Electron Energy-Loss Spectroscopy (EELS)
for the Hitachi HD-2000

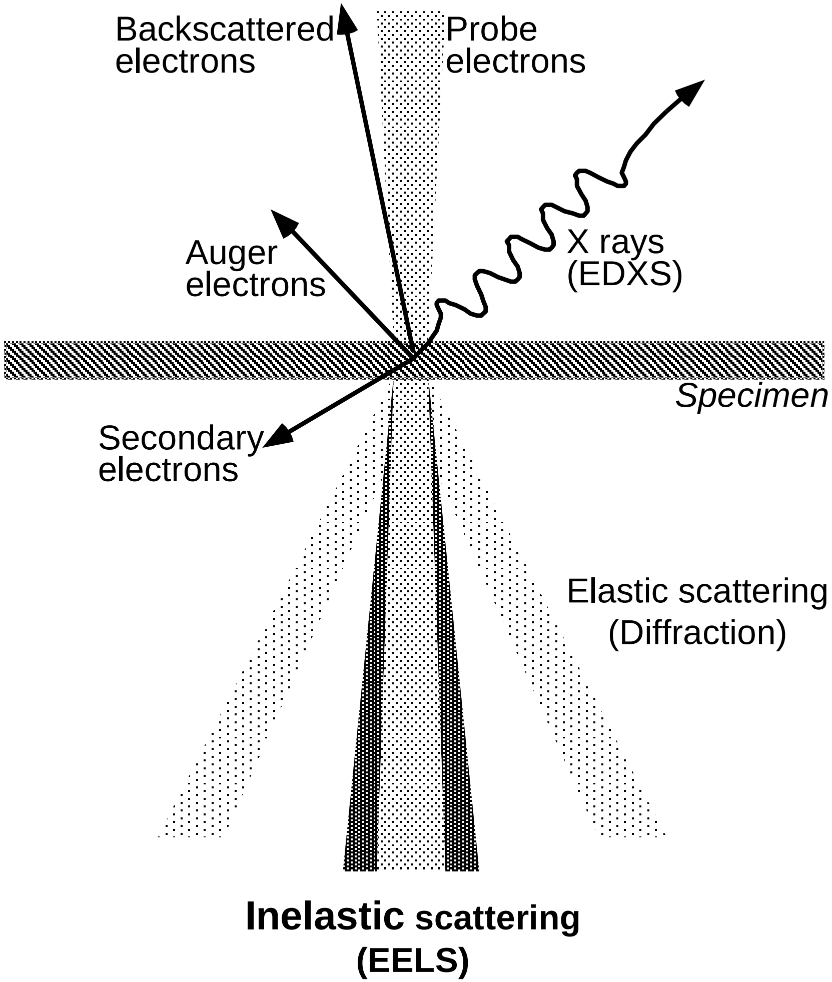
Outline

- Introduction to Electron Energy-Loss Spectroscopy (EELS)
- EELS Spectrum Imaging
- EELS on the HD-2000

Introduction to EELS

- How EELS works
- What can EELS do?
- Spectrum information
- Quantification
- Comparisons to X-ray microanalysis

How EELS works



- (S)TEM probe electrons travel through a thin specimen
- Probe electrons lose energy due to their interaction with the specimen *(inelastic scattering)*
- The energy-losses are characteristic of the elements and chemistry of the specimen
- An EELS Spectrometer can disperse the probe electron beam according to its lost energies into a spectrum

What can EELS do?

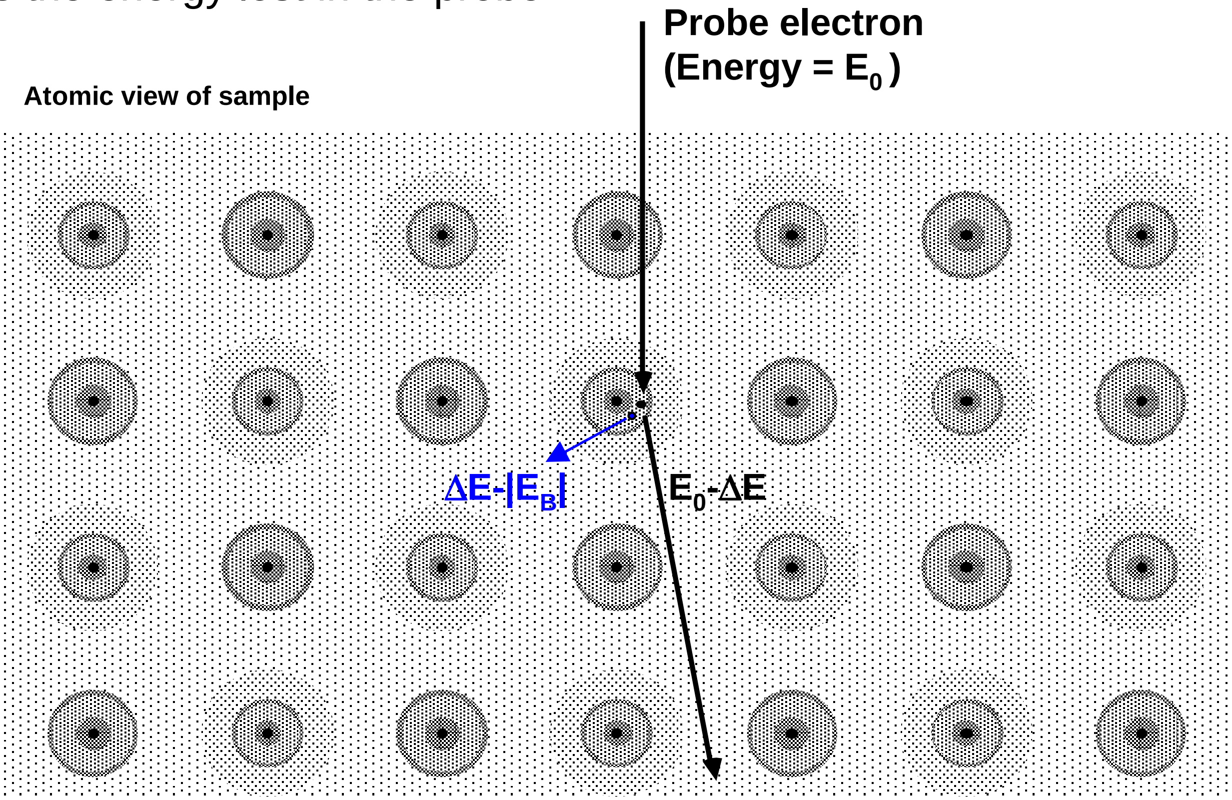
- EELS can provide information about:
  - Elemental composition of all elements Z>1
  - Chemical composition
  - Specimen thickness

- The high sensitivity of EELS makes it ideal for mapping

- Further capabilities:
  - *Chemical bonding*
  - *Band structure*
  - *Valence and conduction electron density*
  - *Atom-specific radial distribution of near neighbors*
  - *Polarization/dielectric response*
  - *Z=1 quantification*

