

# Unprecedented X-CFEG for the Talos (S)TEM

Ultra-high-brightness cold FEG for best quality images  
and analytical information with the least amount of effort

# Introduction

Characterization challenges in materials science involve analyzing a broad range of materials with sub nanometer or atomic resolution with better signal to noise and higher resolution in scanning transmission electron microscopy (STEM), energy dispersive X-ray spectroscopy (EDS), and electron energy loss spectroscopy (EELS).

## Better and faster STEM and analytics

Challenges arise when the high-resolution STEM images are not sharp enough, and/or the analytical results (EDS / EELS) maps are too noisy, making details unclear and difficult to analyze. A typical cause of not being able to image and chemically analyze nanoparticles or (atomic) structures is because they do not get enough EDS/EELS signal from small spots in STEM. And if an acquisition takes too long, the sample may get damaged. This slows down research, publications, and the development or optimization of materials.

To resolve these challenges, a smaller gun source and higher gun brightness are needed. Our new ultra-high-brightness cold field FEG source (X-CFEG) on the Thermo Scientific™ Talos™ (S)TEM provides better spatial resolution and higher quality images and spectral data with the least effort.

## Better energy resolution for better chemistry

To resolve spectra fine enough to extract accurate information about the details of the sample's chemistry, you must be able to perform EELS with an energy resolution better than the standard of 0.8 eV. Our X-CFEG provides an energy resolution of 0.3 eV, greatly surpassing the standard.

## Flexibility

Another challenge is if a sample at 200 kV gets too much knock-on sample damage. Going to lower high tension, typically 80 kV will reduce that damage; however, with a standard gun, too much spatial resolution is lost in HRSTEM. However, a better energy resolution (which is lowering chromatic aberration) and improved brightness, such as the X-CFEG provides, will improve the HRSTEM imaging and analytics at 80 kV.

Moreover, a very high level of automation makes changing modes, beam currents, and other optical settings very intuitive on the Talos (S)TEM. The Velox™ software provides the fastest and easiest way to acquire and analyze multimodal data.

This new gun also provides lower cost of ownership and longer warranty. On the X-CFEG, we will offer a 5-year warranty, instead of a 1-year warranty,

The X-CFEG for the the Talos (S)TEM improves the imaging and analytical performance, productivity, and ease of use of the Talos instrument. This new addition helps material scientists analyze materials with subnanometer resolution

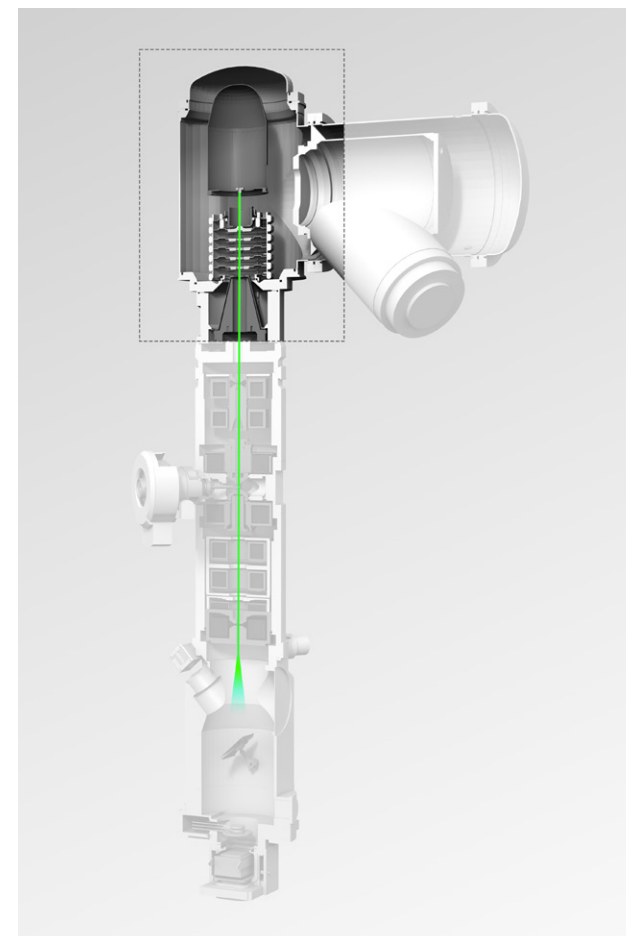


Figure 1. Talos (S)TEM column with ultra-high-brightness cold field emission gun (X-CFEG) for fast, high-quality stem imaging and analytics.

# Performance

much faster with very little effort, and improves the signal-to-noise ratio and resolution in STEM, EDS, and EELS.

The Thermo Scientific Talos F200i and Talos F200X G2 (S)TEMs can now be powered by a new ultra-high-brightness emission gun (X-CFEG). The X-CFEG has an unprecedented high brightness, small source size (tip) and low energy spread, which provides:

- 1. Faster time to STEM imaging** thanks to the sharp tip. Taking STEM images with X-CFEG is at least 30% faster than with Schottky S-FEG / X-FEG.
- 2. Easier and improved high-resolution STEM imaging.** With the powerful combination of X-CFEG and Talos optics, an improved HRSTEM system resolution of 0.136 nm can be achieved.
- 3. High-resolution STEM imaging with high probe currents for high-throughput, fast-acquisition STEM and faster EDS and EELS analytics.** With the powerful combination of X-CFEG and Talos optics and high level of automation, high-resolution STEM imaging and chemical analysis with EDS/EELS with high probe current can be routinely achieved. Proof of high-current STEM imaging on Si[110] and SrTiO<sub>3</sub> (STO) is shown in Figures 3 and 4, and Atomic EDS mapping on SrTiO<sub>3</sub> (STO) in Figures 5

