Testing: Techniques and Examples
Making Evidence-based Decisions

ASQ Reliability Division

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Roch J. Shipley, Ph.D., PE, FASM (Metals)
Few if any commercial laboratories offer all of the testing techniques we will be discussing this afternoon.

Much more can be said about all of the tests we have included.

- Plus there are many more tests.

Hopefully, we will provide a framework to decide what tests are appropriate for your situation.
Welcome

- Timothy M. Hicks, PE (Tim)
  - Mechanical Engineer
    - BS – Michigan Technological University
    - MS – Rensselaer Polytechnic Institute
  - Industry – 35 years experience
    - 27 years in design, testing, and manufacturing
    - 8 years in engineering consulting
Welcome

- Michael G. Koehler, PhD
  - Chemist
    - BS – Loyola Chicago
    - PhD – University of Illinois
  - Industry – 32 years experience
    - 21 years in manufacturing and corporate research
    - 11 years in engineering consulting
Welcome

Roch J. Shipley, PhD, PE, FASM
- Materials Engineer
  - BS and PhD – Illinois Institute of Technology
- Industry – 39 years experience
  - 10 years in manufacturing and corporate research
  - 29 years in engineering consulting
WHY is Testing Important?

- TESTING ESTABLISHES & QUANTIFIES
  - Feasibility
  - Product Specifications

- TESTING VALIDATES
  - Product concepts – Prototypes
  - Product Specifications
  - Product performance
  - Manufacturing processes
  - Aging/Wear–out mechanisms
  - Failure Modes

- TESTING MONITORS
  - Manufacturing Processes
  - Product Aging / Wear
  - Product performance
WHEN: Testing Applies Throughout the Product Lifecycle

Pre-Service
- Pre-feasibility
- Feasibility
- Development
- Manufacturing

In-Service
- Burn-in
- Aging/Wearout

Post-Service
- Failure Analysis
- Product Disposal
Testing – Standards (National)

- ASTM (American Society for Testing and Materials) – 12,500+ documents
- ANSI (American National Standards Institute) 9,500+ documents
- SAE (Society for Automotive Engineers) 10,000+ documents
- IEEE (Institute of Electrical and Electronics Engineers) – 1,100+ documents
Testing – Standards (International)

- ISO (International Organization for Standardization) – 22,600+ documents
- International Electrotechnical Commission (IEC) – 9,000+ documents
- International Telecommunications Union (ITU) 4,000+ documents
Laboratory Accreditations

- ISO/IEC 17025
  - General requirements for the competence of testing and calibration laboratories
- A2LA (American Association for Laboratory Accreditation).
  - Accredits calibration and testing facilities to the ISO 17025 standard.
- NADCAP (National Aerospace and Defense Contractors Accreditation Program)
Types of Testing

- Failure Analysis/Root Cause Analysis
  - Should be called \textit{“product performance analysis”}
  - Materials and components don’t really fail
  - Materials and components react to their environment
    - Corrode in aggressive environments
    - Fracture when overloaded or cyclically loaded
    - Degrade due to unanticipated exposure
  - “Failure” to meet expectations (of designer, producer, user, etc.)
Classes of Testing

- Materials Characterization (Analytical Lab) – Our focus today.
  - Analytical Chemistry
  - Metal chemical composition and microstructure
  - Microscopy
  - Surface Analysis
  - Mechanical Testing

- Product Testing (Mechanical Lab/Field)
  - Functional Testing
  - Stress Testing
  - Usability Testing
  - Performance Testing
Materials and Techniques

- Plastics/Polymer Analysis (covalent bonds)
  - Natural (cotton, wool, wood....) and synthetic.

- Metals Analysis (metallic bonds)

- Ceramics (ionic bonds)

- Composite materials – e.g. fiber reinforced, concrete, ...

- Coatings/Surface Analysis

- Corrosion Analysis (Environmental attack)

  "FAILURE ANALYSIS"
Polymer

- Many “mers”
- From Wikipedia

Isoprene

H₂C=CH₂

CH₃

Vulcanization is an example of cross-linking. Schematic presentation of two "polymer chains" (blue and green) cross-linked after the vulcanization of natural rubber with sulfur (n = 0, 1, 2, 3 ...).
Common Issues

- What elements are present in the material?
  - Contamination?

