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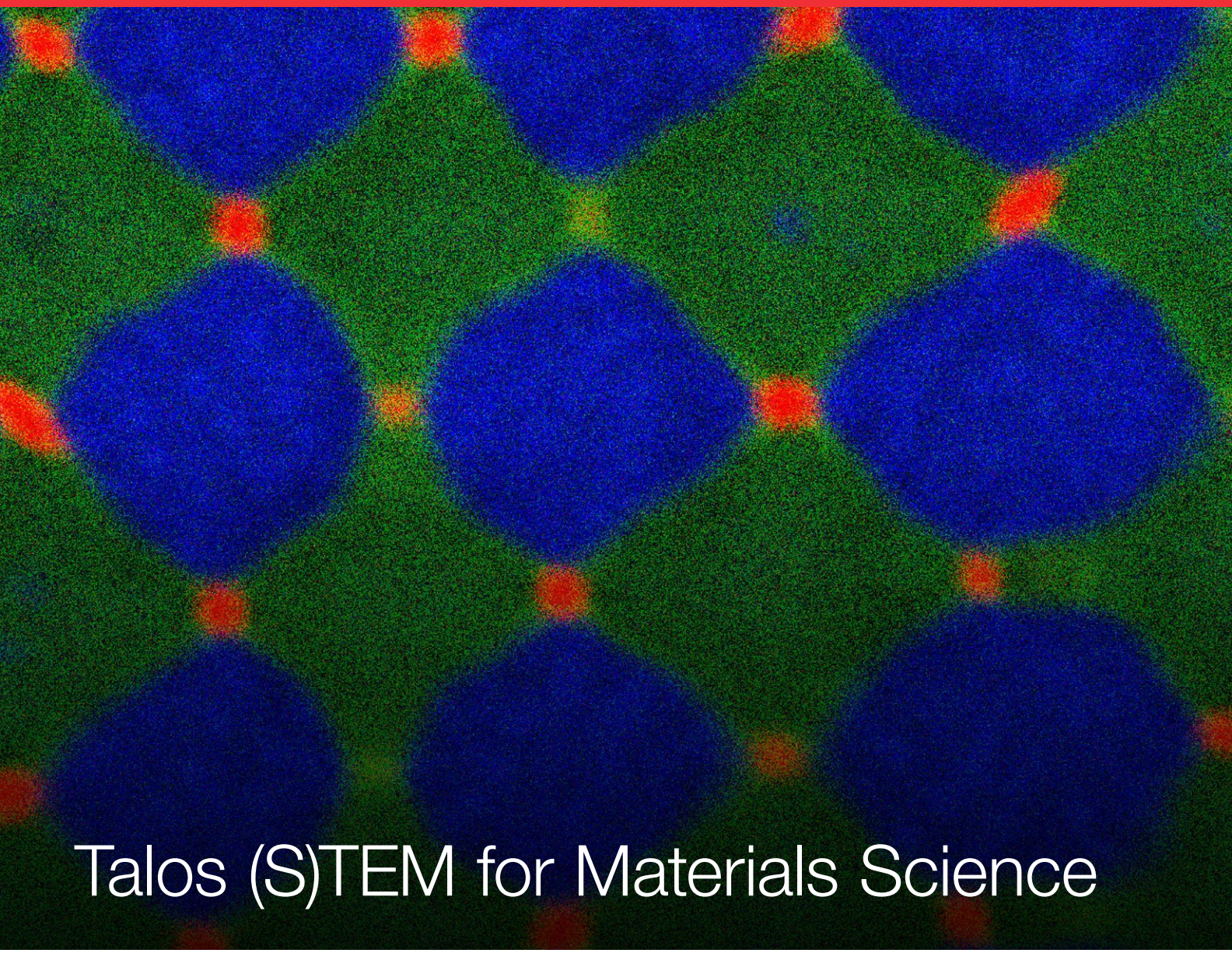


Talos (S)TEM for Materials Science

Accomplish more with fast, precise, quantitative materials characterization in multiple dimensions

ThermoFisher SCIENTIFIC

Introduction

Breakthroughs in materials science research depend on fast, reliable high-resolution information that allows scientists to optimize the material's performance. True understanding of material function and behavior requires advanced visualization and testing alongside compositional and structural data in 2D and 3D, giving materials scientists the information they need to predict the behavior of the material.

4k × 4k EDS map from ALNICO-8. Red: Cu, Blue: Fe, Green: Ni.
Sample courtesy of Prof. Hamish Fraser, The Ohio State University, USA.

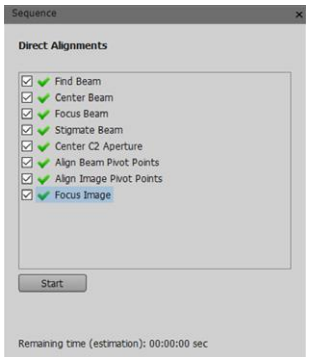
Talos (S)TEM

The Thermo Scientific™ Talos™ Scanning Transmission Electron Microscope (STEM) is designed for fast, precise and quantitative characterization of nanomaterials. It accelerates materials imaging and analysis through higher data quality and faster acquisition with simplified and automated operation. It combines outstanding high-resolution (S)TEM (STEM or TEM) imaging, unparalleled advances in energy dispersive X-ray spectroscopy (EDS) signal detection and 3D chemical characterization with compositional mapping via its unique embedded detector solutions. The Talos (S)TEM is ergonomically designed and remotely operable in daylight conditions.

The Talos portfolio.

Align Genie automation

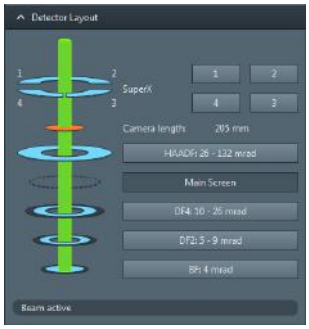
Spend less time setting up the microscope and running alignments with Align Genie. All TEM daily tunings have been automated to provide an optimized, reproducible setup. This automation eases the learning curve for novice operators, reduces overhead in a multi-user environment and improves time-to-data for the experienced operator. This makes the Talos (S)TEM the optimal tool for microscopists and materials scientists seeking to access high-resolution nanoscale data.



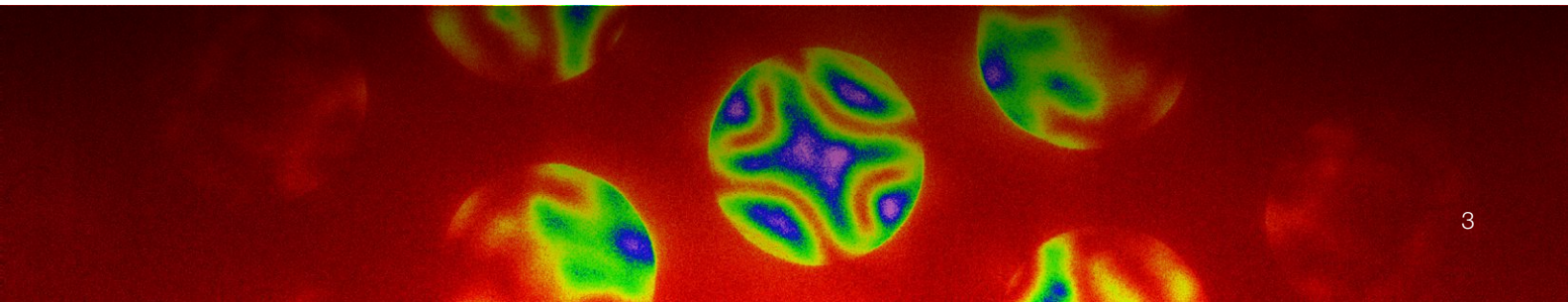
Align Genie for Talos Automation.

