

Access the world of in situ dynamics

# Key benefits

- Dynamically study in situ gas solid and gas liquid reactions with different temperatures in S/TEM at the atomic level
- Determine phase diagrams of nanomaterials as a function of temperature and gas pressure
- Improve the quality of catalytic particles by understanding their function in gas reactions, and analyze the changes in gas composition with a mass spectrometer
- Study phase transitions in situ at the atomic level
- Synthesize new nanomaterials
- Initiate chemical reactions and study their behaviour in situ
- Explore new nanomaterials by creating materials and nanostructures in situ
- Maximise the lifetime of unstable materials by using partial gas pressures during S/TEM examination
- Improve the lateral resolution in your in situ experiments with image C<sub>s</sub> corrector technology and full double tilt capability of the specimen holder

# Titan™ ETEM

Dynamic *in situ* exploration of nanomaterials at the atomic scale with variable gas pressures and temperatures

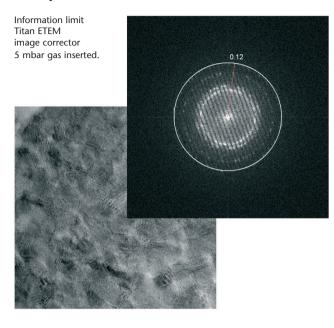
The Titan<sup>TM</sup> ETEM is the ultimate *in situ* high resolution electron microscope to study dynamic behaviour of chemical reactions under the influence of variable temperatures and gas pressures at the atomic level.

The innovative E-cell technology in the objective lens pole piece allows *in situ* S/TEM gas experiments with a mixture of up to four gas inlets with preset partial pressure up to 2 kPa (20 mbar, 15 torr). This unique capability can be combined with the image C<sub>s</sub> corrector and monochromator technology of FEI to explore the dynamic behaviour of the morphology, structure, composition and bonding of nanomaterials down to the atomic level.

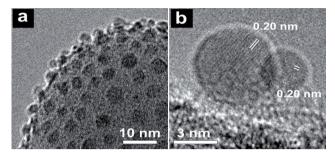
The Titan ETEM is equipped with a gas mixing unit and a mass spectrometer to control and determine the gas composition at the specimen, which permits full control of in situ experiments and gives detailed information of the gases in the microscope. Additionally, a built-in plasma cleaner allows cleaning the column after experiments involving gas. The Titan ETEM in non-ETEM mode has the same lateral S/TEM resolution and energy resolution specifications as a Titan 80-300 without ETEM technology. Therefore, it is a flexible tool that is not solely dedicated to in situ applications. The system is based on the world class modular Titan technology in mechanical, electronic and thermal stability and is designed to deliver the ultimate performance in all TEM, STEM, energy filtered TEM (EFTEM), diffraction and electron energy loss spectroscopy (EELS) modes. The flexibility of operating the Titan 80-300 in the voltage range of 80 to 300 kV allows this important parameter to be optimized to the requirements of the material examined, from ultra-light carbon compounds to ultra-dense heavy metal materials.

Combining all of these enhancements in one, easy to use instrument enables you to explore the dynamic behaviour of the nanoworld, and helps you to expand your boundaries with new pioneering scientific results.

#### **ETEM performance in HR S/TEM**

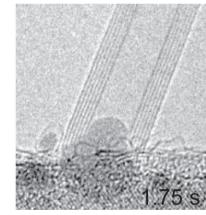


#### **ETEM** applications

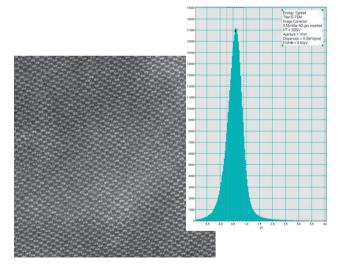


Hoffmann et al (2007) Nano Letters 7(3) pp. 602-608. Reproduced with permission.

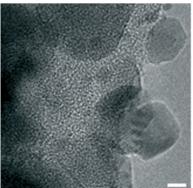
ETEM images showing an initially partially oxidised 1 nm nickel catalyst film on a silica membrane, where the catalyst has restructured into islands consisting of nanocrystals. Treated in ammonia gas at pressure p = 100 Pa (1 mbar, 0.75 torr) and temperatures T = 450 °C (a) and T = 650 °C (b). The image in (b) is a higher magnification view of the nanocrystals.



Courtesy of S.Takeda and H. Yoshida, Osaka University, Japan.



A Young's fringe experiment carried out on gold using nitrogen gas at pressure p = 500 Pa (5 mbar, 3.75 torr) with an aberration corrected ETEM. The information limit in this case is 1.2 Å, as measured from the Fourier transform of the upper figure. In the lower right figure, the zero-loss peak (< 0.7 eV FWHM) in an EELS spectrum, using nitrogen gas at pressure of 55 Pa (0.55 mbar, 0.4 torr) is shown to prove the excellent energy resolution for EELS applications in ETEM mode. The lower left figure shows a HR-STEM image of silicon in the <110> projection in ETEM mode with 50 Pa pressure of nitrogen gas. Growth of a multi wall carbon nanotube from a metal catalyst particle.