Basic Physics of EELS

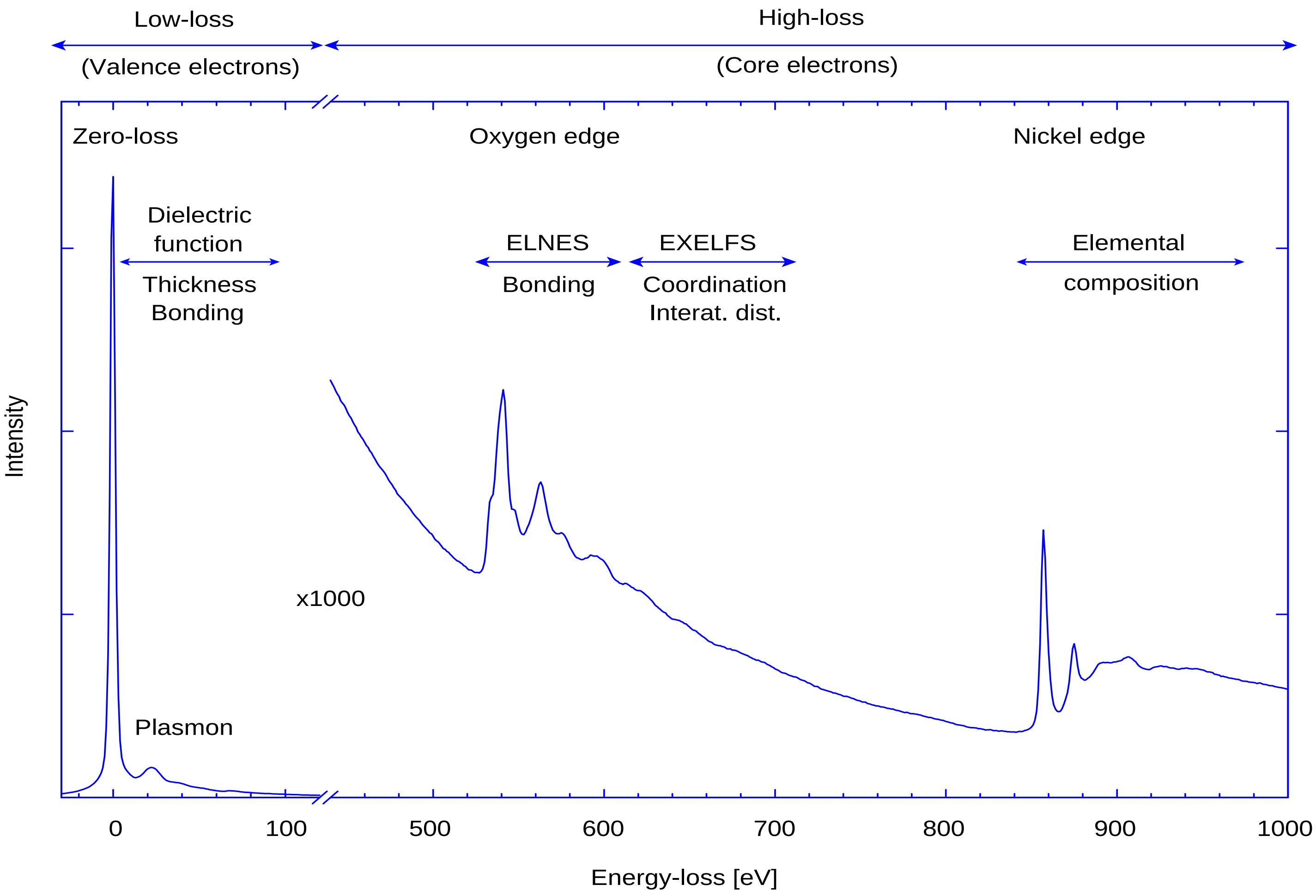
- (S)TEM probe electron donates energy to core or valence electrons in the sample
- EELS analyzes the energy lost in the probe electrons



E_B = Binding energy of electron in sample

ΔE = Energy transferred from probe electron to electron in sample (The "Energy Lost")

EELS Spectrum Features

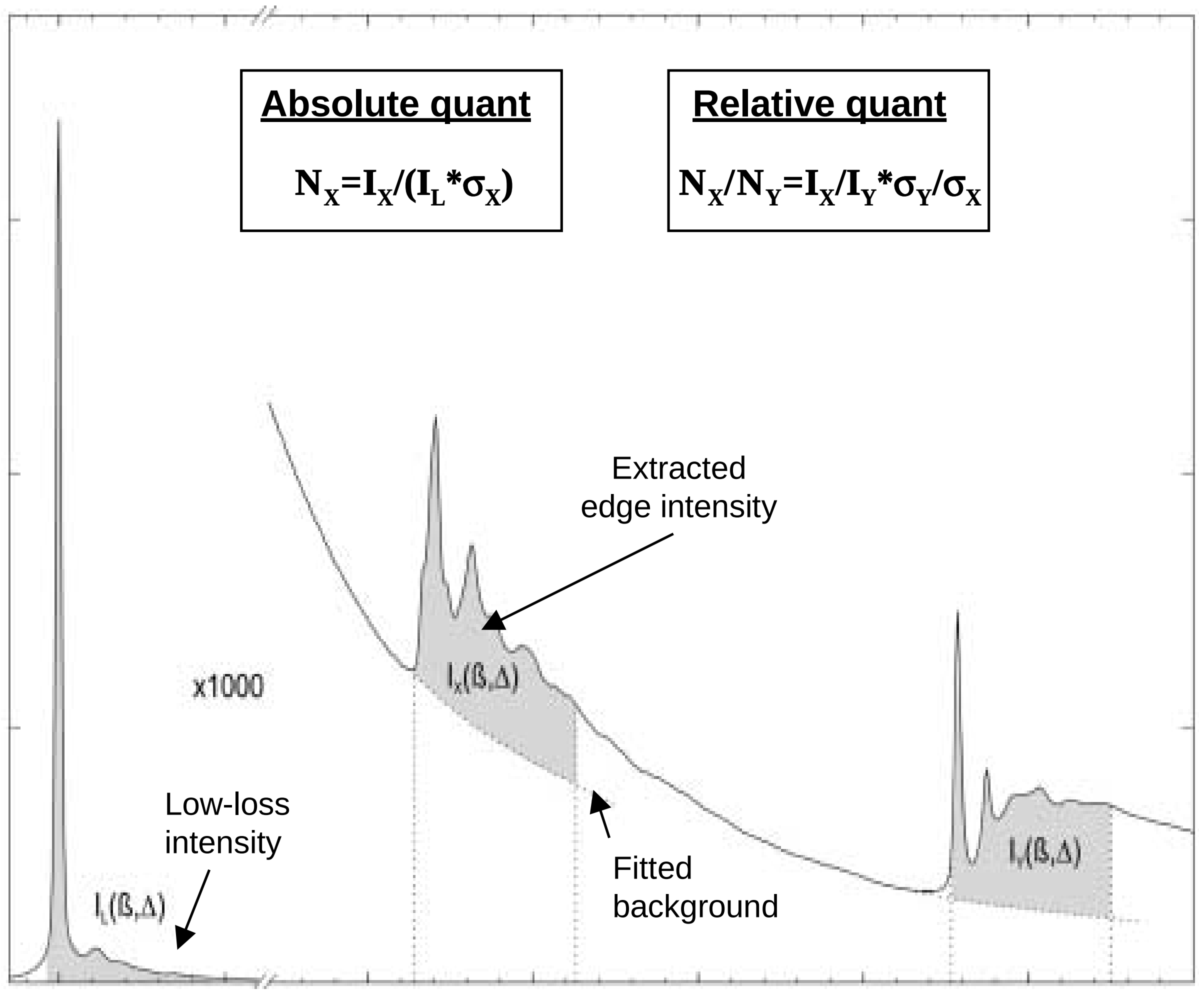


EELS Spectrum Quantification

- Calculate edge intensities by removing background below edge
- Convert edge intensities into concentrations using cross-sections (σ)
- No standards needed!

