

Modern Material Analysis Instruments Add A New Dimension to Materials Characterization and Failure Analysis

by Dr. Binayak Panda

Materials and Processes Laboratory, NASA Marshall Space Flight Center

Overview

Modern analytical tools can yield invaluable results during materials characterization and failure analysis. Scanning electron microscopes (SEMs) provide significant analytical capabilities, including angstrom-level resolution. These systems can be equipped with a silicon drift detector (SDD) for very fast yet precise analytical mapping of phases, as well as electron back-scattered diffraction (EBSD) units to map grain orientations, chambers that admit large samples, variable pressure for wet samples, and quantitative analysis software to examine phases.

Advanced solid-state electronics have also improved surface and bulk analysis instruments:

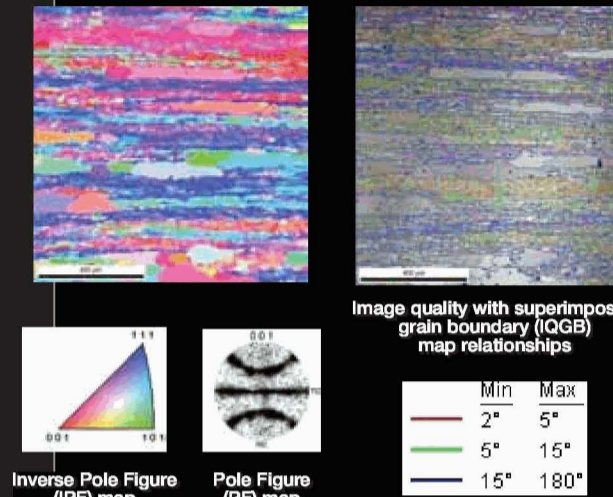
- Secondary ion mass spectroscopy (SIMS) can quantitatively determine and map light elements – such as hydrogen, lithium, and boron – with their isotopes. Its high sensitivity detects impurities at parts per billion (ppb) levels.
- X-ray photo-electron spectroscopy (XPS) can determine oxidation states of elements, as well as identifying polymers and measuring film thicknesses on coated composites. This technique is also known as electron spectroscopy for chemical analysis (ESCA).
- Scanning Auger electron spectroscopy (SAM) combines surface sensitivity, spatial lateral resolution (10 nm), and depth profiling capabilities to describe elemental compositions of near and below surface regions down to the chemical state of an atom.

FE-SEM FEI Quanta 600 F



Provides very high resolution and depth of field. Can examine metallic, non-metallic, and wet samples, up to 12 inches wide. Uses EBSD to evaluate grain orientations. Resolution 2nm@30KV, magnification 7X to 1MX, maximum pressure 20 Torr. Particle analysis systems, SDD energy dispersive spectrometry (EDS).

EBSD images for a partially recrystallized aluminum alloy



Remarks:

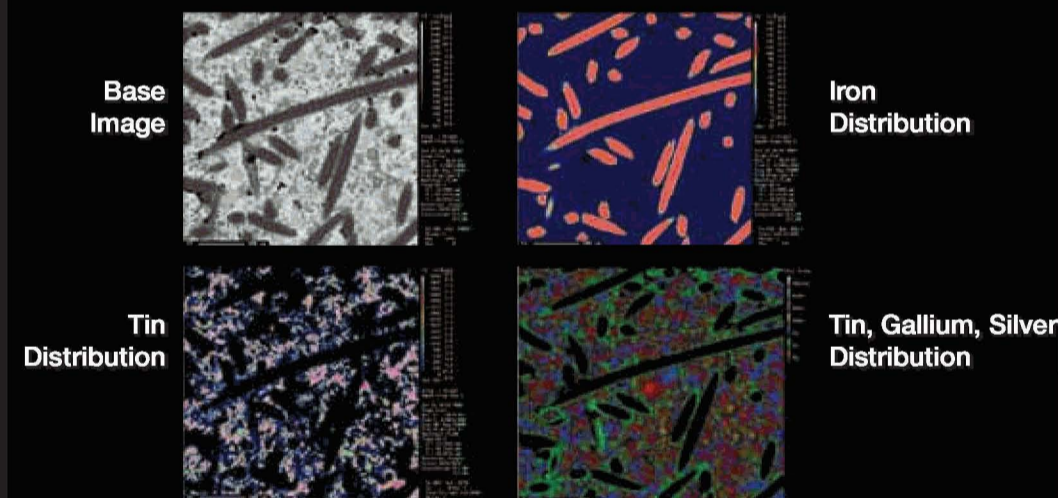
- IPF map shows a mix of multi-oriented, elongated grains with subgrains and large recrystallized grains.
- IQGB map shows no subgrains in recrystallized grains.
- PF map shows a strong “fiber” texture, highly deformed.

