

## Bessel/Butterworth Filter

cyber clones oscillate spontaneously and with considerable stochastic fluctuation. As figure 2 shows, turning on the coupling brings the spontaneous oscillations into phase and mitigates the stochastic fluctuations.

In principle, coupling can account for the cochlear amplifier in mammals, whose ears are more complex than those of the bullfrog and other lower vertebrates. Mammals have two kinds of hair cell, inner and outer. The outer cells are elastically coupled to each other but not to the inner cells, which convey the auditory signal to the brain. It's likely that the outer cells deliver their amplification signal to the inner cells through the intervening fluid via viscous coupling.

But something else happens in the outer hair cells. Embedded in the membrane of each cell at a density of around 4500 molecules/ $\mu\text{m}^2$  is a rod-shaped protein called prestin. Depending on

the voltage across the membrane, the prestin molecules either lie parallel to the membrane surface or bunch together perpendicular to it.

That piezoelectric bunching shortens the cell and provides a second mechanism for converting mechanical energy into electrical energy. At around 1 ms, the membrane's discharge or RC time is too long for the voltage-dependent bunching to play a role in amplifying high-frequency hearing, but it could supplement elastic coupling at lower frequencies.

Charles Day

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## Focus on improving transmission electron microscopes starts to pay off

The latest advance is the chemical identification of closely spaced, lightweight atoms.

Since their invention in the 1930s, transmission electron microscopes (TEMs) have been an invaluable tool, providing highly magnified images of objects that range from biological specimens to electronic materials. Now, researchers are seeking greater performance from those instruments, especially the capability to determine three-dimensional structures with atomic resolution, to chemically identify individual atoms, or to follow the dynamic behavior of atoms within a sample. Of particular interest are materials with low atomic number  $Z$ . Some researchers are keen to explore materials such as carbon nanotubes, graphene, and boron nitride for novel electronic applications. Others are interested in determining the structures of biological molecules, especially ones that aren't amenable to being crystallized and hence aren't candidates for crystallography.

A worldwide 60-year effort to correct spherical and chromatic aberrations has brought about, in the past decade, TEMs with much better resolution.<sup>1,2</sup> Spherical aberration results when electrons traveling at different distances from the axis of an electron beam are focused differently by magnetic-field "lenses." Chro-

matic aberration stems from the different focusing of electrons of different energies. Uncorrected TEMs have operated for many years with electron energies of 200 keV and above because the effect of aberrations grows worse for lower-energy electrons.

TEM designers can compensate for spherical aberration by adding magnetic multipoles along the electron-beam path. It sounds simple in principle but is difficult in practice because the correction for one aberration can unleash a horde of parasitic aberrations. Chromatic aberration can be mitigated by reducing the energy spread of the electron source, typically to a few tenths of an eV. But reduction in energy width can decrease the beam intensity and hence degrade the image quality. Another way is to build a corrector that focuses electrons to the same point regardless of energy.

With aberration correction, TEM resolution has been improved by a factor of two to three. The improvement has been greatest at the electron energies below about 80 keV that are required to avoid radiation damage to materials containing low- $Z$  atoms. The knock-on damage threshold is set by the energy an electron

must have to kick an atom out of its position within a sample; its value is strongly dependent on atomic mass and bonding. Now, even below 80 keV, the new generation of TEMs can resolve interatomic spacings of less than 0.1 nm.

A recent experiment has exploited the high resolution now possible at low energies to identify individual light atoms in a single atomic layer.<sup>3</sup> The showpiece of the work is the experimental image seen in figure 1a, which shows impurity atoms of carbon and oxygen randomly positioned in the regular array of atoms, replacing boron and nitrogen atoms in a BN monolayer. The work, by Ondrej Krivanek and coworkers from the Nion Company in Kirkland, Washington, along with researchers from Oak Ridge National Laboratory, the University of Oxford, and Vanderbilt University, was done on a TEM at Oak Ridge that operated in a scanning mode.

