US Tech – January 2025

PRODUCTION

Supporting Sub-Angstrom Materials Research at Oak Ridge National Laboratory

By Jim McMahon

Advancements in nanofabrication, pharmaceuticals, energy and aerospace follow breakthroughs in the understanding of materials. These breakthroughs unlock unique functionalities that create new pathways to design future devices.

The Center for Nanophase Materials Sciences (CNMS) at Oak Ridge National Laboratory (ORNL) is at the forefront of one of the most powerful capabilities for exploring the nature of materials and energy. CNMS emphasizes discovery of new materials, and the understanding of underlying physical and chemical interactions that enable the creation of nanomaterials. CNMS researchers have access to state-of-the-art microscopy instruments in its Advanced Microscopy Laboratory (AML) for a broad range of nanoscience research, including nanomaterials synthesis, nanofabrication, imaging/microscopy/ characterization, and theory/modeling/simulation.

Amongst the instruments used for materials research in the ALM are some of the most advanced Transmission Electron Microscopes (TEM) and Scanning Transmission Electron Microscopes (STEM).

TEMs utilize a technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen.

The image is magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film, or to be detected by a sensor such as a CCD camera. TEMs use phase-contrast, and therefore, produce results which need interpretation by simulation.

Scanning Transmission Electron Microscopy

A type of TEM which has become highly appealing, the STEM, also permits the electrons to pass through an ultra-thin specimen, however, the STEM focuses the electron beam into a narrow spot which is scanned over the sample in a raster. The rastering of the beam across the sample makes the STEM suitable for analysis techniques, such as mapping, where the signals can be obtained simultaneously, allowing direct correlation of image and quantitative data.

By using a STEM equipped with a high-angle detector, it is possible to form atomic resolution images where the contrast is directly related to the atomic number (Z-contrast image). The directly interpretable Z- contrast image makes STEM imaging with a high-angle detector preferable to TEM in some applications. For example, oxygen can now be visualized in superconductors and colossal magnetoresistant manganites where it plays a dominant role in determining properties.

Electron energy loss spectroscopy (EELS) is a STEM measurement technique made possible with the adaptation of an electron spectrometer. With the addition of EELS, elemental identification is possible, as well as additional capabilities of determining electronic structure or chemical bonding of atomic columns. With EELS, the STEM does not just produce an image, it can also do chemical mapping. Instead of just detecting what scatters from the atom, it can show changes in chemical balance and see an incredible amount of detail about the physics interface when two different materials come together.

“We are using the STEM for different applications,” said Dr. Andrew R. Lupini, R&D staff member in the Center for Nanophase Materials Sciences at Oak Ridge National Laboratory in Tennessee. “As well as the imaging, this STEM can do very high-energy resolution spectroscopy. For example, we can probe proper- ties of materials relating to their bonding and measure their temperature on a very local scale.”

With correction of spherical aberration, the STEM can resolve more than just seeing the atoms more clearly. The high-energy convergent electron beam in STEM provides local information of the sample, even down to atomic dimensions of smaller than one ten-billionth of a meter.

Sensitivity to Vibration

Scanning Transmission Electron Microscopes are more sensitive to ambient conditions, with the addition of field emission guns, imaging filters, and spherical aberration correctors that give higher spatial and energy resolution, and EELS.

The ultimate performance of these extremely sensitive microscopes is strongly influenced by factors such as magnetic fields, barometric pressure changes, room- and chilled-water temperature variations, grounding problems, and quite significantly by floor and acoustic vibrations.
Atomic resolution spectroscopy is particularly sensitive to environmental instabilities as a result of its long acquisition times. The serial nature of the image acquisition in STEM makes the instabilities appear as image distortions. STEMs are most sensitive to low frequency vibration, in the range of a few Hertz. These vibrations are challenging to eliminate from the micro- scope’s environment.

Vibration Isolation

Because of its high vibration isolation efficiencies, particularly in the low hertz frequencies, Negative-Stiffness vibration isolation was selected by ORNL’s Center for Nanophase Materials Sciences for sub- angstrom materials research with its Nion Hermes STEM-EELS. Introduced in the mid-1990s by Minus K Technology, Negative-Stiffness vibration isolation has been widely accepted for vibration-critical applications, largely because of its ability to effectively isolate lower frequencies, both vertically and horizontally.

Negative-Stiffness isolators are unique in that they operate purely in a passive mechanical mode. They do not require electricity or compressed air. There are no motors, pumps or chambers, and no maintenance because there is nothing to wear out.

Selecting the most optimum vibration isolation system is critical when dealing in sub-angstrom measurements. For this application, four Negative-Stiffness isolators were employed, one for each corner supporting the platform upon which rests the Nion Hermes STEM-EELS.

Negative-Stiffness vibration isolation has become a preferred system to analyze atoms in individual nanostructures, permitting the atomic structure of materials to be seen more clearly at sub-atomic levels.

For more information, please contact Minus K Technology, Inc.; 460 Hindry Ave., Unit C, Inglewood, CA 90301; Phone 310-348-9656 or E-mail: sales@minusk.com Web: www.minusk.com.