- Bonding / structure
  - Molecular (polymers)
    - Distribution of molecular weights
  - Crystal (metals, ceramics)
  - Crosslinking

- Coating
  - Same questions as above, plus integrity

- Corrosion/environmental attack
  - Analyze corrosion products
  - Samples from environment (if available)
Choosing the right analytical techniques

- What do you want to know?
  - What sensitivity do you require?
    - Ppm, ppb, ..

- What type samples do you have?

- What is the material?
### Characterization Techniques

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<th>Microscopy</th>
<th>Mechanical Testing</th>
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<td>OES</td>
<td>Stereomicroscopy</td>
<td>Hardness</td>
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<td>TGA</td>
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<tr>
<td>DSC</td>
<td>XPS (ESCA)</td>
<td>Borescopy</td>
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## Coupling Characterization Techniques

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<tr>
<td>GC</td>
<td>XRD/XRF</td>
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<td>Combustion</td>
<td>EDS</td>
<td>SEM</td>
<td>Impact</td>
</tr>
<tr>
<td>IC</td>
<td>MS</td>
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Materials and Techniques

- Plastics/Polymer Analysis – “Holy Trinity” used for initial polymer evaluation
  - Fourier Transform InfraRed (FTIR) – Identifies polymer backbone, i.e. polyethylene (PE), polyvinyl chloride (PVC), polycarbonate, etc.
  - Differential Scanning Calorimetry (DSC) – Refines the type of polymer, e.g. PE could be HDPE, LDPE, PEX, OLMWPE, etc.
  - Thermogravimetric Analysis (TGA) – Identifies specific materials, specifically the filler materials commonly used in polymeric materials.
“SPECTRO” = Light Spectrum  – “SCOPY” = To look at

Molecules absorb IR energy, causing the bonds to vibrate. Each “bond type” has a unique absorption creating a spectrum.

An effective analytical technique for quickly identifying the “chemical family” of a substance, organic and polymeric compounds (and to a lesser degree, inorganic compounds) produce a “fingerprint” IR spectrum, which can be compared to extensive reference databases and the unknown component’s chemical family or actual identity may be determined.
Fourier–Transform Infrared Spectroscopy (FTIR)

Ideal uses:

- Characterization and identification of materials, including gases, liquids and solids
- Identification of organic contaminants (e.g. particles, residues, etc.) on the macro and micro scales.
- Quantification of oxygen and hydrogen in silicon and silicon–nitride wafers
- Determination of organic binders and polymeric backbones of coatings
Polyethylene – Comparisons

PE Pure– In Spec

PE– w/ Contaminants
Fourier–Transform Infrared Spectroscopy (FTIR)

Strengths:

- Capable of identifying organic functional groups and often specific organic compounds
- Extensive spectral libraries for compound and mixture identification
- Analysis performed at ambient conditions
- Capable of analyzing small samples
- Can be quantitative with appropriate standards and sample preparation
Fourier–Transform Infrared Spectroscopy (FTIR)

Limitations:

- Limited surface sensitivity
- Limited to specific inorganic species that exhibit an FTIR spectrum
- Sample quantification requires calibration to standards
- Water interferes with analysis of dissolved or suspended samples
- Simple cations and anions cannot be detected
- Metallic materials cannot be analyzed by this technique
Differential Scanning Calorimetry (DSC)

- A thermo-analytical technique for polymeric and non-metallic materials
- A way to identify polymer materials by measuring the amount of energy required to increase the temperature of a material by a certain amount
  - E.g. water will show a temperature plateau at 0 and 100°C.
- DSC data also used to set operating limits for the material.
- One of the most efficient and cost-effective polymer test methods available
Differential Scanning Calorimetry (DSC)

Ideal uses:

- Characterizing relevant phase transitions (e.g. melting, crystallization, glass transition, etc.)
- Comparing quality of two like samples
- Determining the presence of contaminants
  - E.g. salt in water will shift critical temperatures
- Evaluating formulations, blends and effects of additives
- Determining the effects of aging
- Estimating the degree of cross linking
Differential Scanning Calorimetry (DSC)

Strengths:

- Small sample size – smaller than pencil eraser
- Highly accurate measurement of phase transitions and heat capacities
- Very precise temperature control
- Sensitive measurement of subtle or weak phase transitions
- Ability to separate overlapping thermal transitions
**Differential Scanning Calorimetry (DSC)**

