Handbook Of Analytical Methods For Materials

Practical Solutions to Materials Problems Through Technology and Innovation

Materials Evaluation and Engineering, Inc.

Materials Evaluation and Engineering, Inc.
13805 1st Avenue North, Suite 400
Plymouth, MN 55441-5447

Phone: (763) 449-8870
Fax: (763) 449-8699
Toll Free: (888) 349-8870

Website: www.mee-inc.com
Email: mail@mee-inc.com

Table of Contents

Introduction 5

Atomic Force Microscopy (AFM) 7

Auger Electron Spectroscopy (Auger) 11

Energy Dispersive X-ray Spectroscopy (EDS) 13

Fourier Transform-infrared Spectroscopy (FTIR) 15

Gas Chromatography / Mass Spectroscopy (GC-MS) 17

Ion Chromatography (IC) 19

Light Microscopy (LM) 21

Metallographic Study 23

Microindentation Hardness Testing 25

Nanoindentation Hardness Testing 27

Quantitative Chemical Analysis 29

Rockwell Hardness Testing 33

Scanning Electron Microscopy (SEM) 35

Secondary Ion Mass Spectrometry (SIMS) 39

Thermal Analysis (DSC, TGA) 41

X-ray Photoelectron Spectroscopy (XPS or ESCA) 43

Sample Preservation And Handling 45

About Materials Evaluation And Engineering, Inc. 49

Blank Page

INTRODUCTION

This booklet is a basic primer on selected methods for the characterization and evaluation of materials and products. It has been compiled by the technical staff of Materials Evaluation and Engineering, Inc. (MEE) as an aid to our customers and our colleagues.

The handbook offers basic explanations and practical examples of the analytical methods that we use to find solutions to our customers' materials-related problems. The handbook topics include the methods available in our laboratory, as well as, other common materials characterization techniques. We hope this information helps you choose the evaluation method best suited to solving your materials puzzles and aids in your understanding of the data you receive from the materials characterization work that we provide.

MEE is an independent, privately-owned company that provides practical, cost-effective technical support in materials characterization and engineering. Our lab specializes in the characterization of mechanical, morphological, and chemical properties of materials, especially on the microscopic scale. Our technical staff is also expert at failure analysis and product development activities related to the effective processing and end-use application of materials. We apply traditional and advanced analytical methods, coupled with sound engineering, to provide our customers with practical solutions to materials-related problems.

We will continue to develop this handbook by occasionally updating the current sections and by adding new information. The latest version will be available on our web site at www.mee-inc.com. Please feel free to call on us anytime with your questions on your current projects, and we will do our best to assist you.

Larry D. Hanke, P.E.
CEO/Principal Engineer

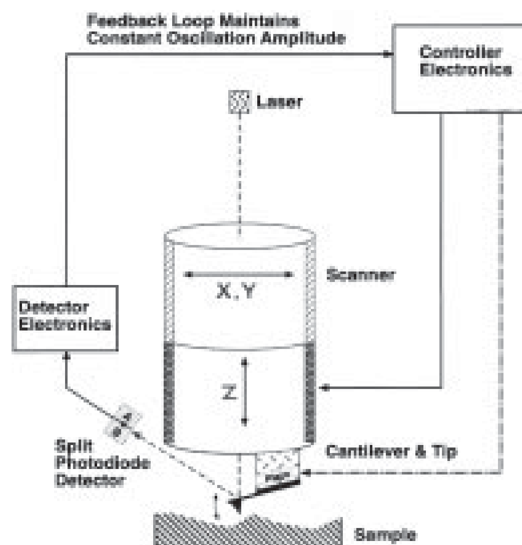
Copyright © 2001 by Material Evaluation and Engineering, Inc. All rights reserved. Printed in the United States of America. Except under the United States Copyright Act of 1976, distribution of this publication is allowed if the entire publication is maintained intact as originally published.

Blank Page

ATOMIC FORCE MICROSCOPY

DESCRIPTION OF TECHNIQUE

Atomic Force Microscopy (AFM) is a form of scanning probe microscopy (SPM) where a small probe is scanned across the sample to obtain information about the sample's surface. The information gathered from the probe's interaction with the surface can be as simple as physical topography or as diverse as measurements of the material's physical, magnetic, or chemical properties. These data are collected as the probe is scanned in a raster pattern across the sample to form a map of the measured property relative to the X-Y position. Thus, the AFM microscopic image shows the variation in the measured property, e.g,. height or magnetic domains, over the area imaged.

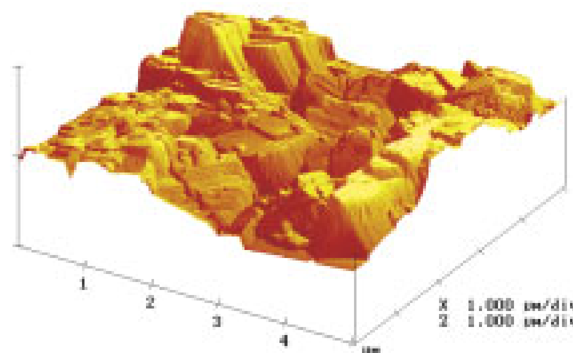


Sketch of AFM Instrument Configuration

The AFM probe has a very sharp tip, often less than 100 Å diameter, at the end of a small cantilever beam. The probe is attached to a piezoelectric scanner tube, which scans the probe across a selected area of the sample surface. Interatomic forces between the probe tip and the sample surface cause the cantilever to deflect as the sample's surface topography (or other properties) changes. A laser light reflected from the back of the cantilever measures the deflection of the cantilever. This information is fed back to a computer, which generates a map of topography and/or other properties of interest. Areas as large as about 100 µm square to less than 100 nm square can be imaged.