## Two types of TEM

In a conventional TEM, a broad electron beam is diffracted by the atoms in a sample. Experimenters record the resulting interference pattern of the transmitted and diffracted electron waves and deduce the atomic structure from the phase contrast in the recorded image. The scanning TEM shown in figure 2 is an alternative design, in which a tightly focused electron beam is scanned across the sample, one small spot at a time. As shown in the figure, an STEM can be equipped with one or more detectors to collect the electrons transmitted from each spot: An

annular dark-field (ADF) detector and an electron-energy-loss spectrometer (EELS).

Each type of TEM has its advantages. A conventional TEM images a wide area of the sample in a shorter time and hence lends itself better to studying dynamic changes. An STEM equipped with either an ADF detector or EELS can provide direct information about individual atoms.

A number of companies and research centers have focused on advanced TEM development. One is Nion, founded by Krivanek and Niklas Dellby in 1997. The US Department of Energy has spearheaded an effort called TEAM to develop ultrahigh-resolution TEMs. TEAM is led by Lawrence Berkeley National Laboratory's National Center for Electron Microscopy and includes FEI Co of Oregon and the Netherlands as well as Corrected Electron Optical Systems GmbH of Germany. In Germany, a TEM development program called SALVE is headed by Ulm University, with the participation of CEOS and Carl Zeiss GmbH. Others involved in TEM development are JEOL Ltd and Hitachi Ltd in Tokyo.

## Which atom is which?

To test the ability of the ultrahigh-resolution TEMs to discriminate between atoms, researchers have turned to imaging BN monolayers, structural cousins to graphene sheets. They're a good choice for such a test because they contain two elements in a well-defined chemical structure. In the past year,

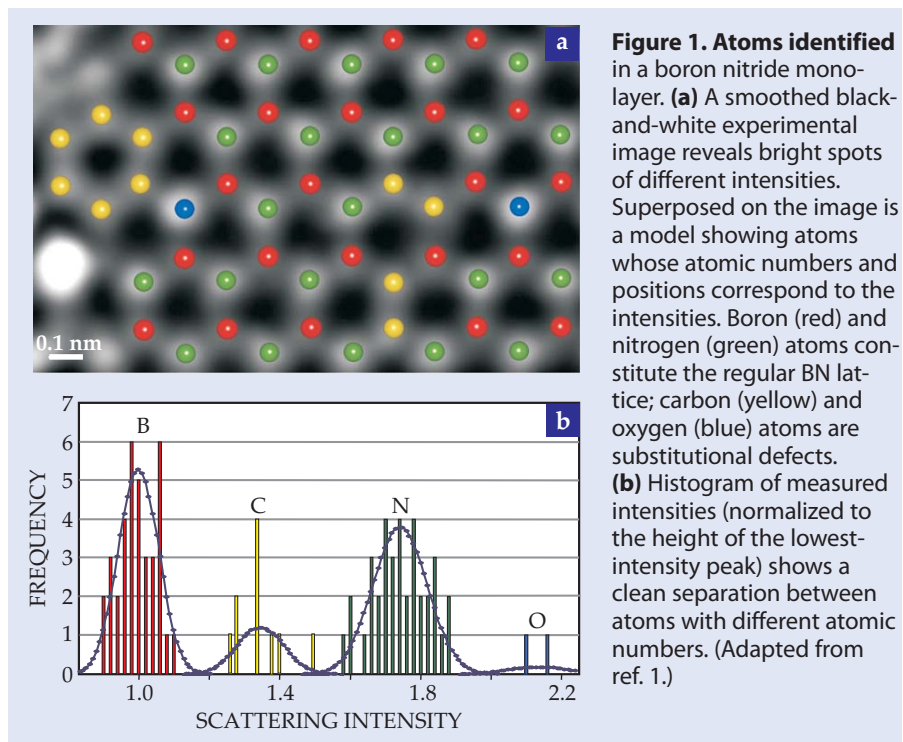
three groups have produced atomic-resolution TEM images of thin BN layers and studied those structures.<sup>4-6</sup> The observed difference in phase contrast between B and N atoms was very small. The only way to discriminate between the two atom types was to combine multiple images and impose prior knowledge of the hexagonal BN lattice.<sup>5-6</sup>