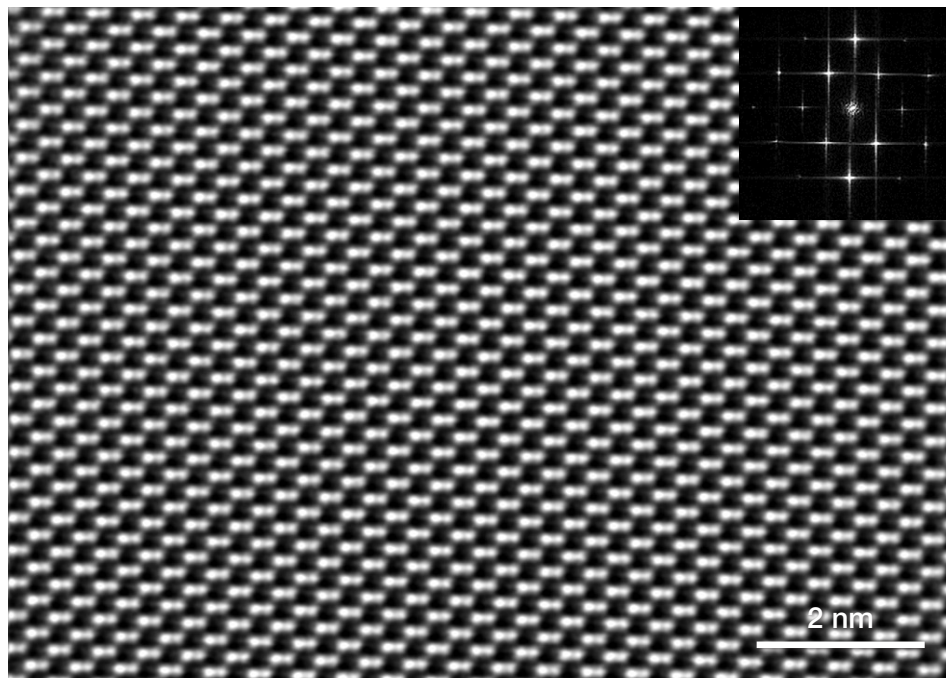


Figure 2. The X-CFEG is superior to other guns with respect to brightness, source size, and energy spread. Shown is a Si[110] HAADF HRSTEM image with an energy resolution of <0.4 eV with Si dumbbells (0.136 nm) clearly visible. With a traditional Schottky FEG, 0.16 nm is visible with 0.8 eV energy resolution. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Gatan Continuum at 200 kV.

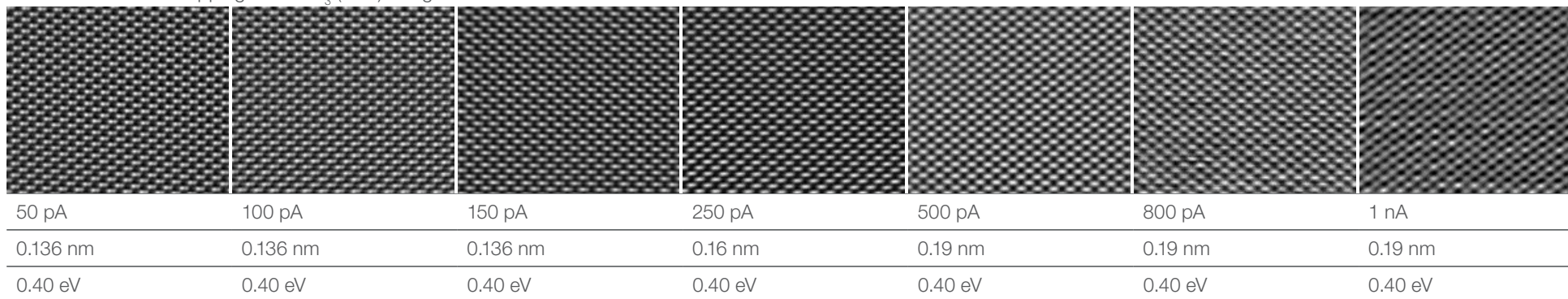


Figure 3a. 200 kV Si[110] HAADF HRSTEM images taken on a Talos F200 X-CFEG with probe currents ranging from 50 pA up to 1 nA while maintaining the indicated STEM resolution. The improved HRSTEM system resolution of 0.136 nm can be achieved with >100 pA of beam current.



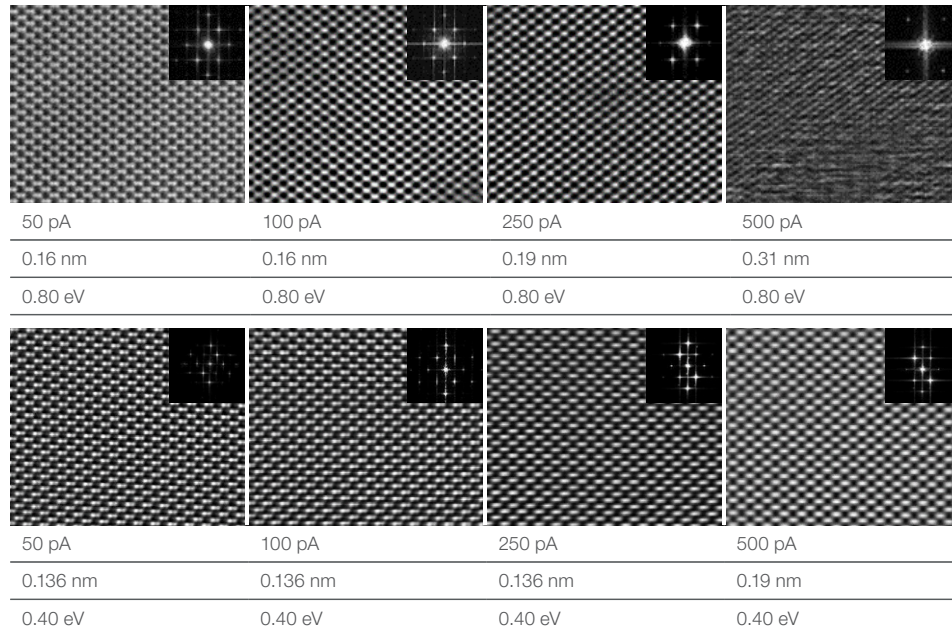


Figure 3b. Comparison of HRSTEM images with 50, 100, 250 and 500 pA current with X-FEG (top) vs X-CFEG (bottom) with typical resolutions.