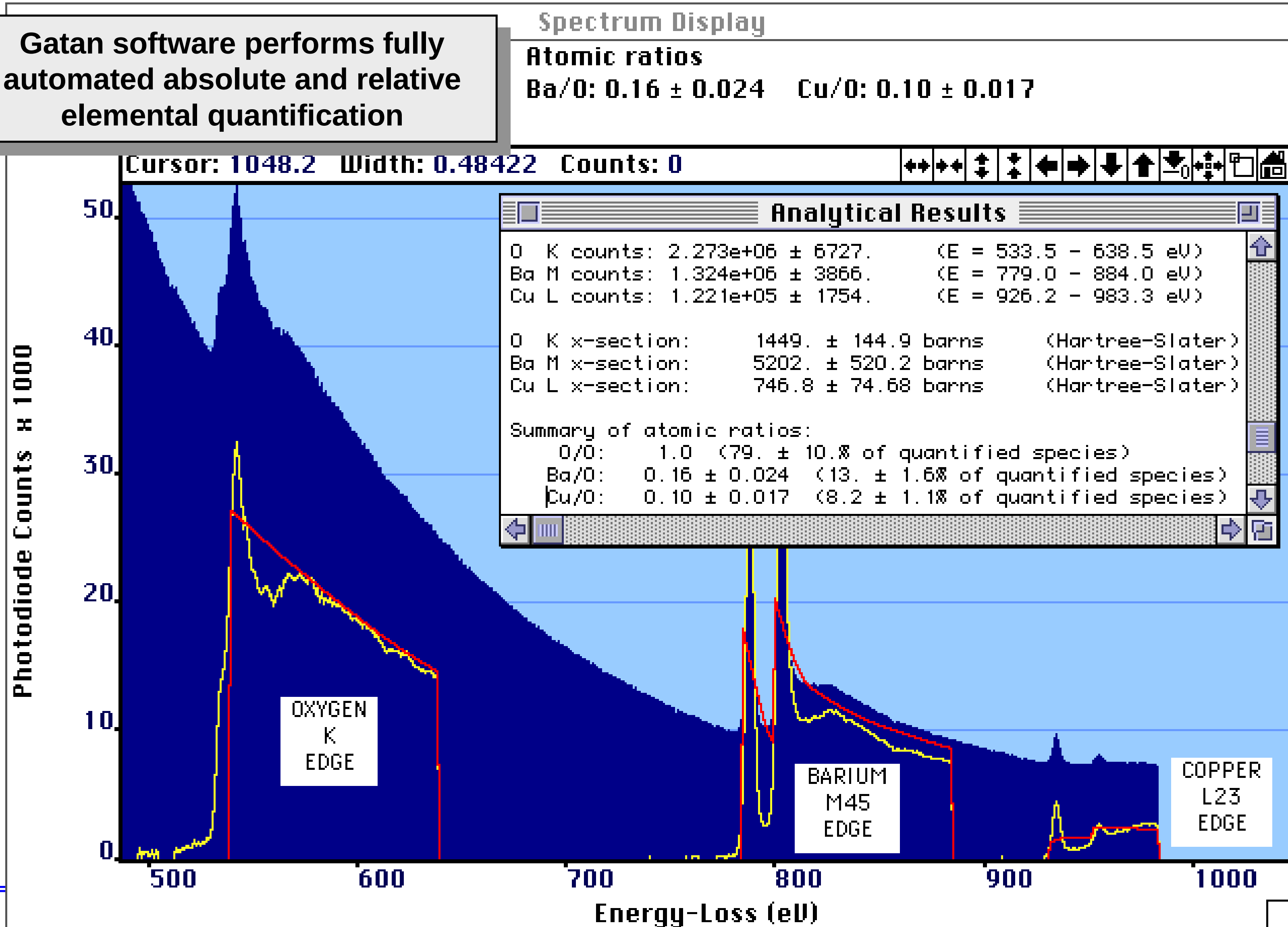
- Absolute quant
  - Use edge and low-loss intensity
  - Result is in atoms or atomic density

- Relative quant
  - Uses ratio of edge intensities
  - Result is in atomic fractions



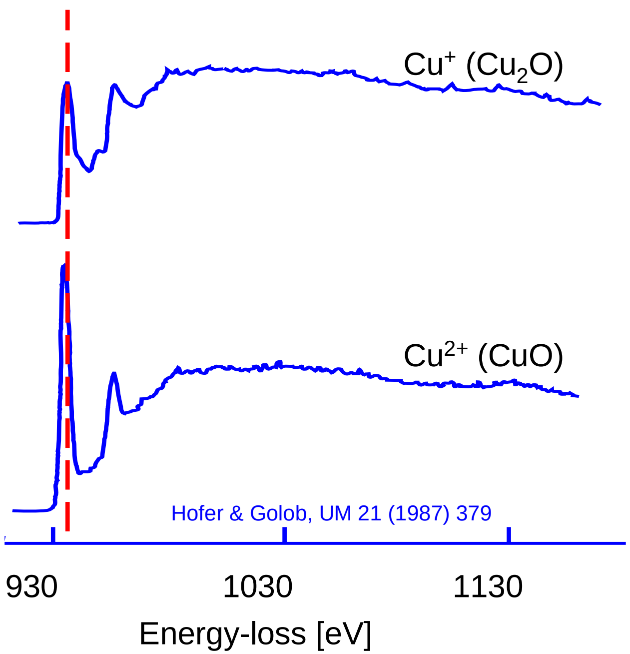
Automated EELS elemental analysis

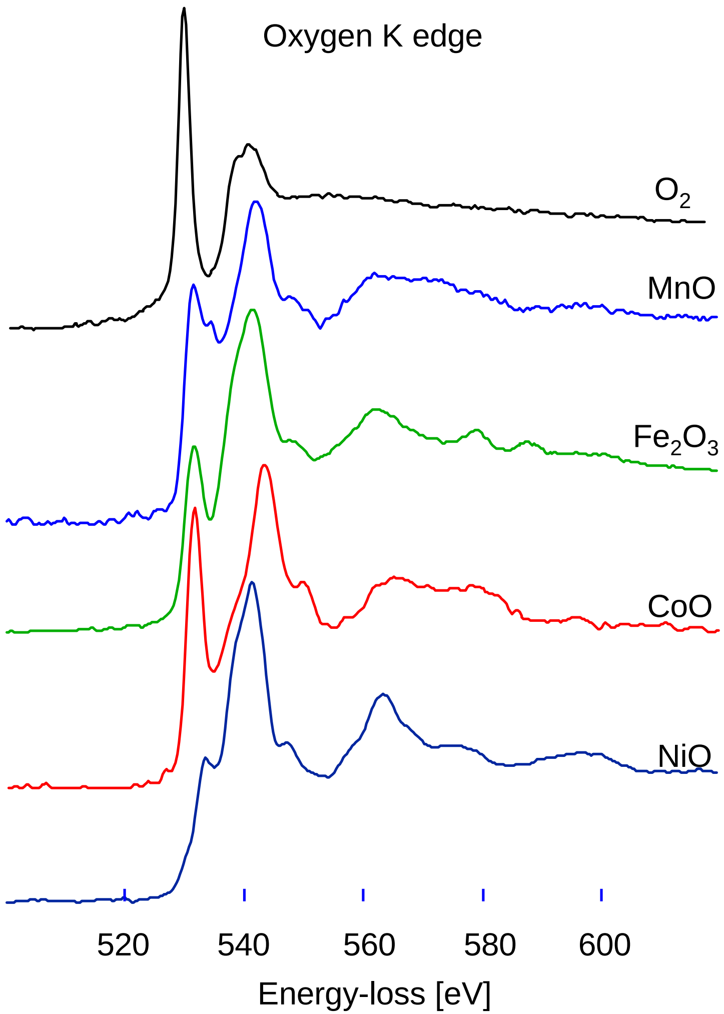
Gatan software performs fully automated absolute and relative elemental quantification



Chemical analysis

- Changes in chemical bonding produce changes in the shape of EELS edges
  - Fingerprinting is the technique of identifying a chemical structure from the edge shape
  - Quantification is possible with references



