# Spectra Ultra (S)TEM for material science

# Our highest-resolution (S)TEM platform for the characterization of a wide range of materials

With the new Spectra Ultra (S)TEM, high tension becomes an adjustable parameter just like probe current, and the massive Ultra-X EDX system enables chemical characterization of materials too beamsensitive for conventional EDX analysis.

# Fast high tension switching: quick access to optimized data for more materials

The objective lens produces magnetic fields that vary wildly depending on the mode and accelerating voltage. With the new objective lens of the Thermo Scientific™ Spectra™ Ultra (Scanning) Transmission Electron Microscope, however, the thermal load remains constant at all times. The stabilization time of the optics and stage when switching between different accelerating voltages has been reduced from several hours to less than five minutes. This provides unique and new capabilities.

You can image specimens at 300 kV to achieve high imaging resolution (50 pm), and then switch to lower accelerating voltages to do STEM EDX mapping from the same area with higher X-ray yields, leading to reduced sample damage.

Additionally, the accelerating voltage can be switched multiple times within a single microscopy session to accommodate specimens that suffer from "knock-on" damage (Figure 1).

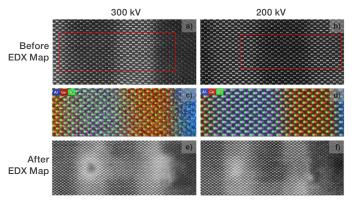


Figure 1. HAADF and EDX maps from an AlGaAs/GaAs interface taken at 300 kV and 200 kV in less than one hour. Reducing the accelerating voltage reduces specimen damage (compare e and f) and improves the EDX signal (compare c and d). The Spectra Ultra (S)TEM can switch and stabilize between any available accelerating voltages in less than five minutes. Specimen courtesy of J. Zweck, University of Regensburg.

### **Key features**

**Full constant power column.** The accelerating voltage can be switched and used within a single microscope session to reduce knock-on damage of beam sensitive specimens or to improve the EDX signal.

**Ultra-X next-generation EDX detector.** Ultra-X provides a unique combination of extremely large solid angle (>4.45 Sr) and cleanliness comparable to the cleanest EDX solution on the market (<1% spurious peaks). Ideal for beam sensitive specimens, Ultra-X provides STEM EDX analysis with less than half the dose required by conventional EDX detectors.

**Damage mitigation.** The combination of fast accelerating voltage switching and the new Ultra-X EDX detector mitigates knock-on and dose-related damage effects in beamsensitive materials.

*In situ* and dynamic research. Fast cameras, chemical detectors, smart software, and our wide gap S-TWIN lens enable *in situ* data acquisition with no compromise on resolution and analytical capabilities.

**Highly repeatable data.** Sophisticated software automation routines, such as OptiSTEM+ and OptiMono+, optimize the system to its peak performance, resulting in more repeatable and quantifiable data.

**High environmental stability.** The redesigned enclosure and ultra-stable base with passive and (optional) active vibration isolation (with iVIS) minimize external environmental influences and ensure high-quality data from long-term and short-term experiments.

# Extremely low dose STEM EDX investigations of beam-sensitive specimens with Ultra-X, our largest EDX system available

The Spectra Ultra (S)TEM brings the next era in EDX detection to the market with the Ultra-X EDX detector, which provides a wide solid angle (>4.45 Sr un-shadowed, 4.04 Sr with a double-tilt analytical holder) as well as cleanliness comparable to the cleanest EDX solution on the market (<1% spurious peaks). Ultra-X opens up new capabilities in STEM EDX analysis of beam-sensitive materials.

Figure 2 shows the difference in sensitivity of Ultra-X compared to the Super-X and Dual-X detectors as a function of tilt. Ultra-X is approximately 2.5x more sensitive than Dual-X and 6x more sensitive than Super-X.

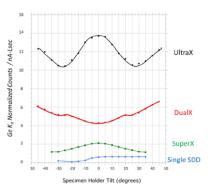


Figure 2. Normalized countrates as function of tilt angle for a single detector: Super-X, Dual-X, and new Ultra-X. Data recorded at 200 kV with optimized specimen holders for each detector configuration. (Zaluzec, et al., submitted to Microscopy and Microanalysis, 2021).

