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Research at Room Temperature

A raft of new technologies has kept Hitachi High Technologies America cold field-emission transmission electron microscopes competitive among top-end, high-voltage instruments.

Among electron microscope manufacturers, Hitachi High Technologies America has paved its own way. Since pioneering the commercial use of field-emission electron microscopes in 1978, the company has made continual improvements to this high-end, high-resolution form of imaging. To keep up in a competitive electron microscope market, the company has overcome inherent limitations in “cold” field-emission technologies, and has augmented their microscopes with a number of unique supporting systems.

Through persistent development, Hitachi High Technologies America, Schaumburg, Ill., has achieved resolution similar to scanning/transmission electron microscopes (S/TEMs) from other vendors.

In the past couple of years this has progressed to the sub-Ångstrom level—true atomic-scale imaging. It has also carved niches for its high-voltage electron microscopes—many of which use cold field-emission electron beams—in the study of nanomaterials, from carbon nanotubes to the newest gated oxides.

Officially introduced earlier this year, the HF-3300 TEM/STEM is the newest iteration of Hitachi’s cold field-emission electron beam technology. Unlike microscopes equipped with thermal guns, the electron emitter of the HF-3300 operates at room temperature (using a “cold” emitter). The selling points are high spatial resolution—achieved with a high-energy 300 kV gun—and high coherence through the use of the cold field-emission. In addition, the HF-3300 avoids the use of a monochromator, which allows full beam current and high energy resolution.

“For any microscope, resolution is always the highest priority, so we are emphasizing that—spatial resolution and energy resolution,” says Xiaofeng Zhang, a senior product development manager at Hitachi High Technologies America.

The S/TEM market, he says, is gravitating toward aberration-correction microscopes, which use one or two aberration correctors and a monochromator to overcome the limitations posed by the aberration effects of the electromagnetic lens when a narrow electron beam passes by. This type of microscope is very complicated, says Zhang, and challenging to use.

“People usually don’t realize you need to invest a lot into room requirements which are extremely strict for these aberration-corrected electron microscopes,” says Zhang. Because the HF-3300 architecture dispenses with aberration-correction, its environmental requirements are substantially relaxed. A single Ångstrom or better spatial resolution can be achieved without substantial investment in building or refurbishing a laboratory.

The other key that Zhang believes will improve the HF-3300’s profile in the marketplace is energy resolution. Currently, he says, users want energy resolution better than 0.5 eV, a level not achievable when using thermally-assisted electron guns. A monochromator is one solution. It disperses an electron beam according to energies of electrons contained in the beam, from low energy to high energy. Then an aperture or slit is used to permit the transmission of only a small part of the electron beam. Because electrons in this very narrow beam all have similar energy, the energy difference among electrons is maintained in a narrow area.

“The problem with this technology is that you keep only one small part of the energy beam, so the beam is very weak,” he says, typically only 20% or less of the original beam.

In contrast, a cold field emitter maintains energy resolution with full beam current. According to Zhang, a thermal field emission gun (or so-called Schottky gun) TEM with a monochromator can achieve a 0.2 eV energy resolution, which is slightly better than the HF-3300 can offer.

“But remember you can only use less than 20% of the beam (with the monochromator on), but HF-3300 uses the full beam,” he says.

Still, there are reasons other manufacturers haven’t adopted cold field emitters. One has to do with convenience.

A commitment to cold field

The HF-3300 TEM/STEM, a high-end electron microscope featuring high spatial and energy resolutions, represents Hitachi's commitment to cold field emission S/TEM technology for nanomaterials studies and industry applications. Image: Hitachi High Technologies America.

Zhang's experience with electron microscopes spans 20 years, but he has been with Hitachi High Technologies America for just two years. Before he joined the company, he was aware that cold field-emission microscopes had fallen out of favor in the marketplace, primarily because of gun contamination.

"If you talk to the older generation customers they will tell you it's troublesome to use cold field emission because of the tip noise. After a while, you will need to flash the (gun) tip, because molecules accumulate on your electron emitter," he says.

Molecular buildup destabilizes the electron emission, causing beam current fluctuation and thereby compromising the analytical performance of the microscope. To get rid of the problem, users must "flash heat" the gun to burn off the accumulation. For a thermal field emission gun, this isn't necessary because the emitter already operates at a high temperature. An instrument like the HF-3300, on the other hand, operates at room temperature.

"People will tell you how troublesome it was because people have had different experiences from the old generation cold field emission instruments. "I would not choose this type of technology if it had this problem," says Zhang.

Refinements have largely eliminated this problem. Instead of experiencing a loss of beam stability every few hours, the typical flash cycle has been extended to 8 to 10 hours. This interval between flashes allows for a full day of study, which can be accomplished in just a minute.

A trio of supporting technologies

Because cold field electron emission guns are little used by other S/TEM manufacturers, Hitachi High Technologies America has pioneered a number of technologies that are more or less unique to its new cold field-emission high-voltage TEMs.

The first, and according to Zhang probably the most important innovation, is electron holography. This is common to TEMs, but innovation in the HF-3300 lies in using more than one biprism to control the balance between resolution and field of view. This gives rise to several benefits for users.

