The Use of Scanning Electron Microscopy (SEM) for Microstructural Characterization and Failure Analysis of Materials

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Introduction to Scanning Electron Microscopy

- SEM uses a focused electron beam to probe specimen surfaces, generating multiple signals that carry information about structure, composition, and electronic properties
- Secondary electrons (SEs) provide surface topographic contrast at high resolution
- Backscattered electrons (BSEs) reveal compositional differences due to atomic number variations
- Characteristic X-rays enable elemental identification via Energy Dispersive Spectroscopy (EDS)
- Cathodoluminescence (CL) provides information about band-gap structure, defects, and impurity distributions
- The integration of multiple signal detection modes gives SEM a unique and irreplaceable role in materials characterization

Jiang, R. et al. (2024). Frontiers of Materials Science, 18(4), Article 240709.

Evolution of SEM: From Imaging to Quantitative Analysis

- SEM has evolved from a qualitative imaging tool into a robust quantitative analytical instrument
- Advances driven by improvements in electron source technology, detector design, and digital image analysis
- Correlative microscopy combines SEM-EDS with X-ray computed tomography (XCT) and nanoindentation for comprehensive characterization
- SEM-based fractography has become increasingly sophisticated for failure analysis
- Computational methods now enable quantitative evaluation of fatigue striations for predictive capability

Huang et al. (2023). Materials Characterization, 205, 113331; Maruschak & Maruschak (2024). MethodsX, 13, 102989.

Scope: Two Applications of SEM

Application 1

Microstructural Characterization
— Correlative microscopy of Aba Panu meteorite (SEM-EDS + XCT + nanoindentation)
— SEM-CL mapping of geological and semiconductor microstructures

Application 2

Failure Analysis & Fractography
— Qualitative SEM fractography of dental ceramics and polymers
— Computational methods for quantitative fatigue striation analysis

Huang et al. (2023); Jiang et al. (2024); Alghazzawi et al. (2023); Maruschak & Maruschak (2024).

SEM Technique: Electron-Matter Interactions

- Primary electron beam interacts with specimen atoms via elastic and inelastic scattering
- Elastic scattering: electrons deflected by atomic nucleus with negligible energy loss → backscattered electrons (BSEs); probability increases with atomic number, providing compositional contrast
- Inelastic scattering: energy transfer to specimen atoms → secondary electrons (SEs); low-energy electrons from shallow surface region, highly sensitive to topography
- Inner-shell ionization produces characteristic X-rays as outer-shell electrons fill vacancies → elemental identification via EDS
- Electron excitation of valence electrons generates cathodoluminescence (CL) photons sensitive to band-gap, defects, and impurities

Jiang, R. et al. (2024). Frontiers of Materials Science, 18(4), Article 240709; Huang et T.-J. et al. (2023). Materials Characterization, 205, 113331.

SEM Detection Modes and Analytical Capabilities

- Everhart-Thornley detector: standard SE imaging for surface topography
- Solid-state diode detectors: BSE imaging for compositional (Z) contrast
- Silicon drift detectors (SDDs): rapid EDS elemental analysis and mapping
- EBSD: electron backscatter diffraction for crystallographic orientation analysis
- FIB: focused ion beam milling for site-specific cross-sectioning and TEM sample preparation
- High depth of field makes SEM ideal for examining rough or fractured surfaces

Huang et al. (2023). Materials Characterization, 205, 113331; Alghazzawi et al. (2023). Journal of Prosthodontics, 32(5), e100–e110.

The SEM-CL System: Principle and Acquisition Modes

- CL detector: aluminum-coated parabolic reflector beneath SEM pole piece collects luminescence photons from beam impact point
- Panchromatic mode: captures total luminescence intensity across all wavelengths
- Monochromatic mode: diffraction grating isolates specific wavelengths for selective phase imaging
- Spectral imaging mode: wavelength-resolved spectra collected at each pixel during area scans
- Angular resolution mode: maps directional emission of CL photons using back-projection from parabolic reflector geometry
- Combined with BSE, EDS, and EBSD for comprehensive multimodal characterization from the same microregion

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SEM-CL Applications Across Material Systems

- Geological mineralogy: mineral identification and classification using CL color variation, e.g., tracing origin of quartz grains based on defect structure
- Halide perovskite research: monochromatic CL imaging distinguishes PbI_2 and perovskite phases that appear identical in SE images but exhibit distinct luminescence bands
- Semiconductor analysis: mapping defect density, dislocation distributions, and local band-gap variations at nanoscale spatial resolution
- Non-metallic inclusions: CL perspective may elucidate impact of oxides on microstructural phenomena such as composition segregation and grain orientation

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SEM for Fractographic Analysis

- Fractography: examination of fracture surfaces to determine failure origins and propagation mechanisms
- SEM's high depth of field and variable magnification make it the instrument of choice for identifying fracture features
- Key features observed: failure origin, mirror region, mist, hackle lines, arrest lines, compression curl, and grain morphology
- EDS at fracture origins can identify inclusions and contaminants responsible for failure initiation
- Quantitative methods now enable automated measurement of fatigue striation parameters from SEM images

Alghazzawi et al. (2023). J. Prosthodontics, 32(5), e100–e110; Maruschak & Maruschak (2024). MethodsX, 13, 102989.