The benefits of such high sensitivity are shown in the improvement in spectrum imaging quality with Ultra-X (Figure 3). A comparison using the same electron dose (8.28 x 108 e/Ų) is shown between Super-X, Dual-X, and Ultra-X on a DyScO3 specimen. The improvements in signal to noise shown in the raw data can be easily seen. Additionally, the oxygen lattice can be directly imaged with Ultra-X, which was not possible with Super-X and Dual-X.

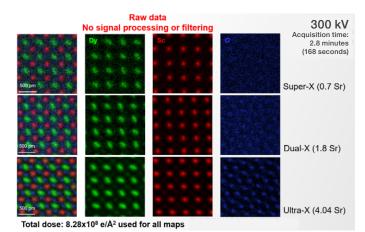


Figure 3. A quantitative comparison between Super-X, Dual-X, and Ultra-X. On a DyScO<sub>3</sub> specimen where the same total dose is used, the improvements in signal to noise with Ultra-X are clearly seen. Sample courtesy of L.F. Kourkoutis, Cornell University. Data collected by Cigdem Ozsoy-Keskinbora.

Additionally, the high sensitivity of Ultra-X means that the same level of chemical information can be obtained with a fraction of the electron dose that would be required for other EDX detector solutions (Figure 4). This unlocks possibilities for STEM EDX analysis from more beam-sensitive specimens and faster mapping for more stable specimens.

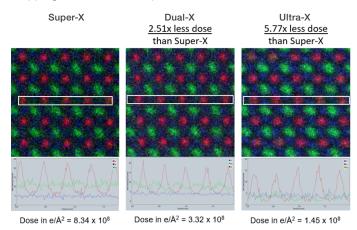


Figure 4. Similar Sc line profiles extracted from the spectrum images. This demonstrates that a similar signal-to-noise ratio can be obtained with Ultra-X with only a fraction of the electron dose needed for Super-X and Dual-X.

# Investigate a wide range of materials at the atomic scale

The Spectra Ultra (S)TEM combines our highest available imaging resolution with the ability to investigate a wide range of materials.

With the combination of its large-gap S-TWIN pole piece and six-fold astigmatism (A5) probe corrector (S-CORR), the Spectra Ultra (S)TEM supports our highest spatial resolution (see Figure 5) and in situ, dynamic, and 3D EDX tomography capabilities in one system.

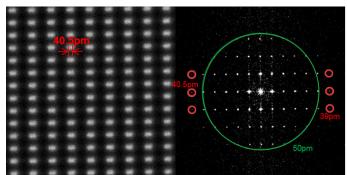


Figure 5. HAADF (DCFI) STEM image of GaN [212] at 300 kV. This shows 40.5 pm Ga-Ga dumbbell splitting and 39 pm resolution in the FFT on a wide-gap (S-TWIN) pole piece. Image collected on a Thermo Scientific Spectra 300 (S)TEM.

The ability to rapidly switch the accelerating voltage from 30 to 300 kV with a stabilization time less than five minutes means you can optimize the accelerating voltage for the specimen and for the experiment during the microscope session; for example, high-resolution imaging at 300 kV and optimized STEM EDX mapping at lower voltages, or simply imaging at a lower accelerating voltage if the specimen is found to be sensitive to knock-on damage.

Extremely low dose STEM EDX mapping capability is unlocked with the massive Ultra-X EDX detector. More than 2x less electron dose can be used compared to the largest current EDX detector solutions. This opens opportunities to image dose-sensitive specimens that previously could not be investigated with STEM EDX.

Atomic resolution imaging from hydrogen to uranium is now possible on the Spectra Ultra (S)TEM with integrated differential phase contrast (iDPC) (see Figures 6 and 7).

Higher quality atomic characterization data is available from more materials types than ever before.

# The Panther Detector: the next generation in low-dose STEM imaging

The Spectra Ultra (S)TEM is equipped with the Thermo Scientific Panther Detector, a segmented STEM detection system and data infrastructure unit. The Panther Detector geometry offers access to advanced STEM imaging capability combined with the sensitivity and detectability to measure single electrons. The entire signal chain has been optimized and tuned to provide unprecedented signal-to-noise-ratioimaging capability with extremely low probe currents (<1 pA). When combined with sensitive STEM imaging techniques such as iDPC, new possibilities are enabled for imaging dose-sensitive samples that have typically been very difficult to characterize with a TEM. Additionally, the completely redeveloped data processing infrastructure offers the future capability of combining detector segments in arbitrary ways and a scalable interface to synchronize multiple STEM and spectroscopic signals.