**Limitations:**
- Destructive in nature
- No direct elemental information
- Accurate data cannot be obtained when a decomposition or reaction event occurs within the same temperature region as the phase transformation
- Limited use for cross linked materials (elastomers), thermosets.
- Mass of sample has to remain consistent for accurate measurement (e.g. no loss of sample to evaporation or sublimation during testing)
DSC for Polyethylene Plastic

**Heating Cycle**
- First Heat
- Enthalpy (normalized): 109.78 J/g
- Onset: 118.67 °C
- Peak temperature: 128.325 °C

**Cooling Cycle**
- Cooling Step
- Peak temperature: 115.729 °C
- Enthalpy (normalized): 189.27 J/g
- Onset: 110.544 °C
- 73.63 °C

Professional Analysis and Consulting, Inc.
ASQ
Thermogravimetric Analysis (TGA)

- Burns or decomposes material.
  - Useful for investigating thermal stability of solid or liquid materials under ramping temperature in an inert gas or oxygen containing atmosphere.

- Measures changes in sample weight in a controlled thermal environment as a function of temperature or time.

- Separate volatiles, non-volatile organics from minerals.

- Can also be conducted at constant temperature to evaluate thermal stability of materials over time.
Ideal uses:

- Identification of material
- Thermal stability/degradation
- Measure volatiles/moisture
- Screening of additives
- Evolved gas analysis (TGA with MS or FTIR)
- Vaporization or sublimation
- Deformulation of organic/inorganic mixtures
- Loss on drying
- Residue/filler content
Thermogravimetric Analysis (TGA)

Strengths:

- Small sample size
- Analysis of solids and liquids with minimal sample preparation
- Qualitative and quantitative analysis
- Detection of multiple mass loss thermal events from physical and chemical changes of materials
- Separation of overlapping mass loss thermal events
Limitations:

- Evolved products are identified only when the TGA is connected to other instrumentation, e.g. spectrophotometer (i.e. MS or FTIR).
TGA – Polyethylene Plastic

Sample: Bottle 3 Material
Size: 15.6180 mg
Method: Ramp
Comment: C002092

TGA

File: J2092T021001
Operator: SWJ
Run Date: 14-Sep-2018 15:20
Instrument: TGA Q50 V20.2 Build 27

6.300% Weight Loss Ambient to 375°C
(0.0275 mg)

86.39% Weight Loss 375°C to 500°C
(13.49 mg)

1.567% Weight Loss 500°C to 600°C
(0.3057 mg)

Residue:
6.382% at 800°C
(0.9935 mg)

Temperature (°C)

Weight (%)

Universal V4.3A TA Instruments

Professional Analysis and Consulting, Inc.
Materials and Techniques

- Plastics/Polymer Analysis
  - Physical and Mechanical Testing
    - GC·MS, LC·MS – A workhorse in deformation and identification of volatile organic compounds (VOC)
    - Tensile Testing – Identifies the strength, elasticity and plasticity of the material
    - Hardness – Shore A scale and D scale identify the resistance to indentation
Gas Chromatography/Mass Spectrometry (GC–MS)

- Chromatography is a separation method. eg. Dyes in ink spot on paper can separate by allowing solvent to wick up the paper. The different dyes migrate at different rates.

- In Gas Chromatography (GC), a sample is volatilized and carried by an inert gas through a coated capillary column, causing the various components in the mixture to separate.

- Combined with other “spectroscopy methods” you can separate … and identify.

- In the Mass Spectrometry (MS) step, the separated compounds leave the GC column and are identified.
Liquid Chromatography-Mass Spectroscopy (LC-MS)

Sample Prep

Liquid Chromatography
Separation of components
# Mass Spectroscopy Identification of each component

Table 2. Analytes detected in polyethylene samples S1 front and S1 back.

