

Titan ETEM G2

Environmental Transmission Electron Microscope for dynamic *in situ* exploration of functional nanomaterials and devices at the nanometer and atomic scale

Characterizing new, improved functional nanomaterials for energy and environmental technologies, requires a detailed understanding of their structure-performance relationships and atomic-scale insight into their geometric and electronic structures and chemical composition.

FEI's Titan™ ETEM G2 atomic-resolution Scanning/Transmission Electron Microscope (STEM) is an all-in-one solution for time-resolved *in situ* studies of dynamic behavior of nanomaterials during exposure to reactive gas environments and elevated temperatures. Built on the world class Titan TEM platform, which delivers the ultimate performance in mechanical, electronic and thermal stability, Titan ETEM is a flexible solution for imaging of static specimens, observations of nanomaterials' dynamic response to the applied stimulus (gas and temperature), observations of growth (kinetics) as well as function, reliability and breakdown studies of nanodevices. Titan ETEM can be combined with optional image Cs corrector, FEI's X-FEG module and monochromator technology to further extend it to meet the high standards in atomic-resolution S/TEM imaging and spectroscopy expected from FEI's Titan 80-300 kV technology.

Titan ETEM G2 features an innovative differentially pumped objective lens, uniquely designed for the ETEM platform. This lens design enables all the same features you would expect from a standard Titan S/TEM, like window-free imaging and compatibility with Titan heating holders for easy sample insertion, while also allowing ample chamber space for full double tilt capability to support 3-D tomography. Gas inlets allow operators to safely add inert and reactive gas to the chamber. Gas pressures in ETEM experiments can be accurately preset from 10⁻³ Pa up to 2000 Pa (for N₂). The new software-controlled user interface offers a range of settings to accommodate both handling by novice (automatic mode) as well as advanced (manual control) operators.

The ETEM is equipped with a mass spectrometer to determine gas composition either in the gas inlet system or in the specimen area. A built-in plasma cleaner allows for cleaning of the specimen area after using a gas. For safe and reliable use the Titan ETEM G2 features built-in hardware and software protections.

KEY BENEFITS

Observe functional nanomaterials' time-resolved (dynamic) response to gas and temperature stimuli *in situ*.

Study gas-solid interactions at the nanometer and atomic scale, including shape and morphology, and interaction at surfaces and interfaces.

Gain insight at the atomic-scale into the geometric and electronic structure, and chemical composition of functional nanomaterials.

Redesigned ETEM user interface and full software control of all operational parameters.

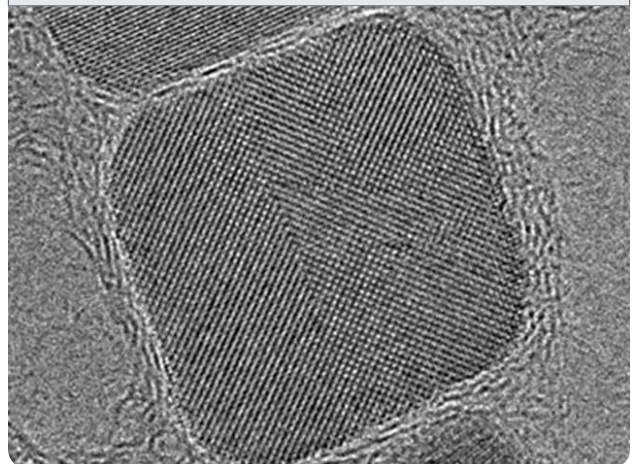
Rapid interchange between ETEM and high vacuum mode.

Large polepiece gap allows full tilt capability of the specimen holder for optimal orientation and electron tomography.

Compact and bakeable gas inlet system.

Linear gas flow regimen to minimize cross-contamination.

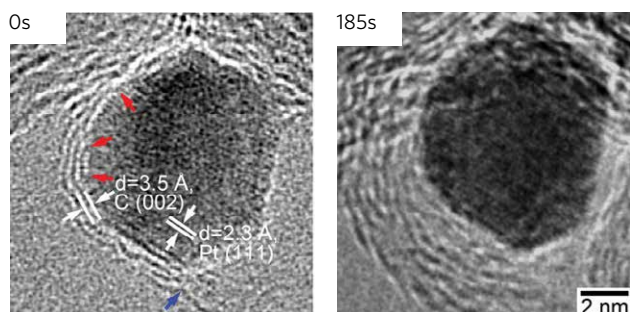
Built-in protection assures a safe working environment and safe use of flammable gases.