ANALYTICAL INFORMATION

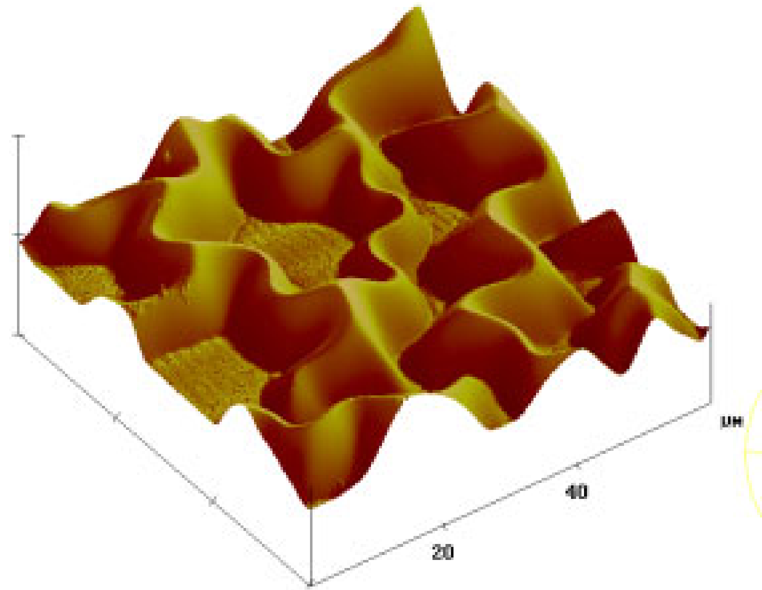
Contact Mode AFM - The AFM probe is scanned at a constant force between the probe and the sample surface to obtain a 3D topographical map. When the probe cantilever is deflected by topographical changes, the scanner adjusts the probe position to restore the original cantilever deflection. The scanner position information is used to create a topographical image. Lateral resolution of <1 nm and height resolution of <1 Å can be obtained.



Surface Profile of Crystalline Material

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

Intermittent Contact (Tapping Mode) AFM - In this mode, the probe cantilever is oscillated at or near its resonant frequency. The oscillating probe tip is then scanned at a height where it barely touches or "taps" the sample surface. The system monitors the probe position and vibrational amplitude to obtain topographical and other property information. Accurate topographical information can be obtained even for very fragile surfaces. Optimum resolution is about 50 Å lateral and <1 Å height. Images for phase detection mode, magnetic domains, and local electric fields are also obtained in this mode.

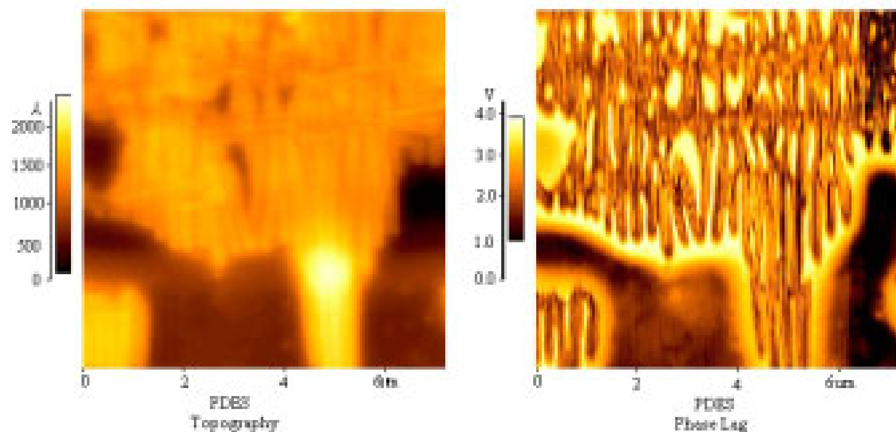


AFM Image of Defect on Coated Glass

Lateral Force Microscopy - This mode measures the lateral deflection of the probe cantilever as the tip is scanned across the sample in contact mode. Changes in lateral deflection represent relative frictional forces between the probe tip and the sample surface.

Phase Detection Microscopy - With the system operating in Tapping mode, the cantilever oscillation is damped by interaction with the sample surface. The phase lag between the drive signal and actual cantilever oscillation is monitored. Changes in the phase lag indicate variations in the surface properties, such as viscoelasticity or mechanical properties. A phase image, typically collected simultaneously with a topographical image, maps the local changes in material's physical or mechanical properties.

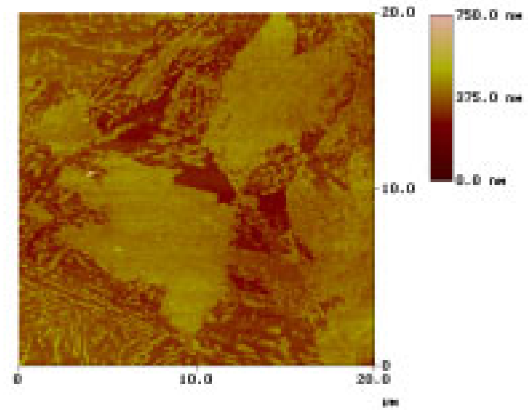
Magnetic Force Microscopy - This mode images local variations in the magnetic forces at the sample's surface. The probe tip is coated with a thin film of ferromagnetic material that will react to the magnetic domains on the sample surface. The magnetic forces between the tip and the sample are measured by monitoring



Height and Phase Mode Image of a Polymer Sample

 Copyright © 2001 by Materials Evaluation and Engineering, Inc.

