



MICRO-CONTAMINATION DEPARTMENT PRESENTS : *FAILURE ANALYSIS*

What is Failure Analysis and what is it designed for? - Failure Analysis is a process of evaluating and comparing the design property parameters as they relate to load, temperature, fatigue, etc. Failure analysis is designed to: identify the *failure modes* (the way the product failed); identify the *failure site* (where in the product failure occurred) ; identify the *failure mechanism* (the physical phenomena involved in the failure); determine the *root cause* (the design, defect, or loads which led to failure); and recommend failure prevention methods.

Why is Failure Analysis important? - Failure analysis and prevention are important functions to all of the engineering disciplines. The materials engineer often plays a lead role in the analysis of failures, whether a component or product fails in service or if failure occurs in manufacturing or during production processing. In any case, one must determine the cause of failure to prevent future occurrence, and/or to improve the performance of the device, component or structure.

Visually inspection the external condition of the device is very important as it often provides valuable information for subsequent analysis using the equipments as listed below :

Scanning Electron Microscopy (SEM) -A focused beam of electrons is rastered across a sample surface, the raster scan being synchronous with that of a cathode ray tube (CRT). The brightness of the CRT is modulated by the detected secondary electron current from the sample, such that the viewing CRT displays an image of the variation of secondary electron intensity with position on the sample. This variation is largely dependent on the angle of incidence of the focused beam onto the sample, thus yielding a topographical image. Different detectors can be used to provide alternative information, e.g., a backscattered electron detector will provide average atomic number information. An auxiliary energy dispersive X-ray EDS detector provides elemental identification analyses from boron to uranium.

Specimen Requirements

- Insulating samples must be coated with a conductive film
- Vacuum compatible
- <125 mm diameter, <50 mm height

Unique Advantages

- High resolution with little sample preparation
- Large depth of field allows use with rough samples
- Rapid qualitative analysis of particles and small areas by EDS

Energy Dispersive X-ray Spectrometry (EDS) -An EDS attachment to an SEM permits the detection and identification of the x-rays produced by the impact of the electron beam on the sample thereby allowing qualitative and quantitative elemental analysis. The electron beam of an SEM is used to excite the atoms in the surface of a solid. These excited atoms produce characteristic X-rays which are readily detected. By utilizing the scanning feature of the SEM, a spatial distribution of elements can be obtained.

Specimen Requirements

- Insulating samples must be coated with a thin conductive film
- <125 mm diameter, <40 mm height
- Quantitative analysis requires standard materials of known concentration be available for comparison for best results
- Quantitative analysis requires flat, polished homogeneous samples

Unique Advantages

- Rapid identification of particles, films, and unknown bulk materials
- Very fast elemental images and line scans
- Excellent first look technique for new problems

Jeol SEM equipped with Oxford EDS



Field Emission Scanning Electron Microscopy (FE-SEM) -The Field Emission Scanning Electron Microscope (FE-SEM) is similarly configured to a conventional SEM except that a cold field emission electron source is used, which permits higher image resolution to be attained, increased signal to noise ratio, and increased depth of field.

Specimen Requirements

- Insulating samples must be coated with a conductive film, or imaged at very low keV
- Ultra-high vacuum compatible
- <32 mm diameter, <20 mm height

Unique Advantages

- High resolution at low accelerating voltages
- High resolution with little sample preparation
- Large depth of field technique compatible with rough samples image



Hitachi FE-SEM equipped with Oxford EDS

Fourier Transform Infrared Microscopy (micro-FTIR) - Individual chemical bonds, as well as groups of bonds, vibrate at characteristic frequencies. When exposed to infrared (IR) radiation, molecules selectively absorb radiation at frequencies that match those of their allowed vibrational modes. Measurement of the absorption of IR radiation by the sample as a function of frequency produces a spectrum that can be used to identify functional groups and consequently structure. However, vibrations that do not yield a change in dipole moment do not absorb IR radiation. For example, O₂ and N₂ do not absorb IR radiation. Consequently, IR spectra can be obtained in air. FTIR provides specific information about chemical bonding and molecular structure, making it useful for analyzing organic materials and certain inorganic materials.