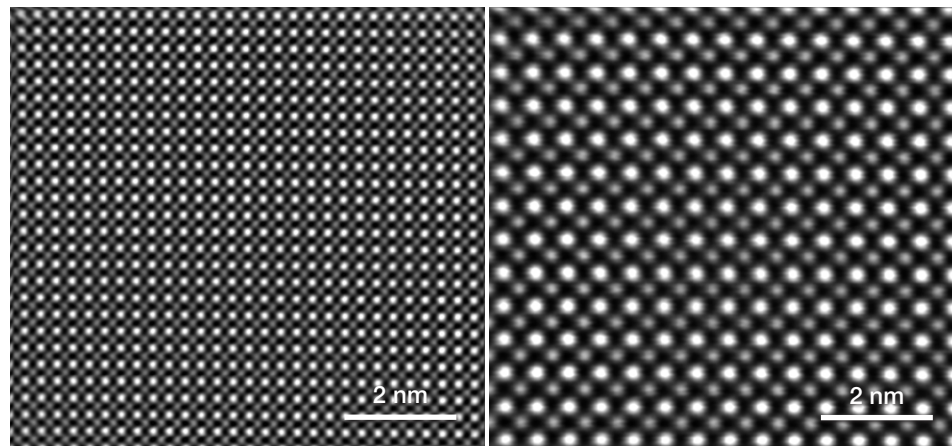


Figure 4. Here a HAADF STEM  $\text{SrTiO}_3$  (STO) image is shown with an energy resolution of  $<0.40$  eV at  $<0.19$  nm with 75 pA. Results were shot on a Talos F200 (S)TEM with X-CFEG and Panther STEM at 200 kV.

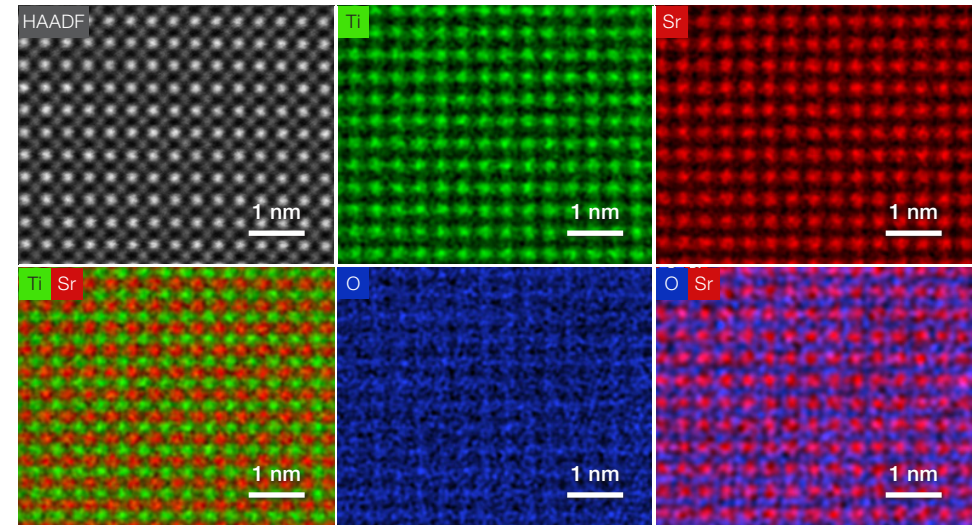


Figure 5a. An STO HAADF STEM image and 512x512 EDS maps are shown with 75 pA. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Bruker Dual-X at 200 kV.

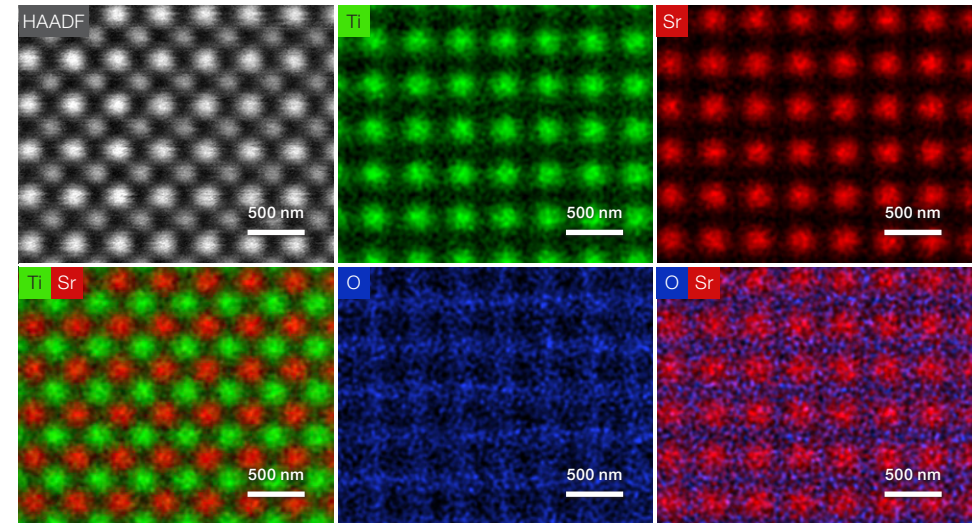


Figure 5b. Here an STO STEM image and 512x512 EDS maps are shown at higher magnification with 75 pA. Oxygen is clearly visible. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM, and Bruker Dual-X at 200 kV.



