



Materials Characterisation: SEM, XRD, Spectroscopy and Thermal Analysis



Materials Characterisation: SEM, XRD, Spectroscopy and Thermal Analysis

Introduction:

Materials characterisation decides whether a cracked bracket, a blistered coating or a suspect batch of resin is explained by evidence or by guesswork. Laboratories often run the wrong technique first, destroy the only fracture surface or report spectra that nobody can link to a cause. This Core Concept course trains laboratory and engineering practitioners to choose between optical microscopy, SEM with EDS, XRD, XRF, spark OES, FTIR, DSC and TGA, prepare specimens correctly and interpret the data together. Participants produce a Characterisation Plan and Results Interpretation Report for a case failed component.

Course Objectives:

- Select a sequence of non-destructive and destructive techniques that answers a defined material question while preserving critical evidence
- Prepare sectioned, mounted, polished and etched specimens and coated SEM samples free of preparation artefacts
- Operate imaging and microanalysis by reading secondary and backscattered electron contrast and EDS spectra, maps and line scans
- Identify crystalline phases from XRD patterns and confirm elemental chemistry with XRF and spark OES results
- Characterise polymers, adhesives and paint films using FTIR spectra, DSC thermograms and TGA mass-loss curves
- Integrate multi-technique data into a defensible characterisation report for failure investigation or incoming material acceptance

Target Audience:

- Engineers who investigate cracked, corroded or worn components and must prove the root cause with laboratory evidence
 - Quality control staff who accept or reject incoming metals, polymers and coated parts against specification
 - Laboratory analysts who prepare specimens and run microscopy, spectroscopy and thermal instruments
 - Research and development staff who compare candidate materials, formulations and surface treatments
 - Materials and corrosion specialists who commission external laboratory work and review the resulting data
-

Course Outline:

Day 1: Characterisation Strategy, Sampling and Optical Microscopy

- Material Question Definition and Technique Selection Matrix
- Non-Destructive First: Visual, Stereo Microscope and Photographic Records
- Sampling Plan, Chain of Custody and Evidence Preservation Log
- Sectioning, Mounting, Grinding, Polishing and Etchant Choice
- Reflected-Light Microscopy: Bright Field, Dark Field and Polarised Light

Day 2: Electron Microscopy, EDS Microanalysis and X-ray Diffraction

- SEM Imaging with Secondary and Backscattered Electron Detectors
- Sputter Coating, Charging Control and Low-Vacuum Imaging of Insulators
- EDS Point Spectra, Elemental Maps and Line Scans with Detection Limits
- XRD Powder Diffraction: Bragg Angle, Peak Positions and Reference Pattern Matching
- Semi-Quantitative Phase Analysis, Residual Austenite and Scale Identification by XRD

Day 3: Bulk Chemistry, FTIR Spectroscopy and Thermal Analysis

- XRF Elemental Screening and Positive Material Identification of Alloys
- Spark OES for Carbon, Sulphur and Trace Alloying Elements
- FTIR with ATR: Polymer, Adhesive and Paint Film Fingerprinting
- DSC Thermograms: Glass Transition, Melting, Crystallinity and Degree of Cure
- TGA Mass-Loss Curves: Filler Content, Moisture and Thermal Stability

Day 4: Hardness, Surface Analysis and Data Interpretation Pitfalls

- Microhardness Traverses on Welds, Case Layers and Decarburised Zones
- Coating Thickness, Cross-Section Adhesion and Porosity Measurement
- Surface-Sensitive Methods Overview: XPS, Profilometry and Contact Angle
- Preparation Artefacts, Overlapping EDS Peaks and Misleading XRD Matches
- Measurement Uncertainty, Reference Materials and Cross-Checking Between Techniques

Day 5: Case Study: Characterisation Plan and Results Interpretation Report

- Case Failed Component Briefing and Service History Review
 - Characterisation Plan Build: Technique Sequence and Sample Map
 - Interpretation Session: Micrographs, Spectra, Diffractograms and Thermograms
 - Incoming Material Acceptance Decision Using Case Batch Data
 - Results Interpretation Report Assembly and Peer Review
-

Skills You Will Gain:

- Characterisation Technique Selection
- Metallographic Specimen Preparation
- Electron Micrograph Interpretation
- EDS Spectrum Evaluation
- Diffraction Pattern Phase Matching
- Infrared Spectral Fingerprinting
- Thermogram Evaluation
- Laboratory Evidence Reporting

Why Attend This Course:

- Leave with a Characterisation Plan and Results Interpretation Report built around a case failed component
- Avoid wasting samples and budget by ordering laboratory techniques in the sequence that protects fracture and deposit evidence
- Challenge external laboratory reports by recognising preparation artefacts, peak overlaps and unsupported conclusions
- Compare laboratory practice with peers from oil and gas, utilities, manufacturing, construction and research organisations

Conclusion:

Sound conclusions about a material come from choosing the right techniques, preparing specimens without artefacts and reading the data together. The week moves from characterisation strategy and optical microscopy, through SEM, EDS and XRD, to XRF, spark OES, FTIR, DSC and TGA, then to hardness, surface methods and interpretation pitfalls. The final day applies the full sequence to a case failed component, and each participant completes a Characterisation Plan and Results Interpretation Report ready for their next investigation or acceptance decision.