Easiest way to acquire multimodal data with Velox Software

Designed for multi-user, multi-disciplinary environments and equipped with the Thermo Scientific Velox™ Software user interface, the Talos (S)TEM is ideal for novice users. Velox Software provides the fastest and easiest way to acquire and analyze multimodal data. Moreover, the detector panel provides a graphical overview of all optical settings and will be updated live upon detector selection (e.g. when camera lengths are changed or when blanking the beam). This allows the scientist to always be in control of the experimental conditions during operation. These conditions are stored with the images, so results can easily be reproduced. Additionally, all relevant information for subsequent image simulations is documented.



Velox' live optics and detector layout.



Large area image and analytical data acquisitions at high resolutions with Maps Software

Modern materials science requires multimodal, statistically meaningful data to obtain sample properties across platforms on multiple length scales and modalities. This has become increasingly more important for research on nanoparticles, catalysis samples and for precipitates in metals. Thermo Scientific Maps™ Software for TEM and EDS automates navigation across multiple length scales to characterize large volumes quickly and reproducibly, and provides pinpoint, contextual analysis on relevant areas.

System automation

- Acquire large area images that preserve the context of your observations
- Easily set up multi-resolution acquisition on a single or multiple samples
- Make the most of overnight or weekend microscope usage

Correlative microscopy

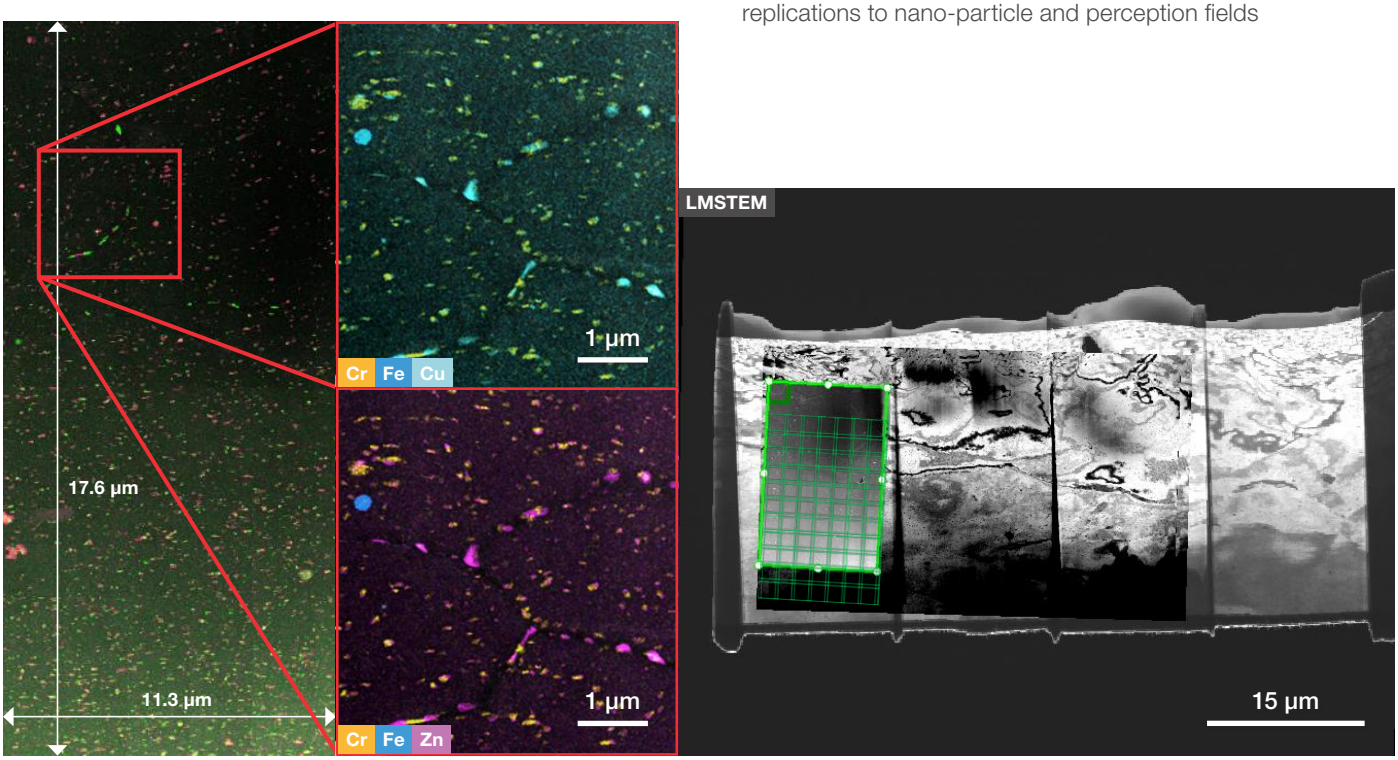
- Import imagery from any source
- Manage multi-scale, multi-modal data with ease
- Explore and interpret all your data efficiently and with context

Automated Particle Workflow

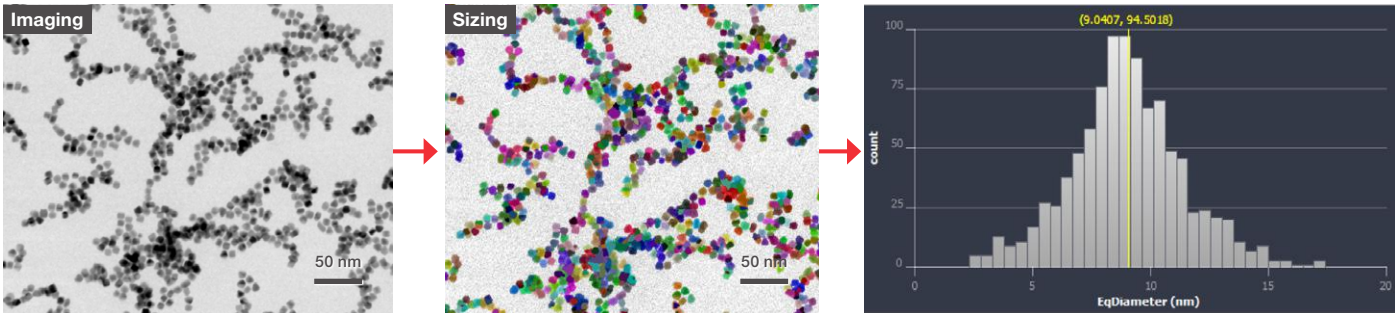
The APW Pack has all of the benefits described in the previous section about Maps software, and adds unique processing on a dedicated processing PC with Avizio2D Software. You can get nanoparticle parameters like size, area, perimeter, shape, factor, contacts, etc., in an automated way.

The fully automated and unattended software pack enables you to:

- Use the Talos 24/7
- Get better statistics (typically >500 particles/hour)
- Significantly improve the repeatability because the operator bias is not present
- Do easy and flexible recipe setup in Avizo2D to enable many replications to nano-particle and perception fields



Left: Correlated large area EDX map of precipitates in aluminum (acquired on the indicated area of the rightmost image) showing precipitate chemistry at the nanometer scale. Right: 70 x 30 micron Ga-free TEM section of precipitates in aluminum, prepared in the Thermo Scientific Helios PFIB DualBeam.
Sample courtesy of University of Manchester/University of Trento.