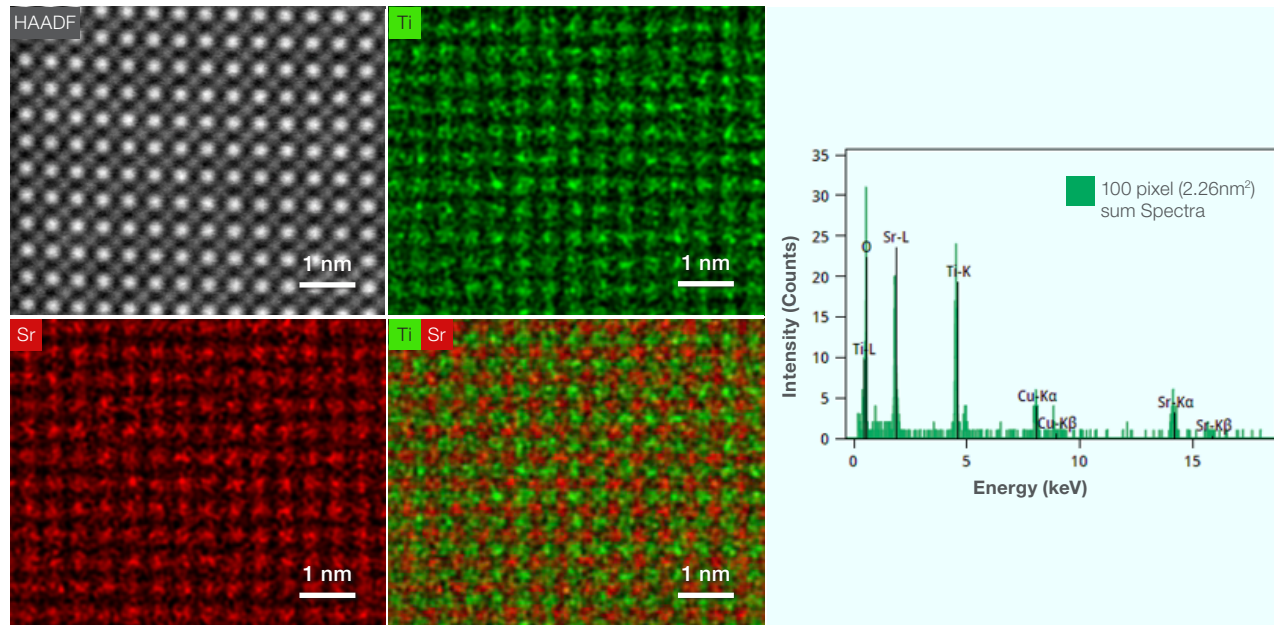


Figure 6. An STO STEM image and a superfast 512x512 EDS map with 400 pA. Results were shot in only 40 seconds on a Talos F200 (S)TEM with X-CFEG, Panther STEM, and Bruker Dual-X at 200 kV.

and 6.

#### High currents in small probes:

4. Improved EELS energy resolution (compared to conventional FEG) resolves more information about the chemistry of the sample. Figure 7 shows an example of a zero-loss peak with  $<0.40$  eV and  $>14$  nA of beam

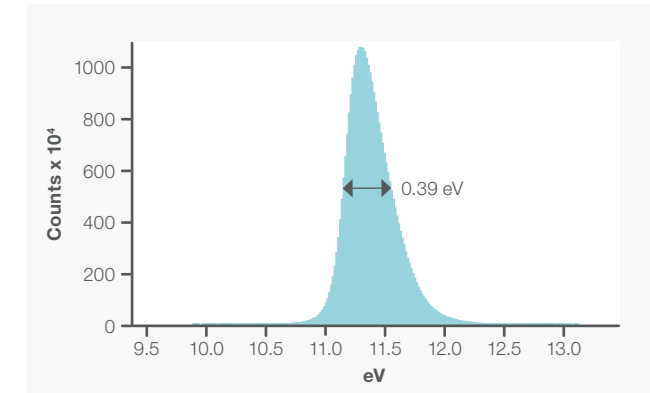


Figure 7. An energy resolution of  $<0.40$  eV is shown with  $>14.0$  nA beam current. Results were shot on a Talos F200 (S)TEM with X-CFEG and Gatan Continuum at 200 kV.

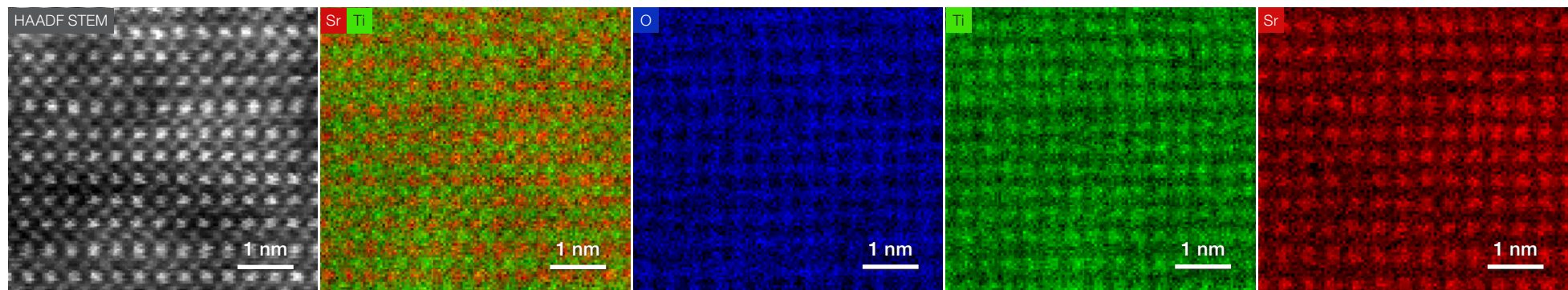


Figure 8. Atomically resolved STO STEM and EELS spectrum images are shown with an energy resolution of  $<0.40$  eV with 220 pA. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Gatan Continuum at 200 kV in only 2 minutes.

current, enabling high signal to noise imaging and fast high resolution EELS spectroscopy, and Figure 8 shows atomic EELS mapping on STO.

Figure 9 shows HREELS spectra of cobalt oxide ( $\text{Co}_3\text{O}_4$ ).  $\text{Co}_3\text{O}_4$  is an attractive earth-abundant catalyst for CO oxidation. Optimization of nanostructured metal oxide catalysts by substituting inactive cations near the active sites and thereby increasing the overall activity of the exposed surfaces. The EELS L peaks in transition metals