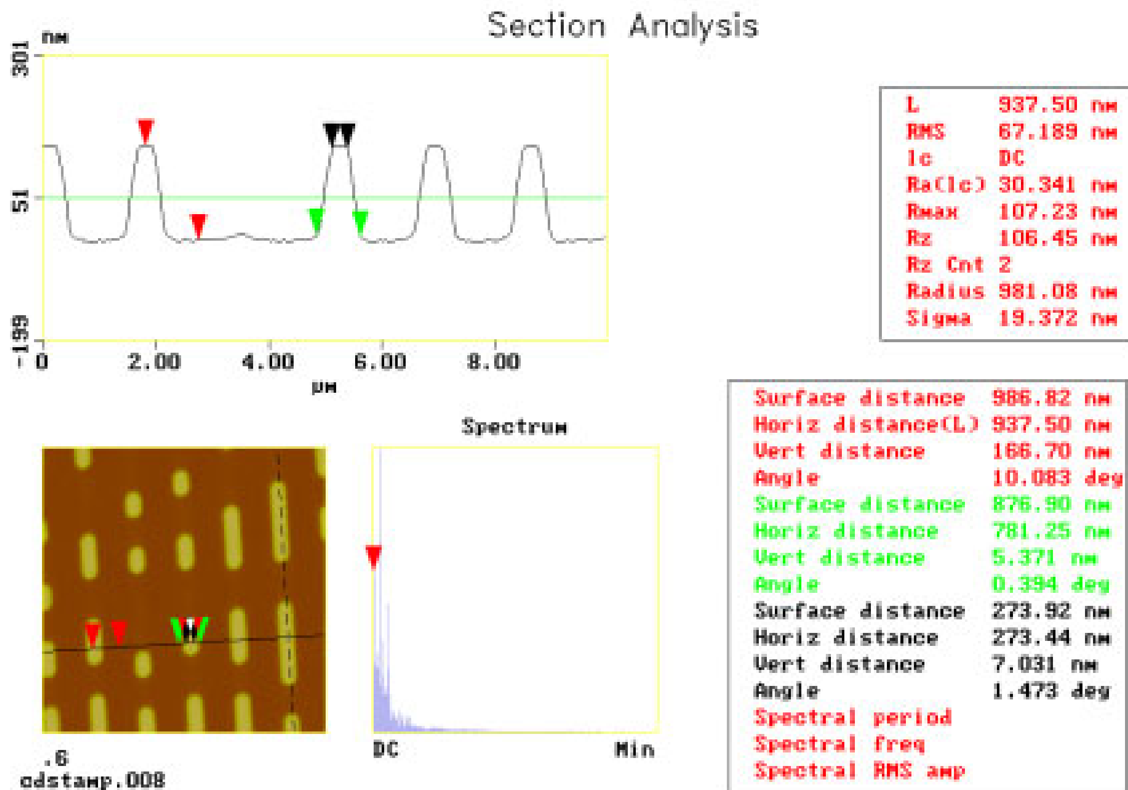
cantilever deflection while the probe is scanned at a constant height above the surface. A map of the forces shows the sample's natural or applied magnetic domain structure.



Top View AFM Image of Steel Microstructure

Image Analysis - Since the images are collected in digital format, a wide variety of image manipulations are available for AFM data. Quantitative topographical information, such as lateral spacing, step height, and surface roughness are readily obtained. Images can be presented as two-dimensional or three-dimensional representations in hard copy or as digital image files for electronic transfer and publication.

Nanoindentation - A specialized probe tip is forced into the sample surface to obtain a measure of the material's mechanical properties in regions as small as a few nanometers. (See the Handbook section on Nanoindentation Hardness Testing.)