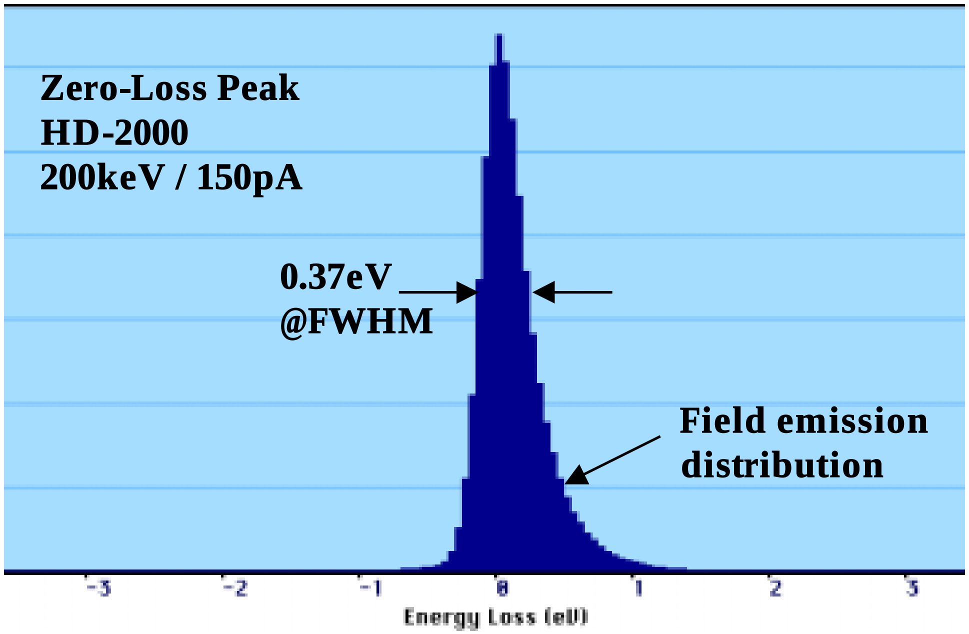


EELS Resolution

- EELS information has excellent energy and spatial resolution
  - Spatial resolution for EELS edges is limited only by probe size
  - Energy resolution is limited by the probe energy distribution

- Typical energy resolution *(depends on gun current)*
  - LaB_6 : >1 eV
  - Warm FEG: 0.6-0.9 eV
  - Cold FEG: 0.35-0.5 eV

- Cold FEG STEM has best energy and spatial resolution



EDXS and EELS comparisons

- EELS and X-ray are complementary techniques
  - EELS excels at lower Z
  - X-ray excels at higher Z
  - EELS and X-ray data can be acquired simultaneously

- Quantification
  - EELS processing doesn't need standards (no k-factors)
  - Standards-based processing is easier with X-ray (currently less developed for EELS since it is less widely used)
  - EELS has greater sensitivity for Z<30 (and most of periodic table)
  - EELS can do absolute quant or relative quant

- Mapping
  - EELS mapping is much faster because of greater signal generation and detection efficiency
  - EELS maps have greater S/N and more pixels for the same mapping times

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EDXS and EELS comparisons

(S)TEM EDS X-ray

- X-rays provide elemental information only
- Inefficient signal collection; inefficient low Z signal generation & detection
  - Slow mapping or poor S/N
- X-ray spectra can contain artifact information from column and other parts of sample
- High detection efficiency for higher Z elements
  - Poor sensitivity to Z<10
- Energy resolution > 120eV causes frequent overlaps
- No sample thickness limitations

(S)TEM EELS

- EELS provides elemental, chemical & dielectric information
- Very efficient in all aspects
  - Higher sensitivity to most elements
  - Very fast mapping technique
- EELS information is highly localized and does not contain sample or column artifacts
- High detection efficiency for lower Z elements
  - Poor sensitivity to a few high Z elements
- Energy resolution 0.3-2eV gives far fewer overlaps (overlaps when edges ~<30eV apart)
- Sample thickness is important - should be less than ~100nm @ 200keV

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Spectrum imaging

- What is Spectrum Imaging?
- Gatan spectrum imaging
  - Overview
  - Correcting for instabilities
  - Basic processing
- Examples

What is Spectrum Imaging?

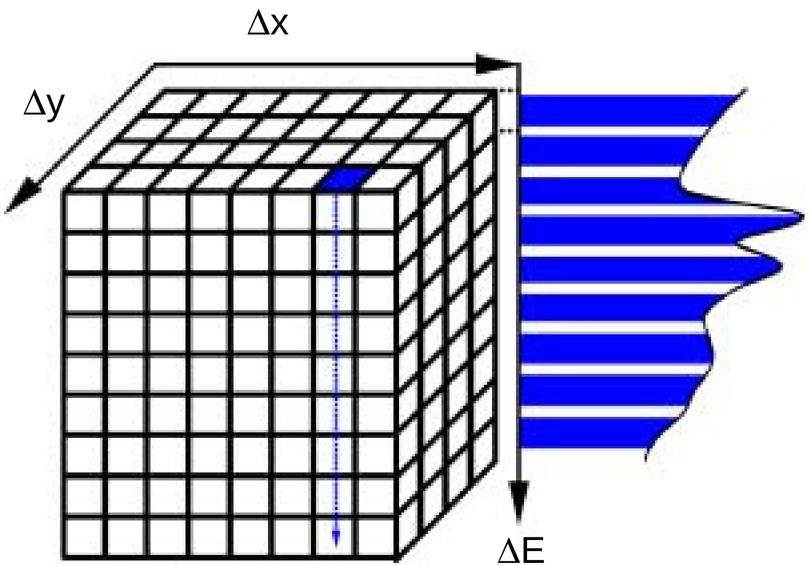
- Spectrum imaging is the technique of acquisition, storage, processing, and analysis of spectroscopic data at each pixel in a digital image
- Conventional image pixels contain one intensity value only
- Spectrum image pixels contain complete spectra
- Produces quantitative elemental maps and profiles

Spectrum image schematic:

Δx, Δy	Spatial dimensions
ΔE	Energy-loss dimension

Vertical columns are spectra

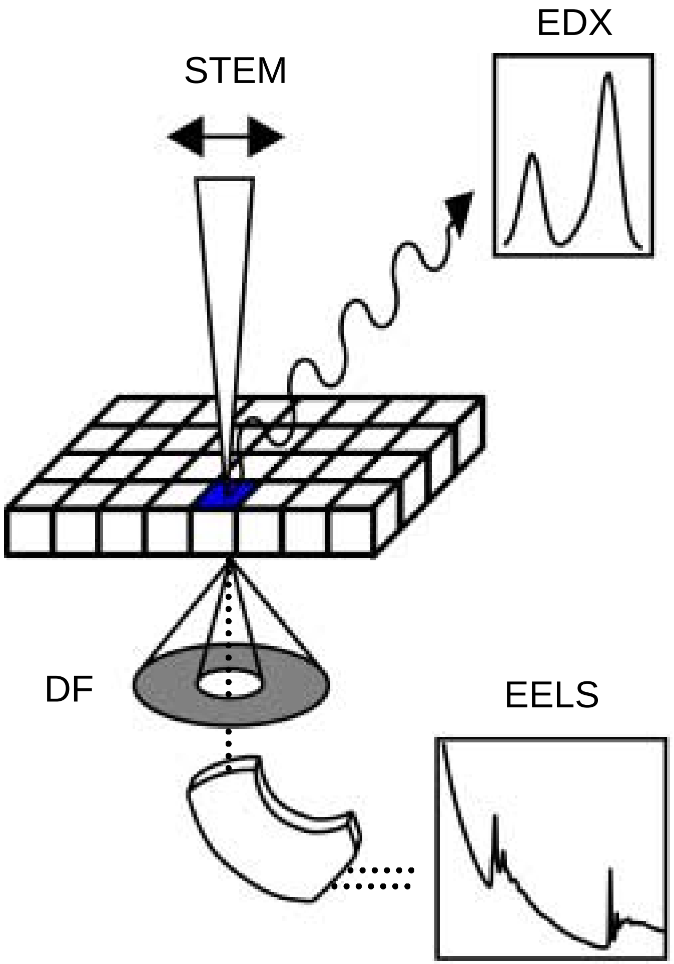
Horizontal planes are energy filtered images



spectrum at pixel Δx_i , Δy_i

Acquisition of Spectrum Images

- A spectrum image is typically acquired in STEM mode by stepping a focused electron probe from one pixel to the next
- The spectrum image data cube is filled one spectrum column at a time
- In STEM it is possible to collect EELS, X-ray or both spectra simultaneously
- Use of the DF or SE signal during acquisition permits spatial drift correction and assurance that information is coming from the desired area