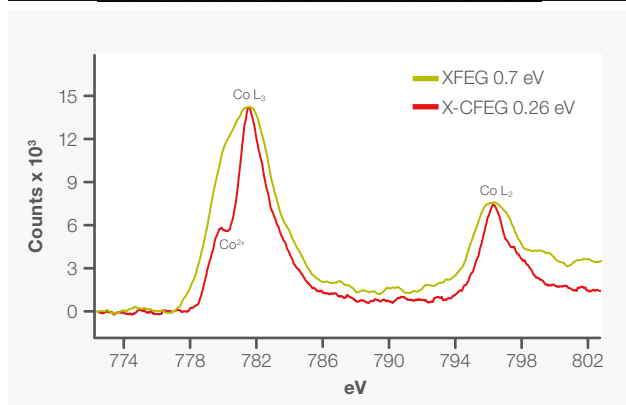
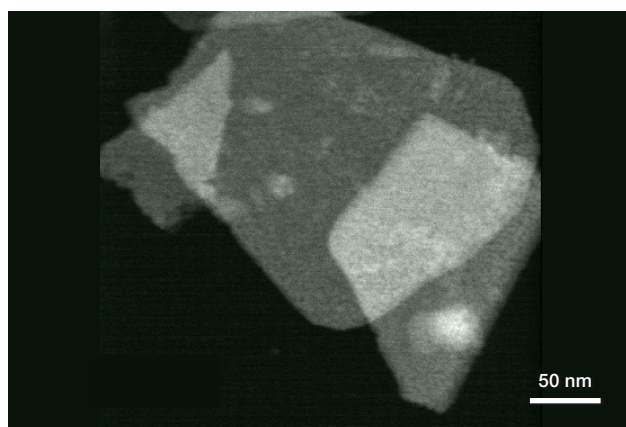
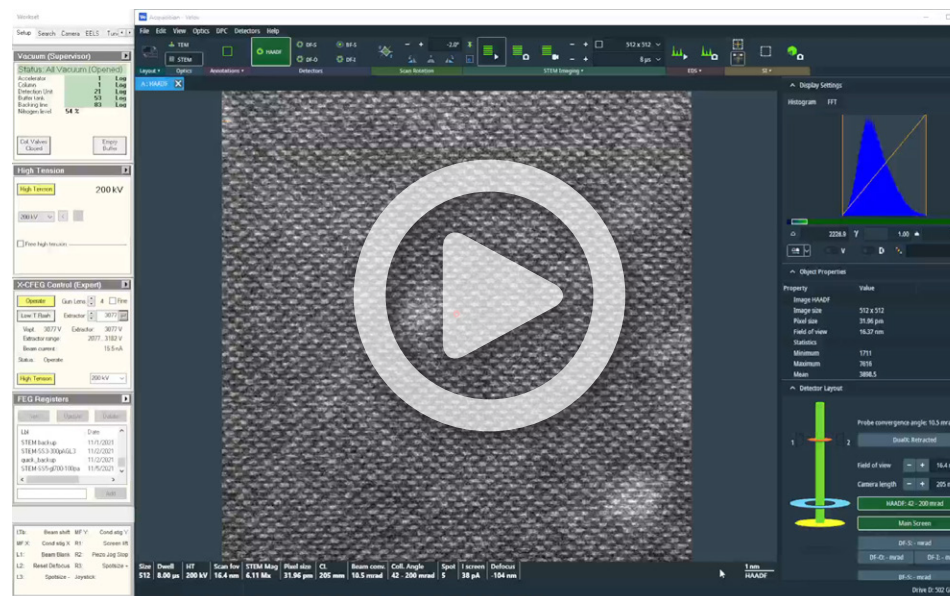


Figure 9. Cobalt oxide STEM image and EELS spectra with an energy resolution of  $<0.26$  eV (red). The same sample was analyzed on a conventional FEG with  $0.7$  eV energy resolution (green). Clearly the X-CFEG fine structure (red) is much more visible, revealing a shoulder on the  $\text{Co L}_3$  which is  $\text{Co}^{2+}$ . This shoulder is not visible on the green spectrum. Results were shot on a Talos F200 (S)TEM with X-CFEG (red) and X-FEG (green), respectively, and Panther STEM with Gatan Continuum at  $200$  kV.



Video 1. Tip flashing (video) on the X-CFEG.  $0.14$  nm resolution (with  $0.40$  eV) at  $200$  kV is maintained before and after a tip flashing without adjustment of the optics. Video was shot on a Talos F200 (S)TEM with X-CFEG and Panther STEM at  $200$  kV. Duration 1.33

are originated by excitations of electrons from  $2p$  to  $3d$  orbitals. The shape and weight of the  $L_{2,3}$  peaks depend on the occupancy of the  $3d$  orbitals.

##### 5. The X-CFEG can be operated from 20 to 200 kV.

It helps improve the resolution in HRSTEM at  $80$  kV. An improved energy resolution (lowering chromatic aberration  $C_c$ ) together with a much higher brightness improves the HRSTEM imaging and analytics.

##### 6. The ultra-high-brightness X-CFEG speeds up long experiments like tomography (EDS), Maps Software (EDS), APW, dopant materials, etc.

##### 7. Longer lifetime tip with lower cost of ownership and longer 5-year warranty.

STEM probe currents can be flexibly set up from  $<1$  pA

up to several nA range with fine control of the gun and condenser optics, so that the widest range of specimens and experiments can be accommodated.

As with all cold field emission sources, the sharp tip requires a periodic regeneration (called flashing) to maintain the probe current. With the X-CFEG, the tip requires flashing only once or twice per working day, a process that takes less than a minute. There is no measurable impact on the image even in the highest resolution imaging conditions (see video below), and the daily tip flashing process has no impact on the tip lifetime.

This new-generation X-CFEG also produces enough total beam current ( $>14$  nA) to support standard TEM imaging experiments (e.g., *in situ*) with large parallel probes, making it a uniquely all-purpose, yet high-performance, cold FEG.

Adding to the flexible nature of the X-CFEG is the capability

# Example: Batteries

to adjust the energy resolution by varying the extraction voltage. The lifetime of the tip is unaffected by the extraction voltage chosen to perform the experiment.

Figure 10a shows an example of a rechargeable battery

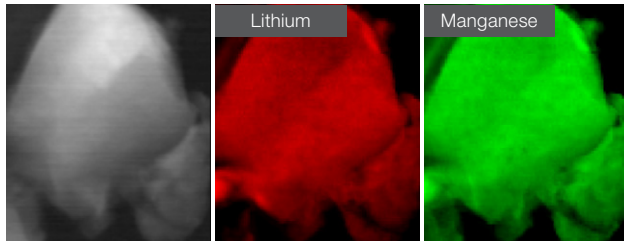


Figure 10a. A STEM image and corresponding EELS spectrum images are shown with an energy resolution of  $<0.28$  eV. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Gatan Continuum at 200kV. Sample courtesy Dr. Chongmin Wang, PNNL.

cathode material, containing Mn, Li and Ti, analyzed with EELS. This new battery material is more environmentally friendly than its predecessors.

The exceptionally good energy resolution makes fine structure of the beam-sensitive lithium, and also the manganese fine, and titanium (Figures 10b and 10c, respectively) structure visible. With conventional FEGs, this fine structure that is directly related to the different valence states of the elements in the battery, is not visible. This battery example for a conventional FEG with 1.0 eV EELS resolution fails to capture the splitting of both the Ti  $L_2$  and the  $L_3$  peaks due to the limited energy resolution. X-CFEG has more than enough resolution (0.28 eV) to capture the splitting of both peaks (see Figure 10b).

