

NCFL's FEI Titan 300

Features and Specifications

- 300kV X-TWIN corrector platform (currently software-limited to 200kV)
- STEM system for FEG configuration
- Scanned Image Technique
- HAADF Detector
- EDX Spectroscopy Technique
- EDAX Digital Pulse Processor
- r-TEM retractable SUTW Detector Unit for Titan X-TWIN (0.3 Sr collection angle)
- Xplore3D
- STEM Tomography data acquisition software (Inspect3D)
- TrueImage Professional
- EFTEM Technique
- EFTEM EELS Module
- Gatan Model 863 Tridiem GIF
- Gatan 794 MUltiScan Camera (EFTEM): <200 kV/YAG/B/1 K
- Low-Dose Exposure Technique
- Lorentz lens for 300 kV X-TWIN
- Titan k-Space Control
- Titan Smart Tilt
- Titan Compucentricity
- Titan Free Lens Control
- CompuStage Double-Tilt Specimen Holder
- CompuStage High-Visibly, Low-Background, Double-Tilt Specimen Holder
- CompuStage Cold Stage, Double-Tilt Specimen Holder
- Tomography Single-Tilt Specimen Holder

New Column The Titan consists of a state of the art support frame, which supports a revolutionary new column design. The diameter of the new column is larger, which gives extra room for optimizing the electron optical performance, and novel new designs are aimed at eliminating the effects of cross talk and interference, which was becoming a limiting issue with the existing column designs. The column is also designed in a modular fashion, allowing easier modification and tailoring to highly specialized application requirements. The lens modules themselves no longer perform supporting tasks anymore, making the alignment of the column more reliable. This design in addition allows for a stepwise approach to corrected microscopy. The Titan system, when originally delivered without correctors, can be upgraded in the field with a single corrector. This is unique. All lenses are based on the ConstantPower design for ultimate stability, high controllability and reproducibility: allowing switching of modes, magnifications, conditions and techniques to be fast and with minimized stabilization time. Unique SmartOptics — real-time modeling of the optical characteristics of the microscope, for each and every actual microscope setting — guarantees maximization of performance, control and reduction of alignments and tuning. A 3 condenser system

in combination with SmartOptics extend the experimental parameter space, greatly and smartly: illuminated area can be controlled at will, while keeping the beam current constant (TEM); coherence can be varied, while keeping the illuminate area constant (TEM); convergence angle can be controlled while keeping the beam current constant (STEM); probe size can be set, while keeping the convergence angle fixed (STEM). Importantly, this control and flexibility is all available without much need to change condenser apertures: all resulting in flexibility at ultimate stability. The Titan 300 kV platform can be operated over a large range of acceleration voltages (currently from 80 kV up to 200 kV).

The column contains a field emission gun with a factory-aligned Schottky field emission and isolated vacuum system for the gun, three condenser lenses, the X-TWIN objective lens with a computerized 5-axes eucentric goniometer, diffraction lens, intermediate lens and two projector lenses and a housing for cameras, energy filters and other accessories. All apertures are fully computer controlled and motorized. Alignments and positions are automatically recalled. The control includes a motorized mechanism, the electronics and the software needed to fully automate the use of the apertures.

Vacuum System The vacuum system ensures a clean, oil-free, high-quality vacuum throughout the column, with special attention being paid to the specimen and emission chambers. The vacuum in the projection chamber is separated from the high vacuum in the column by a differential pumping aperture. A cooling device with cold trap and liquid nitrogen Dewar is fitted in the objective block to maintain the cleanliness of the vacuum, even under critical conditions such as observation of specimens at low temperatures. A cryo-cycle option is included, which provides an automated routine allowing the column to be pumped by a turbo molecular pump. The result is a very clean column and vacuum system. An automatic vacuum valve between the gun and the column is present to allow the high tension and emitter to remain on, even when the sample is exchanged.

Control of the Microscope Control of the microscope and all its accessories is through one common graphical user interface operating under Windows XP. For complete control of all microscope functionality, logical combinations of controls are grouped on two movable control panels. Some of these controls are user-definable in order to enhance throughput and usability. The microscope desk is ergonomically designed to provide essentially two workspaces: one in front of the column for normal transmission microscopy and one in front of the display monitor(s) for data management.

HAADF Detector for 200 kV The High-Angle, Annular Dark Field detector (HAADF) is used in TEM/STEM systems to generate (atomic resolution) dark-field STEM images. This HAADF detector fits into the wide-angle port (below the lowest lens and above the projection chamber) of the microscope.

