

**MICROSCOPY AND MICROANALYSIS LAB**

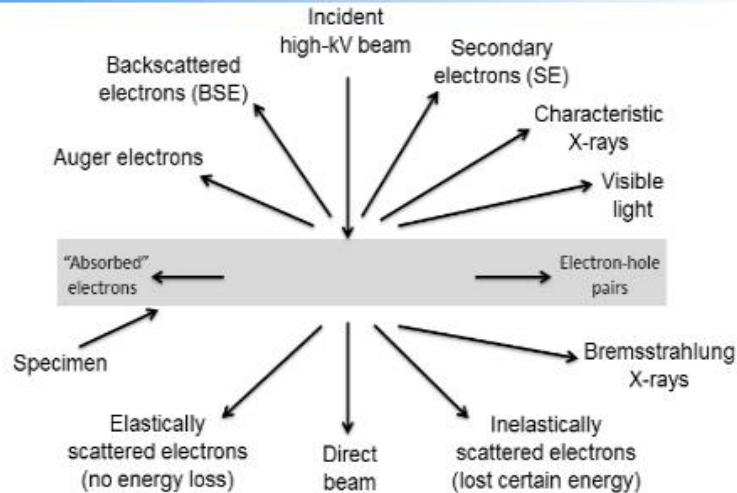
# **TRANSMISSION ELECTRON MICROSCOPY (TEM)**

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through a specimen to form an image. The specimen is most often an ultrathin section less than 100 nm thick or a suspension on a grid. An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device, such as a fluorescent screen or a sensor, such as a scintillator attached to a charge-coupled device (CCD camera). Transmission electron microscopes are capable of imaging at a significantly higher resolution, enabling the instrument to capture fine detail—even as small as a single column of atoms. Transmission electron microscopy is a major analytical method in the physical, chemical, and biological sciences. TEMs find application in materials science, such as nanotechnology, semiconductor research, devices, and catalysis, and in biological sciences, such as cancer research, virology, bacteriology, and other fields.

TEM instruments boast an enormous array of operating modes, including conventional imaging, scanning TEM imaging (STEM), diffraction, spectroscopy, and combinations of these. Even within conventional imaging, there are many fundamentally different ways that contrast is produced, called "image contrast

mechanisms." Contrast can arise from position-to-position differences in the thickness or density ("mass-thickness contrast"), atomic number ("Z contrast"), crystal structure or orientation ("crystallographic contrast" or "diffraction contrast"), the slight quantum-mechanical phase shifts that individual atoms produce in electrons that pass through them ("phase contrast"), the energy lost by electrons on passing through the sample ("spectrum imaging"), and more. Each mechanism provides the user with a different kind of information, depending not only on the contrast mechanism but also on how the microscope is used. This means that a TEM is capable of returning an extraordinary variety of nanometer- and atomic-resolution information, in ideal cases revealing not only where all the atoms are but also what kinds of atoms they are and how they are bonded to each other. For this reason, TEM is regarded as an essential and, probably, the only tool for nanoscience in both biological and materials fields.

## Electron-specimen interactions



Thin samples, image using transmitted electrons

The capabilities of the TEM can be further extended by additional stages and detectors incorporated on the same microscope. Some of these are:

### ■ ANALYTICAL MICROSCOPY

TEMs could be equipped with elemental analysis capabilities (EDS) where elemental qualitative, quantitative and mapping could be conducted on atomic scale

### ■ SCANNING TEM

A TEM can be modified into a scanning transmission electron microscope (STEM) by the addition of a system that rasters a convergent beam across the sample to form the image, when combined with suitable detectors. By correlating the electron count to the position of the scanning beam (known as the "probe"), the transmitted component of the beam may be measured. The non-transmitted components may be obtained either by beam tilting or by the use of annular dark field detectors.

### ■ CRYO-TEM

Cryogenic transmission electron microscopy (Cryo-TEM) uses a TEM with a specimen holder capable of maintaining the specimen at liquid nitrogen or liquid helium temperatures. This allows imaging specimens prepared in vitreous ice, the preferred preparation technique for imaging individual molecules or macromolecular assemblies, imaging of vitrified solid-electrolyte interfaces, and imaging of materials that are volatile in high vacuum at room temperature, such as sulfur.