Specimen Requirements

- The FTIR microscope can be used to analyze samples as small as ~15 microns.
- FTIR is normally a bulk analysis technique. However, using Attenuated Total Reflectance (ATR), FTIR can acquire a spectrum from the top 1-2 mm of material
- Detection limits are 0.1-1 wt %

Unique Advantages

- Chemical bonding information
- Molecular "fingerprint"

Shimadzu FTIR Micro-scope



Ion Chromatography (IC) - It is the separation and quantification of anions and cations using High Performance Liquid Chromatography (HPLC). HPLC is an analytical technique based on the separation of the components of a mixture in solution by selective adsorption. There are basically three modes of separation: liquid/liquid, liquid/solid, and molecular size. Once the components have been separated, they are measured by a conductivity detector.

Analytical Information

- Material identification - Reference materials are needed
- Quantification - Concentrations of components can be determined by establishing a standard curve of known concentrations.
- Sensitivity - detection limits down to part per billion levels.

Sample Requirements

- Sample must be able to go into solution with a solvent compatible to the IC system, or the components of interest must be able to be extracted from the sample
- Components of interest must cause a change in the conductivity of the solute to be detected.

Unique Advantages

- Ion Chromatography allows the separation and detection of:
 - trace ionic species such as F⁻, Br⁻, Cl⁻, NO₂⁻, NO₃⁻, HPO₄⁻², SO₄⁻², Li⁺, Na⁺, K⁺, Mg⁺², Ca⁺², Sr⁺², Ba⁺²
 - ammonium and low molecular weight amines
 - quaternary ammonium compounds
 - oxyhalides
 - weak organic acids
 - silicates
 - aliphatic and aromatic sulfonic acids
 - carbohydrates
 - amino acids

Shimadzu IC



Dionex IC



Gas Chromatography/Mass Spectrometry (GC/MS) -In GC/MS, the sample is injected into a gas chromatograph which separates the various components of the sample based on size and/or polarity. The separated components then go into a mass selective detector. The resulting mass spectrum allows for the identification of the components using standard reference libraries

Analytical Information

- Material identification - The mass spectrum of the unknown compound can be searched against our computerized reference library of over 275,000 spectra for identification
- Quantification - The concentration of the various compounds in a sample can be quantified by establishing a standard curve of known concentrations of each material.

Sample Requirements

- Physical State - Sample must be either a liquid or a gas for a direct injection. The compounds offgassed by a solid can be analyzed using head space analysis.
- Sample size - several microliters for a liquid or a gas, between 0.5 - 1 gram for headspace analysis of a solid.

GC/MS

Unique Advantages

- Separates and identifies the components of a mixture, also quantifies known compounds

Of the many non-destructive evaluation (NDE) techniques available during failure analysis, **optical microscopy** is the most used, because it is rapid and convenient in locating and identifying most external defects



Optical Microscopy

Once the *failure site* is located, usually through electrical testing and visual inspection, **micro-sectioning** is often necessary to identify the exact *failure mechanism and root cause*. The first step in microsectioning often involves potting in an epoxy resin, to ensure planarity and prevent any additional damage from occurring. The potted sample is then cut or ground to reach the area of interest. Final polishing with diamond or alumina particles removes any surface damage from previous steps. Analysis of the polished micro-section can be performed using a variety of techniques, such as optical and electron microscopy and energy dispersive spectroscopy (EDS).

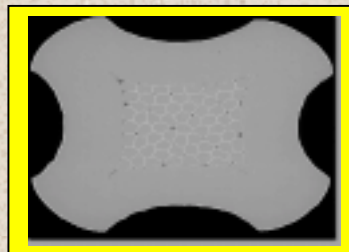
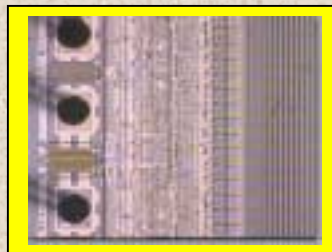
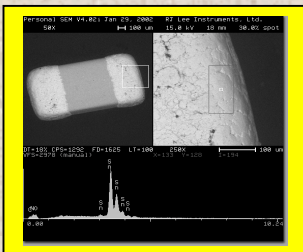


Photo of images taken by SEM/EDX and cross section

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