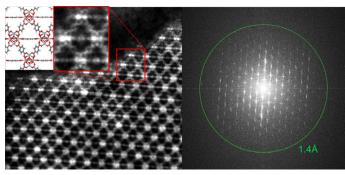


Figure 6. Extreme low-dose imaging (166 e/Ų) of the metal organic framework (MOF) UiO 66, collected on a Thermo Scientific Spectra 300 (S)TEM. A probe current of <0.5 pA was used in combination with iDPC and new sensitive STEM detectors to image atomic-level details in this highly dose-sensitive material. Specimen courtesy of Professor Y. Han, King Abdullah University of Science and Technology.

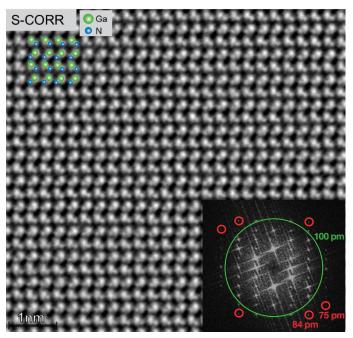


Figure 7. GaN[110] imaged with iDPC STEM at 60 kV with X-FEG/Mono on a wide-gap S-TWIN pole piece. Both Ga and N columns are simultaneously revealed using iDPC at 75 pm resolution, as demonstrated in the FFT. Image collected on a Thermo Scientific Spectra 300 (S)TEM.

Spectra Ultra (S)TEM	Energy spread*	Information limit	STEM resolution
Uncorrected	0.2-0.3 eV**	100 pm	• 136 pm
Probe corrector	0.2-0.3 eV**	100 pm	50 pm (with 30 pA of probe current)
			• 125 pm at 30 kV (with 20 pA of probe current)
Probe + image corrected X-FEG/Mono	0.2-0.3 eV**	60 pm	• 50 pm (with 30 pA of probe current)
			• 125 pm at 30 kV (with 20 pA of probe current)
Probe + image corrected X-FEG/UltiMono	0.025 eV***	60 pm	• 50 pm (with 30 pA of probe current)
			• 125 pm at 30 kV (with 20 pA of probe current)
Probe + image corrected X-CFEG	0.4 eV or 0.3 eV****	70 pm	• 50 pm (136 pm at 30 kV)
			<ul> <li>with 100 pA of probe current</li> </ul>

<sup>\*</sup> For X-FEG/Mono unless otherwise specified.

<sup>\*\*</sup> Depending on energy filter options.

<sup>\*\*\*</sup> Specification for 60 kV

Note: All specifications are at 300 kV using an S-TWIN lens (unless otherwise noted).

## **Technical highlights**

#### Source

- X-FEG Mono: High-brightness Schottky field emitter gun and monochromator with a tunable energy resolution range between 1 eV and <0.2 eV</li>
- X-FEG UltiMono: High-brightness Schottky field emitter gun with ultra-stable monochromator and accelerating voltage with a tunable energy resolution range between 1 eV and <0.025 eV</li>
- X-CFEG: Ultra-high brightness with an intrinsic energy resolution of <0.4 eV (energy resolution of <0.3 eV is possible with a reduced extraction voltage)
- Flexible high-tension range from 30 to 300 kV

## Optical column and correctors

- Three-lens condenser system with indication of convergence angle and size of illuminated area for quantitative measurement of electron dose and illumination conditions
- S-CORR probe corrector provides sub-angstrom imaging resolution at 60 kV as specification and an order of magnitude improvement in optical stability over our previous DCOR technology. The S-CORR corrects A5 for all accelerating voltages
- New CEOS Auto S-CORR auto alignment software makes probe corrector tuning easy, fast, and fully automated up to 5th order aberrations
- Patented mechanical stacking of column modules minimizes instabilities caused by excessive deflector excitations
- Thermo Scientific™ ConstantPower™ Lens and corrector are designed for extremely high thermal stability in accelerating voltage and in mode switches, minimizing image drift
- Low-hysteresis design to minimize crosstalk between optical components for high reproducibility
- Symmetric S-TWIN objective lens with wide-gap pole piece design of 5.4 mm with "space to do more" allows the use of special holders, such as heating, cooling, indentation, and electrical probing holders
- Objective aperture in the back focal plane of the objective lens for optimized TEM dark field application work
- Automatic apertures for remote control operation and reproducible recall of aperture positions during aperture change
- Field upgradeable probe and image Cs-corrector
- Rotation-free imaging for easy operation and a clear orientation relationship between the imaging and diffraction
- Deep sub-angstrom resolution for all accelerating voltages (60–300 kV) with low specimen drift
- Field-free imaging in TEM Lorentz mode with 2 nm resolution for magnetic property studies and option for Cscorrected Lorentz with <1 nm resolution</li>