Detector dimensions: The YAP is fixed to an aluminum-coated quartz tube, which makes, at the detector end, an angle of 45 degrees with the electron beam. In the plane perpendicular to the beam, the inner diameter of the hole is 4 mm and the outer diameter is 20 mm.

Construction: the detector can be retracted pneumatically, with the possibility of mechanical alignment to center it around the electron beam. The quartz tube links the YAP to the PMT, which is mounted directly onto this tube. The re-positioning accuracy after retraction and reinsertion is < 0.1 mm.

EDX Spectroscopy Technique The EDX Spectroscopy Technique provides all hardware and software required for both qualitative and quantitative EDS analysis as well as EDS spectrum imaging. This module operates in conjunction with the EDX pulse processor, EDX detector and Techni Imaging and Analysis modules. The EDX Spectroscopy Technique module imports data from the EDX Pulse Processor into the Tecnai Imaging and Analysis (TIA) data acquisition and handling system, communicating with the EDX Pulse Processor via an interface card in the main workstation.

It provides all of the functionality necessary for qualitative and quantitative EDX analysis:

- it acquires spectra directly from the EDX detector
- Allows live viewing of spectra, fixed-length acquisition using presets, and acquisition of time-series of spectra
- Qualitative analysis features include automatic peak identification and manual identification using the Periodic Table
- Quantitative analysis features nonlinear peak fitting for deconvolution, and quantification using standard or standardless Cliff-Lorimer k-factors including specimen thickness corrections
- When combined with the Scanned Imaging Module, gives full EDX spectrum imaging and profiling capabilities, including acquisition with compensation for specimen drift. An unlimited number of elemental maps or profiles can be extracted from any spectrum image or profile
- The EDAX Digital Pulse Processor is an acquisition module for collecting and digitizing the x-ray signal input from the Si(Li) detecting unit. It includes the acquisition boards and high voltage bias supply in a mini-tower housing cabinet. Also included is the r-TEM motorization board, suitable for r-TEM EDAX detectors. The digital pulse processor is capable of handling output count rates up to 50 kcps, selectable processing times (0.4 microseconds to 102.4 microseconds) and three eV/channel selections (5, 10, and 20).

r-TEM SUTW Detector Unit Motorized retractable Si(Li) type TEM Detecting Unit with Super Ultra Thin Window for detection of all elements down to and including beryllium. Resolution of 136 eV or better, measured at MnK α , 1000 cps and with the time constant 100 μ s. Peak to background is 18,000:1 or better as measured with a Mn γ -source. Active detector area is 30 mm². The take-off angle is 25 degrees and the solid angle is 0.30 steradians; the design results in no significant spurious system peaks present in the spectra. The crystal is automatically protected against warm-up.

EFTEM EELS Module An Gatan Image Filter (GIF) energy filter is positioned below the viewing chamber. It enables elemental mapping, zero loss and non-zero loss filtering as well as spectroscopy. The software is integrated into the main User Interface, through which all main functions on the filter can be controlled. EELS and CCD spectrum imaging is available. A second CCD (Gatan 794 MultiScan camera) is mounted below the projection chamber and above the GIF to enable digital imaging for non-EFTEM applications. The EFTEM EELS Module operates in conjunction with the Tecnai Imaging and Analysis module and imports data from the Gatan Energy Mer into the TIA Tecnai Imaging and Analysis Software.

It provides all of the functionality necessary for qualitative and quantitative Parallel EELS analysis:

- It acquires spectra directly from a Gatan Imaging Filter;
- It allows live viewing of spectra, summed acquisition, and acquisition of time-series of spectra. Peak identification is performed using the Periodic Table;

- Quantitative analysis features include background modeling using several models and quantification using hydrogenic cross sections;
- When combined with Scanned Imaging Module, gives full Parallel EELS spectrum imaging and profiling capabilities, including acquisition with compensation for specimen drift. An unlimited number of elemental maps or profiles can be extracted from any spectrum image or profile including full analytical background subtraction for each spectrum in the series. It allows simple viewing and recall of spectra from any position in the spectrum image or profile.

Parallel EELS Spectroscopy Capabilities

- Spectral display — no software limit on number of spectra, energy cursors, or energy windows per display or number of display windows; all display parameters (colors, styles, etc.) user-definable;
- Peak annotation/labeling (KLM markers);
- Graphical or manual energy calibration;
- Analytical background fitting using various supplied analytical models;
- Quantitative analysis using standards;
- Data export to spreadsheets for user-defined processing;
- Data storage in portable formats for import into other software packages;
- User-selectable mode - immediate/view; add/average;
- Continuously-adjustable dwell time and preset time (0.025 – 1000 s);
- Beam current can be recorded during series acquisition.