## ■ ENVIRONMENTAL/IN-SITU TEM

In-situ microscopy enables conducting experiments in TEM using differentially pumped sample chambers, or specialized holders. Types of in-situ experiments include heating and cooling experiments studying different materials including nanomaterials, chemical reactions using liquid-phase electron microscopy, and The Climate system enables the nano-world to be investigated in the real-world 1 bar pressure, gas and heating environment.

TEM gas cell specimen holder allows researchers to study material behavior in gases and at elevated temperatures ( $>1000^{\circ}\text{C}$ ), obtaining atomic resolution images of gas-solid interactions at real-time reaction temperatures and pressures. Sample research applications for which realistic reaction conditions include: Gas catalysis, Fuel-cell research, Growth of nano-structures. Thin film deposition.

The HBKU Core Labs is equipped with two Thermo Fisher Scientific's (formerly FEI company, Netherlands). Transmission Electron Microscope one configured for material science and the other for biological science.

# APPLICATIONS

## ▪ **STRUCTURAL ANALYSIS:**

- Crystallographic structure determination
- Lattice spacing and unit cell dimensions
- Grain boundaries, interfaces, and defects

## ▪ **ELEMENTAL AND CHEMICAL ANALYSIS:**

- Elemental mapping and compositional distribution
- Chemical state identification using electron energy loss spectroscopy (EELS)
- Detection of light elements and trace elements

## ▪ **MATERIALS SCIENCE:**

- Phase identification and phase transformations
- Dislocation dynamics and plastic deformation
- Nanoparticle and nanostructure characterization

## ▪ **SEMICONDUCTOR INDUSTRY:**

- Defect analysis and dopant profiling
- Device structure and interconnect analysis

- Thin-film and multilayer structure characterization

## ▪ **BIOLOGICAL AND BIOMEDICAL RESEARCH:**

- High-resolution imaging of cellular structures and organelles
- Protein structure determination and macromolecular complexes
- Virus morphology and structural biology

## ▪ **POLYMER SCIENCE:**

- Morphology of polymer blends and composites
- Crystallinity and lamellar structure of semicrystalline polymers
- Microphase separation in block copolymers

## ▪ **GEOLOGY AND MINERALOGY:**

- Crystal structure and orientation of minerals
- Amorphous and crystalline phase identification in geological samples

# EQUIPMENT AND TECHNICAL SPECIFICATIONS

## ▪ FEI TALOSF200X TEM

This High Resolution analytical Transmission Electron Microscope (HRTEM) is a powerful tool for materials analysis to study variety of materials, including catalysts, minerals, interfaces, nanocomposites, ceramics, polymers and metallic clusters to determine structure, crystallinity, crystalline defects and chemistry.

The Thermo Scientific TalosF200X scanning/transmission electron microscope (S/TEM) combines outstanding high-resolution STEM and TEM imaging with industry-leading energy dispersive x-ray spectroscopy (EDS) signal detection and 3D chemical characterization with compositional mapping.

The main parameters of this instrument:

## ➤ ACCELERATION VOLTAGE:

This TEM is routinely operated at 200kv. Specimens that are immune to radiolysis suffer knock-on damage at high current densities, and this form of radiation damage can be reduced or avoided by choosing a low accelerating voltage of 80Kv.

## ➤ ELECTRON SOURCE:

- high-intensity, X-FEG space emission radiation source, brightness  $1.8 \times 10^9$  A / cm<sup>2</sup> srad (@ 200 kV);
- beam current  $\geq 50$  nA;
- current density of 1.5 nA at a beam diameter of 1 nm (probe current)

## ➤ MAGNIFICATION:

- TEM magnification range: 25x- 1.5Mx
- STEM magnification range: 150x-230Mx
- Camera length (mm): 12- 5100

## ➤ RESOLUTION:

- STEM high angle annular dark field (HAADF): spatial resolution (nm) 0.16
- 0.12nm TEM information limit
- 0.25nm TEM point resolution