Automated Particle Workflow example: Pt-Rh particle analysis: size, area, perimeter, shape factor, contacts, etc.
Sample courtesy of Prof. B Gorman and Prof. R. Richards, Colorado School of Mines.

Talos X-TWIN lens for best-in-class (S)TEM resolution and analytical capability for all materials

The large X-TWIN pole piece gap (providing high flexibility for the widest range of applications) combined with a reproducibly performing electron column opens new opportunities for high-resolution 3D and *in situ* dynamic observations. The X-TWIN column is available on the 20-200 kV Thermo Scientific Talos F200X, F200S and F200i FEG (S)TEM. The Talos product line is equipped with the fast 4k resolution Thermo Scientific Ceta™ 16M Camera, which provides, in combination with drift compensated frame integration (DCFI) in TEM and (S)TEM modes, large field-of-view, fast, drift-compensated imaging with high sensitivity and precise sample navigation on a 64 bit platform. The Talos (S)TEMs also include the workflow-based Velox Software for intuitive acquisition and analysis. All these features combine to make the Talos product line the most productive (S)TEM system available.

Talos C-TWIN lens for high-contrast and cryo-TEM for soft matter

Two other members of the Talos family, the 20-200 kV Thermo Scientific Talos F200C FEG and the 20-120 kV L120C thermionic (S)TEM, have a very large C-TWIN pole piece gap making them powerful, versatile, high-resolution systems for 3D characterization of biomaterials, with a special emphasis on high-contrast and cryo-TEM imaging for cell biology and nanotechnology research. The large distance between the C-TWIN pole pieces also enables a large tilt range for diffraction applications as well as special sample holders. The C-TWIN lens is also available with a phase plate to enhance contrast even further.

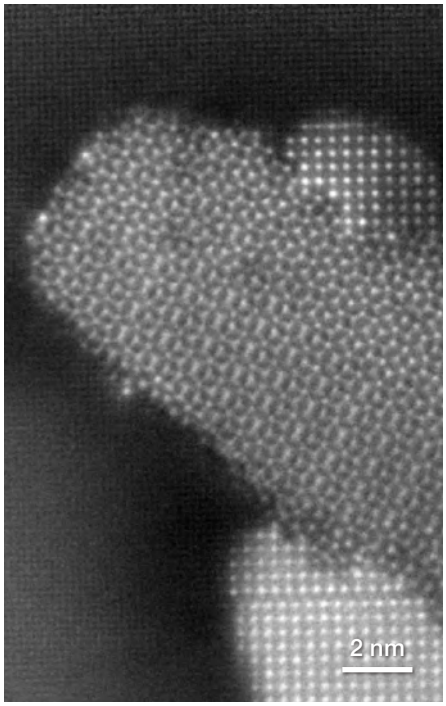
Easy to use – fast mode and high tension switching

Swift mode switches like HRTEM to HRSTEM are essential for high productivity in multi-user, multi-material environments. The constant power objective lenses and low hysteresis design, coupled with easy to use pre-stored settings, allow for straightforward, reproducible mode and high-tension switches. Simply loading a pre-alignment (called a FEG register) which contains the desired conditions required for a certain application allows immediate data collection.

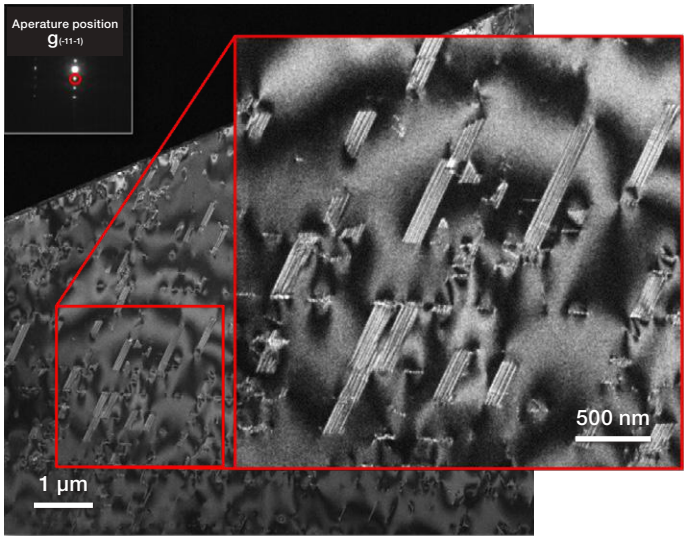
Its variable high tension, enabled by constant power objective lenses, allows for the widest sample variety. For example, switching from 80 kV to 200 kV is achieved via a single button click with the beam visible during the high-tension switch.

Fast reproducible tilting to zone axis

Thermo Scientific CrystalPack™ Software enables users to do compucentric ß tilting, rapid toggling between diffraction conditions and can automatically tilt to the relevant zone axes. The user can simply enter the zone axis and with a single mouse click the sample is tilted automatically and reproducibly to the desired zone axis.



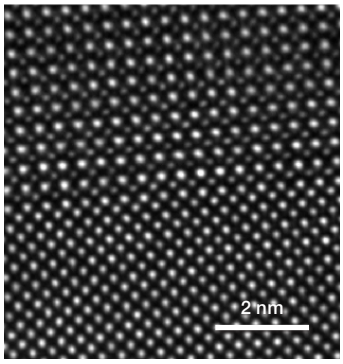
High-resolution DCFI STEM image of aluminum-zirconium alloy microstructure.
Sample courtesy TU Delft, the Netherlands.



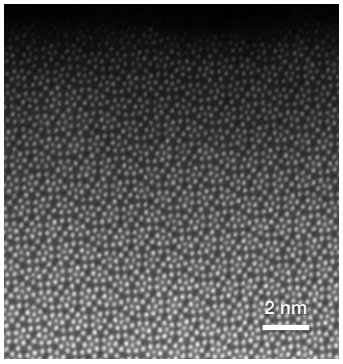
Weak-beam dark-field TEM of nickel alloy.
Sample courtesy Prof. G. Burke, Manchester University.

AutoSTEM Software for fast optimization on Panther STEM imaging

Panther STEM is the newest generation STEM engine with improved detectors and amplifiers. It has an optimized mechanical alignment and detector geometry for better multi-signal acquisition and mechanical alignment accuracy. It has a higher throughput and easier operation with linear response of gain/offset and more flexibility in signal processing. Visualize more details with up to 16 segments (for future applications) and a new amplifier design with ultrahigh electron sensitivity for low-dose STEM. Thermo Scientific AutoSTEM Software provides automated focus and astigmatism correction to reproducibly obtain best quality (HR)-STEM images with push-button operation. It simplifies HRSTEM and reduces the needed level of expertise. It works on crystalline areas of interest, simplifying tuning since there is no need to find an amorphous area, which is typically required for such routines.