Feature Measurements for CD Stamper

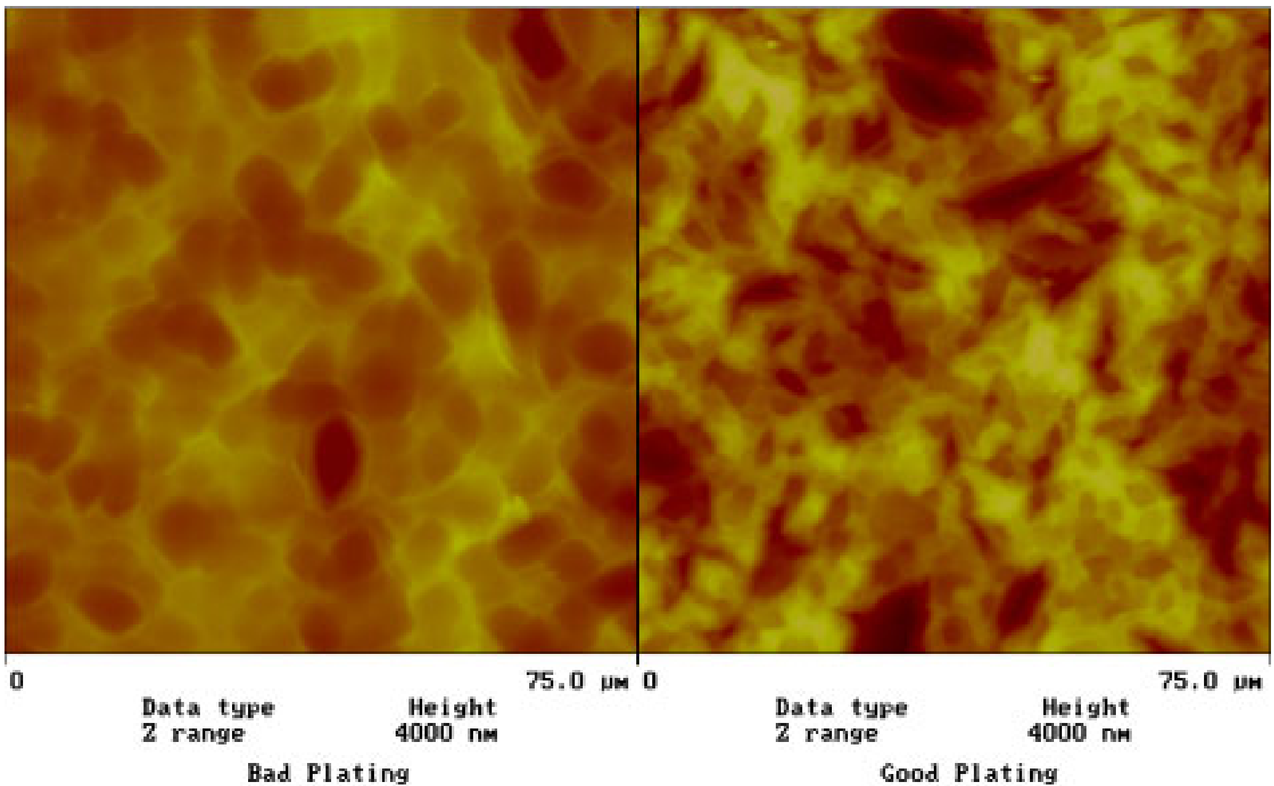
 Copyright © 2001 by Materials Evaluation and Engineering, Inc.

TYPICAL APPLICATIONS

- 3-dimensional topography of IC device
- Roughness measurements for chemical mechanical polishing
- Analysis of microscopic phase distribution in polymers
- Mechanical and physical property measurements for thin films
- Imaging magnetic domains on digital storage media
- Imaging of submicron phases in metals
- Defect imaging in IC failure analysis
- Microscopic imaging of fragile biological samples
- Metrology for compact disk stampers

SAMPLE REQUIREMENTS

No sample preparation is typically required. Samples can be imaged in air or liquid. Sample height is limited to about 1.5 inches. Areas up to 8 inches in diameter can be fully traversed without repositioning. Larger samples can be fixtured for imaging within a limited area. Total surface roughness in the image area should not exceed about 6 μm.



AFM Images of Gold Plating for Wire Bond Failure Analysis

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

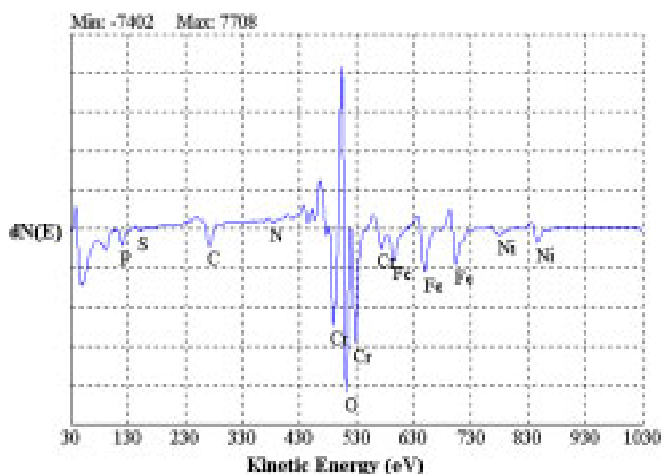
AUGER ELECTRON SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

Auger Electron Spectroscopy (AES) provides information about the chemical composition of the outermost material comprising a solid surface or interface. The principal advantages of AES over other surface analysis methods are excellent spatial resolution (< 1 µm), surface sensitivity (~20 Å), and detection of light elements. Detection limits for most elements range from about 0.01 to 0.1 at%.

AES uses a primary electron beam to excite the sample surface. When an inner-shell electron is ejected from a sample atom by the interaction with a primary electron, an electron from an outer shell fills the vacancy. To compensate for the energy change from this transition, an Auger electron or an x-ray is emitted. For light elements, the probability is greatest for the emission of an Auger electron, which accounts for the light-element sensitivity for this technique.

The energy of the emitted Auger electron is characteristic of the element from which it was emitted. Detection and energy analysis of the emitted Auger electrons produces a spectrum of Auger electron energy versus the relative abundance of electrons. Peaks in the spectrum identify the elemental composition of the sample surface. In some cases, the chemical state of the surface atoms can also be determined from energy shifts and peak shapes.