The EFTEM EELS module additionally provides the following Parallel EELS Mapping capabilities:

- Real-time display of processed elemental maps and line profiles;
- Any portion of raw spectral data can be stored at each scan position (spectrum images) for postprocessing;
- Imaging signals and spectroscopic signals from all detectors can be acquired synchronously.

Gatan GIF Tridiem The GIF Tridiem is Gatan's 3rd generation of post-column energy filters. It combines 3rd-order spectrometer aberration correction with a multi-port, high-speed, high-resolution CCD sensor to yield a system that captures highly-detailed EELS and EFTEM data sets with high throughput. This GIF combined with its associated software can generate rich data sets including EFTEM and EELS STEM spectrum images, EFTEM tilt and tomography series, and time series. The key features of the GIF Tridiem system include:

- Large collection semi-angle for energy-filtered diffraction: 120 mRad, full azimuth
- Large field-of-view for energy-filtered TEM imaging and mapping: 20 μm , diagonal
- Excellent isochromaticity over the entire EFTEM field-of-view: deviations ≤ 1.25 eV
- Very large searchable field-of-view in unfiltered TEM mode: >200 μm , diagonal
- "cinema mode" readout for smooth, high-quality real-time viewing: > 10 frames/sec
- Rich EELS detail with each spectrum readout: >2000 channels (e.g. capture 600 eV range at 0.3 eV/channel)
- High spectrum readout rate for STEM spectrum imaging: >30 spectra/sec

The GIF Tridiem is optimized for TEM beam energies from 200 to 300 keV and, as is true for all Gatan post-column energy filters, is attached to the bottom flange of any modern TEM to form energy filtered images and diffraction patterns, as well as electron energy loss spectra. The post-column design provides maximum flexibility in the choice of imaging optics (objective lens, correctors, TEM and STEM mode). Furthermore, Gatan's patented quadrupole optics ensure that spectra are always aligned with the detector, can be captured with a broad range of dispersions, and are optimally coupled to a rectangular area of the CCD detector to guarantee the best dynamic range and sensitivity possible.

The electron optics of GIF Tridiem fully correct focusing aberrations of the prism through second order and also correct the most important third order prism aberration. This advance permits the use of a large 5 mm aperture at the entrance of the spectrometer, giving the GIF Tridiem a large field of view. Distortions and resolution are excellent across the entire field of view. Distortion is below 1.75%, chromaticity is less than $1.5 \mu\text{m}/\text{eV}$, and deviations from perfect isochromalinity are below 1.25 eV in magnitude over the entire image field.

The GIF Tridiem comes standard with a 2K x 2K UltraScan 1000 FT (Frame Transfer) camera as its primary detector. This 4-port readout camera is 2 times faster than the 1K x 1K MSC of the GIF 2001 and 4 times faster than the 2K x 2K MSC of the GIF 2002 for imaging applications. The new detector is particularly advantageous for EELS data acquisition, offering truly live viewing of full spectra collected across the entire 2048-channel extent of the CCD. The GIF Tridiem sensor also offers a frame-transfer "cinema" mode that provides a high-quality live display for specimen searching and EFTEM setup. This same mode can be harnessed for rapid spectrum readout, 5-10 times faster than previous GIF models, which is especially beneficial for STEM spectrum imaging.

Xplore3D Xplore3D is FEI's software for 3D imaging (by means of electron tomography). The Data Acquisition Software provides a user-friendly and fast way for the acquisition of tilt series for three-dimensional reconstruction. In brief, a sample is tilted along a single axis over a large angular range (typically +/- 70 degrees) with small angular tilt increments (typically 1 degree) and an image is recorded at every tilt angle.

High Field-of-View Tomography Holder Designed specifically to provide high tilt capabilities in narrow gap objective lens pole pieces at room temperature, this holder can tilt from -80 degrees to +80 degrees in the S-TWIN pole piece. It easily fits in the TWIN lens pole piece with or without cryo-shields/box. By minimizing the shadowing associated with most holders at high tilt angles in tomography experiments, the holder's low profile and unique specimen damping mechanism extends the visible area to 7.5 times of what is achievable with FEI's regular +/-70 degree single-tilt tomography holder. Within the range of -70 degrees to +70 degrees, the specimen area that is visible is approximately 2 mm along the tilt axis and 1.5 mm perpendicular to the tilt axis. The damping mechanism is a set of two spring-loaded clamps that are tightened and released by Philips head screws.