The environmentally friendly battery material in this example is being developed without the use of scarce natural resources such as cobalt. Since nickel is not sustainable, the researchers try to use earth-abundant materials (such as manganese) to tailor the battery structure towards high capacity. For this, high-resolution EELS, as provided by the X-CFEG, is a must.

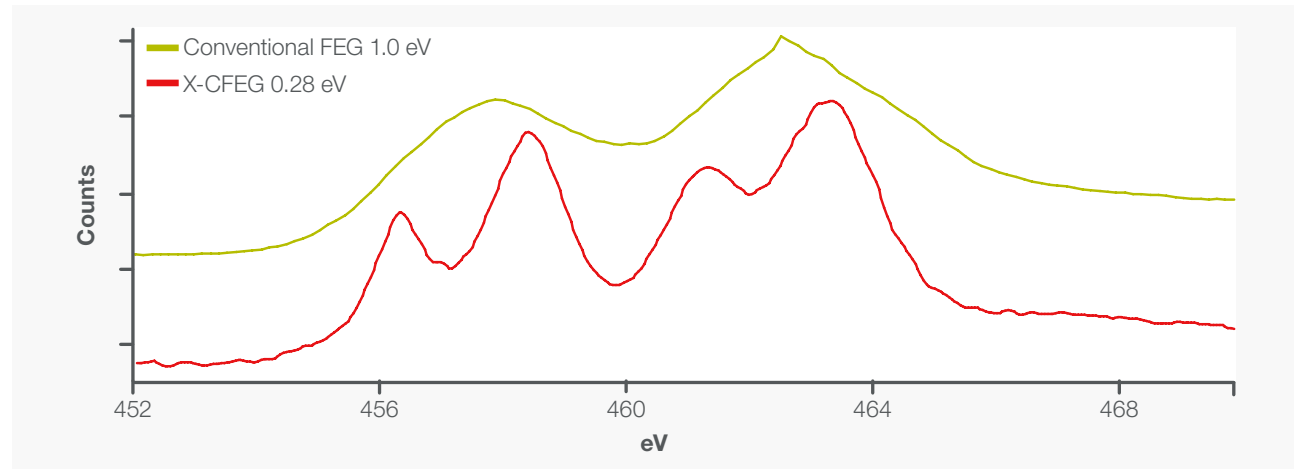


Figure 10b. The Ti- $L_{2,3}$  edges in the Li battery. EELS spectra are shown from the same sample as in Figure 10a. The difference is shown between 0.28 eV (X-CFEG, red) and 1.0 eV (conventional-FEG, green) energy resolution. Clearly more peaks and more details are visible in the X-CFEG spectrum (red). Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Gatan Continuum at 200 kV. Sample courtesy Dr. Chongmin Wang, PNNL.

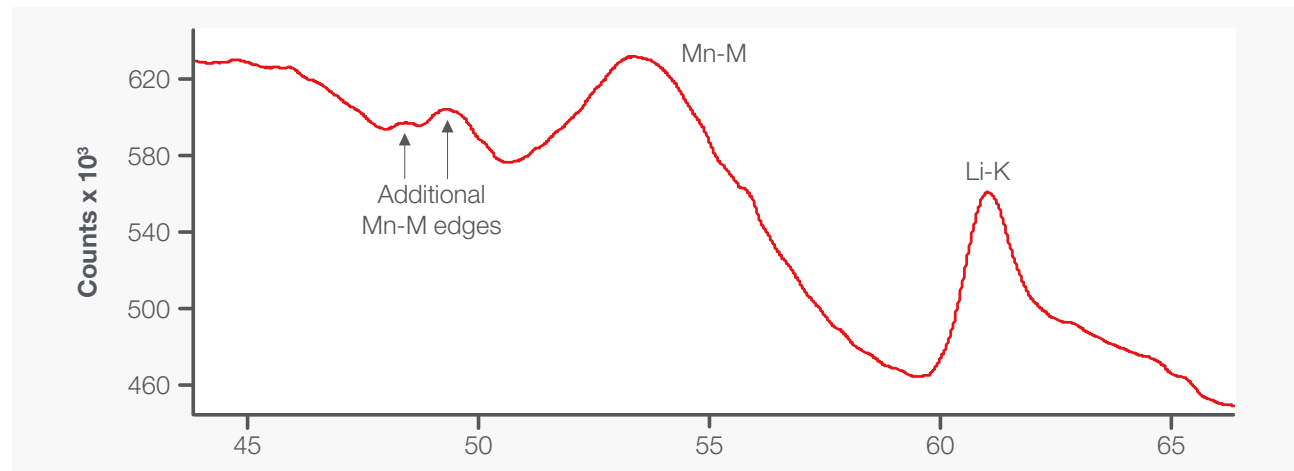


Figure 10c. An EELS spectrum from the same sample but at lower energies is shown with an energy resolution of  $<0.28$  eV. The Mn-M edge fine structure and Li-K edge in the Li battery are distinguished. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Gatan Continuum at 200 kV. Sample courtesy; Dr. Chongmin Wang, PNNL.