<table>
<thead>
<tr>
<th>PEAK NO.</th>
<th>RETENTION TIME (min)</th>
<th>ION (m/z)</th>
<th>MODE</th>
<th>TENTATIVE MATCH</th>
<th>CAS NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.00</td>
<td>282.32</td>
<td>(+H+)</td>
<td>Irganox 5057 -- component 1, additive</td>
<td>68411-46-1 component one</td>
</tr>
<tr>
<td>2</td>
<td>5.03</td>
<td>563.44</td>
<td>(+H+)</td>
<td>Dimer of Irganox 5057 -- component 1, additive</td>
<td>68411-46-1 component one</td>
</tr>
<tr>
<td>3</td>
<td>5.25</td>
<td>501.46</td>
<td>(-H)</td>
<td>Durad AX38 Component 4, additive</td>
<td>171090-93-0 component four</td>
</tr>
<tr>
<td>4</td>
<td>5.62</td>
<td>338.48</td>
<td>(+H+)</td>
<td>Irganox 5057 -- component 3, additive</td>
<td>68411-46-1 component three</td>
</tr>
<tr>
<td>5</td>
<td>5.62</td>
<td>675.55</td>
<td>(+H+)</td>
<td>Dimer of Irganox 5057 -- component 3, additive</td>
<td>68411-46-1 component three</td>
</tr>
<tr>
<td>6</td>
<td>6.53</td>
<td>1175.91</td>
<td>(-H)</td>
<td>Irganox 1010</td>
<td>6683-19-8</td>
</tr>
<tr>
<td>7</td>
<td>6.80</td>
<td>504.62</td>
<td>(+H+)</td>
<td>RELATED TO CRODAMINE 203</td>
<td>N.A.</td>
</tr>
<tr>
<td>8</td>
<td>6.80</td>
<td>1007.78</td>
<td>(+H+)</td>
<td>Dimer of 504.62</td>
<td>N.A.</td>
</tr>
<tr>
<td>9</td>
<td>7.12</td>
<td>506.75</td>
<td>(+H+)</td>
<td>Crodamine 203, slip agent</td>
<td>96674-02-1</td>
</tr>
<tr>
<td>10</td>
<td>7.12</td>
<td>1011.85</td>
<td>(+H+)</td>
<td>Dimer of Crodamine 203, slip agent</td>
<td>96674-02-1</td>
</tr>
<tr>
<td>11</td>
<td>7.55</td>
<td>705.79</td>
<td>(+H+)</td>
<td>Oxidized Tris(nonylphenyl) phosphite (TNPP), heat stabilizer</td>
<td>26523-78-4 (TNPP)</td>
</tr>
<tr>
<td>12</td>
<td>7.55</td>
<td>1409.83</td>
<td>(+H+)</td>
<td>Dimer of oxidized TNPP</td>
<td>Dimer of oxidized 26523-78-4 (TNPP)</td>
</tr>
<tr>
<td>13</td>
<td>7.66</td>
<td>703.68</td>
<td>(-H)</td>
<td>Oxidized TNPP, heat stabilizer</td>
<td>26523-78-4 (TNPP)</td>
</tr>
<tr>
<td>14</td>
<td>8.66</td>
<td>508.72</td>
<td>(+H+)</td>
<td>RELATED TO CRODAMINE 203</td>
<td>N.A.</td>
</tr>
<tr>
<td>15</td>
<td>8.66</td>
<td>1015.87</td>
<td>(+H+)</td>
<td>Dimer of 508.72</td>
<td>N.A.</td>
</tr>
</tbody>
</table>
Gas (or Liquid) Chromatography/Mass Spectrometry (GC–MS or LC–MS)

Ideal uses:

- Deformulation of polymers, paints, pharmaceuticals, coatings, other mixtures.
- Identifying and quantifying volatile organic compounds in mixtures.
- Outgassing studies.
- Identifying trace impurities in liquids and gases.
- Evaluating extracts from plastics.
- Evaluating contaminants on semiconductor wafers or other technology products.
Materials and Techniques

- Plastics/Polymer Analysis
- Metals Analysis
- Coatings/Surface Analysis
- Corrosion Analysis

“FAILURE ANALYSIS”
Materials and Techniques

- **Metals Analysis**
  - OES – Determine the elemental composition of the metal (e.g. Arc/Spark, ICP, Glow Discharge)
  - Metallography – Microstructural analysis and sample preparation of additional testing
  - Electron Microscopy – High magnification examination and qualitative elemental analysis
  - Hardness – Evaluate heat treatment and other thermal effects, correlated with tensile strength
  - Tensile Testing – Identifies the strength, elasticity and plasticity of the material
  - Impact Testing – Determine material toughness (energy to fracture)
Optical Emission Spectroscopy (OES)

- Elemental composition of a broad range of metals and materials.
- Arc/Spark–OES
- Glow Discharge (GD–OES)
- Inductively–Coupled Plasma (ICP–OES)
OES Marks on Surface
Optical Emission Spectroscopy (OES)

Limitations:

- The sample portion to be analyzed must be completely digested or dissolved prior to analysis (ICP), (Arc/Spark), or flat and able to hold a seal (Glow Discharge).