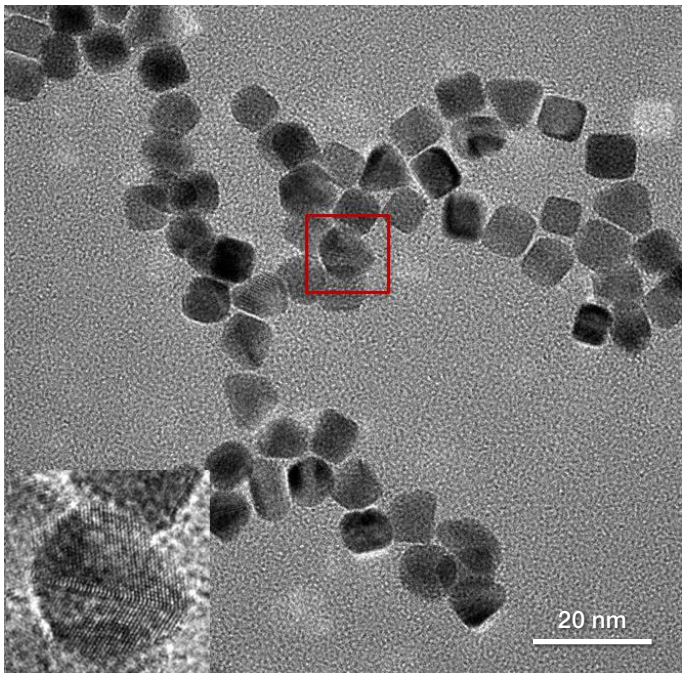
HRTEM image of $BaTiO_3/SrTiO_3$ interface. *Sample courtesy of Prof. Di Wu, Nanjing University.*



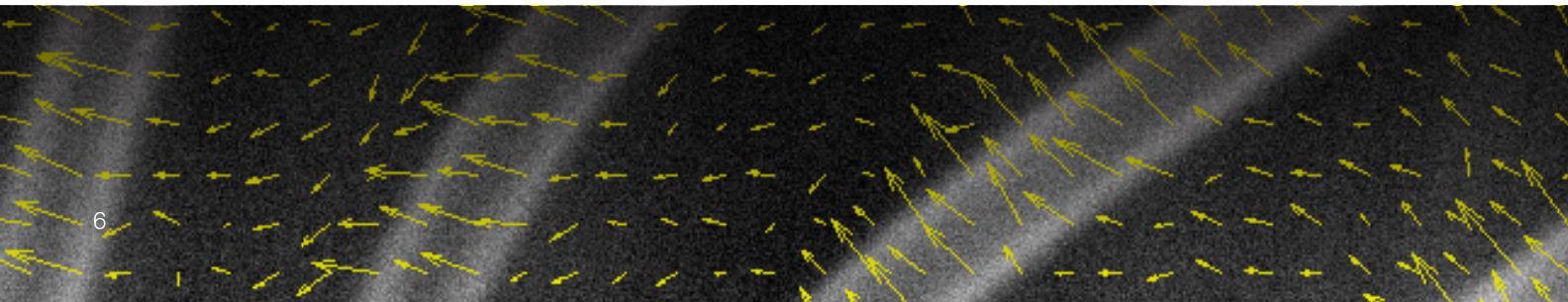
HAADF HRSTEM on potassium tungsten niobate.

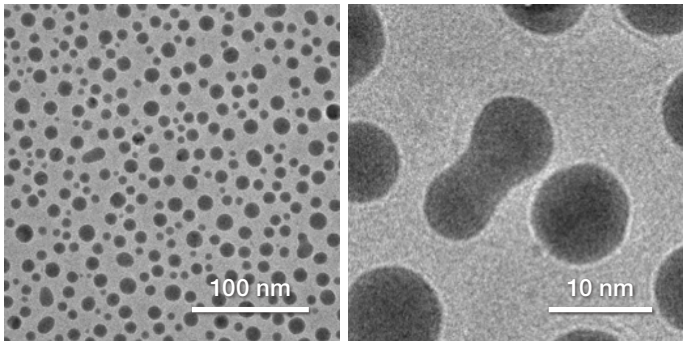
Imaging results

This image shows a 16 Mpixel acquisition capturing all information at once; atomic level details can be extracted using digital zoom. The 4k by 4k high-speed Ceta Camera with large field of view enables live digital zooming with high sensitivity and high speed over the entire high-tension range. In this example, platinum/rhodium catalyst nanoparticles are shown in a large field of view; digital zoom allows details to be extracted down to the atomic level. The embedded piezo-enhanced stage enables precise sample navigation and drift correction.



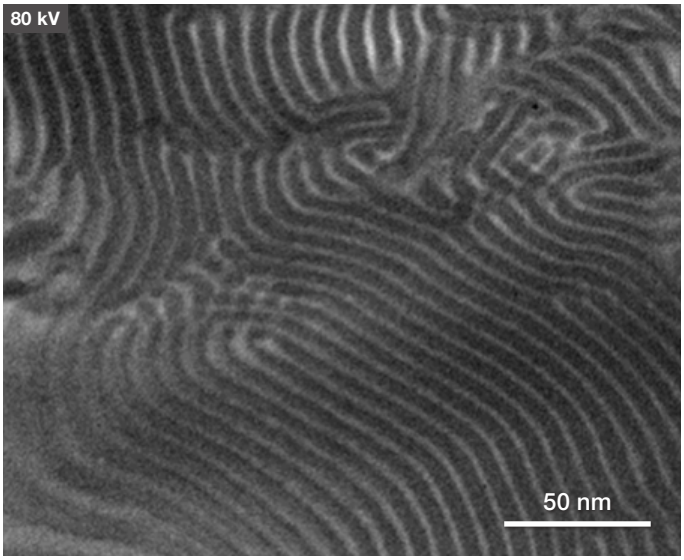
Platinum/rhodium catalyst nanoparticles. *Sample courtesy of Prof. B. Gorman and Prof. R. Richards, Colorado School of Mines.*



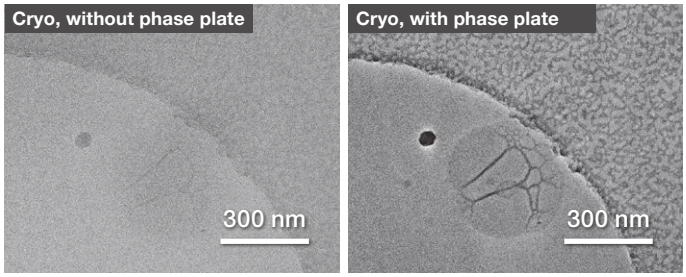


Gold nanoparticles coalescing at 1000°C with the NanoEx i/v MEMS heating holder, full 4k × 4k resolution image (left) with close-up (right).

The Ceta speed enhancement package enables recording of 4k by 4k field of view TEM images with 30 frames per second allowing all relevant information to be captured. Large field-of-view imaging enables the monitoring of multiple *in situ* events simultaneously, providing statistical information.



PS-PMMA block copolymer: Lamellar structure with PMMA units exhibiting dark contrast at 80 kV. *Sample courtesy: A.Prof. Kevin Jack and A.Prof. Idriss Blakey, University of Queensland.*



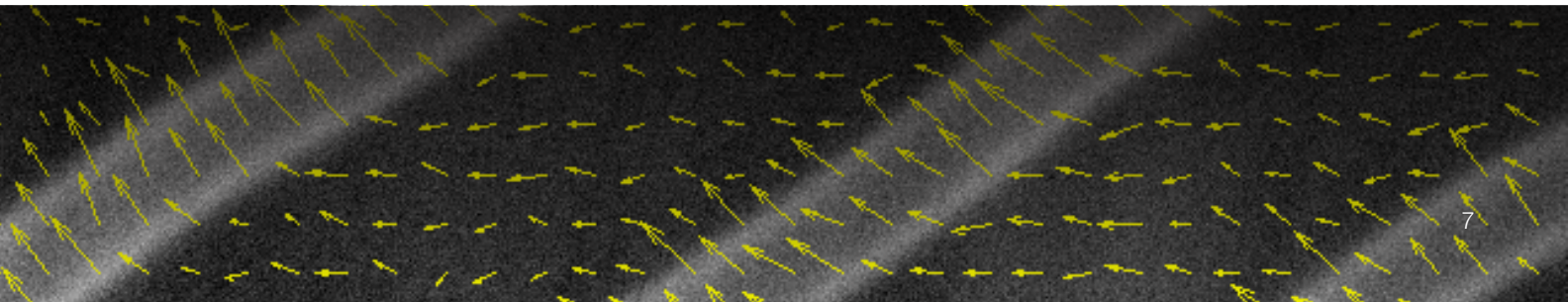
Polymer vesicles in water. *Sample courtesy: Institute of Molecular Genetics of the ASCR, v. v. i., Vlada Filimonenko, Pavel Hozak.*

High contrast imaging with C-TWIN lens at low voltage

One of the challenges in polymer TEM characterization is the rather low contrast between structural features. Various strategies are available to enhance the contrast, for example; sample staining or the use of lower beam energies.