# Example: Gold Nanorods

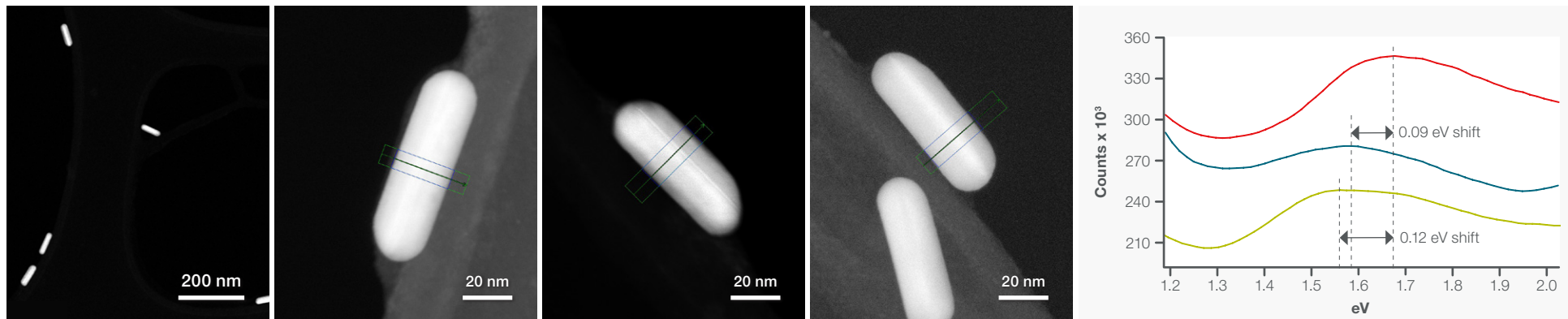


Figure 11: STEM images and EELS spectra are shown with three different diameters and resonances. Indicated are very small but relevant shifts of 0.09 and 0.12 eV with respect to the smallest diameter. Results were shot on a Talos F200 (S)TEM with X-CFEG, Panther STEM and Gatan Continuum at 200 kV. *Sample courtesy Prof. Sara Bals From University of Antwerp*

Surface plasmon resonances find use in a wide range of applications, from biomedical applications, such as cellular imaging, molecular diagnosis, and targeted therapy, to photonic, semiconductor, and polymeric applications. Noble metal nanoparticles and nanorods are used most often. The electron beam can excite the surface plasmon resonances, making it possible to measure, image, and analyze them with high-spatial and high-energy resolutions.

Figure 11 shows an example of gold nanorods with various diameters analyzed with HREELS. The resonance mode of the gold nanorods shifts with the diameter of the Au nanowire; hence, the frequency and the amount of energy losses that were absorbed change. Three diameters were measured: 23.0, 23.5, and 24.0 nm, which resulted in shifts of 0.09 and 0.12 eV, relatively.

The high energy resolution and high brightness that the X-CFEG provides on the Talos (S)TEM reveals the diameter of gold nanorods and how their resonance mode shifts with diameter. These elements are not visible with conventional FEGs. Thus, the X-CFEG on the Talos (S)TEM brings researchers closer to understanding how these nanorods behave.

Criterion	Talos L120C Thermionic LaB <sub>6</sub>	Talos F200i/S Schottky S-FEG	Talos F200X Schottky X-FEG	Talos F200i/X X-CFEG
Tip lifetime (yrs)	~0.5	>1	>1	>5
HRTEM infor limit (nm)	0.2 (only line res.)	0.12	0.12	0.11
HRSTEM (nm)	1.0	0.16	0.16	0.14
Brightness 200 kV (A/cm <sup>2</sup> /sr)	Low	4 10 <sup>8</sup>	1.8 10 <sup>9</sup>	2.4 10 <sup>9</sup>
Energy resolution 200 kV (eV)	~1.5	0.8	0.8	0.3

Table 1. Comparison overview of the most important specifications.



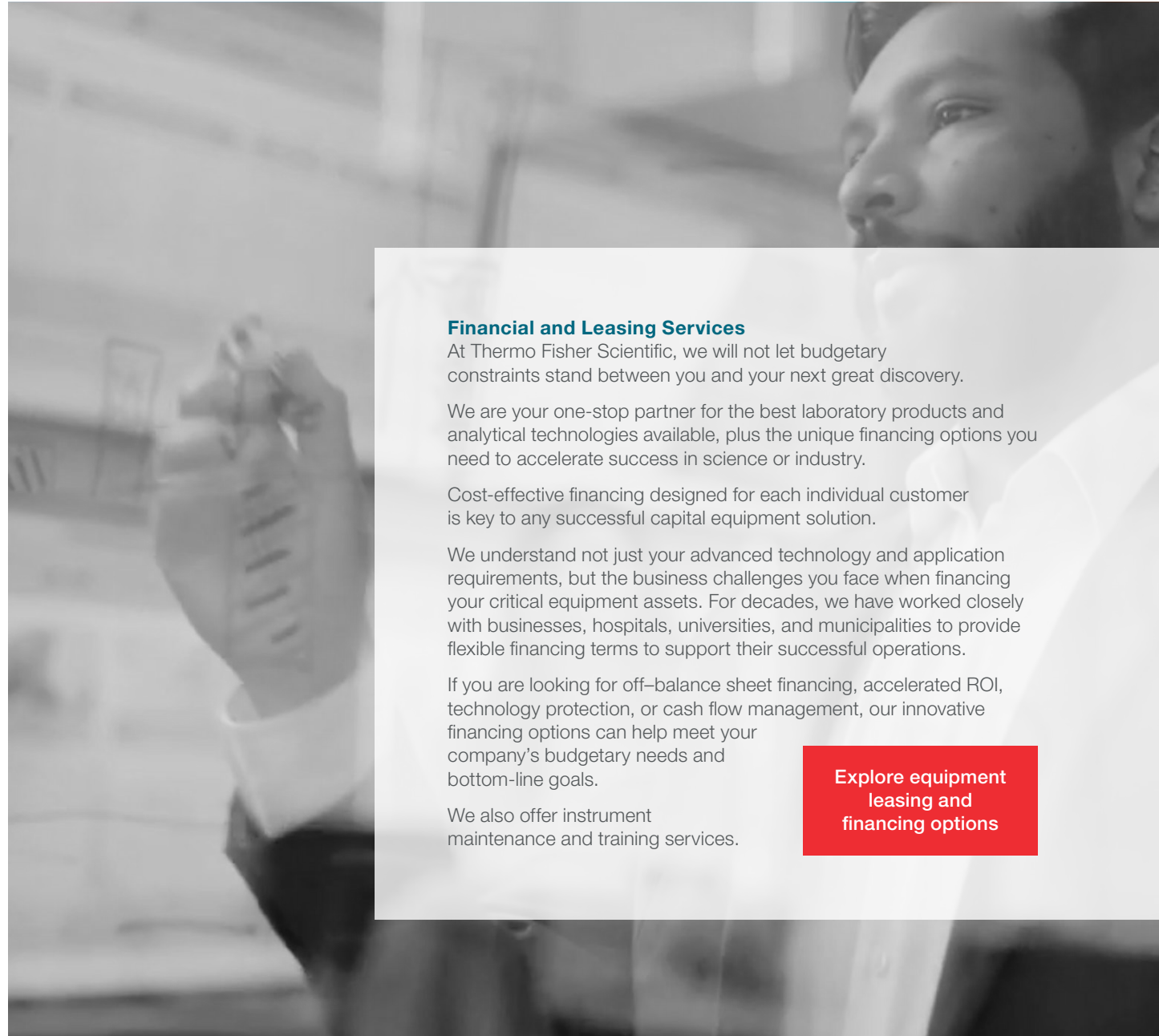
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We are the world leader in serving science. Our mission is to enable our customers to make the world healthier, cleaner and safer.