- Emission spectra can be complex and spectral interferences are possible if the wavelength of the element of interest is very close to, or overlaps that of another element.

- Matrix related effects can create challenges in quantitation.

- Carbon (accurately), nitrogen, hydrogen, oxygen and halogens cannot be determined using this technique.
Example

- Metal content in drinking water.
- Chemistry of steel, aluminum, other structural materials.
Specimens are prepared by mounting a piece of the metal in a resin and polishing the sample to examine metal grain structure, plating layers, secondary phases, corrosion paths, and other material properties.

Specific etchants are used to highlight features such as grain boundaries, phases, and inclusions.

Examination of the polished specimens is done with reflected-light microscopes or scanning electron microscopes.
Metallography

Ideal uses:

- Examining microstructures of materials in cross-section, including cleanliness, grain size, phase identification, porosity, etc.
- Evaluating coating/plating thicknesses/case depth
- Evaluating joint configuration and quality (i.e. soldering, brazing, welding)
- Identifying processing issues
Strengths:

- Provides relatively simple, quick method for evaluating material properties by examining the structure and phases present.
- Properly prepared samples provide a wealth of information regarding the quality of the raw material, as well as how the material was formed, shaped, and finished.
Limitations:

- Requires highly trained and skilled personnel to properly prepare specimens without creating false or misleading structures.

- Requires significant knowledge to properly interpret the microstructural features and relate these features to material properties.

- Requires cutting, grinding and polishing of specimens, which means it is destructive and important data can be lost during sample preparation.
Stereomicroscopy

- First step, especially fracture surfaces
- 5X – 150X
- Three-dimensional visual perspective with exceptional depth of field
- Retain color information
Scanning Electron Microscopy (SEM)

- Provides high-resolution and high depth-of-field images of the sample surface and near-surface.
- One of the most widely used analytical tools due to the extremely detailed images it can quickly provide.
- Coupled to an auxiliary Energy Dispersive X-ray Spectroscopy (EDS) detector, SEM also offers elemental identification of nearly the entire periodic table.
Scanning Electron Microscopy (SEM)

Ideal uses:

- High resolution surface topography images
- Defect identification and mapping
- High magnification, high depth-of-field imaging
- Powder morphological analysis
- Coating/Plating thickness measurements
Scanning Electron Microscopy (SEM)

Strengths:

- Rapid, high-resolution imaging
- Excellent depth of field (100 times optical microscopy)
- Versatile platform that supports other analytical techniques (EDS, WDS, BSE, etc.)
- Low vacuum mode enables imaging of insulating and hydrated samples
Scanning Electron Microscopy (SEM)

Limitations:

- May need to etch planar samples for contrast
- Size restrictions may require cutting sample
- Ultimate resolution is a strong function of the sample chemistry and stability of the electron beam
- For best resolution, the samples should be conductive or sputter-coated with a conductive material
- Electron beam interaction with the surface may alter delicate surface features
SEM Exam Finds Surface Cracks
A chemical analysis method that can be coupled with the major electron beam based techniques:

- Scanning Electron Microscopy (SEM)
- Transmission Electron Microscopy (TEM)

The impact of the electron beam on the sample produces x-rays that are characteristic of the elements present on the sample.
EDS – Cu contamination carried through lubrication system and deposited on surface of steel part
Energy Dispersive Spectroscopy (EDS)

Ideal uses:

- Elemental microanalysis and particle characterization
- Elemental composition of small areas using SEM/TEM imaging
- Identification of coatings and plating materials
Energy Dispersive Spectroscopy (EDS)

Strengths:

- Quick identification of elements present
- Quick, “first look” compositional analysis
- Versatile, inexpensive and widely available
- Quantitative results may be possible with proper sample preparation and standards.
Energy Dispersive Spectroscopy (EDS)

Limitations:

- Chamber size limitations for samples
- Generally, semi-quantitative results, at best
- Beam penetration affects results when looking at thin films or contaminants on surfaces
- Numerous elemental peak overlaps are possible, requiring knowledgeable analysts to evaluate spectra
Hardness applies to—metals and nonmetals alike—and is defined as the resistance of the material to deformation, penetration, scratching, or other physical force.