Case Study 1: Correlative Microscopy of the Aba Panu Meteorite

Background & Motivation

- Aba Panu: ordinary L3 chondrite meteorite that landed in Nigeria in 2018
- Classified as minimal weathering (W0) and shock stage S4 — pristine condition ideal for microstructural investigation
- Meteorite samples provide information about the formation history and environment of the early solar system
- Multimodal, multiscale characterization is necessary to understand complex heterogeneous mineral assemblages
- Approach: SEM-EDS combined with X-ray computed tomography (XCT) and nanoindentation

Huang, T.-J. et al. (2023). Materials Characterization, 205, Article 113331.

Case Study 1: Experimental Setup

SEM-EDS:

- Nova 200 NanoLab FEG SEM/FIB; specimens polished to 0.25 µm and Au-Pd coated
- EDS mapping identified five mineral phases: olivine, pyroxene, chromite, kamacite, and troilite

XCT:

- Zeiss Versa 520 X-ray microscope at 3.1 µm voxel size
- 3D reconstruction correlated with 2D SEM data to eliminate sampling bias

Nanoindentation:

- Diamond Berkovich tip with continuous stiffness measurement (CSM)
- Measured modulus and hardness of individual mineral phases

Huang, T.-J. et al. (2023). Materials Characterization, 205, Article 113331.

Case Study 1: Key Results

Phase	Vol. %	Modulus (GPa)	Hardness (GPa)
Olivine	~83.5% (combined)	202.7	16.4
Pyroxene	with olivine	175.9	12.7
Kamacite	~1.9%	211.3	3.3
Troilite	~2.0%	108.0	3.9

Key findings:

- 3D XCT revealed kamacite and troilite clusters preferentially distributed along chondrule rims
- EDS line scans showed compositional gradient: decreasing Mg from olivine cores to dendritic matrix
- Correlative approach provided comprehensive structural, compositional, and mechanical understanding

Huang, T.-J. et al. (2023). Materials Characterization, 205, Article 113331.

Case Study 2: SEM Fractography for Failure Analysis

Background & Motivation

- Fractography uses SEM to examine fracture surfaces and determine failure origins and mechanisms
- Alghazzawi et al. studied dental ceramics (zirconia, glass-ceramics) and polymers (PEEK, PEKK, glass fiber composites) under standardized mechanical tests
- Maruschak & Maruschak reviewed computational methods for quantifying fatigue striations on SEM images of polycrystalline materials
- Goal: understand how SEM reveals manufacturing defects and predicts residual life under cyclic loading

Alghazzawi et al. (2023). J. Prosthodontics, 32(5); Maruschak & Maruschak (2024). MethodsX, 13, 102989.

Case Study 2: Methods and Key Results

Qualitative Fractography

- Tests: 4-pt bend, 3-pt bend, biaxial, crunch-the-crown
- Zirconia bars: fracture origins at processing/sintering defects
- Glass-ceramics: failure at surface flaws or fabrication voids
- Polymers: PEKK showed ductile dimples then brittle propagation; fiber composites exposed glassy fibers
- Critical flaws are a function of both material composition and loading geometry

Quantitative Striation Analysis

- Multi-scale analysis: macro-, meso-, and micro-level fracture description
- Striation step ∝ macroscopic crack length (linear in elastic-plastic FCG regime)
- Machine learning methods now rank among the most promising for automated striation recognition
- Fourier analysis, stereocouple methods, and 3D profile analysis address manual measurement limitations
- Corrective D-factor needed when crack front growth is non-uniform

Alghazzawi et al. (2023). J. Prosthodontics, 32(5); Maruschak & Maruschak (2024). MethodsX, 13, 102989.

Summary

- SEM provides a versatile analytical platform capable of probing morphology, composition, crystallography, and optoelectronic properties from the same specimen region
- Case Study 1: Correlative microscopy (SEM-EDS + XCT + nanoindentation) yielded comprehensive phase, compositional, and mechanical characterization of the Aba Panu meteorite; SEM-CL extends analytical reach to band-gap and defect mapping
- Case Study 2: SEM fractography identified failure origins and manufacturing defects in dental materials; computational methods are transforming fractography from descriptive to predictive
- As computational image analysis matures, SEM-based characterization will increasingly serve as both diagnostic and predictive across engineering, geological, biomedical, and semiconductor applications

References

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Thank You

Thank you for your time and attention.

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