ETEM applications

Alkene producing catalysts: deactivation by carbonaceous layer growth

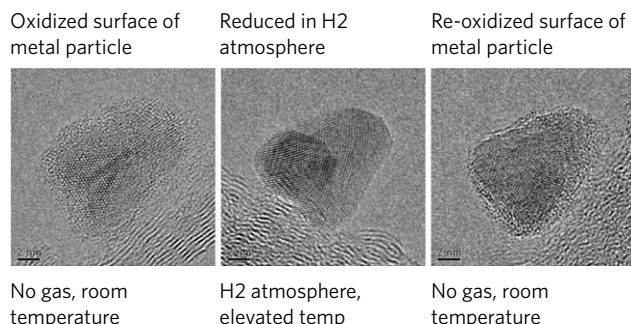
In situ: Haldor Topsøe A/S, FEI Titan ETEM (300 kV)



↑ Surface-step as growth centers for graphene on a Pt / MgO alkane dehydrogenation catalyst - dynamic *in-situ* ETEM study (1.3 mbar C₄H₈, 475°C) with atomic-scale resolution. Z. Peng, et al., *J. Catal.* 286 (2012) 22; Courtesy of A. Bell (UC Berkeley), C. Kisielowski (LBNL) & S. Helveg (HTAS).

Reduction and oxidation reactions over nanoparticle catalysts

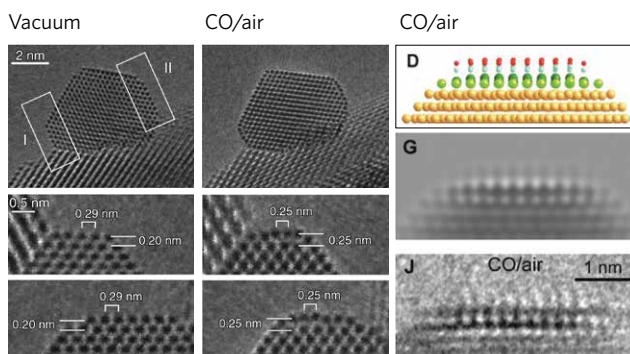
In situ: DTU Cen, FEI Titan ETEM (300 kV)



↑ Atomic resolution images of an oxidized metal nanoparticle undergoing reduction in hydrogen gas followed by subsequent re-oxidation. Courtesy of T. Hansen & J. Wagner, DTU Cen, and J. Nielsen. DTU Cinf.

Low-temperature CO oxidation catalysts—example: Au/CeO₂

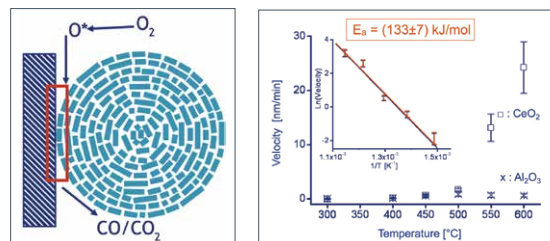
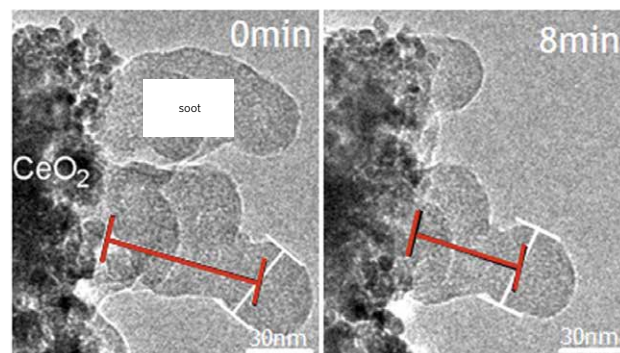
In situ: Osaka University, FEI Titan ETEM (80 & 300kV)



↑ Dynamic ETEM study (1 vol% CO in air gas mixture at 0.45mbar at room temperature) of carbon monoxide oxidation on Au/CeO₂. Visualizing of surface reconstruction and CO gas molecules interaction with surface of Au nanocatalysts at reaction conditions. H. Yoshida, et al., *Science* 335 (2012) 317; Courtesy of S. Takeda (Osaka University).