Two types of hardness testing: bulk hardness and microhardness

<table>
<thead>
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<th>Microhardness</th>
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<tr>
<td>Rockwell</td>
<td>Knoop</td>
</tr>
<tr>
<td>Brinell</td>
<td>Vickers</td>
</tr>
<tr>
<td>LEEB</td>
<td></td>
</tr>
<tr>
<td>Shore</td>
<td></td>
</tr>
<tr>
<td>Barcol</td>
<td></td>
</tr>
</tbody>
</table>
Ideal uses:

- Quality Control/Quality Assurance check for heat treatment and processing
- Non-destructive, indirect method for determining approximate tensile strength (Hardness $\propto$ Tensile Strength)
- Method for determining total and effective case depth (microhardness)
- Identifying components or equipment that has been altered or damaged by heat exposure
Hardness

Strengths:

- Quick and convenient test of material properties
- Portable
- Inexpensive
- Requires minimal training to provide accurate, reproducible results
Hardness

Limitations:

- Hardness values are provided using arbitrary scales.

- No direct correlation between individual scales. All conversions are based on empirical data, and are material-specific.

- Hardness testing is material-specific, as well as size and shape dependent, requiring the operator/coordinator to understand the limitations of each method and appropriateness.

- Accuracy and repeatability of the results are highly dependent upon the operator.
Vickers Microhardness Impression
Example

- Manufacturer of steel parts unilaterally increased hardness by changing heat treatment.
- Higher strength might be considered good, except
  - Susceptible to stress corrosion cracking / hydrogen embrittlement
  - Delayed failure.
Tensile Testing

- A destructive mechanical testing method – uniaxial tension until failure
- Universal load machine equipped with extensometers and load cells measures load/deflection data vs. load (stress)
Tensile Testing

Ideal uses:

- Directly determining material properties including: ultimate tensile strength ($\sigma_{uts}$), yield strength ($\sigma_{ys}$), percent elongation, and reduction of area

- Indirectly determining materials properties Young’s Modulus, Poisson’s ratio, strain hardening characteristics

- Verifying material meets design requirements for strength and ductility
Tensile Testing

Strengths:

- Highly repeatable and widely relied upon
- Relatively simple, inexpensive test
- Commonly used to determine materials properties specified by designers
- Tensile strength is a material property, independent of size, so a reduced section can be tested
Tensile Testing

Limitations:

- The test method is destructive, therefore material will be consumed.
- Assumes an isotropic material. If the material is non-isotropic, multiple specimens must be tested.
- Data is only valid at the temperature the test was performed.
- Testing performed at a constant strain rate or a constant loading rate, so it does not provide dynamic information.
- Typically only used with ductile materials.
Materials and Techniques

- Plastics/Polymer Analysis
- Metals Analysis
- Coatings/Surface Analysis
- Corrosion Analysis

“FAILURE ANALYSIS”


Materials and Techniques

Coatings/Surface Analysis

- **SIMS** – Secondary Ion Mass Spectroscopy – Highly sensitive surface analysis technique for evaluating coating compositions and surface features

- **XPS/ESCA** – (X-Ray Photoelectron Spectroscopy / Electron Spectroscopy for Chemical Analysis)

- **Electron Microscopy** – High magnification examination and qualitative elemental analysis

- **Metallography** – Microstructural analysis and sample preparation of additional testing
Secondary Ion Mass Spectroscopy (SIMS)

- Detects very low concentrations of dopants and impurities.

- Provides elemental depth profiles over a wide depth range from a few angstroms (Å) to tens of micrometers (µm).

- The sample surface is sputtered/etched with a beam of primary ions (usually O$_2^+$ or Cs$^+$) while secondary ions formed during the sputtering process are extracted and analyzed using a mass spectrometer (quadrupole, magnetic sector, or Time of flight.)