Electron Probe Microanalyzer (EPMA) Cameca SX50

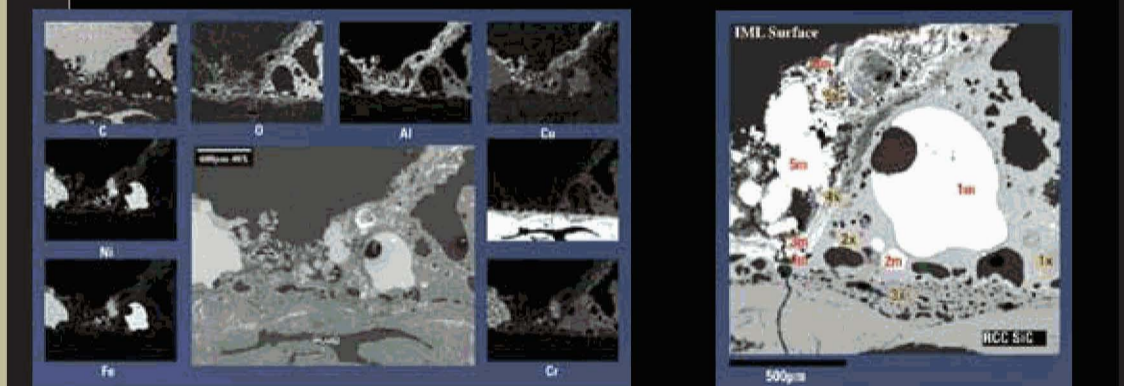
Allows precise elemental analysis at parts per million (ppm) levels. Uses three or more wavelength spectrometers and several crystals for chemical analysis using X-rays. Requires flat polished samples and standards.



Microprobe analysis of an amalgam composite



Perform qualitative and quantitative chemical analysis of slag cross sections



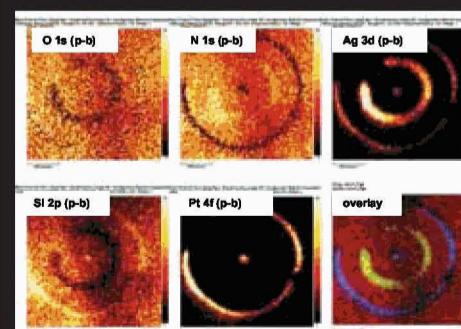
Identify elemental distributions in slag using SEM with x-ray mapping.

Identify precise quantitative chemical composition of slag feature, using electron microprobe.

ESCA – Auger System Kratos AXIS Ultra Delay Line Detector (DLN)

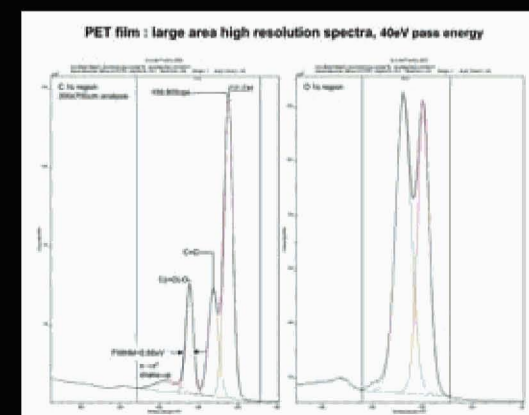


Uses white/monochromatic X-rays for surface chemical analysis, including depth profiling. Can identify elements and their chemical states. Applications include elemental mapping, imaging, and identifying compounds, films, and polymers.

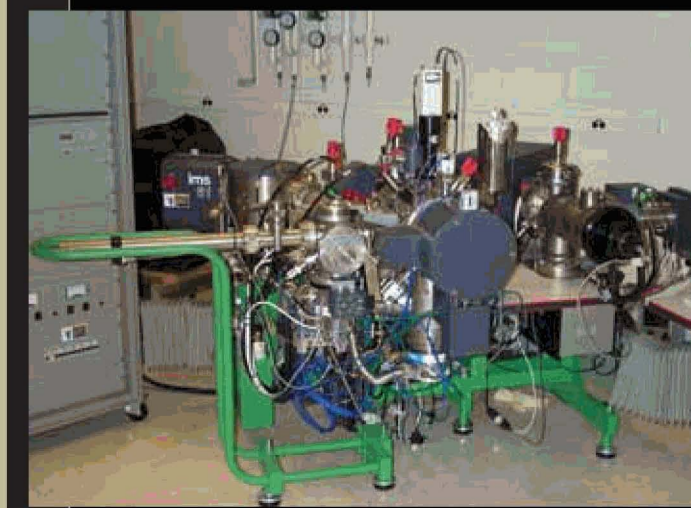


X-ray parallel imaging on a patterned sample.

High-resolution spectra of poly ethylene terephthalate, with three carbon and two oxygen peaks corresponding to different electronic binding levels in the polymer.



SIMS Cameca IMS-6F



Uses secondary ions from sample surfaces to identify elements and their isotopes. Sensitive to all elements and crystal impurities at ppm levels. Can map all elements, including hydrogen. Incorporates elemental distribution, depth profiling, and quantitative analysis. Requires flat polished samples and very high vacuum.