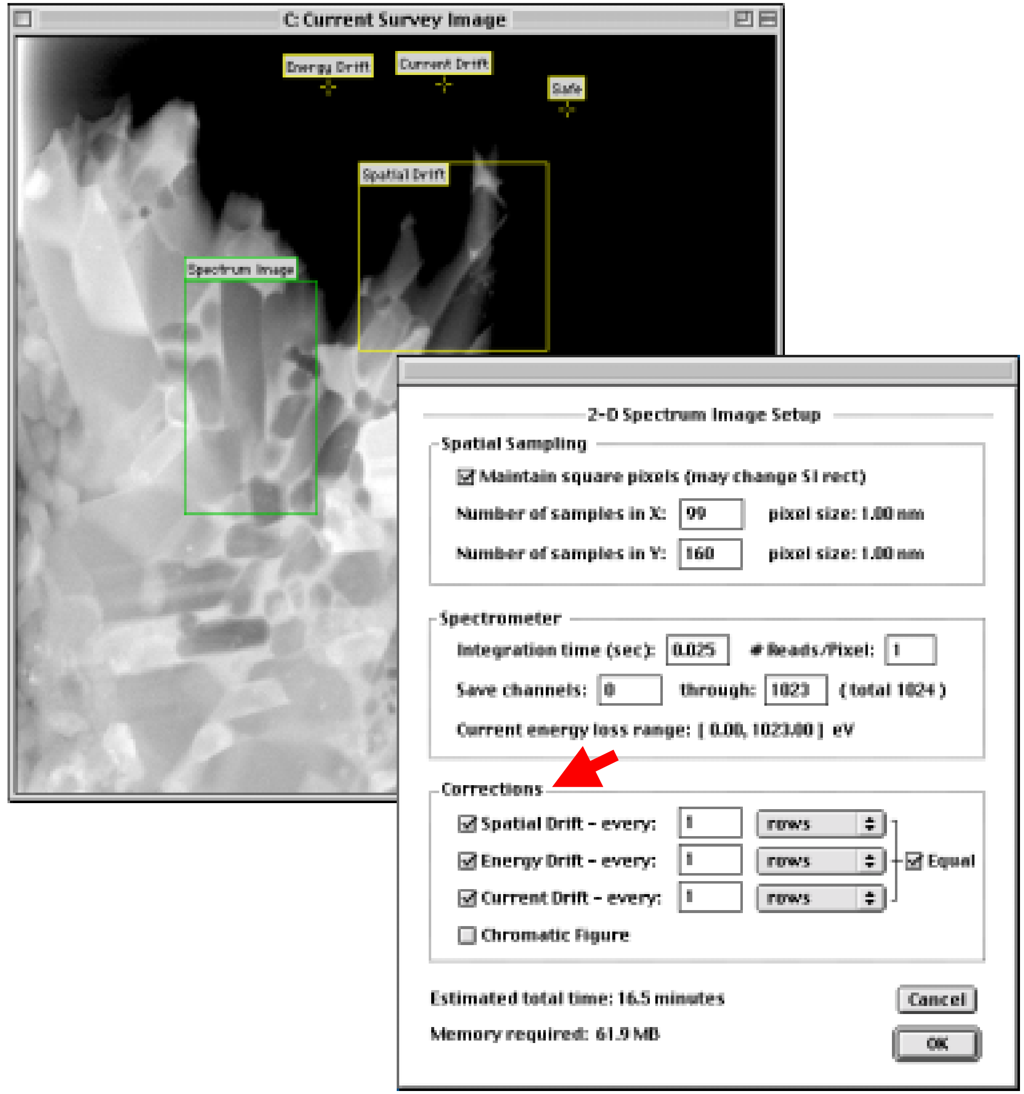
Gatan Spectrum Imaging Overview

- Powerful and highly automated software for producing quantitative maps and line profiles
  - Supplied as plug-ins to Gatan DigitalMicrograph scientific-grade image processing software
  - Intuitive acquisition and analysis features
  - EELS spectrum imaging can map up to 50 pixels/second
  - Corrects for detector artifacts and system instabilities
  - X-ray spectrum imaging available in Fall '99 for many brands

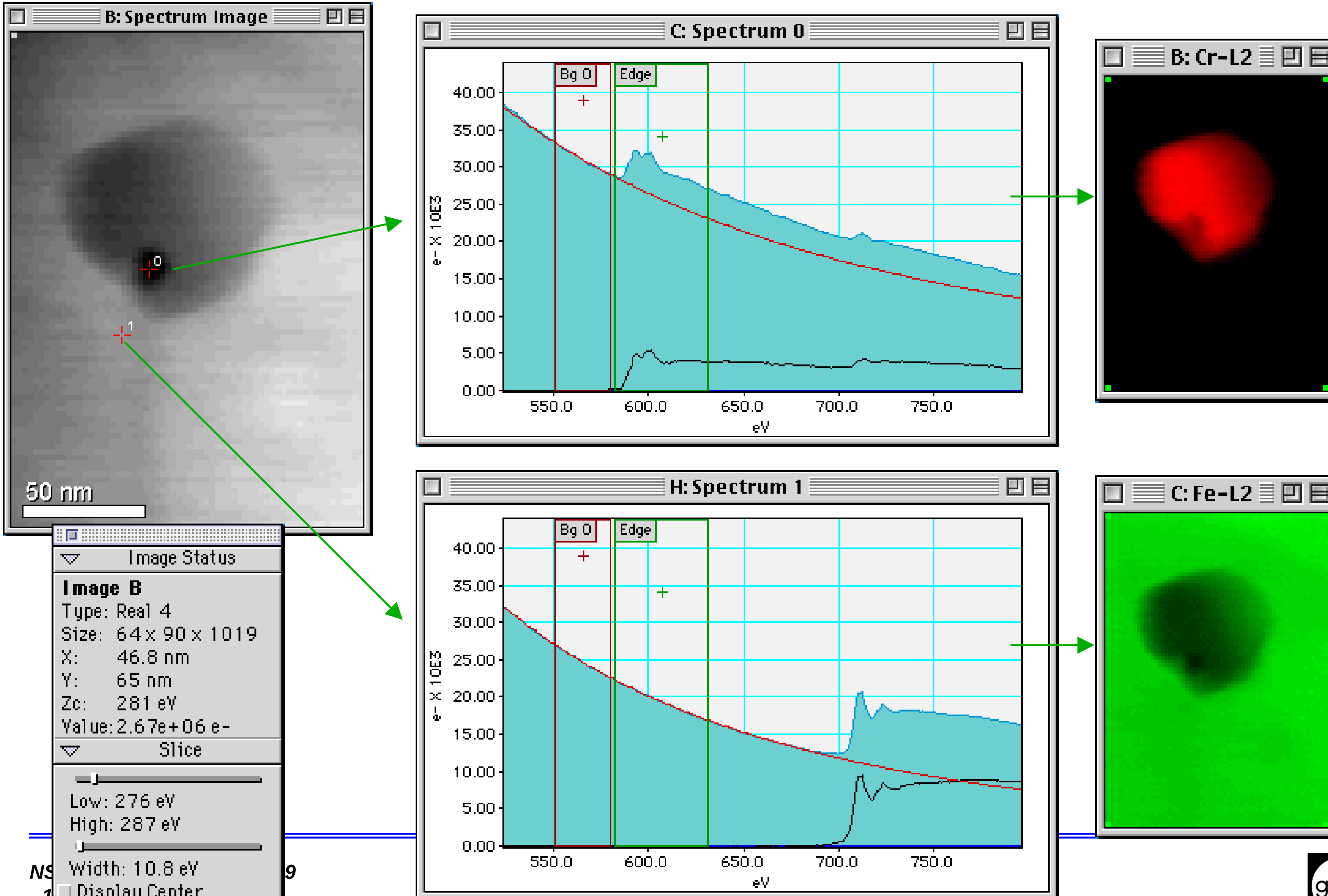
- Requires Gatan DigiScan hardware
  - Controls beam raster
    - 16 bit DACs (max image size 8K*8K)
  - Simultaneous acquisition of multiple signals (BF, DF, SE, etc.)
    - 12-bit ADC samples at 5MHz, can oversample to >16 bits
  - Flexible control of all acquisition parameters (size, speed, aspect ratio …)