- The secondary ions can range in concentration from matrix levels down to sub–ppm trace levels.
Secondary Ion Mass Spectroscopy (SIMS)

Ideal uses:

- Dopant and impurity depth profiling
- Composition and impurity measurements of thin films
- Bulk chemical analysis including boron, carbon, oxygen and nitrogen in silicon
Primary Ion Mass Spectroscopy (SIMS)

Strengths:

- Excellent detection sensitivity for dopants and impurities in the ppm to ppb range
- Depth profiles with excellent detection limits and depth resolution
- Small area analysis (≥ 5μm)
- Detection of all elements and isotopes
Limitations:

- Destructive analysis
- No chemical bonding information
- Analysis is element-specific
- Sample must be solid and vacuum-compatible
X–Ray Photoelectron Spectroscopy (XPS)
Also known as Electron Spectroscopy for Chemical Analysis (ESCA)

- Used to determine quantitative atomic composition and chemistry.

- A surface analysis technique with a sampling volume that extends from the surface to a depth of approximately 50–100Å. The process works by irradiating a sample with monochromatic X–rays, resulting in the emission of photoelectrons whose energies are characteristic of the elements within the sampling volume.

- An elemental analysis technique that is unique in also providing chemical state information for the detected elements, such as distinguishing between sulfate and sulfide forms of sulfur.
Ideal uses:

- Surface analysis of organic and inorganic materials, stains, or residues
- Determining composition and chemical state information from surfaces
- Depth profiling for thin film composition
- Thin film oxide thickness measurements
X-Ray Photoelectron Spectroscopy (XPS)

Strengths:

- Chemical state identification on surfaces
- Identification of all elements except for hydrogen (H) and helium (He)
- Quantitative analysis, including chemical state difference between samples
- Applicable for a wide variety of samples, including insulating materials (i.e. paper, plastics, glass, etc.)
- Depth profiling with matrix-level concentration
- Oxide thickness measurements
X-Ray Photoelectron Spectroscopy (XPS)

Limitations:

- Detection limits are usually about 0.1 at%
- Smallest analytical area is approx. 10 μm
- Limited specific organic information
- Sample compatibility with UHV environment necessary
Materials and Techniques

- Plastics/Polymer Analysis
- Metals Analysis
- Coatings/Surface Analysis
- Corrosion Analysis

“FAILURE ANALYSIS”
Corrosion Analysis

- IC – Water chemistry
- XRD – Identification of crystalline species and determination of residual stress using x-rays
- XRF – Identification of elements present based on the fluorescence created by interaction with x-rays
- Electron Microscopy – High magnification examination and qualitative elemental analysis
- Metallography – Microstructural analysis and sample preparation for additional testing
Materials and Techniques

- Plastics/Polymer Analysis
- Metals Analysis
- Coatings/Surface Analysis
- Corrosion Analysis

“FAILURE ANALYSIS”
Failure Analysis

ANY AND ALL THE TECHNIQUES
Material what it is supposed to be in all respects?
- Chemical composition?
- Structure and properties (heat treatment)?
- Manufacturing process?

Environment what designer anticipated?
- Applied stresses well below material capability?
  - Including contact, vibratory (fatigue)?
- Temperature below material capability? Or too low?
- Chemical environment not aggressive to material?

If material and environment pass, then consider operation, maintenance, combination of factors, ...
Failure Analysis Approach Example

- Plastic tubes carrying water based solution.
- Leaking after some time in service. Why?
- Examine fracture surface.
  - Evidence of environmental stress cracking.
  - Material not compatible.
- Material confirmed to meet specification by FTIR, DSC, TGA.
- Extract contaminants.
  - Identify – GC MS
- Literature review identifies aggressive chemicals.
- Confirmed presence in the water.
- Root cause – water system was not sealed to prevent contamination.
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Summary of Capabilities

Materials / Metallurgical Engineering  
Mechanical Engineering  
Chemistry  
Electrical Engineering  
Structural / Civil  
Aviation  
Agricultural

Accident Reconstruction  
Failure Analysis  
Fire Origin and Cause  
Machinery and Equipment  
UAS – Drone Scene documentation  
All Types of Vehicles  
Consumer Products  
Manufacturing Processes

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