## ➤ SAMPLE HOLDERS:

- Single tilt holders
- Double tilt holder: analytical holder optimized for EDS with low background, inclined  $\pm 30^\circ$
- TEM/STEM tomography holder: sample holder for tomographic imaging with an inclination of  $\pm 70^\circ$
- Gatan Model 652 Heating holder: to heat sample up to maximum temperature of 850°C
- Gatan Model 636 Liquid Nitrogen Cooling holder: to image sample less than of -170°C
- Protochip in-situ gas holder: to study samples in gas environments within the TEM

## ➤ IMAGING CAMERA:

- Ceta 16 Mpixel, 4k x 4k CMOS camera with wide viewing angle, 16 bit color depth and 20 fps.
- Wide angle ring, dark field of view (HAADF) detector
- Bright Field/Dark Field detector
- SmartCam digital search camera

## ➤ ENERGY DISPERSIVE X-RAY SPECTROMETER (EDS):

- “Super-X” windowless or ultra-thin window detector system with 4 silicon drift detectors (SDD) integrated in the microscope (which can be switched on and off individually)
- Signal acquisition solid angle  $\geq 0.9$  sr
- 140 eV spectral resolution at the peak of Mn K $\alpha$
- Fast EDS mapping (10 ds dwell time / pixel)

## ■ FEI TALOSF200C TEM

This Transmission Electron microscope is dedicated to the ultra-structure analysis of a variety of biological sample analysis such as 2D and 3D imaging of cells, organelles, asbestos, polymers, and soft materials, both at ambient and cryogenic temperatures.

This TalosF200C TEM elevates the imaging quality of beam-sensitive materials with its optional, motorized, retractable Cryo-box and low-dose technique. The large C-Twin pole piece gap, which provides high application flexibility, combined with a reproducibly performing electron column, opens new opportunities for high-resolution 3D characterization, in situ dynamic observations, and diffraction applications, with a special emphasis on high-contrast imaging and Cryo-TEM.

The main parameters of this instrument:

### ➤ ACCELERATION VOLTAGE:

This TEM is routinely operated at 200kv. Specimens that are immune to radiolysis suffer knock-on damage at high current densities, and this form of radiation damage can

be reduced or avoided by choosing a low accelerating voltage of 80Kv.

### ➤ ELECTRON SOURCE:

Schottky field emission gun(S-FEG) having brightness of about  $10 \times 10^8 \text{ Acm}^{-2}\text{sr}^{-1}$

### ➤ MAGNIFICATION:

- TEM magnification range: 25x- 910kx
- STEM magnification range: 150x-230Mx
- Camera length (mm): 12- 5100

### ➤ RESOLUTION:

- STEM high angle annular dark field (HAADF): spatial resolution (nm) 0.21
- 0.14nm TEM information limit
- 0.3nm TEM point resolution



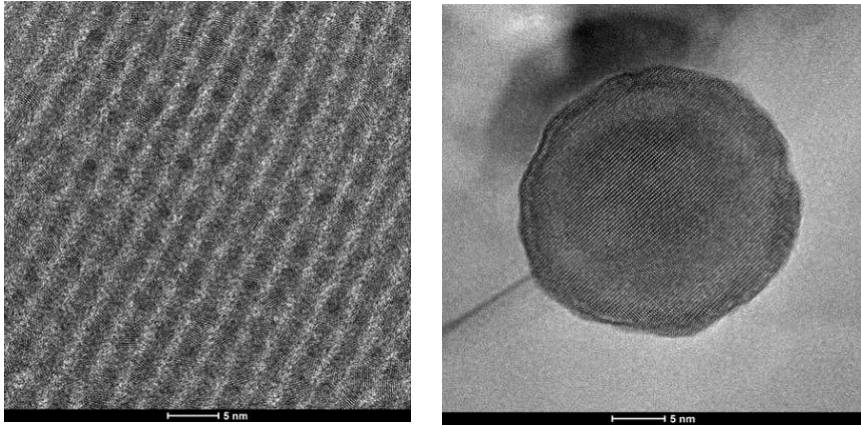
## ➤ **SAMPLE HOLDERS:**

- Single tilt holders
- TEM/STEM tomography holder: sample holder for tomographic imaging with an inclination of  $\pm 70^\circ$
- Gatan Model 626 Cryo transfer holder