When employing two biprisms, artifacts such as Fresnel fringes and the vignetting effect are eliminated. In addition, because the biprisms are adjustable independently, the field of view and interference fringe spacing can now be controlled independently. The result is that the area of study can be widened without compromising the resolution level. In contrast, expanding the interference range with a single biprism would hurt resolution. This extends the holography flexibility and helps speed the rate of study, which in turn helps the prototyping process for certain industries such as semiconductors.

Another important feature of the HF-3300 impacts the applications in crystallographic study which relies on electron diffraction.

“Traditionally, if you want to obtain an electron diffraction pattern with sharp diffraction spots, you want to get a parallel beam. You can measure the locations of diffraction spots to calculate lattice spacing and dimension of the unit cell with high accuracy,” says Zhang.

However, if diffraction is desired for only a limited, or nanoscale, area, the beam must be focused. The focusing usually results in beam convergence, which means the diffraction spots are no longer sharp. Instead, they present as disks. Subsequent spot location measurement is not accurate because of the difficulty of finding the center of the disks. Determining structural parameters such as lattice spacing becomes harder. Parallel nanobeam electron diffraction technology in the HF-3300 allows users to form a beam to the nanoscale, but still maintains parallel paths of the electron. Therefore sharp diffraction spots can be obtained even if the beam size is in a nanoscale range. This yields precise information about crystallographic structure, orientation, and lattice dimension in nanoscale areas.

“Industry is extremely interested in this function as well. If you come to a junction area” when studying a semiconductor transistor, says Zhang, “there is lattice stress and industry wants to know the stress distribution. You have to use electron microscopy with this parallel beam nanodiffraction technology to get accurate stress mapping.”

The precision level for stress measurement when using the HF-3300, says Zhang, is within the desired level for developers of technologies such as next-generation silicon-based devices and self-assembled nanomaterials.

Spatially-resolved electron energy loss spectroscopy (EELS) is also unique to Hitachi High Technologies America. When using EELS, a beam of electron of a known energy is focused onto the sample. By measuring the loss in energy after electrons are transmitted through the sample, the valence nature and quantity of chemical elements in the sample can be inferred. S/TEM users typically use straightforward EELS when profiling a sample along a line. When doing this, the scanning mode is typically used. The electron beam halts at each dwell stop to collects EELS spectra, and to characterize the chemical composition.

“The problem for this method is sample drift, which may cause you trouble,” says Zhang, who is referring to the tendency for some materials to be adversely affected by the illumination beam, which is instrumental for mechanical and environmental stability and sample sensitivity to the beam. Spatially-resolved EELS minimizes this problem by collecting the EELS spectra in one snapshot, without resorting to the scanning mode. “You still need an EELS spectrometer, but we managed to get a group of EELS spectra simultaneously along a line,” says Zhang.

Finally, potential customers of the HF-3300 may be attracted to the ability of this microscope to perform electron tomography without the limitations in tilting angle endemic to other TEMs. A 0 to 360-degree range is possible.

“You can get an idea of the 3-D profile without having to rely on the computational 3-D reconstruction software,” says Zhang. Structural reconstruction software compensates for a limited dataset—the result of a limited tilting angle—by calculating the missing portions. Invariably, however, artifacts are still generated. The ability to build 3-D tomography images without the intervention of modeling software results in time savings in the lab and also precise information. The possibilities for the semiconductor industry are significant, especially now that Intel is talking about 3-D transistors, says Zhang.

The HF-3300 in the lab

To save users still more time, the new cold field-emission S/TEM’s sample holder is designed to be directly compatible with Hitachi High Technologies America’s focused-ion beam (FIB) sample preparation instrument. Relocating the sample to another cartridge is unnecessary when using these systems in tandem. According to Zhang, this can save 5 minutes or more per sample, a time difference that can add up in high throughput operations.

A more important advantage of using a common sample holder is orientation. The sample is usually cut in a specific direction in FIB in an attempt to orient certain crystallographic direction with the electron beam in the S/TEM. Using the TEM/FIB holder preserves the angle, and reduces disturbances to the sample.

The HF-3300 TEM/STEM has been available since May of this year (a custom model was built for a Canadian lab last year), and the first unit in the United States has been placed in the High Temperature Materials Laboratory Microscopy Group at the Oak Ridge National Laboratory, Oak Ridge, Tenn. The microscope is the newest instrument in the U.S. Department of Energy’s Office of Basic Energy Sciences’ Shared Research Equipment Program at ORNL. A research team led by Karen More will be using the microscope to study nano- and biomaterials, as well as magnetic phenomena using holography.

Academic research core facilities, metrology control laboratories and defect analysis laboratories in particular are the target users for the HF-3300’s performance and sample throughput level, and Zhang expects future interest from research centers and thin film industries.

“Users of cold field emission instruments typically know what kind of bonus it provides,” says Zhang. “They want to enjoy the extended capability inherited from the high brightness, high coherence, and high energy resolution of electron beam imaging.”

The HF-3300 TEM/STEM is a good example of Hitachi’s commitment to cold field-emission technology, but Zhang believes it may not go unchallenged forever.

“So far Hitachi has been the only one to stay with the cold field-emission TEM/STEM technology. Other manufacturers will go to it soon, I think. They will want to compete in this market as they have found that the technology has matured and is more accepted by customers."

--Paul Livingstone

Resources: *Hitachi High Technologies America Inc., Schaumburg, Ill., 847-273-4141, www.hitachi-hta.com*