STEM Tomography Data Acquisition Software The tomography software automatically determines the optimum acquisition position, at which sideways displacements and focus changes caused by the tilting are minimized. At this optimized position, the behavior of the holder (shift and focus changes) is calibrated (by calibration) and the result is stored. Hence, images are acquired with

minimum changes in shift and focus. Depending on the required accuracy, additional tracking of image shift and focus can be done at user-defined intervals. Use of the holder calibration strongly reduces the typical tilt-series acquisition time to around 25 minutes (on a 1K camera for a series from -70 degrees to +70 degrees in 1 degree intervals).

Customer Hardware Specification after Holder Calibration At a magnification corresponding to a field of view or $2\ \mu\text{m}$ at zero degrees tilt, the residual displacement between individual images of an acquired tilt series (performed after a proper holder calibration but without tracking), may not exceed 20% of the total field of view. In addition to this new hardware calibration, the software is capable of compensating for residual (hardware-related) displacements. A calibration is typically carried out in ca. 30 minutes; recalibration of the holder only needs to be carried out approximately once every four weeks.

Inspect3D The software package Inspect3D provides a user-friendly method for aligning a tomographic series of images, 3D reconstruction of the data, as well as full visualization functionality. For visualization purposes, the software package Amira is included, which is an advanced visualization and data analysis tool. Inspect3D involves an x-y alignment, which is based on image cross-correlations. These cross-correlations can be adjusted by using extra tapering, a Hanning window, a Sobel filter and a band pass filter. The alignment of the tilt axis can be carried out interactively. The resulting reconstructed volumes can be read into the software package Amira (version 3.0) via a proprietary plug-in for further analysis. The plug-in allows for importing the Inspect3D output into an Amira-compatible format. The plug-in, which was developed by FEL, as well as the software package Amira, are included with Inspect3D. Inspect3D can be run on a dual-processor system: the reconstruction algorithm can make use of multiple processors in parallel.

TrueImage TrueImage is a software package for obtaining directly interpretable results from HR-TEM beyond the point resolution. It reconstructs the electron wave function of the thin TEM sample, based on a series of high-resolution TEM images. The reconstruction is performed by a combination of linear, non-linear and maximum likelihood approaches. As a result of the reconstruction, the spherical aberration of the microscope is eliminated and therefore delocalization effects resulting from the FEG source are removed. The professional version of TrueImage allows for automatic correction of defocus and 2-fold astigmatism, and for manual correction of coma and 3-fold astigmatism. TrueImage (Professional) consists of two parts: (1) Focus Series Acquisition and (2) Focus Series Reconstruction. The package aids the user in determining all important acquisition and reconstruction parameters.

Low Dose Low-dose exposure technique helps to protect the sample from damage by the electron beam. Fast SpotScan is also available; this is a mode of operation where the beam is scanned in a serial fashion across the sample to reduce the total dose in the resulting image. The Low-Dose technique helps the microscope operator in minimizing the electron dose needed for the total sequence from searching for specimen areas, through focusing, to the final exposure. The Low-Dose technique has three states, Search, Focus, and Exposure, which have independent settings for various electron optical parameters such as spot size, Intensity and magnification. Switching between settings only requires a single button to be pressed.

- The Search state is typically employed at low magnifications or in defocused diffraction and with a low-intensity beam.

- Focus is typically executed at high magnifications but on an area that lies adjacent to the area of interest.
- The Exposure is automatically recorded under the proper conditions, started by pressing a single button and with 'high' (user-defined) intensity illuminating the specimen only during the actual exposure itself.

A beam blanker is used during Low-Dose state transitions and for the actual exposure. The beam blanker is also under direct user control. Each of the three states can make use of various media (viewing screen, CCD or TV-rate camera for Search and Focus; CCD for Exposure) with automatic switching to the required operational state (screen up/down, camera inserted/retracted, etc.). The Low-Dose technique includes the facility for recording through-focus series.

Lorentz lens for 300 kV X-TWIN Lorentz microscopy is based on the Lorentz force that acts on the electrons passing through a magnetic field. Because of the Lorentz force, the electrons leaving the specimen are deflected over a certain angle. The polarity of the magnetic domains in the specimen determine the direction of the deflection. So the deflection angle is a function of the magnetic properties of the specimen. Focusing the Lorentz lens allows imaging of magnetic domains with different properties. The basic requirement for imaging magnetic structures is a specimen environment free of magnetic fields. Such a magnetic-free environment can be obtained by switching off the objective lens and exciting the Lorentz lens. The operator can switch for focusing of the specimen between the objective lens and Lorentz lens. The focal length of the Lorentz lens is 23 mm.