Low HT imaging with the C-TWIN lens also enhances contrast significantly and has successfully shown the inner morphology of a co-polymer structure in detail (left). In this example, low-voltage TEM images reveal the morphology of an annealed polymer film made of the block co-polymer polystyrene-b-poly(methyl methacrylate) at 80 kV, showing a lamellar structure with light-contrast PS regions and dark contrast PMMA layers.

In order to enhance the contrast even more a phase plate is available on the C-TWIN lens.

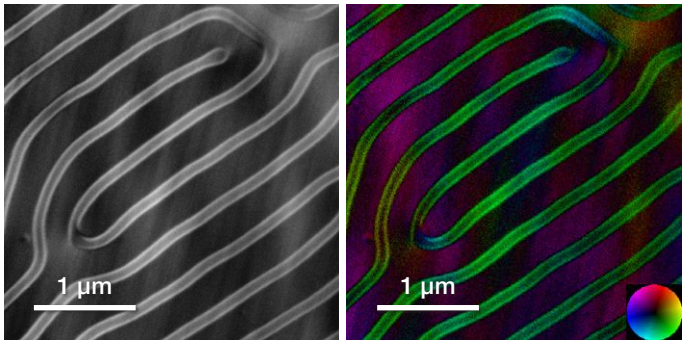


Differential phase contrast STEM imaging of magnetic structure

Differential phase contrast STEM imaging (DPC-STEM) is used to image the strength and distribution of magnetic fields in and around a sample, thereby directly displaying the magnetic domain structure. DPC-STEM uses the four segments of the dark-field detector. The signals are simultaneously acquired to measure shifts of the diffraction pattern. The Velox Software on Thermo Scientific TEM instruments has an option for live DPC acquisition fully integrated in the user interface.

Here we look at domain structure and domain walls (Bloch walls) in an M-type barium hexaferrite, which is a promising hard magnetic material for data storage devices. The relative strength and orientation of the magnetic field in each domain is visible in the map, and the variation in magnetic field across the domain wall indicates what type of wall is present, the wall thickness and observe defects and variation in the wall structure and how these would affect the overall performance of the material. Because the technique works with in-focus STEM images and provides quantitative information about the field strength as well as the field orientation, it is a powerful technique for visualizing magnetic structure.

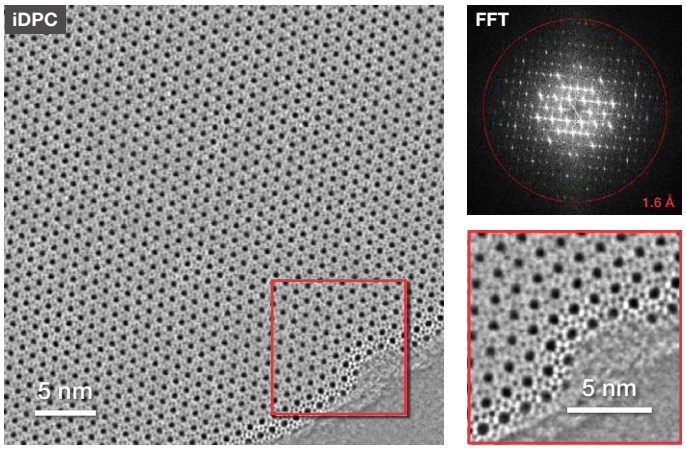
Various other visualizations are also possible to highlight different aspects of the magnetic structure.



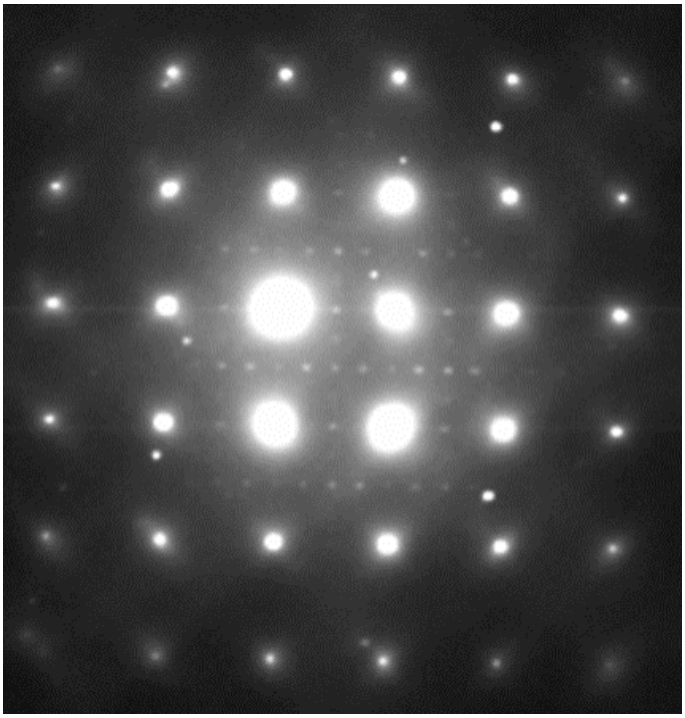
Domain structure and domain walls in an M-type barium hexaferrite. Different aspects of the magnetic structure can be highlighted, such as magnitude of the magnetic field at each point (left) or color wheel representation of field orientation (right). ***Sample courtesy H. Nakajima and S. Mori, Osaka Prefecture University.***

Low-dose high-contrast imaging with integrated differential phase contrast (iDPC) STEM

The innovative integrated differential phase contrast method delivers the best contrast and signal-to-noise ratio with extremely low electron doses. This enables imaging of beam sensitive materials containing light elements at atomic resolution. The example to the right is taken on a zeolite sample with a beam current of only 2 picoamperes while still retaining a resolution down to 0.16 nanometers.



iDPC image of a zeolite sample (left), fast Fourier transform (top right), iDPC image of zeolite with digital zoom (bottom right).