Video 2. Step ahead. Step beyond. Duration 1:33

Our innovative solutions for electron microscopy, X-ray tomography, surface analysis, and microanalysis help materials science researchers advance their sample characterization to gain deeper insight into the physical and chemical properties of materials (morphological, structural, magnetic, thermal, and mechanical) from the macroscale to the nanoscale. Our multiscale, multimodal solutions cover a broad range of applications (discovering new materials, solving analytical problems, improving processes, and assuring product quality) across dozens of industries and research fields, serving customers in academia, government, and industry. Our TEMs, DualBeam™ FIB-SEMs, comprehensive portfolio of SEMs, microCT, XPS, and microanalysis solutions, combined with software suites, take customers from questions to usable data by combining high-resolution imaging with physical, chemical, elemental, mechanical, and electrical analysis across scales and modes.



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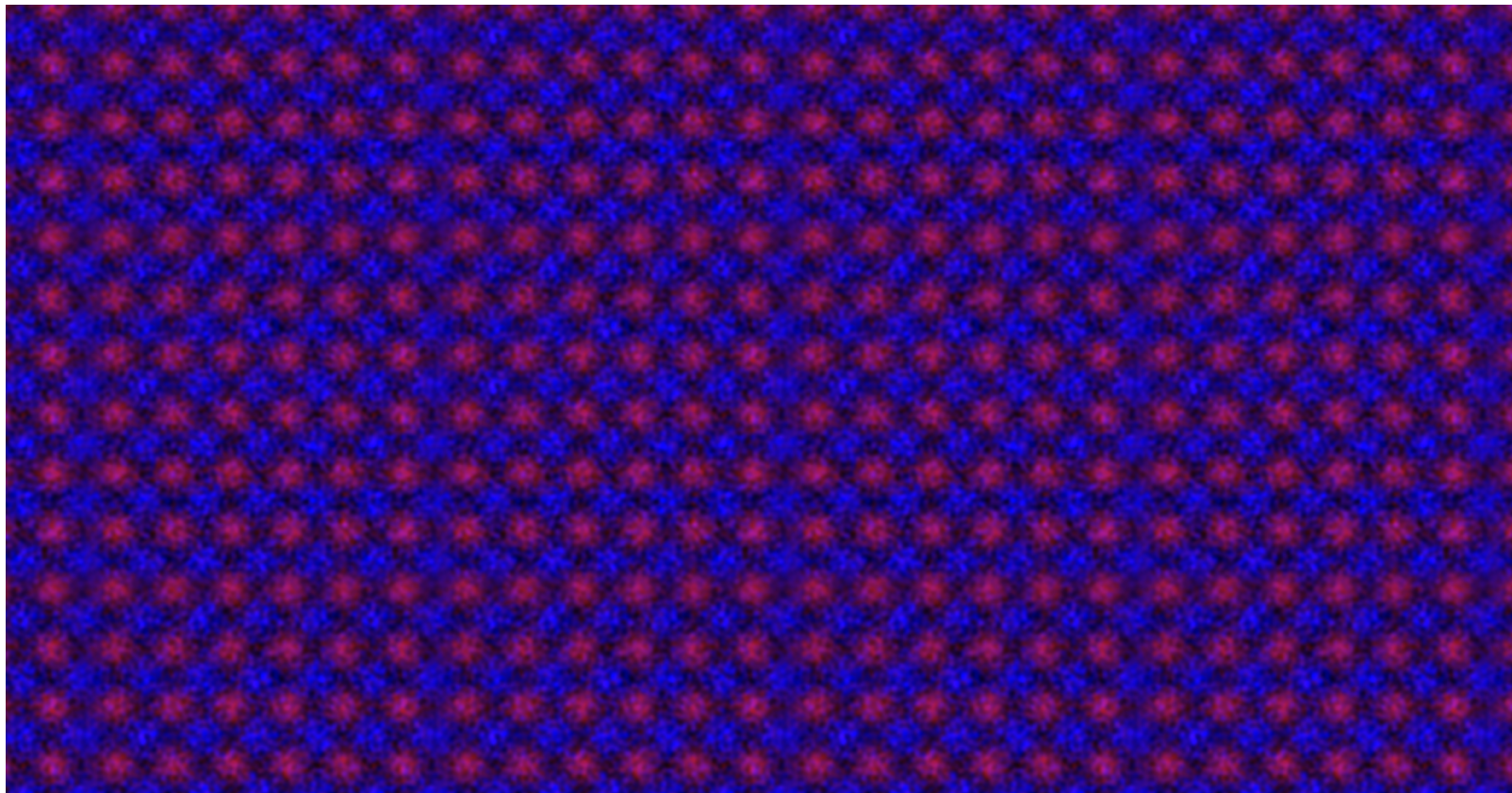
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# Unprecedented X-CFEG for the Talos (S)TEM

Ultra-high-brightness cold FEG for best quality images and analytical information with the least amount of effort



## Brightness of an electron gun

- The current density of an electron gun is the number of electrons (or charge) per unit area per unit time.
- The gun brightness of an electron gun is the current density per unit solid angle of the source.
- Electron sources differ in their size. As a result, the electrons leave the source diverging from each other with a range of divergent angles. This angular distribution of the electrons is relevant for the brightness.
- In STEM and analytical EDS and EELS, brightness is particularly important when using very small convergent probes. The concept of brightness is less important in conventional TEM where we use a large, defocused beam, but it is still relevant to the intensity we see on the screen, and so it affects how easy it is to operate the microscope and see our images and diffraction patterns.
- The most coherent electron sources for (S)TEMs have the sharpest tips in field emission electron guns due to the remarkably high coherence and brightness.

Find out more at [thermofisher.com/talos](https://thermofisher.com/talos)

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