## ➤ **IMAGING CAMERA:**

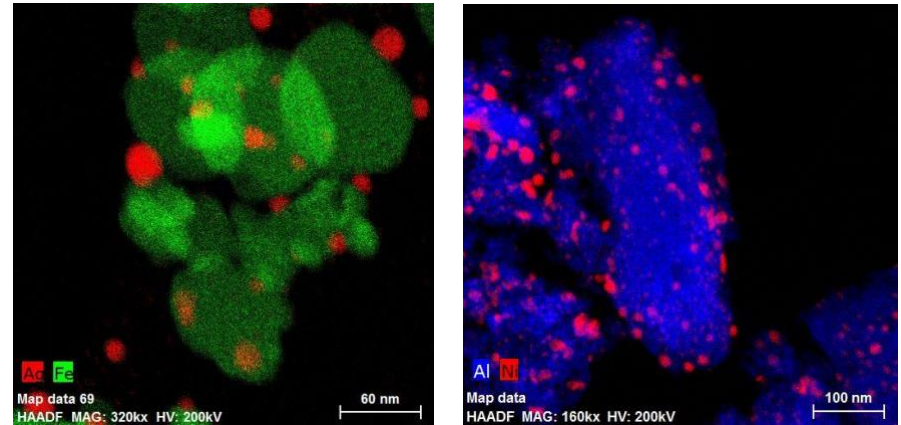
- Ceta 16 Mpixel, 4k x 4k CMOS camera with wide viewing angle, 16 bit color depth and 20 fps.
- Wide angle ring, dark field of view (HAADF) detector
- Bright Field/Dark Field detector
- SmartCam digital search camera

# EXAMPLES OF WORK DONE

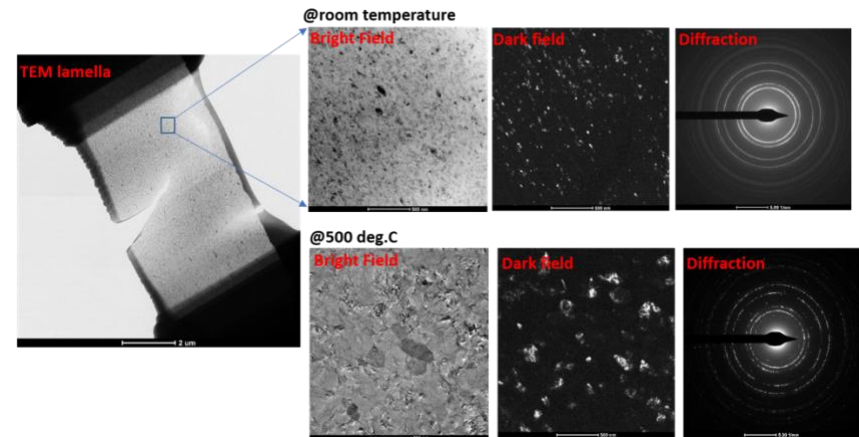


HR-TEM imaging of a) Pb nanoparticles b) Al-Sc Memory shape alloy

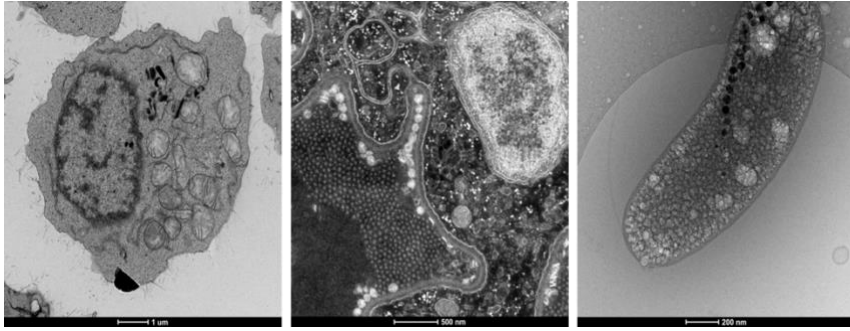
The energy dispersive X-ray spectrometer (EDS) features a “Super-X” windowless or ultra-thin window detector system with four silicon drift detectors (SDD) integrated into the microscope, a signal acquisition solid angle of  $\geq 0.9$  srad, a spectral resolution of 140 eV at the peak of Mn K $\alpha$ , and fast EDS mapping with a 10 ds dwell time per pixel.