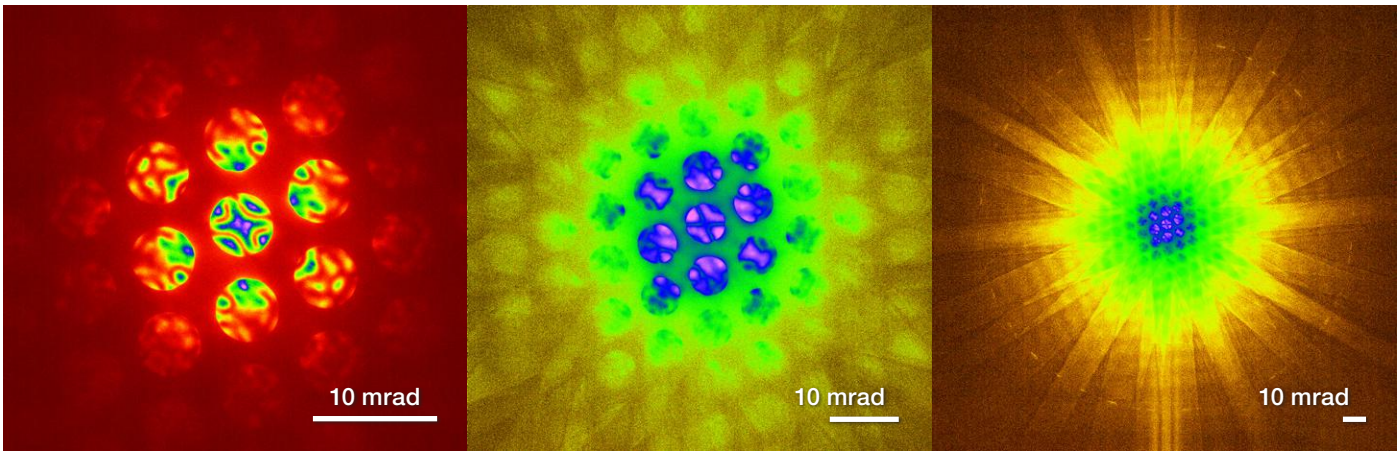


Selected area diffraction on aged super-alloy Inconel 625. Only in the [100] zone axis orientation of the matrix material is the structural information of small precipitates visible as weak reflections. *Sample courtesy Prof. G. Burke, Manchester University.*

Diffraction results

Selected area electron diffraction is a complementary technique to X-ray diffraction when structural information on nanocrystalline materials is required. The high dynamic range of the Ceta Camera enables detection of these weak intensities next to the strong signal of the base material (see example to the left).

The flexible condenser system of Talos (S)TEMs allows the formation of a wide range of convergent beams. Convergent beam electron diffraction (CBED) can reveal the full 3D symmetry of a crystalline sample and can enable crystallographic point group determination and thickness measurements.



CBED patterns on silicon [011] acquired with different camera lengths, showing the flexibility of the Talos F200 (S)TEM and the superior dynamic range of the Ceta 16M Camera.

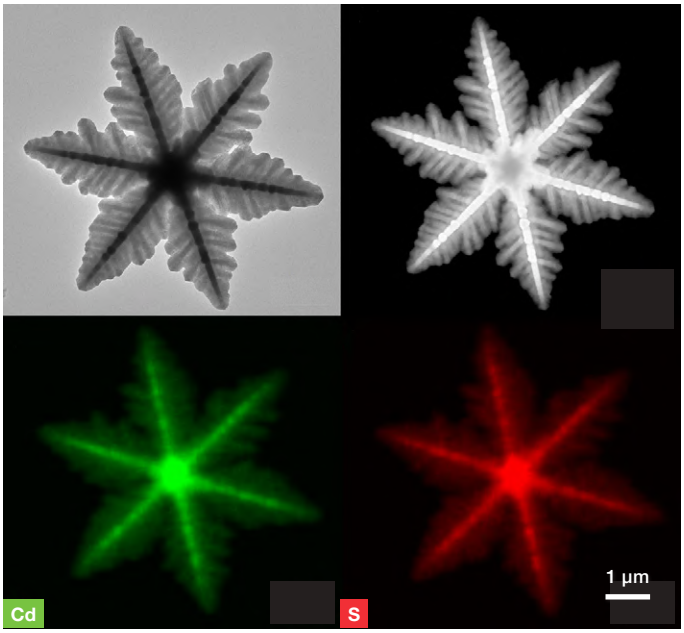
Spectroscopy results

Side-entry retractable EDS detectors can be added to the instrument configuration to enable chemical analysis. The Talos (S)TEM can be configured with a wide range of EDS solutions, ranging from single 30 mm^2 or 100 mm^2 to symmetrical large dual 100 mm^2 racetrack ("Dual-X") to in-column Super-X detectors. They are all fully embedded in Velox Software to enable unique absorption correction for accurate quantification. Super-X and Dual-X Detectors with Velox Software also enable automated EDS tomography.

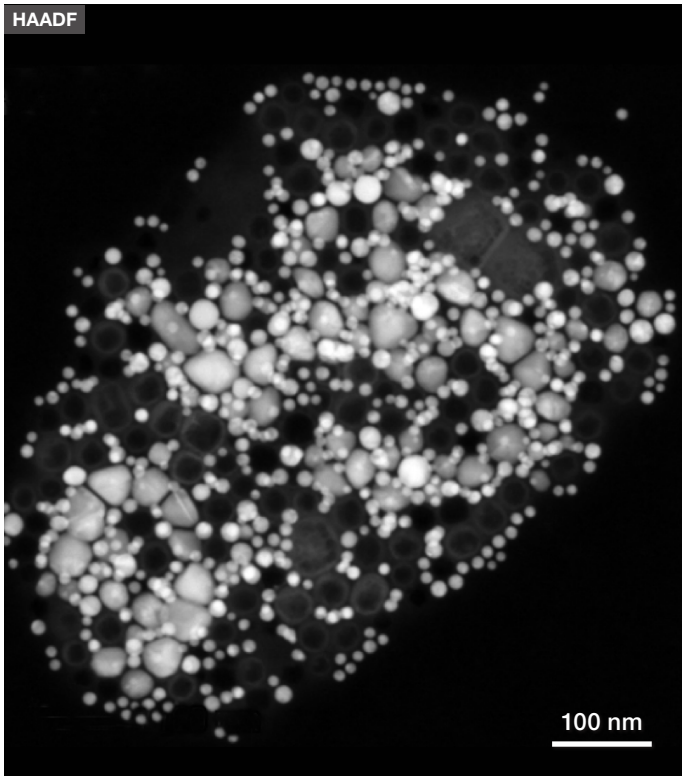
The Super-X EDS with 4 symmetrically positioned windowless SDDs enables fast access to the highest quality spectra. In compositional mapping applications EDS is the easiest technique to visualize elemental distribution. The high collection efficiency combined with minimization of unwanted system signals, like spurious system peaks and high background contributions, are necessary for compositional identification with high lateral resolution.

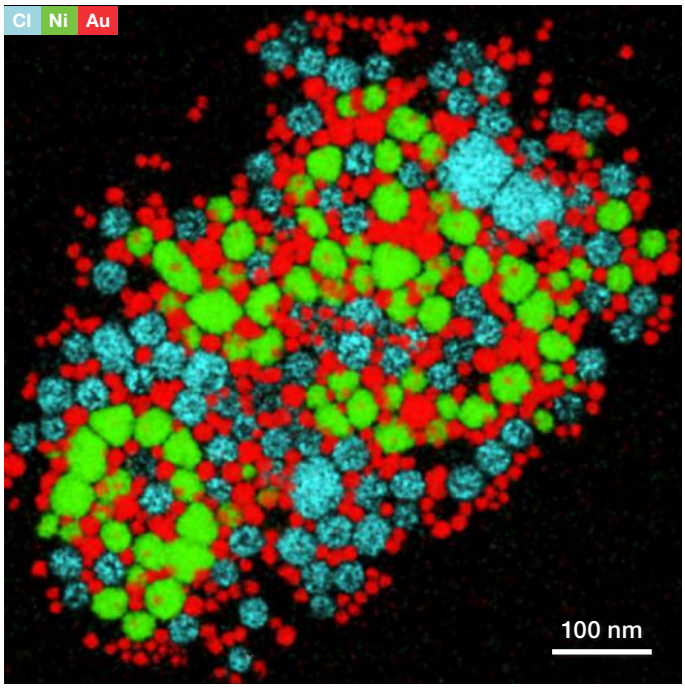
Super-X and Dual-X Detectors are optimized for materials science and have a low sensitivity to tilt, delivering an enhanced chemical signal under all tilt conditions. The unique Velox absorption correction scheme takes into account the detector geometry (no longer 1 take-off angle) and the holder geometry (shadowing changes dramatically with sample tilt). As a result the chemical composition data are consistent across the entire tilt range.