AES Spectrum for Passivated Stainless Steel

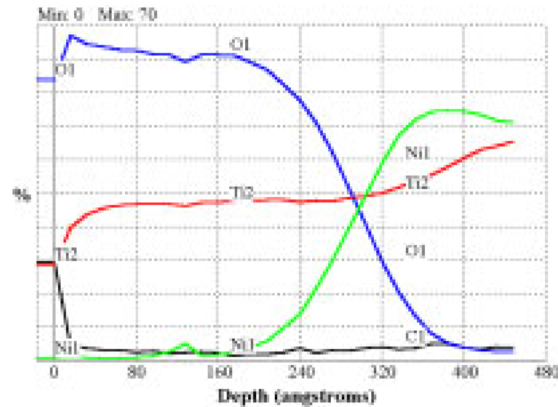
Auger electrons have relatively low kinetic energy, which limits their escape depth. Any Auger electrons emitted from an interaction below the surface will lose energy through additional scattering reactions along its path to the surface. Auger electrons emitted at a depth greater than about 2 - 3 nm will not have sufficient energy to escape the surface and reach the detector. Thus, the analysis volume for AES extends only to a depth of about 2 nm. Analysis depth is not affected by the energy of the primary electron energy.

The AES instrumentation can include a tungsten filament or field emission electron gun for the primary electron beam. The instruments are equipped for secondary electron imaging (SEM) to facilitate location of selected analysis areas, and micrographs of the sample surface can be obtained. The sample chamber is maintained at ultrahigh vacuum to minimize interception of the Auger electrons by gas molecules between the sample and the detector. Some instruments include special stages for fracturing samples to examine interfaces that have been freshly exposed within the vacuum chamber. A computer is used for acquisition, analysis, and display of the AES data.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

ANALYTICAL INFORMATION

Survey Scan - The position of the peaks in the AES spectrum obtained in a survey scan identifies the elemental composition of the uppermost 20 Å of the analyzed surface.



Composition Profile for Ni-Ti Alloy

Multiplex Scan - A higher resolution analysis of the Auger spectrum in the region around a characteristic peak is used for determination of the atomic concentration of the elements identified in the survey scans and, in some cases, chemical state information.

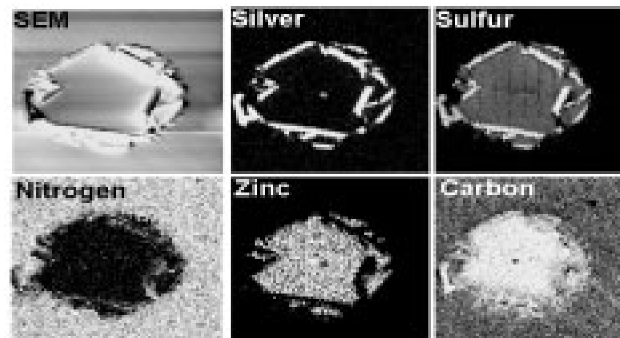
Quantitation - The AES analysis results can be quantified without standards by using the area under the peaks in the AES spectrum and corrections based on elemental sensitivity factors.

Mapping and Line Scans - These are imaging techniques that measure the lateral distribution of elements on the surface. The electron beam is scanned across the sample surface, either along a fixed line (line scan) or across a given area (mapping) while the AES signal is analyzed for specific energy channels. The AES signal intensity is a function of the relative concentration of the element(s) corresponding to the selected energy channel(s). Spatial resolution is approximately 0.3 µm.

Depth Profile - Material is removed from the surface by sputtering with an energetic ion beam concurrent with successive AES analyses. This process measures the elemental distribution as a function of depth into the sample. Depth resolution of < 100 Å is possible.

TYPICAL APPLICATIONS

- Microscopic particle identification
- Passive oxide film thickness
- Contamination on integrated circuits
- Quantitation of light element surface films
- Mapping spatial distribution of surface constituents



Auger maps of defect in glass coating (250x250 mm)

SAMPLE REQUIREMENTS

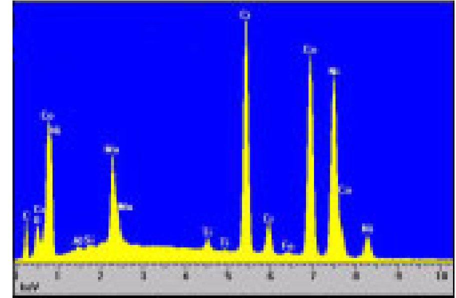
Samples should be no larger than approximately 3/4 in. by 1/2 in. (18 mm by 12 mm). Height of samples should not exceed 1/2 in. (12mm). Samples must be conductive or area of interest must be properly grounded. Insulating samples, including thick insulating films (>300 Å), cannot be analyzed. Samples must also be compatible with a high vacuum environment ($<1x10^{-9}$ Torr).

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

ENERGY DISPERSIVE X-RAY SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

Energy Dispersive X-Ray Spectroscopy (EDS or EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). (See Handbook section on SEM.)The EDS technique detects x-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analyzed volume. Features or phases as small as 1 µm or less can be analyzed.



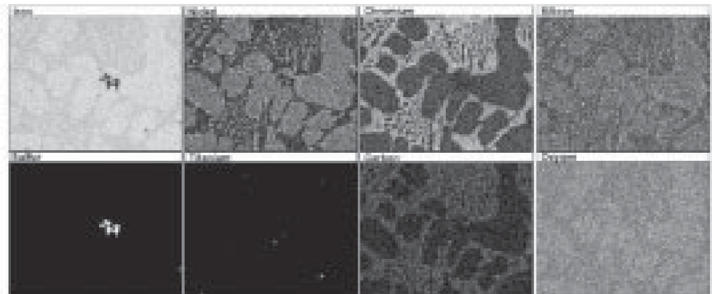
EDS Spectrum for Alloy MP35N

When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electron vacancies are filled by electrons from a higher state, and an x-ray is emitted to balance the energy difference between the two electrons' states. The x-ray energy is characteristic of the element from which it was emitted.