Correcting system instabilities

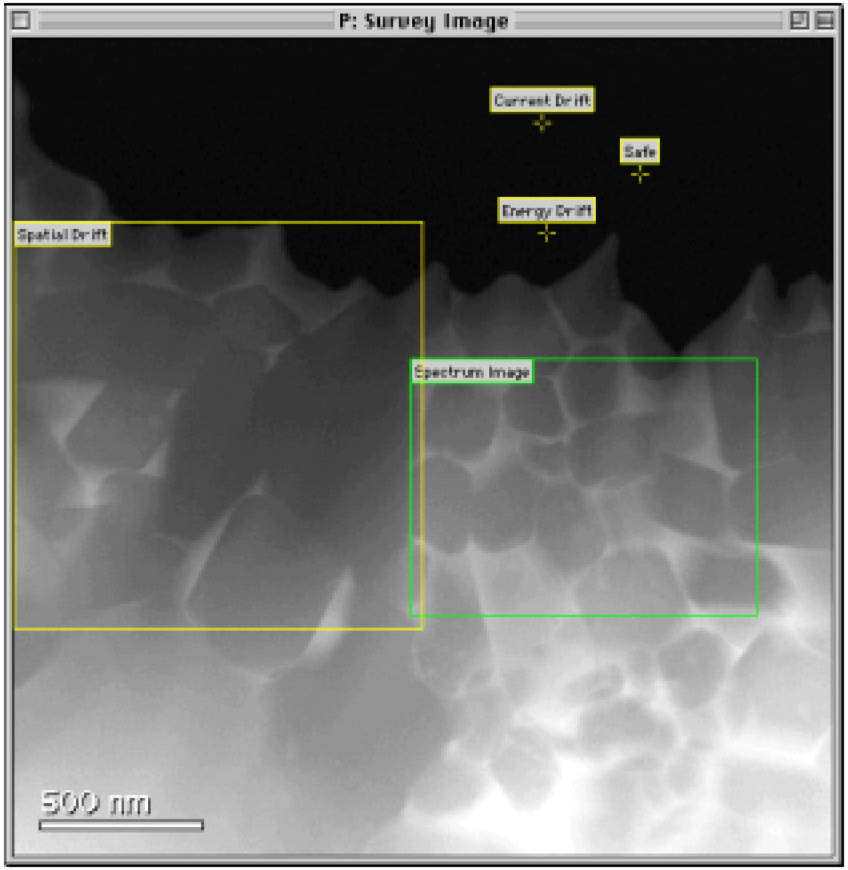
- Gatan software can automatically correct:
  - All detector artifacts
  - System instabilities
    - Spatial drift
    - Energy drift
    - Current drift
  - User needs only to enable correction
  - User can modify defaults for improved control
    - Reduce dose to analyzed area
    - Increase correction frequency