The Dual-X Detector also enables chemical analysis on beam sensitive material.

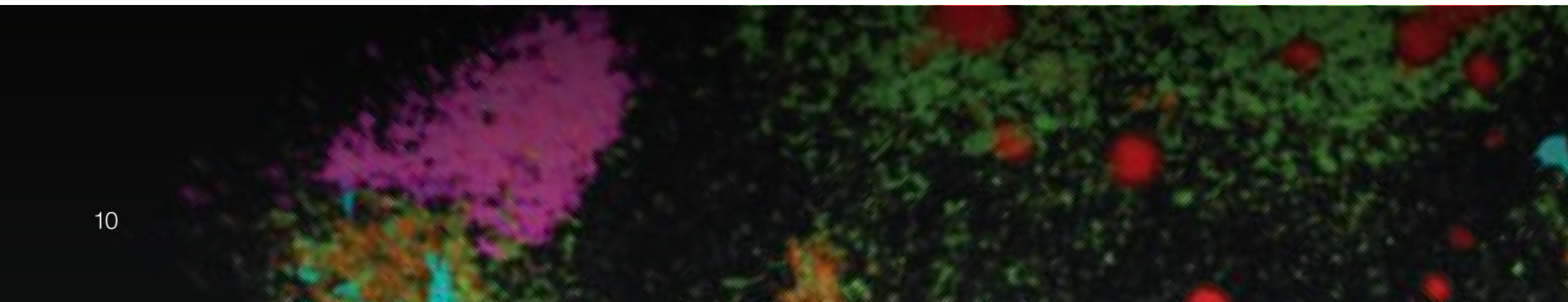


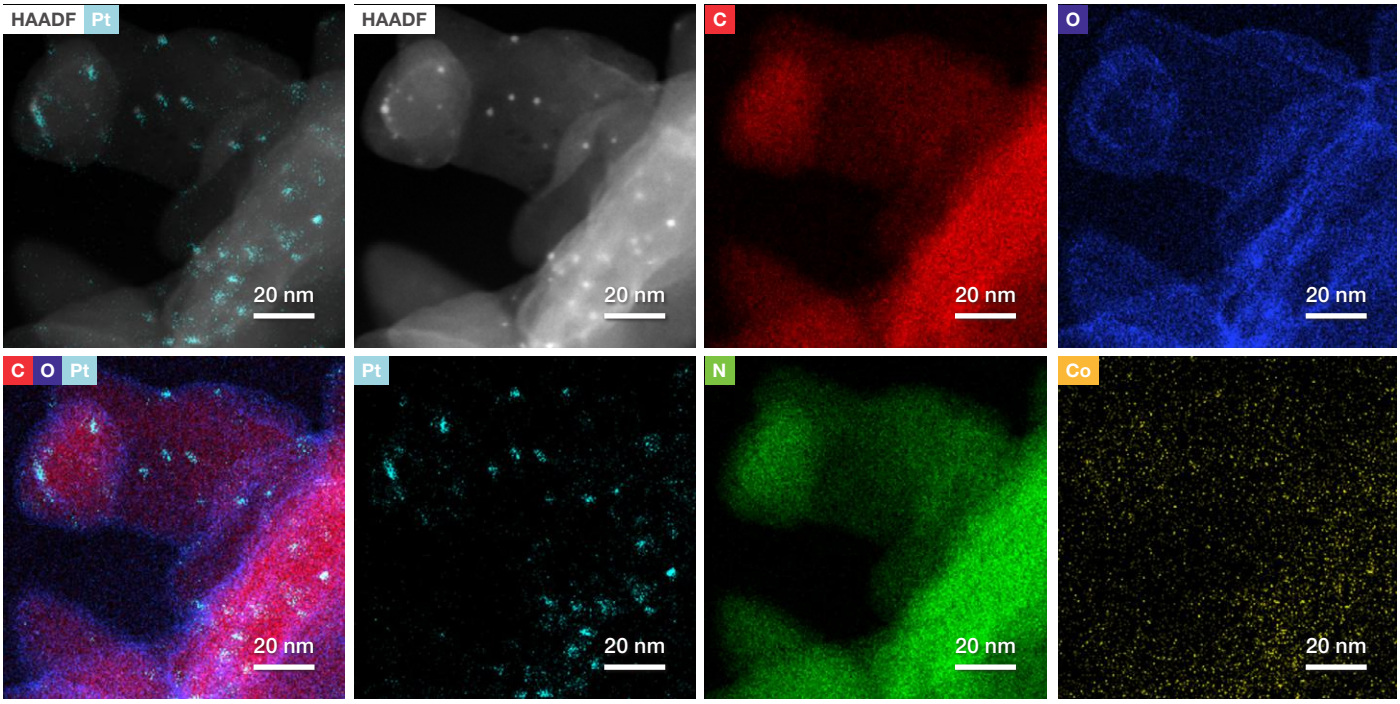
Cadmium sulfide sample, mostly used for optical applications, analyzed with side entry EDS. *Sample courtesy Prof. Li Haidong, Qingdao University.*



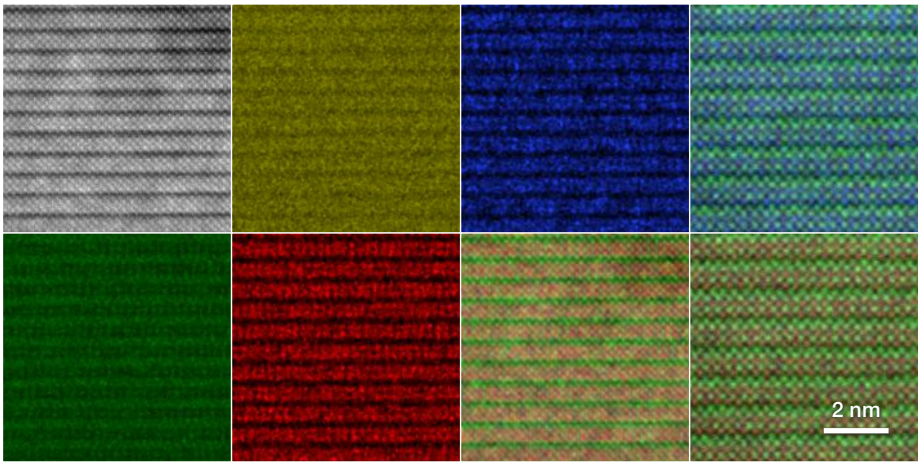


Example of large-area, high-resolution EDS mapping with Dual-X on gold-nickel nanoparticles, acquired in less than one minute. *Sample courtesy: J. Bursik, Institute of Physics of Materials, Brno.*