Diesel automotive exhaust clean-up catalyst role of CeO₂ surface in soot oxidation

In situ: Haldor Topsøe A/S, Philips CM300 ETEM (300kV)



↑ Dynamic ETEM study (2.0mbar O₂, 475°C) of soot oxidation near the soot-CeO₂ interface. Soot particles observed to move with constant velocity towards CeO₂. S.B. Simonsen, et al., *J. Catal.* 255 (2008) 1; Courtesy of S. Simonsen & S. Helveg (HTAS).

	STANDARD MODE		ETEM MODE (< 0.5 mbar nitrogen)	
	No corrector	C _s image corrected	No corrector	C _s image corrected
TEM information limit (nm)	0.10	0.10 (0.09 mono on)	0.12	0.12
TEM point resolution (nm)	0.20	0.10	0.20	0.12
Probe current @ 1 nm (nA*)	0.6	0.6	0.6	0.6
System energy resolution*	0.7 eV	0.7 eV	0.8 eV	0.8 eV
STEM resolution (nm)	0.136	0.136	0.16	0.16

Note: All specifications are at 300kV. For a list of specifications at other acceleration voltages please contact your FEI sales representative.

* With SFEG. The ETEM is also optionally available with X-FEG and gun monochromator. Depending on the energy filter option the energy resolution could be 0.20 eV (0.25eV in ETEM mode).

Technical highlights

- Ultra-stable schottky field emitter gun
- New three lens condenser system with quantitative indication of convergence angle and size of illuminated area
- Flexible high tension from 80 to 300 kV
- X-FEG high brightness gun (optional)
- Gun monochromator (optional)
- Image C_s correction (optional)
- Proven sub-Ångström performance
- Modular column design
- Patented accurate mechanical stacking system
- ConstantPower™ lens design
- Low hysteresis design to minimize crosstalk between optical components
- Symmetric S-TWIN objective lens with wide, 5.4 mm pole piece gap and objective aperture in the back focal plane of the objective lens for TEM dark field applications
- Automatic apertures
- Rotation free imaging
- Computerized 5 axis specimen stage
- Tilt range +/- 35 degrees for analytical double tilt holder and with tomography holder +/- 70 degrees
- Field free imaging in Lorentz mode
- Holography mode
- TrueImage™ ATLAS focus series software for quantitative HR-TEM applications
- Xplore3D™ software for automated tomography S/TEM experiments and Xpress3D for ultra-fast 3D reconstructions

Detector options

- On axis triple BF/DF detector (DF1/DF2/BF)
- HAADF detector
- Gatan US1000XP camera
- Eagle™ series cameras
- Gatan energy filter series
- EDS detector 0.13 srad solid angle (for detailed EDS performance please contact sales and service organization)

