(1) Introduction

Energy-Dispersive X-Ray Spectroscopy (ED-XRS)
- Easy-to-use, well-established technique for elemental analysis.
- Uses incident X-rays or Electrons to induce X-Ray Fluorescence (XRF).
- Scanning Electron Microscope (SEM) used most commonly for e-beam ED-XRS.

SEM-based Microanalysis
- ED-XRS is a common addition to an SEM to provide in-situ x-ray analysis.
- SEM typically configured with sample stage, automation, imaging & x-ray detector.
- Adding x-ray tube provides x-ray induced XRF analysis with low additional cost & no extra lab space.

Combining E-beam and X-ray Source for ED-XRS
- Use advantages of e-beam for light elements & sample imaging.
- Advantage of x-ray excitation for heavier elements, better detection limits, thicker multilayer samples.
- Combine both techniques into common software platform.
- Sample scanning can be used for both e-beam & x-ray sources for elemental x-ray mapping.
- Samples can be analyzed without vacuum if necessary.

(2) Integration

Internal Microtube
- Low-power tube must be small to get close to the sample for good efficiency, & clear pole piece.
- External collimator provides small spot (<2 mm) & clean spectra with minimal contamination lines.

External Tube with Transfer Optic
- Polycapillary optic with microfocus x-ray tube & external passive cooling allows higher power.
- Automatic shutter for beam stability, dual adjusts to align x-ray & e-beams.

Miscellaneous Issues
- Implementation must provide electrostatic, electromagnetic and heat shielding.
- XRF software designed to integrate with standard ED-XRS software for the SEM.
- Provide stage scanning to make multipoint and mapping using x-ray signals.

(3) Spectral Purity – Scatter Spectra

(4) X-beam Spot Size Scan

(5) Implementation

(6) Comparison of E-beam & X-ray ED-XRS Spectra

(7) Glass Standard MDL's

(8) X-ray Mapping with X-beam & SEM x-y Stage

(9) Standardless Analysis of 316 Stainless Steel

(10) Summary: Re-entrant Microtub e

- Tested 3 different types of transmission-target microtubes.
- Only 1 has met the requirements for insertion into an SEM:
  - Target must be thin (1-2 microns) for good low-energy XRF.
  - OD of tube must be small and long enough to get close to sample.
  - Design must efficient to avoid defocusing in long, narrow tube.
  - Heat conduction must be passive to remove heat from inside SEM.
  - Transformers must not interfere with SEM imaging.
  - Ag-anode tube has been successfully used, at 35 kV, 0.1 mA.
  - Beam sizes (~0.5 - 5 mm) determined by aperture size.
  - Collimator must block x rays from walls of tube & SEM.
  - Tube interlocks to vacuum sensor and/or flange microswitches.
  - Critical to adjust sample within 100 microns of focal point.
  - Optimal performance gives 50 x 80 micron (FWHM) spot size.
  - Use stage & tube rotor to co-align x & e-beams.
  - Use in-situ slide on tube to bring to the correct focal point.
  - XY stage scans allow recording of x-ray line scans & mapping.

(11) Summary: External X-beam with Optic

- Use standard “off-the-shelf” microbeam x-ray tube.
- Higher power usable (up to 20 watts, or more with fans).
- Use custom (polycapillary) optic to transport beam to sample.
- Polycapillary transfer function is not uniform.
- Reduced radiation at low and high energies relative to the mid-range.
- Shutter enables beam to be on continuously for stability.
- Automatically closed if interlock failure occurs.
- Interlocked to both vacuum sensor and/or flange microswitches.
- Gas detectors on SEM.
- Shelf” microbeam x-ray tubes.
- Use standard “off-the-shelf” microbeam x-ray tubes.
- Use custom (polycapillary) optic to transport beam to sample.
- Polycapillary transfer function is not uniform.
- Reduced radiation at low and high energies relative to the mid-range.
- Shutter enables beam to be on continuously for stability.
- Automatically closed if interlock failure occurs.
- Interlocked to both vacuum sensor and/or flange microswitches.
- Critical to adjust sample within 100 microns of focal point.
- Optimal performance gives 50 x 80 micron (FWHM) spot size.
- Use stage & tube rotor to co-align x & e-beams.
- Use in-situ slide on tube to bring to the correct focal point.
- XY stage scans allow recording of x-ray line scans & mapping.

(12) Conclusions

- (a) Have successfully integrated both re-entrant and external x-ray tubes within, and attached to, a typical commercial SEM.
- (b) Have demonstrated the feasibility of doing practical XRF within the SEM, and the advantages of XRF over e-beam ED-XRF analysis for many elements, especially traces (lower MDL’s).
- (c) Have integrated XRF software into a standard e-beam microanalysis software package, and demonstrated quantitative standardless analysis, line scans and mapping for both techniques on the same sample.
- (d) The next step will be to fully integrate the e-beam and XRF analysis to automate the analysis of the same sample, where the elements are each analyzed by the most appropriate method (e-beam or x-ray).
- (e) Further options will be explored for future x-ray sources, and improved stage scanning for high-speed x-ray mapping.

(13) References


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