
<thead>
<tr>
<th>System</th>
<th>Analytical Technique</th>
<th>Typical Applications</th>
<th>Lateral Resolution</th>
<th>Special Features</th>
</tr>
</thead>
<tbody>
<tr>
<td>JEOL 6320F</td>
<td>Field-emission scanning electron microscopy</td>
<td>Micro- and nanoscale characterization of topography, composition and phases</td>
<td>1.2 nm @ 1.5 kV, 2.5 nm @ 1 kV</td>
<td>HKL NORDLYS II EBSD, THERMO EDS, EBIC</td>
</tr>
<tr>
<td>JEOL JSM-5800</td>
<td>Scanning electron microscopy</td>
<td>Microstructure, EBIC, cathodoluminescence</td>
<td>3.5 nm</td>
<td>Cathodoluminescence spectrum imaging, cryostage (15 to 300 K), CCD, InGaAs PDA</td>
</tr>
<tr>
<td>Customized SPM</td>
<td>STM, AFM, NSOM</td>
<td>Nanoscale characterization and manipulation of nanostructures</td>
<td>&lt; 1 nm</td>
<td>Scanning tunneling luminescence, electroluminescence, lateral transport measurements, NFCL</td>
</tr>
<tr>
<td>JEOL JXA-8900L</td>
<td>Electron probe microanalysis (EPMA)</td>
<td>Quantitative compositional analysis</td>
<td>100 nm to 5 mm</td>
<td>± 0.2 at.% detection limit</td>
</tr>
</tbody>
</table>

1.08 eV

Electron-beam-induced current image of a GaAsP-on-Si solar cell revealing threading dislocations crossing the p-n junction.
the junction in a device, shunts, electrical activity of grain boundaries, and diffusion length.

- **Cathodoluminescence (CL).** CL is the photon emission stimulated by the electron beam. CL can be used to investigate the distribution of recombination centers in semiconductors, including extended defects (dislocations, grain boundaries), stress fields, and compositional fluctuations. At NREL, we have combined spectroscopy and imaging into a single mode: spectrum imaging. Basically, spectrum imaging acquires a spectrum for each pixel on the image at high speed (typically 10 ms/pixel). This is achieved by synchronizing the scanning of the electron beam with the spectrum acquisition. In our system, CL observations can be performed at temperatures between 15 and 300 K.

- **Electron backscattered diffraction (EBSD).** EBSD indexes diffraction patterns for each pixel on the image, providing maps of the orientation of crystalline phases, misorientation between grains, texture, grain distribution, deformation, and strain.

- **Scanning-probe microscopy (SPM) platform.** We have developed a very compact SPM integrated with SEM. Combining these two platforms, the following operation modes are available:
  - **Scanning tunneling luminescence**—Based on scanning tunneling microscopy (STM), the photon emission is stimulated by tunneling electrons and can be applied to nanostructures.
  - **Lateral transport**—Based on a combination of STM and SEM, e-h pairs excited by the electron beam at specific locations are detected through the STM tip and the lateral electron diffusion can then be measured.
  - **Electroluminescence mapping**—Based on conductive atomic force microscopy (C-AFM), individually injected current pulses during intermittent contact are used to stimulate electroluminescence when the p-n junction is under forward bias.
  - **Near-field cathodoluminescence (NFCL)**—Based on near-field scanning optical microscopy (NSOM), this SPM is designed for surface-sensitive cathodoluminescence. The luminescence is excited by the electron beam and detected through the optical fiber positioned in the near field.

**DUAL-BEAM FOCUSED-ION-BEAM (FIB) WORKSTATION**

This dual-beam instrument consists of a focused-ion-beam (FIB) column and a scanning electron microscope (SEM) column on the same platform. Uses include ion milling, metal deposition, ion imaging, and electron imaging on the micrometer and nanometer scale of advanced photovoltaic materials and devices. Supporting other analytical tools such as TEM, the dual-beam FIB performs precise, site-specific sample preparation for both TEM and SEM. Acquiring chemical spectra and elemental maps is another feature of this system with energy-dispersive spectroscopy (EDS). 3D chemical reconstructions can be obtained by combining controlled ion milling with chemical mapping. The FIB is equipped with a gas injection system (GIS) Pt metal deposition capability that can be used with either ion-beam-assisted or electron-beam-assisted chemical vapor deposition. Using a digital patterning generator also allows for complete FIB milling or deposition of complex structures with software-supplied parameters and/or by direct input of bitmap files.
A group of scanning probe microscopy (SPM) techniques uses very sharp tips that scan extremely close (several nm) to or in contact with the material being analyzed. The signal used to map and analyze the surface may be generated in a number of ways: attractive/repulsive forces, electric current, or even friction force. The techniques we use include scanning tunneling microscopy (STM) and atomic force microscopy (AFM), with variants such as scanning Kelvin probe microscopy (SKPM), conductive AFM (C-AFM), scanning capacitance microscopy (SCM), and scanning tunneling spectroscopy (STS).

**Features**

- Spatial resolution to atomic scale.
- Digital data acquisition, allowing real 3D imaging and measurements such as roughness and surface linescan.

**Applications**

- **Surface imaging.** Scans the surface of materials to produce topographic images with up-to-atomic resolution.
- **Electrical and electronic properties.** Can obtain these properties of samples with excellent spatial resolution. Analysis of properties that include surface potential (SKPM), type and concentration of charge carriers (SCM), electric conductivity (C-AFM), and local density of states (STS).
- **Various materials.** Can be used to analyze most types of materials, from conductors to insulators. Can provide information on the properties of semiconductor devices—e.g., potential profile along p-n junctions by analyzing their cross sections.

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**MAJOR INSTRUMENTATION FOR SCANNING PROBE MICROSCOPY**

<table>
<thead>
<tr>
<th>System</th>
<th>Analytical Technique</th>
<th>Typical Applications</th>
<th>Lateral Resolution (Å)</th>
<th>Vertical Resolution (Å)</th>
<th>Vacuum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Veeco AutoProbe CP</td>
<td>AFM, STM, SKPM, SCM</td>
<td>Nanoscale characterization of sample morphology and electrical properties</td>
<td>&lt; 1</td>
<td>&lt; 0.1</td>
<td>No</td>
</tr>
<tr>
<td>Veeco Dimension 3100 – Nanoscope IIIa</td>
<td>AFM, STM, SKPM, SCM, C-AFM</td>
<td>Nanoscale characterization of sample morphology and electrical properties</td>
<td>&lt; 1</td>
<td>&lt; 0.1</td>
<td>No</td>
</tr>
<tr>
<td>Veeco Dimension 3100 – Nanoscope V</td>
<td>AFM, STM, SKPM, SCM, C-AFM</td>
<td>Nanoscale characterization of sample morphology and electrical properties, including close-loop tip positioning</td>
<td>&lt; 1</td>
<td>&lt; 0.1</td>
<td>No</td>
</tr>
<tr>
<td>Omicron VT-STM/AFM</td>
<td>AFM, STM, SKPM, C-AFM</td>
<td>Nanoscale and atomic characterization of sample morphology and electrical/electronic properties</td>
<td>&lt; 1</td>
<td>&lt; 0.1</td>
<td>10⁻¹¹ torr range</td>
</tr>
</tbody>
</table>

Electrical potential image of a III-V multijunction cell from scanning Kelvin probe microscopy.

- Nondestructive.
- Can be performed in air or in ultra-high vacuum.
- Field of view is from atoms to about 100 μm (vertical limit of 7 μm).
- AFM uses the force between the tip and sample surface; STM uses the tunneling current between the tip and sample surface.