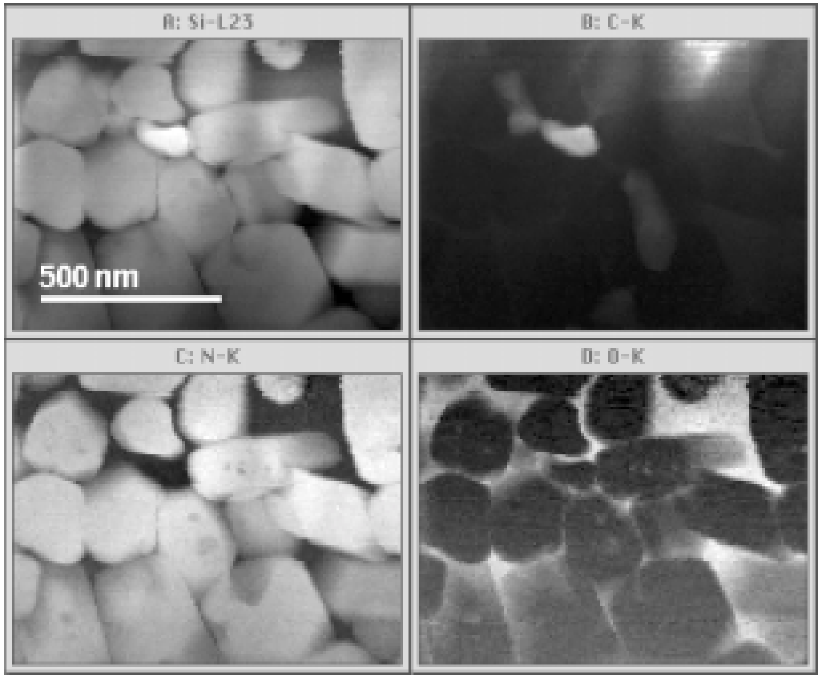


Basic spectrum image processing



Example - Spectrum imaging

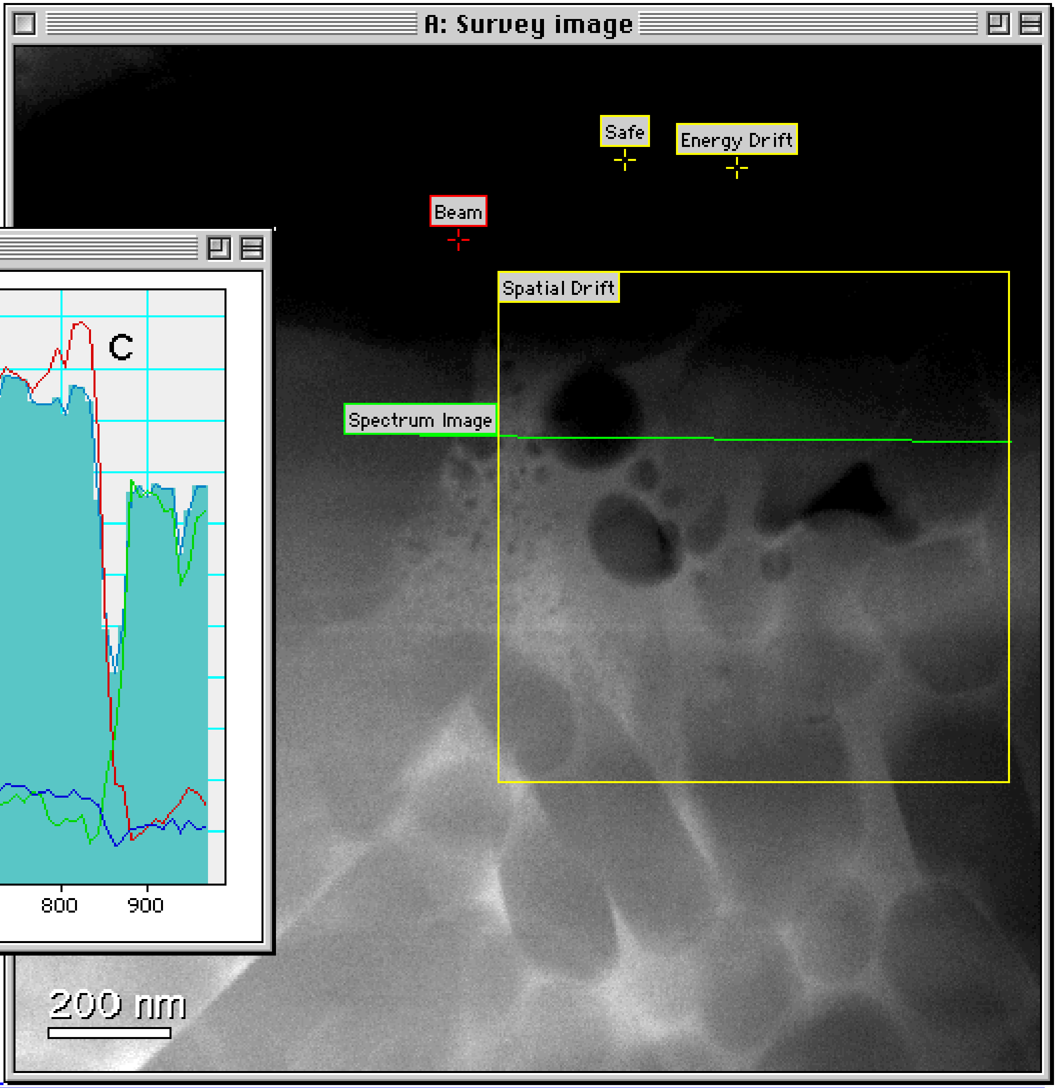


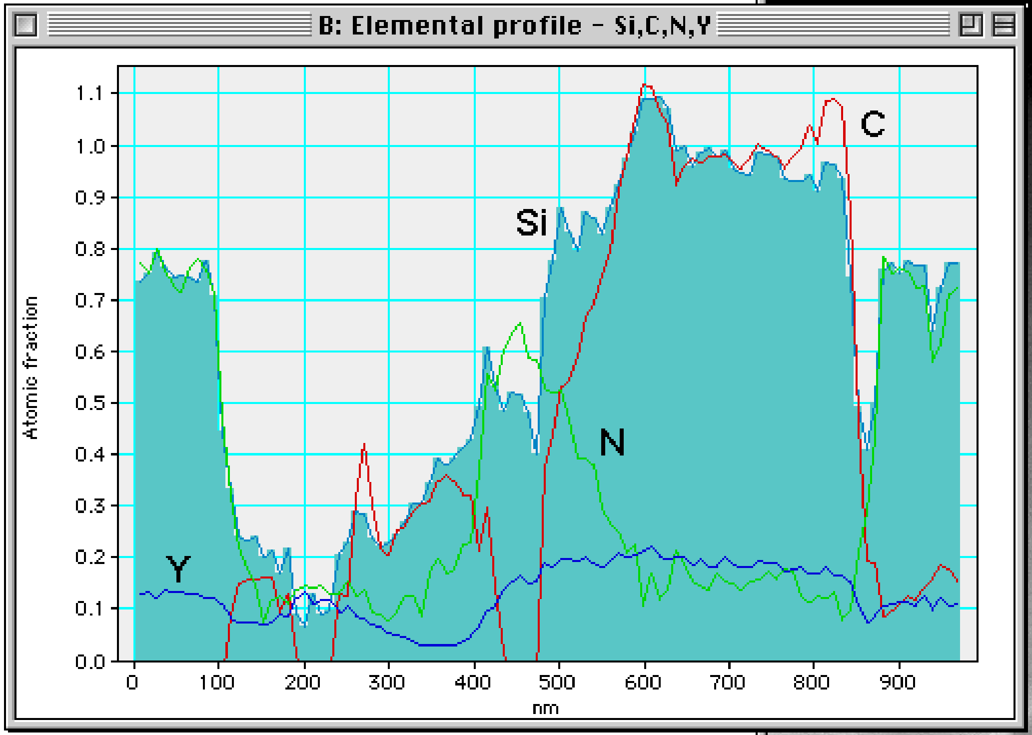


- EELS spectrum image [160,90] pixels, 1022 energy channels
- Acquired on HD-2000 at 200keV

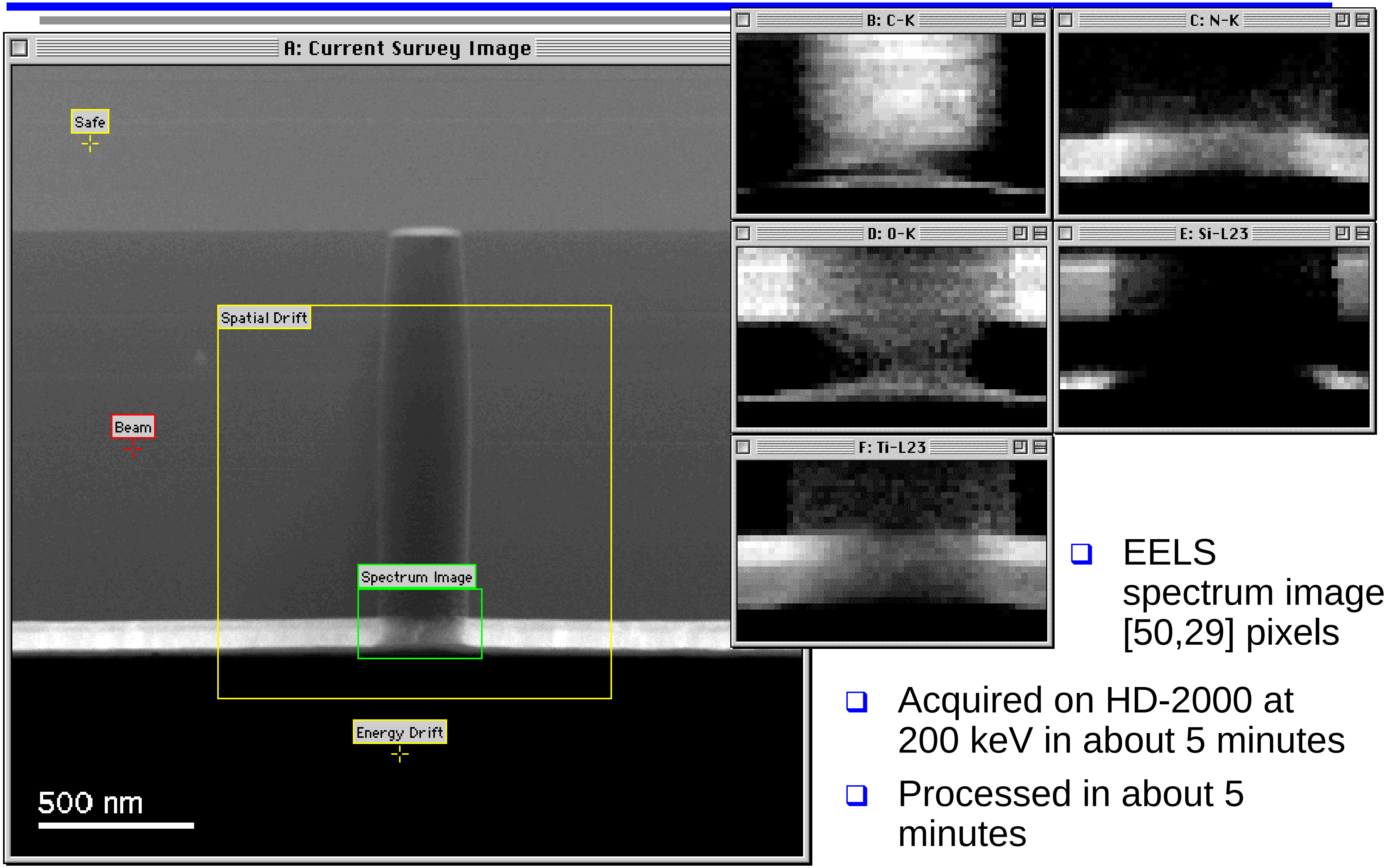
Example - Line profiles

- 100 points acquired in < 1 min
- Acquired on HD-2000 at 200keV





Example - Spectrum imaging



- EELS spectrum image [50,29] pixels
- Acquired on HD-2000 at 200 keV in about 5 minutes
- Processed in about 5 minutes