The EDS x-ray detector measures the relative abundance of emitted x-rays versus their energy. The detector is typically a lithium-drifted silicon, solid-state device. When an incident x-ray strikes the detector, it creates a charge pulse that is proportional to the energy of the x-ray. The charge pulse is converted to a voltage pulse (which remains proportional to the x-ray energy) by a charge-sensitive preamplifier. The signal is then sent to a multichannel analyzer where the pulses are sorted by voltage. The energy, as determined from the voltage measurement, for each incident x-ray is sent to a computer for display and further data evaluation. The spectrum of x-ray energy versus counts is evaluated to determine the elemental composition of the sampled volume.

ANALYTICAL INFORMATION

Qualitative Analysis - The sample x-ray energy values from the EDS spectrum are compared with known characteristic x-ray energy values to determine the presence of an element in the sample. Elements with atomic numbers ranging from that of beryllium to uranium can be detected. The minimum detection limits vary from approximately 0.1 to a few atom percent, depending on the element and the sample matrix.



Elemental Map for the Microstructure of a White Iron Casting

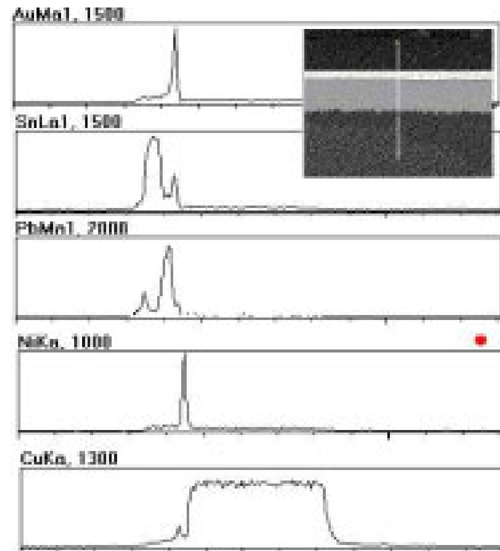
Quantitative Analysis - Quantitative results can be obtained from the relative x-ray counts at the characteristic energy

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

levels for the sample constituents. Semi-quantitative results are readily available without standards by using mathematical corrections based on the analysis parameters and the sample composition. The accuracy of standardless analysis depends on the sample composition. Greater accuracy is obtained using known standards with similar structure and composition to that of the unknown sample.

Elemental Mapping - Characteristic x-ray intensity is measured relative to lateral position on the sample. Variations in x-ray intensity at any characteristic energy value indicate the relative concentration for the applicable element across the surface. One or more maps are recorded simultaneously using image brightness intensity as a function of the local relative concentration of the element(s) present. About 1 µm lateral resolution is possible.

Line Profile Analysis - The SEM electron beam is scanned along a preselected line across the sample while x-rays are detected for discrete positions along the line. Analysis of the x-ray energy spectrum at each position provides plots of the relative elemental concentration for each element versus position along the line.



EDS Linescan Across Plated PCB Layers

TYPICAL APPLICATIONS

- Foreign material analysis
- Corrosion evaluation
- Coating composition analysis
- Rapid material alloy identification
- Small component material analysis
- Phase identification and distribution

SAMPLE REQUIREMENTS

Samples up to 8 in. (200 mm) in diameter can be readily analyzed in the SEM. Larger samples, up to approximately 12 in. (300 mm) in diameter, can be loaded with limited stage movement. A maximum sample height of approximately 2 in. (50 mm) can be accommodated. Samples must also be compatible with a moderate vacuum atmosphere (pressures of 2 Torr or less).

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

FOURIER TRANSFORM-INFRARED SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

Fourier Transform-Infrared Spectroscopy (FTIR) is an analytical technique used to identify organic (and in some cases inorganic) materials. This technique measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify molecular components and structures.

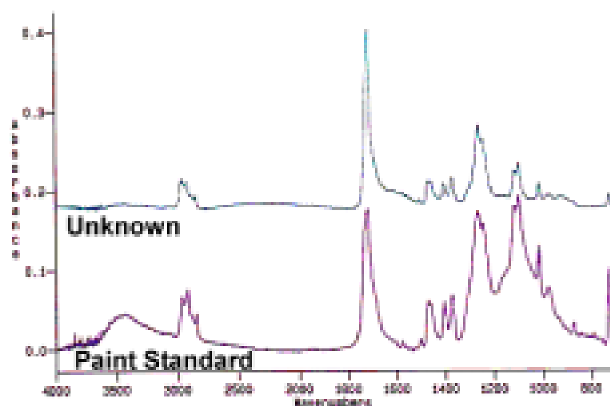
When a material is irradiated with infrared radiation, absorbed IR radiation usually excites molecules into a higher vibrational state. The wavelength of light absorbed by a particular molecule is a function of the energy difference between the at-rest and excited vibrational states. The wavelengths that are absorbed by the sample are characteristic of its molecular structure.