Courtesy of R. Sharma, Arizona State University.

Nickel oxide particles imaged in nitrogen gas at pressure p = 720 Pa (7.2 mbar, 5.4 torr) and T = 470 °C. Scale bar = 1 nm.

## Main specifications at 300 kV acceleration voltage

	Standard mode		ETEM mode (< 0.5 mbar nitrogen)	
	No corrector	C <sub>s</sub> image corrected	No corrector	C <sub>s</sub> image corrected
TEM information limit (nm)	≤ 0.1	≤ 0.1	≥ 0.12	≥ 0.12
TEM point resolution (nm)	0.20			
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

Detailed main specifications list is also available for 80 and 200 kV acceleration voltage.

\*The ETEM is also available with a monochromator for UHR-EELS. In this configuration the energy resolution is 0.2 eV.

### **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
- Gun monochromator
- Image C, correction
- Proven sub-Ångström performance
- Modular column design
- Patented accurate mechanical stacking system
- ConstantPower<sup>™</sup> lens design
- Low hysteresis design to minimize crosstalk between optical components
- Symmetric S-TWIN objective lens with wide pole piece gap design of 5.4 mm and objective aperture in the back focal plane of the objective lens for TEM dark field application
- Ready for on site retrofit of corrector
- Automatic apertures
- Rotation free imaging
- Computerized 5 axis specimen stage
- Tilt range +/- 40 degrees for analytical double tilt holder and with tomography holder +/- 80 degrees
- Field free imaging in Lorentz mode
- Holography mode
- TrueImage<sup>™</sup> focus series software for quantitative HR-TEM applications
- Xplore3D<sup>™</sup> software for automated tomography S/TEM experiments and Xpress3D for ultra-fast 3D reconstructions

# Detectors

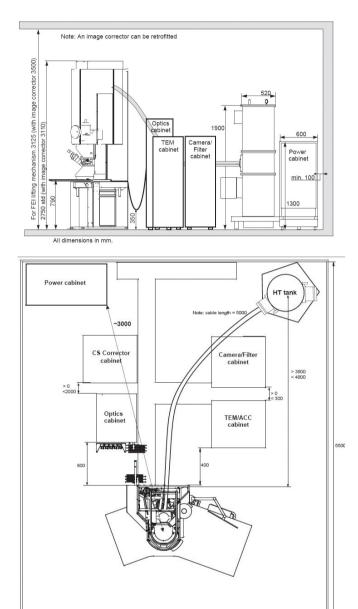
- On axis triple BF/DF detector (Df1/DF2/BF)
- HAADF detector
- Gatan US1000/4000 camera
- Gatan Orius camera
- Eagle<sup>™</sup> series cameras
- Energy filter Tridiem 863/865/866
- Plate camera
- EDS detector 0.13 srad solid angle (for detailed EDS performance please contact sales and service organization)

# **ETEM technology**

- Environmental cell (E-cell) in S-TWIN pole piece with max 2 kPa (20 mbar, 15 torr)
- Normal Titan operation in E-cell switched off mode (see table)
- HAADF STEM scattering angle < 70 mrad
- 4 different gases via 4 gas inlets with preset partial pressure
- Reactant gas analysis via mass spectrometer
- Integrated plasma cleaner to clean the column after ETEM experiment
- Gas control unit to mix different gases, and pressure indicators to monitor and control the gas pressure in the ETEM

#### Holders

- Single tilt holder
- Analytical double tilt holder
- Tomography holder
- Single/double tilt cryo-holder (L-N2)
- Single/double tilt heating holder
- Straining holder
- STM/AFM holder



#### 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: 1050 mm
- Ceiling height: 3500 mm (max. configuration)
- Required floor space for microscope 4500 x 5500 mm
- Weight distribution max 465 kg/m<sup>2</sup>
- Power voltage: 3 phase incl. neutral and earth 398 V (+6 %, -10 %)
- Frequency 50 or 60 Hz (+/- 3 %)
- Power consumption with all microscope options max. 10 kVA
- 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. 5 bar max. 7 bar
- Nitrogen N<sub>2</sub>, pressure min. 1 bar max. 3 bar
- SF<sub>6</sub> gas proper ventilation required
- Pre-vacuum pump outlet
- Liquid nitrogen LN<sub>2</sub>
- 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.

Floorplan



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TÜV Certification for design, manufacture, installation and support of focused ionand electron-beam microscopes for the NanoElectronics, NanoBiology, NanoResearch and Industry markets.

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