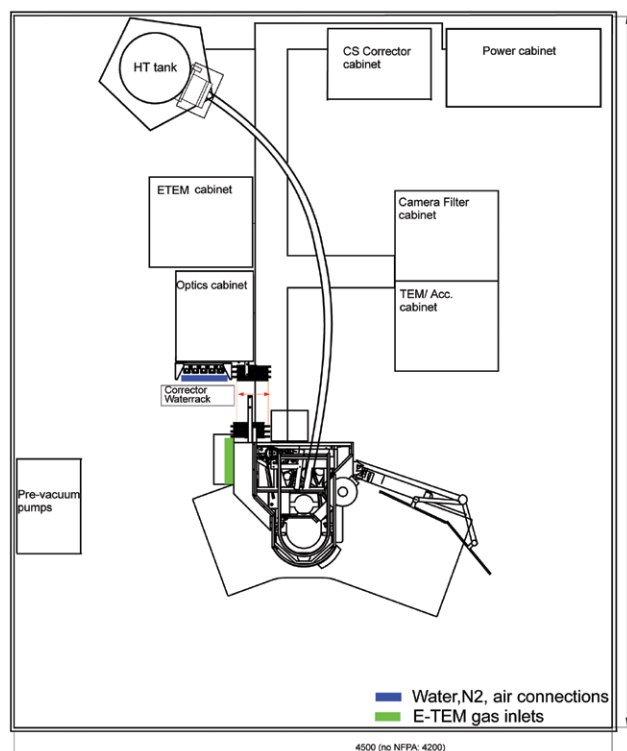
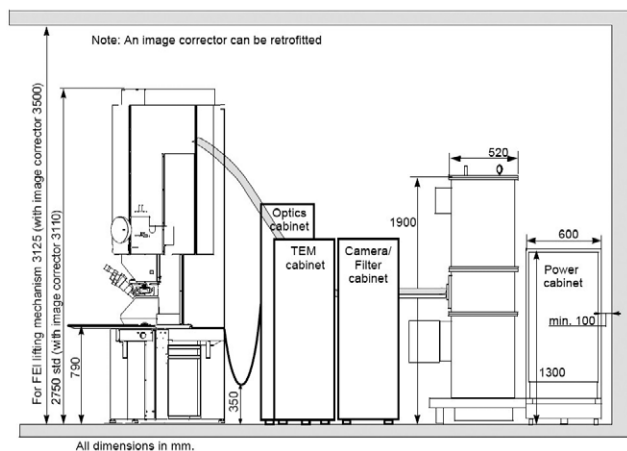
ETEM technology

- Maximum protection for FEG emitter with differential pumping apertures (window-free imaging)
- Differentially pumped S-TWIN pole piece, space for full double tilt capability
- Regular Titan operation in non-ETEM mode (see table)
- HAADF STEM scattering angle up to 70 mrad
- Compatible with regular Titan TEM holders for simple sample insertion
- Fast switching between ETEM and high vacuum modes < 4 minutes
- Accurate and computer control of gas pressure in ETEM experiments, from 10⁻³ Pa up to 2000 Pa (20 mbar, 15 torr; N₂) & efficient pumping of gases (incl. H₂)
- 3 different gases via 3 gas inlets with preset partial pressure
- Reactant gas analysis via mass spectrometer (RGA)
- Effective cleaning of the column with built-in plasma cleaner
- Built in safety features (for hardware protection and safe working environment)

Holders options (requires gas compatibility in ETEM gas experiments)

- Single tilt holder
- Analytical double tilt holder
- Tomography holder
- Single/double tilt cryo-holder (LN₂)
- Single/double tilt heating holder
- Straining holder
- STM/AFM holder

Floorplan



↑ Schematic drawing of Titan ETEM room layout. In addition to standard TEM room requirements (room survey, etc.), please consider space, installation, and safety requirements for gas supply, gas storage, gas inlet/mixing units and gas exhausts. Please contact your FEI representative.

Installation requirements

- Environment temperature 18 °C to 23 °C temperature stability 0.2 °C/h heat dissipation into air nominal 4300 W
- Door height: 2275 mm (depends on version)
- Door width: 1320 mm
- Ceiling height: 3500 mm (max. configuration)
- Required floor space for microscope 4500 x 5500 mm
- Max weight microscope column: 1800 kg max
- Max point loading: 105 N/cm²
- Power voltage: 3 phase including neutral and earth 398 V (+6 %, -10 %)
- Frequency 50 or 60 Hz (+/- 3 %)
- Power consumption with all microscope options max. 14330 W
- Electrical connection single phase for water cooler 230 V, 4 kVA
- Cooling water required depending on ordered water cooling unit
- Double earth connection required
- Compressed air supply, pressure min. 6 bar max. 7 bar
- Nitrogen N₂, pressure min. 1 bar max. 3 bar
- SF₆ gas—proper ventilation required
- Gas exhaust system
- Liquid nitrogen LN₂
- LAN connection for remote diagnostics telephone line

Please contact your sales and service organization for more detailed information and for a complete pre-installation requirement document.

Certain gases may not be approved for use with the ETEM or their use may be restricted. Please contact FEI for additional information on approved gases and our gas approval process.

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