The FTIR spectrometer uses an interferometer to modulate the wavelength from a broadband infrared source. A detector measures the intensity of transmitted or reflected light as a function of its wavelength. The signal obtained from the detector is an interferogram, which must be analyzed with a computer using Fourier transforms to obtain a single-beam infrared spectrum. The FTIR spectra are usually presented as plots of intensity versus wavenumber (in cm^{-1}). Wavenumber is the reciprocal of the wavelength. The intensity can be plotted as the percentage of light transmittance or absorbance at each wavenumber.

ANALYTICAL INFORMATION

Qualitative Material Identification - To identify the material being analyzed, the unknown IR absorption spectrum is compared with standard spectra in computer databases or with a spectrum obtained from a known material. Spectrum matches identify the polymer or other constituent(s) in the sample. Absorption bands in the range of 4000 - 1500 wavenumbers are typically due to functional groups (e.g., -OH, C=O, N-H, CH_3, etc.). The region from 1500 - 400 wavenumbers is referred to as the fingerprint region. Absorption bands in this region are generally due to intramolecular phenomena and are highly specific to each material. The specificity of these bands allows computerized data searches within reference libraries to identify a material.

Quantitation - Quantitative concentration of a compound can be determined from the area under the curve in characteristic regions of the



FTIR Spectrum for Paint Analysis

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

IR spectrum. Concentration calibration is obtained by establishing a standard curve from spectra for known concentrations.

TYPICAL APPLICATIONS

- Identification of foreign materials
    - Particulates
    - Fibers
    - Residues
- Identification of bulk material compounds
- Identification of constituents in multilayered materials
- Quantitation of silicone, esters, etc., as contamination on various materials

SAMPLE REQUIREMENTS

Sample requirements vary depending on the sample form and instrument. Samples may be in liquid, solid or gaseous form. When using a microscope attachment on the spectrometer, the analysis area can be as small as 10 µm. Thin organic films on a reflective surface (e.g., gold) can be analyzed in situ using the microscope's reflectance mode. The outer 1-10 µm of a material can be analyzed using attenuated total reflectance (ATR).

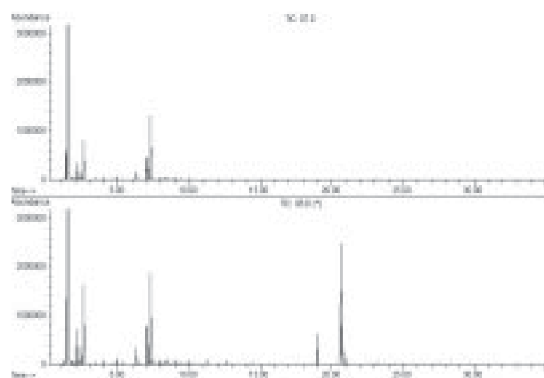
Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

GAS CHROMATOGRAPHY / MASS SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

Gas chromatography / mass spectrometry (GC/MS) is the marriage of two analytical methods into a versatile technique for the identification of complex volatile materials. Gas chromatography (GC) effectively separates the different constituents of the sample for subsequent analysis and identification by mass spectrometry (MS).

The chromatographic separation relies on the interaction of the sample with a mobile phase and a stationary phase within the GC instrument column. The sample is carried through the column by the mobile phase, typically an inert gas. However, the sample is slowed in its travel through the column as the sample molecules repeatedly adsorb and desorb from the stationary phase in the column. The affinity of a particular molecule for the stationary phase determines the retention time of that constituent in the column. The molecules for each component of the sample will travel through the column at nearly the same rate and exit (elute) from the column within a narrow time band that is specific to that component. Thus, compounds with different retention times in the column are physically separated for presentation to a detector and analyzer.



Total Ion Chromatogram for Two Vegetable Oils

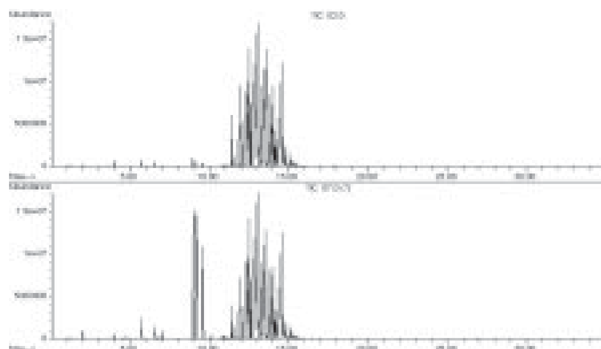
The typical GC capillary column consists of a small-diameter tube with a thin film of a high-molecular-weight polymer coated on the inside. The polymer is the stationary phase for the chromatographic process. The mobile phase can be any inert gas, but is typically helium. The instrument also includes a heated injection port to vaporize all volatile constituents of the sample and an oven to keep the constituents in gas form as they pass through the column.

As a sample constituent elutes from the GC column, it enters the ionization chamber of the mass spectrometer where the molecules are ionized, typically by electron impact. When an electron impact with a sample molecule results in the loss of an electron from the molecule, a positive ion is formed. Some of the molecular ions are further fragmented into daughter ions and neutral fragments. The positive ions are then repelled out of the ionization chamber by a small positive charge within the chamber. Negative ions are also formed by the electron impact, but are not analyzed.