To provide a more direct identification of the B and N atoms, Krivanek and his group used an STEM with 0.1 nm resolution at 60 keV. The instrument was equipped with an annular dark-field detector, which was first developed in the 1970s by Albert Crewe and his colleagues. The ADF detector is a scintillation counter that surrounds the scattered beam and, in the instrument used by Krivanek's team, collects electrons scattered at half angles ranging from 58 to 200 mrad. The only electrons likely to be scattered to such angles are those undergoing Rutherford scattering with the partially screened atomic nuclei. The scattering intensity increases with  $Z^\alpha$ , where  $\alpha$  is around 1.7.

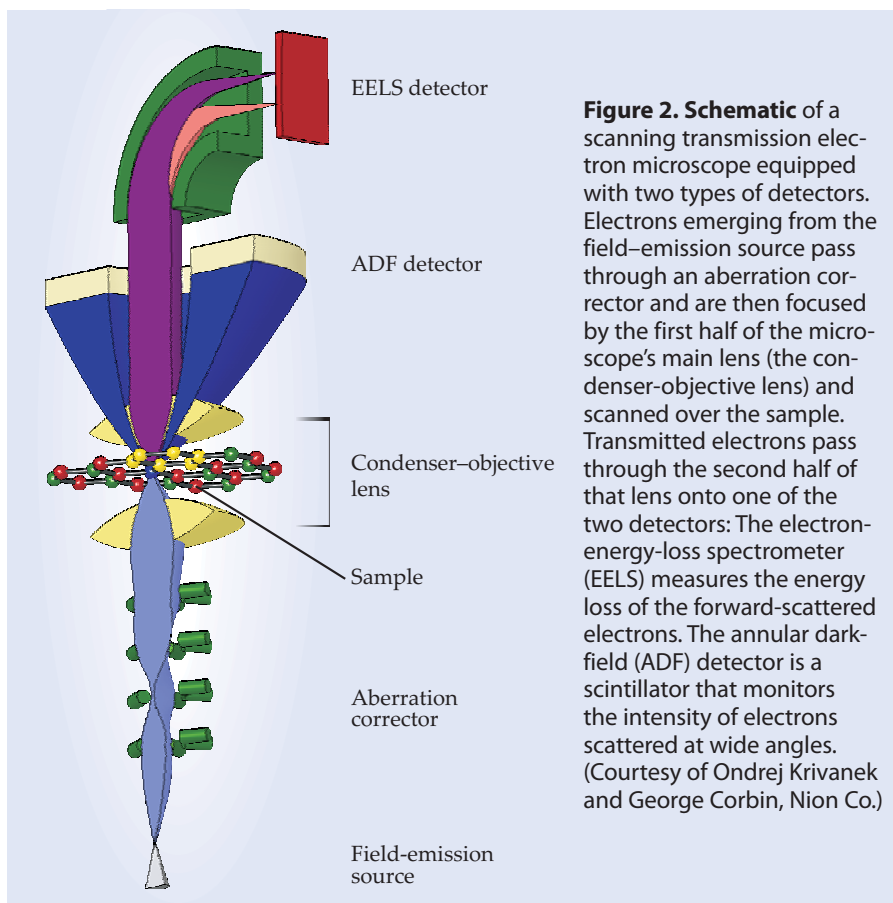
Researchers have used STEMs with ADF detectors in the past to identify heavy elements such as uranium and gold. They have, for example, identified high-Z dopants at grain boundaries and in semiconductors. It was not clear, however, whether the signal from lighter atoms would be strong enough to distinguish them from one another. Krivanek's team showed that it can be.

In a plot of the raw data, the hexagonal pattern of B and N atoms is discernible, but researchers can't directly interpret the deviations from the pattern to find out which impurity atom sits where. That's because the tail of the electron probe centered on one atomic site adds to the scattering from the nearest neighbor sites. Krivanek and his colleagues removed the probe-tail contributions computationally and smoothed out the statistical noise to generate the black-and-white portion of the image in figure 1a. Superposed on it are colored atoms from a density-functional-theory simulation of a BN monolayer with C and O impurities. The C atoms substitute in pairs for B-N pairs and not for individual B or N atoms, whereas single O atoms substitute only for N atoms. The bright spot on the left edge is a higher-Z impurity.