Super-X EDS” Elemental mapping showing nanoparticles of size less than 30nm

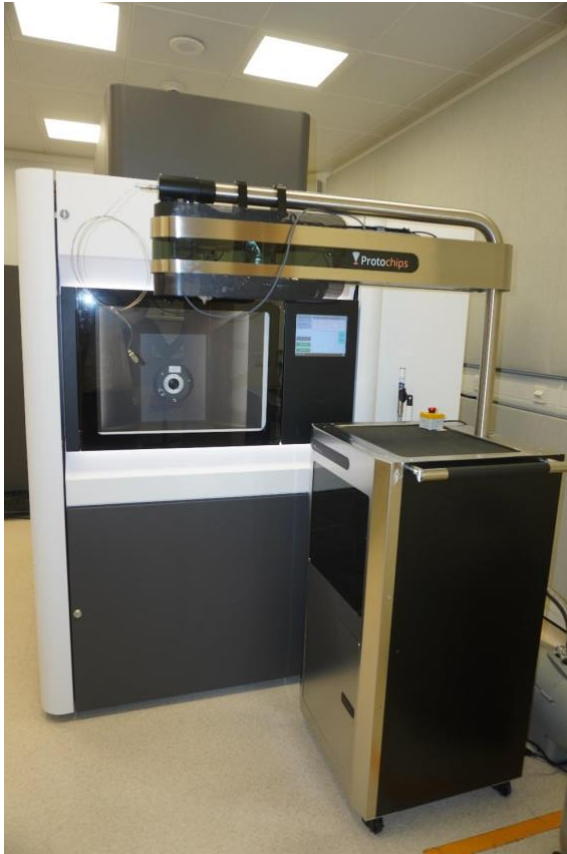


In-situ heating- TEM analysis of Aluminum-Graphite composite

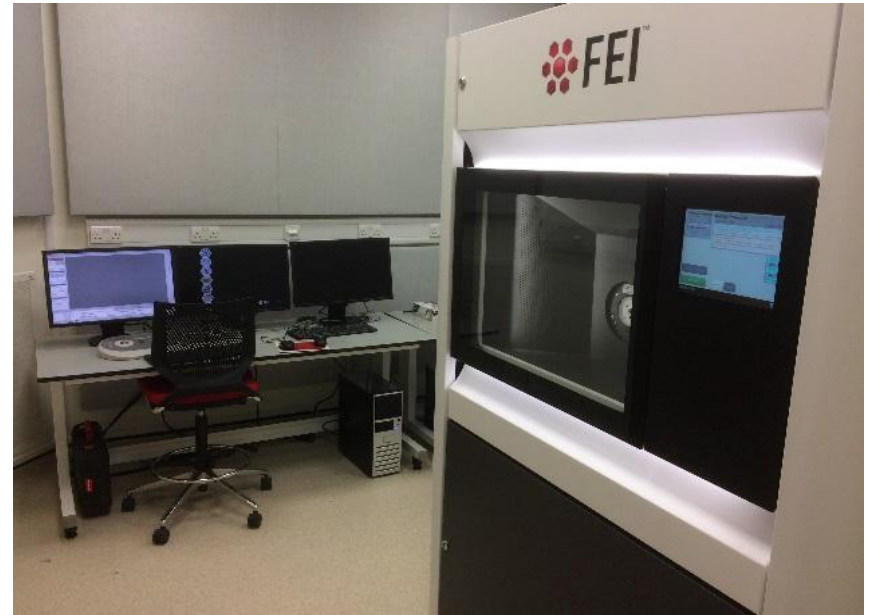


*TEM imaging of a) Mitochondria b) cellular structure c) magnetic nanoparticles on bacteria*

# PICTURES OF EQUIPMENT



*FEI TalosF200X TEM*



*FEI TalosF200C TEM*