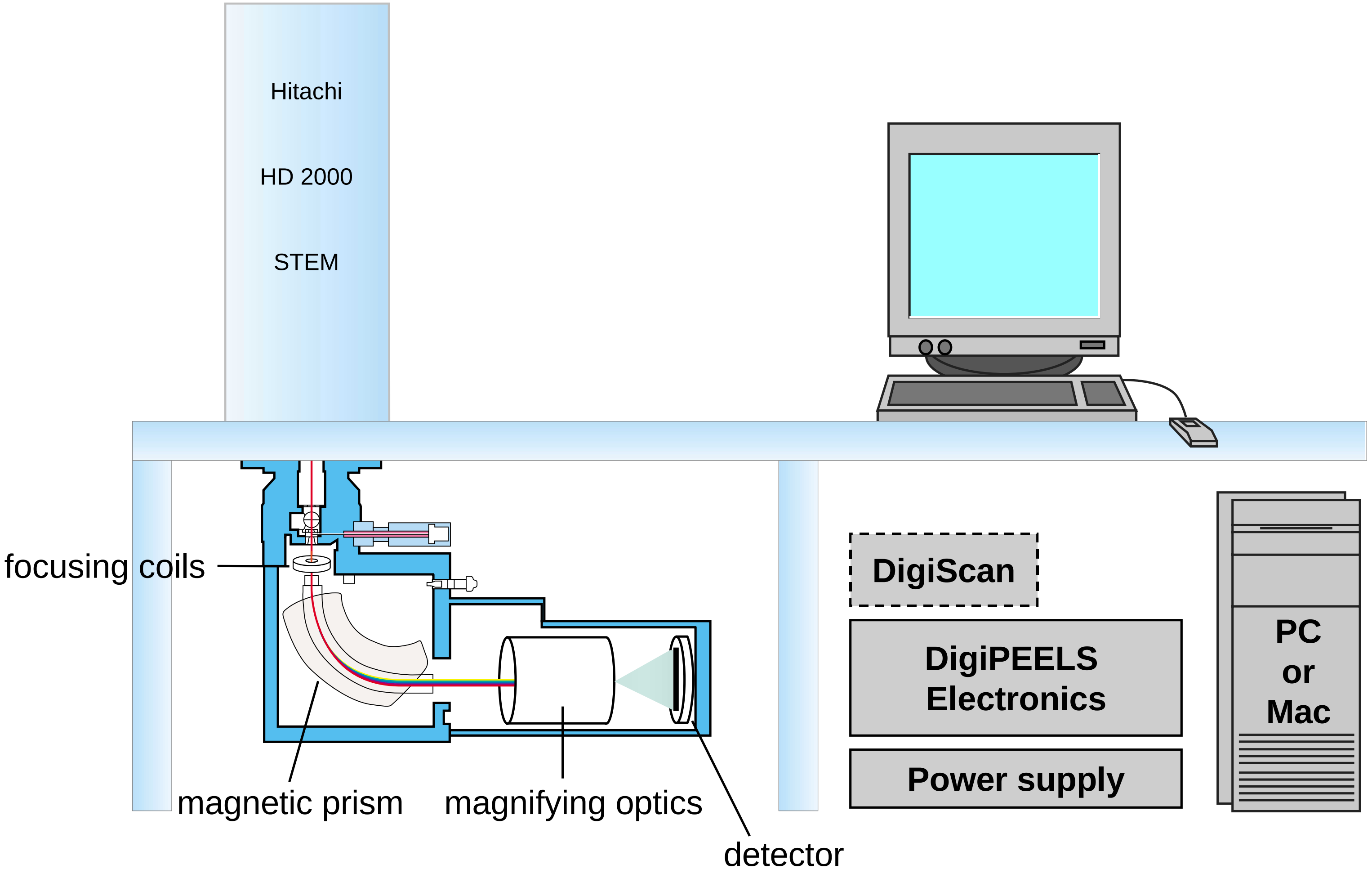
EELS on the HD-2000

- Main DigiPEELS features
- DigiPEELS on the HD-2000
- Configurations for HD-2000
- Major selling points of EELS on HD-2000

Main DigiPEELS features

- DigiPEELS is a parallel-detection energy-loss spectrometer
  - EELS spectrum channels acquired in parallel
- All spectrometer features fully computer controlled
- Linear photodiode array detector
  - 1024 channels
  - 16-bit digitization
  - Maximum of 50 spectra/sec
- Automated EELS processing software

DigiPEELS on HD-2000



Not to scale

EELS is Simplified on HD-2000

- The most difficult issues of EELS data acquisition have been simplified or eliminated on HD-2000 systems !!!
  - Due primarily to lack of TEM viewing chamber and short camera lengths
  - Virtually all scattering angles acquired which simplifies quantification

Tips for good EELS data acquisition

- Work in diffraction or STEM modes
  - Stay away from image mode - chromatic aberration risks
- Control your collection angles
  - Know your collection and converg[illegible] angles
  - Don't use an aperture too [illegible] admit desired electrons
  - Center diffraction [illegible] spectrometer aperture
- Use valid detector artifact corrections

Not necessary on HD-2000 + DigiPEELS systems!

DigiPEELS use on HD-2000 Details

- On HD-2000, EELS is done with projector lens turned off
  - Camera length is extremely short
  - Spectrometer focussing virtually unnecessary
  - Switching between EELS apertures unnecessary
  - Essentially all of the inelastic signal is collected (>100mrad)
  - Precise knowledge of collected scattering angles unnecessary
  - Precise centering of beam in DigiPEELS unnecessary
- HD-2000 has no projector chamber
  - Very good immunity to external fields that affect EELS resolution
- Microscope alignment
  - Installation engineer mechanically aligns DF detector to DigiPEELS
  - Thereafter, user alignment of microscope for imaging also aligns DigiPEELS

EELS Configurations for HD-2000

- DigiPEELS *(available on HD-2000 in Fall '99)*
  - HD-2000 configured DigiPEELS
  - HD-2000 dark field detector with mechanical alignments and hole >= 3mm
  - Recent Macintosh or Windows (NT/98) computer
  - Options
    - Diffraction pattern observation camera mounted above PEELS
      - TV rate camera - Gatan model 692
      - 1K*1K large pixel cameras: Gatan models 792/794

- EELS Spectrum Imaging *(available on HD-2000 in Fall '99)*
  - HD-2000 configured DigiPEELS
  - HD-2000 configured DigiScan (controls beam scan & ultra high-quality imaging)
  - STEM EELS spectrum imaging & DigitalMicrograph image processing software
  - Options
    - X-ray spectrum imaging software (not all brands available Fall '99)

Major selling points of EELS on HD-2000

- Point Analysis
  - Low-Z elements that X-ray does poorly (Li,Be,B,C,N,O,F)
  - Detection of very small concentrations (<100 ppm possible for most elements)
  - Use when X-ray sample/column/detector artifacts are a problem
    - EELS can be used on FIB made specimens without the artifacts X-ray has from around FIB box
  - Use when overlaps are a serious problem (such as separating N, Ti, O)

- Mapping with spectrum imaging
  - All advantages of point analysis
  - Very fast mapping times

- Simplicity
  - HD-2000 has made EELS more simple and accurate than ever

Contacts at Gatan

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