The histogram in figure 1b plots the number of pixels where the intensity, after subtraction of the tails, had a particular value. It reveals four clearly separated peaks. Contributions to the first and third peaks come from the respective positions that B and N atoms occupy







**Figure 2. Schematic** of a scanning transmission electron microscope equipped with two types of detectors. Electrons emerging from the field-emission source pass through an aberration corrector and are then focused by the first half of the microscope's main lens (the condenser-objective lens) and scanned over the sample. Transmitted electrons pass through the second half of that lens onto one of the two detectors: The electron-energy-loss spectrometer (EELS) measures the energy loss of the forward-scattered electrons. The annular dark-field (ADF) detector is a scintillator that monitors the intensity of electrons scattered at wide angles. (Courtesy of Ondrej Krivanek and George Corbin, Nion Co.)

in the BN lattice. The other two peaks appear to come from C and O impurities. Indeed, when experimenters assume that the average intensity of each of the four peaks corresponds, in increasing value, to atomic numbers of  $Z = 5, 6, 7,$  and  $8,$  respectively, a curve of intensity versus  $Z$  is consistent with the theoretically expected power-law dependence.

Krivanek identified two key components that produced those results: One was the high resolution that resulted from introducing a spherical aberration corrector as well as using a cold field-emission gun with a high electron current and a small energy spread (0.3 eV). The second was the extreme stability of the microscope. The key parts of the 2.7-meter-tall instrument do not move more than 0.01 nm with respect to each other. Krivanek and his team also had very stable computer-controlled power supplies for the many optical elements that can deflect the electron beam. They put as much effort into achieving that level of mechanical stability as they did into the aberration correction.

The presence of the C and O impurity atoms was serendipitous, Krivanek says. He and his coworkers set out to demonstrate element identification on a BN monolayer. Even though they operated well below the knock-on damage threshold, some damage mechanism

caused holes to open in the BN lattice in one of their many samples. Those holes were quickly filled by C and O impurity atoms, whose presence enhanced the experimental demonstration of element identification.

"This work is remarkable for the image resolution it achieves at 60 keV," comments Jian-Min Zuo of the University of Illinois, Urbana-Champaign. Although other groups have obtained similar resolution at slightly higher voltages, the advantage in identifying atoms came from combining an STEM with an ADF detector.

### The next steps

Kazu Suenaga of the National Institute of Advanced Industrial Science and Technology in Tsukuba, Japan, whose group performed one of last year's TEM studies of BN monolayers, agrees that the work of Krivanek's team represents great progress. Still, he adds, neither ADF nor TEM imaging is really element selective. To discriminate between atoms unambiguously, he feels, one should use spectroscopic techniques such as EELS.

Some of the electrons monitored by EELS will lose energy during inelastic collisions that excite an electron from the inner shell of an atom to an unoccupied state. The energy difference



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provides a distinct signature of each particular element. Both Suenaga's group and Krivanek's team have reported using EELS to study nanotubes that contain nanopods filled with single atoms such as erbium.<sup>7,8</sup>

Electromechanical probes such as scanning tunneling microscopes (STMs) and atomic force microscopes (AFMs)

are alternative instruments for imaging molecules on a surface, but unlike TEMs, they cannot say anything about deeper layers. Alex Zettl of the University of California at Berkeley is interested in the exotic combination of manipulators like STMs and AFMs and detectors like TEMs. When embedded in a TEM, an STM or AFM can manipulate objects on

a surface as the TEM dynamically images the resulting changes. Such manipulators have been embedded in the older generation of TEMs.<sup>9</sup> Zettl is among those working to incorporate them in the new generation of TEMs. The bottom line, says Zettl, is that "technology advances are opening many new doors."

**Barbara Goss Levi**

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