Integrated Faraday cup and calibrated fluscreen current.
 Readout is linear over whole beam current range

### Stage

- Computerized 5-axis, ultra-stable specimen piezo stage for accurate recall of stored positions and tracking of the areas visited during sample navigation. The piezo stage allows for movements as fine as 20 pm for centering of feature of interest in the field of view
- Tilt range of ±35 degrees alpha and ±30 degrees beta with the Ultra-X optimized analytical double tilt holder to access the maximum number of zone axes of each crystal in polycrystalline materials. With tomography holder, the tilt range is ±70 degrees to minimize the missing wedge in 3D reconstructions
- Linear drift compensation provided by piezo stage can be used to mitigate limitations caused by thermal drift, which is unavoidable during in situ heating or cooling experiments

## Analytics and detectors

- Ultra-X EDX options, integrated software, and the Gatan Ultrafast EELS/DualEELS options together provide up to 1,000 sp/s of simultaneous EDX and EELS data acquisition
- Live peak identification and background fitting during ultrafast EDX acquisition
- Symmetric EDX detector design allows for combined tomographic EDX

## **EDX** detector portfolio

 EDX quantification using Thermo Scientific<sup>™</sup> Velox<sup>™</sup> Software (featuring dynamic correction of holder shadowing as a function of tilt)



- Ultra-X: high-sensitivity, windowless EDX detector system with high solid angle and high cleanliness
  - Output count rate: up to 1.5 Mcps
  - Energy resolution
    - ≤136 eV for Mn-Ka and 10 kcps (output)
    - ≤140 eV for Mn-Ka and 100 kcps (output)
  - 4.45 srad solid angle (without specimen holder)
  - 4.04 srad solid angle (with analytical double tilt holder)
  - High P/B ratio (Fiori number) >2,500
  - Excellent in-hole performance (<1% hole counts)</li>
  - Low system background in EDX (<1% Fe and Co spurious peaks)</li>

#### Available detector options

- HAADF detector
- New ultra-low-noise Panther Detector; on-axis solid state, 8 segmented BF and ADF detectors (16 segments in total)
- Thermo Scientific<sup>™</sup> Ceta<sup>™</sup> S or Ceta M Camera (optionally with speed enhancement)
- Gatan OneView/OneView IS cameras
- Gatan energy filter series
- Electron microscope pixel array detector (EMPAD)

#### Available holders

- Single tilt holder
- Double tilt holder
- Tomography holder
- Thermo Scientific and third-party in situ holders
- Please ask for a list of functional holders

#### Software

 The Electron Dose Control (EDC) module predicts beam current, dose, and dose-rate live based on gun and optics settings. The EDC calibration is done with a built-in Faraday cup. Users can set electron dose without knowledge of TEM optics. The beam current, dose, and dose-rate are reported in Velox Software along with image metadata.

- Differential phase contrast (DPC) STEM technique enables live measurements of intrinsic magnetic and electric fields
- Integrated DPC (iDPC) software for high imaging contrast in STEM on materials across the whole periodic table. This low-dose technique expands the use cases of STEM in materials science and replaces annular bright field as the technique of choice for light elements. Invaluable when applied to samples that are typically damaged under short exposures to the electron beam
- OptiSTEM+ software for single-click correction of 1st and 2nd order probe-forming aberrations to deliver extremely high STEM resolution to all users on our probecorrected tools\*
- OptiMono+ software for completely automated monochromator alignment and tuning to the highest achievable energy resolution on monochromated systems from 1 eV down to <25 meV</li>
- Thermo Scientific<sup>™</sup> Truelmage<sup>™</sup> Atlas focus series software for quantitative HR-TEM applications. (For more details, see separate product datasheet.)
- Fully digital system for remote controlled operation using the SmartCam suite
- Advanced, integrated software enables fast and simultaneous signal acquisition (up to five STEM signals)
- Smart scanning technology for high image quality in STEM

#### Other features

- Environmental enclosure to relax the acoustic and room temperature variation requirements
- Cold trap design for up to three days of operation to maximize uptime



Learn more at thermofisher.com/spectra-ultra



<sup>\*</sup> High performance guaranteed in combination with S-CORR STEM probe corrector.