Feature: **Microscopy**

THE AUTHOR

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Seeing the invisible

New generation high-resolution electron microscopes provide information at the atomic scale allowing researchers to see materials in an unprecedented detail

The latest generation of high-resolution (scanning) transmission electron microscopes (HR S/TEMs) allows material scientists to cross a critical threshold in their efforts to understand the properties of materials down to the most fundamental level - the atomic level. The current nanotechnology revolution is driven primarily by the realisation that atomic and molecular-scale interactions determine the large-scale behaviour of all materials. Ready access to imaging and analytical techniques with the atomic-scale (sub-Angstrom) spatial resolution crosses a critical threshold in the nanotechnology roadmap. New instruments, such as FEI's Titan, can now provide clear, unambiguous images of individual atoms. Though HR S/TEM long ago demonstrated atomic resolution, it was available only in a very limited range of samples and conditions, and then only with great operational and analytical difficulty. All of that has changed. Scientists can now focus on what they see, rather than how they think they may have seen it. And the results are, in many cases, no less than astonishing.

The new generation of HR S/TEMs addresses a range of important operational and analytical issues that have limited the technique's acceptance for routine imaging and analysis applications. The new systems integrate highly stable electronics and mechanics with extensive setup and control automation to eliminate much of the operational difficulty of previous generations. Aberration correctors eliminate image blurring and delocalisation to allow direct interpretation of high-resolution images. Monochromators reduce the energy spread of the incident electron beam to permit more detailed spectral analysis. Improved performance over the full range of accelerating voltages allows beam energy to be freely selected to optimise performance for a particular material or analysis mode.

Though atomic scale HR S/TEM resolution is not new, the ability to achieve sub-Ångström-resolution and observe it directly in a TEM image is. Historically, the difficulty of realising a TEM's ultimate resolution led scientists and engineers to develop two separate definitions of resolution. Image or point resolution was that which could be interpreted directly from an image and was typically two or three Angstroms. The information limit was the ultimate resolution of the instrument, but usable and interpretable information could be achieved at the information limit only with great effort. The availability of aberration correctors and significant improvements in stability and control systems now makes routine achievement of directly interpretable image resolution down to the information limit a practical reality. What you see is indeed what you get.

Spherical aberrations have long interfered with TEM's ability to clearly image material interfaces with atomic detail. In effect, the spherical aberration spreads the information from a single point in the object over an extended region surrounding the corresponding point in the image. This delocalisation is particularly troubling at an interface since the structure of each material extends across the boundary creating confusion and sometimes generating artifacts that are easy to misinterpret as real structure.

Many catalytic processes involve atomic scale interactions at an interface. Often the catalyst is present as a small particle in a liquid or gas environment that includes the chemical reactants. The catalytic activity involves the actions of atoms and molecules at the interface, the particle surface, between the particle and its environment. In an uncorrected TEM, delocalisation makes it very difficult to observe differences among surface atoms that may be critical to understanding the catalytic mechanism. Aberration correctors now available on advanced HR S/TEMs can provide clear, unambiguous images of subtle differences in atomic structure at the interface (Figure 1).

Compositional analysis in a TEM is usually performed in scanning mode (STEM) in which the beam is focused to a small spot that is scanned over the thin sample, allowing point-by-point collection of various analytical signals. The two most important are characteristic X-rays, which carry elemental information, and energy loss in the transmitted electrons, which carries a multitude of important spectral information. The spatial resolution of the analytical information is to a certain degree determined by the size of the spot and the extent to which the beam spreads as it passes through the sample. Just as spherical aberration spreads information from a point in the object over a larger spot in the image, it imposes a lower limit on the spot size in STEM mode.

Electron energy loss spectroscopy (EELS) can derive the identity, chemical and electronic state of sample atoms by measuring the amount of energy lost in interactions with beam electrons. A monochromator at the beam source reduces the energy spread in the beam improving the resolution of the energy loss spectrum, revealing valuable details like fine structure, bonding, valence state and electronics properties with the highest spatial resolution.

The resolution of an electron optical system generally improves with higher beam energies. However, higher energy electrons interact less strongly with the sample, resulting in weaker contrast in the image. In various cases, higher beam energies also tend to cause more damage to the sample, limiting the time it can be exposed to the beam. The improved electron optical capabilities of advanced HR S/TEMs now permit high resolution imaging at lower beam energies with improved contrast and sample stability.

Many of the most interesting new materials are composed of lighter elements that are difficult to image at high accelerating voltage. Carbon nanotubes, for instance, have extraordinary mechanical and electrical properties. The image in Figure 2 was acquired at 80kV and the atomic structure of the nanotube is apparent. The ability to achieve high resolution at lower kV holds great promise for the application of HR S/TEM in soft matter as well as biological sciences.

The availability of directly interpretable atomic level image resolution promises to revolutionise material science. In a practical sense, the ease of operation and interpretation provides access to new information and new results directly to scientists in their chosen fields, allowing them to spend their time and effort applying results rather than obtaining them - seeing things they've never seen before. LN

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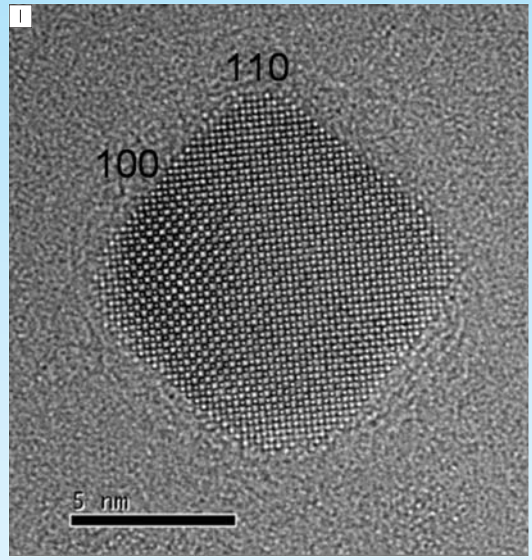

Image Courtesy: Dr. Bert Freitag, FEI Company.

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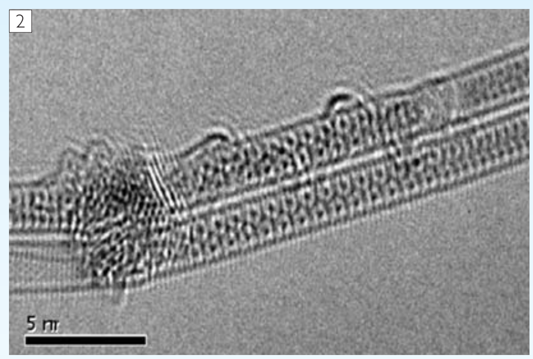

Image Courtesy: Dr. Bert Freitag, FEI Company.

Figure 1: Catalyst Research - High-resolution image of CeO_2 catalyst nanoparticles used in Solid Oxide Fuel Cells (SOFC). With Titan 80-300 Image Corrected, these particles and their crystal surface were visualised clearly. (Acknowledgement: Dr. Jing Zhang, Dr. Satoshi Ohara and Prof. Tadafumi Adschiri from Tohoku University and Prof. Kenji Kaneko from Kyushu University, Japan.)

Figure 2: Carbon Nanotube Research - High-resolution image of Carbon Nanotubes filled with Copper Iodine. (Courtesy and Acknowledgement: Prof. N. Kiselev, Institute of Crystallography, Moscow, Russia.)

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