Example of high-resolution EDS mapping of beam-sensitive material used for photocatalytic hydrogen evolution. The small nanoparticles (Pt) act as active sites for the photocatalytic reaction. These nanoparticles are studied to observe the structure of Co-g-C_3N_4 loaded with Pt nanoparticles and find the relationship between the position of Co atoms and Pt nanoparticles to analyze the reason for improved photocatalytic activity of this sample. *Sample courtesy Prof. ShengChun Yang, Xi'an Jiaotong University, China.*

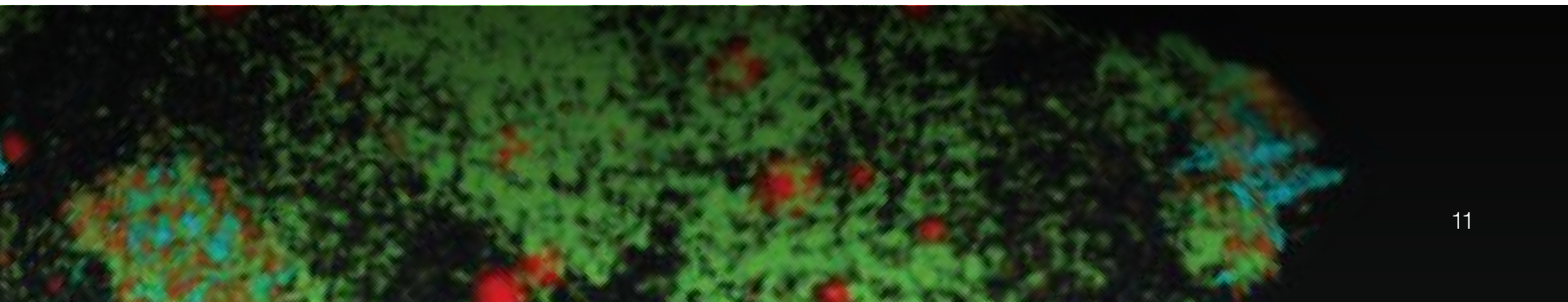


Atomic resolution EDS Mapping on SrRuMnO. *Sample courtesy of Professor Rongying Jin, Louisiana State University, with funding by the NSF.*

$Sr_3(Ru_{0.84}Mn_{0.16})_2O_7$

Atomic EDS

The unique design of the Super-X and Dual-X Detectors even enables the acquisition of atomic-resolution EDS maps, formerly only possible with Cs corrected tools, especially if the high-brightness X-FEG gun is added to the configuration.



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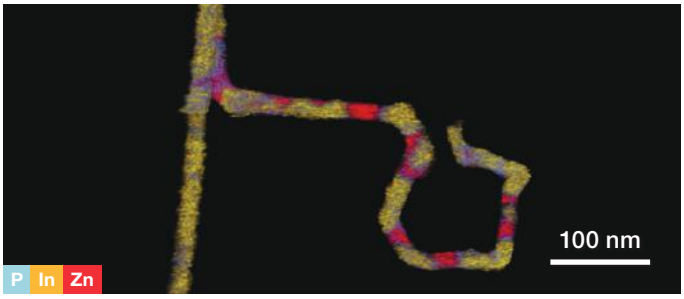
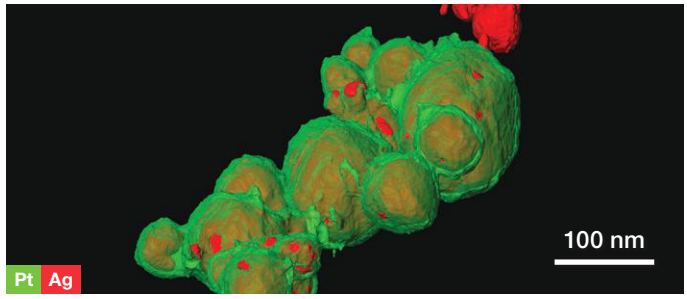


P-Zn-In nanotubes: This example shows nanotubes used as electrode material for sodium-ion and lithium-ion batteries. The segregation of zinc is not very well known during the synthesis. The elemental maps and EDS tomography, however, very clearly reveal the distribution of the zinc relative to the other elements. It also becomes evident that there is almost no zinc in the straight nanotubes. *Sample Courtesy of Dr. Reza Shahbazian Yassar, Michigan Tech University.*



Ag-Pt core-shell EDS tomography study of silver-platinum core-shell nanoparticles with elemental resolution down to a few nanometers. Silver cores are shown in red, covered by green-colored platinum shells, only a few nanometers in thickness. The EDS tomography technique makes visible the pores that partially expose the cores. In this use case, it was of great importance to determine if the platinum layer was continuous or possessed pores. This determination could not be made from a single 2D EDS map; therefore, EDS tomography in three dimensions was employed. *Sample Courtesy Prof. Yi Ding and Prof. Jun Luo, Center for Electron Microscopy, Tianjin University of Technology.*

EDS tomography

The geometries and positioning of the Super-X and Dual-X EDS systems are ideal for providing the best 3D chemical composition information at high lateral resolutions – an essential application for any materials science lab. We offer a fully automated and comprehensive EDS tomography application package including the tomography holder, the symmetrical Super-X/Dual-X Detector, the software to acquire and analyze tilt series automatically and our Thermo Scientific Inspect3D Reconstruction Software and Avizo Visualization Software.

Thermo Scientific Automated EDS Tomography Software enables fast and easy setup with flexible conditions for optimizing throughput and data quality. A user can set up tilt range and scheme, increment steps and choose which detector(s) to use. The auto-functions in the Tomography Software allow for fully automated acquisition, which in turn allows unattended data collection, particularly useful for overnight experiments.

Comparison table of all Talos models:

	Talos F200i			Talos F200S	Talos F200X	Talos L120C	Talos F200C
Pole piece	X-TWIN					C-TWIN	
Gun type	S-FEG	S-FEG	X-FEG	S-FEG	X-FEG	LaB_6/W	X-FEG
Gun brightness at 200 kV [A/cm²/sr]	$4.0 \cdot 10^8$	$4.0 \cdot 10^8$	$1.8 \cdot 10^9$	$4.0 \cdot 10^8$	$1.8 \cdot 10^9$	na	$1.8 \cdot 10^9$
EDS type / area	Side-entry 30 mm²	Side-entry 100 mm²	Side-entry Symmetrical Dual-X (200 mm²)	In-column 2SDD Super-X (60 mm²)	In-column 4SDD Super-X (120 mm²)	Side-entry 30/100 mm²	Side-entry 30/100 mm²
EDS net solid angle [sr]	0.19	1.28	1.65#	0.45	0.90	0.18/0.80	0.18/0.80
Velox absorption correction for most accurate quantification	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Automated/unattended 3D EDS	No	No	Yes	No	Yes	No	No
Maps / Automated particle workflow	(S)TEM and EDS	(S)TEM and EDS	(S)TEM and EDS	(S)TEM and EDS	(S)TEM and EDS	(S)TEM and EDS	(S)TEM and EDS
Cleanest EDS; Fiori P/B (on system)				highest			
HRTEM info limit	0.12					na	0.18
HRTEM line resolution	0.10					0.20	0.10
HRSTEM [nm]	0.16					1.0	0.20
Align Genie	Yes					Not yet	Yes
CrystalPack, iDPC	Yes					Yes	
AutoSTEM	Yes					No	Yes
Enclosure	Compact			Big with touch screen	Big with touch screen	Compact	Big with touch screen
Cryo quality						best	
TEM contrast						highest	
Phase plate	No					Yes	

#=the geometric angle is 2.55

Find out more at **thermofisher.com/talos**

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