The positive ions are separated according to their mass by a mass analyzer. The mass analyzer most commonly used in GC/MS is the quadrapole filter, in which the ions pass by four hyperbolic magnetic poles created by a radio frequency field. The magnetic poles separate the ions by their mass/charge ratio, successively focusing ions with increasing mass onto a detector for counting. The analyzer scans step-wise through a set range of mass values to evaluate the relative abundance of ions at each mass value. The quadrapole filter can perform a complete mass scan within the duration of a single GC elution band.

ANALYTICAL INFORMATION

Material Identification - The first result from the compiled data is a total-ion chromatogram (TIC), which is a plot of the total mass eluting from the GC and detected by MS as a function of time. Each peak or band in the chromatogram represents a discrete chemical compound, or a mixture of compounds with identical retention times. The retention times in the chromatogram provide the first indication of the sample constituents.


Chromotagram for Outgassing Compounds from Two Epoxies

More specific identification of the compound(s) for each band can then be made from the mass spectrum corresponding to the band. Compounds are identified from the mass spectrum by their unique ion fragmentation patterns. This compound identification analysis is performed by a computerized comparison of the mass spectra for the sample with spectra library for known compounds.

Quantitation - The analysis results can be quantified using the data from the chromatogram. The area under each peak in the chromatogram is proportional to the concentration of the compounds represented by that peak. The concentration for each compound in the sample is calculated from a standard curve of known concentrations established for that compound. The analysis sensitivity can be as low as a few nanograms.

TYPICAL APPLICATIONS

- Identification of foreign material contamination
- Analysis of outgassing products for disk drive components
- Identification of polymer additives
- Analysis of polymer cure by-products

SAMPLE REQUIREMENTS

The samples for GC/MS can be gases, liquids, or solids. However, only those constituents that are gaseous and stable at the analysis temperature can be analyzed. Gases and liquids can be injected directly into the sample injector. Volatile compounds from solid materials are collected using a headspace sampler. The sample from a headspace attachment can be collected and injected at one time (static headspace), or the sample can be collected and analyzed continuously over a set time period (dynamic headspace).

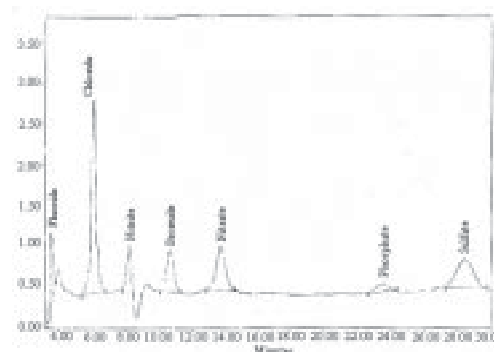
Copyright © 2001 by Materials Evaluation and Engineering, Inc.

ION CHROMATOGRAPHY

DESCRIPTION OF TECHNIQUE

Ion chromatography (IC) is the separation and quantitative analysis of anions and cations in an ionic solution using the ion exchange method of liquid chromatography (LC). The chromatographic process separates the different ions within the sample. The amount of an anion/cation is measured by the change in conductivity as the species passes through the detector.

The ions in the sample solution are carried through the system by an ionic solution, or eluent (mobile phase). The different ions in the eluent are separated in a column packed with an ion exchange resin (stationary phase). The resin has a thin surface layer of active material with limited ion exchange sites. If anions are to be analyzed, the active sites will have a fixed positive charge to attract the anions. Resins for cation analysis will have a negative charge. Individual ions attach and detach from the resins at a rate depending on the affinity of the specific ion to the active sites. Ions with greater affinity for the stationary phase are retained in the column for a longer time than those with less affinity for the stationary phase.



Ion Chromatogram

Thus, the ions of particular chemical species in the solution exit or elute from the column within a narrow time band specific to that ion. The detector at the end of the column continuously measures the conductivity of the eluent to determine the quantity of the eluting ions as a function of time. The data from the detector are compiled into a plot of ion abundance versus time, referred to as a chromatogram. The position of a peak in the chromatogram is characteristic of a specific ion. The peak size is a function of the concentration for the ion represented by that peak.

ANALYTICAL INFORMATION

Material Identification - The presence of specific anions and cations in the sample can be determined through comparison of the sample chromatograms against results for known reference materials.

Quantitation - Anion and cation concentrations can be quantified by establishing a standard curve of known concentrations for each species. The peak height or the area under the peak in the chromatogram for each anion or cation is compared to the standardization curve to determine the concentration of the ion the sample.

Sensitivity - Minimum concentration detection limits can be as low as a few parts per billion (ppb).

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

TYPICAL APPLICATIONS

- Determination of ionic species in plating baths
- Determination of acidic thermal decomposition products of polymer materials
- Determination of ionic contamination on critical components

MINIMUM DETECTION LIMITS FOR IC

SPECIES	MDL		SPECIES	MDL
ANIONS			**CATIONS**	
Fluoride (F^-)	4 ppb		Lithium (Li^+)	1 ppb
Chloride (Cl^-)	20 ppb		Sodium (Na^+)	5 ppb
Nitrite (NO_2^-)	20 ppb		Ammonium ($NH4^+$)	5 ppb
Bromide (Br^-)	20 ppb		Potassium (K^+)	8 ppb
Nitrate ($N0_3^-$)	20 ppb		Magnesium (Mg^{+2})	7 ppb
Phosphate (PO_4^{-2})	20 ppb		Calcium (Ca^{+2})	7 ppb
Sulfate (SO_4^{-2})	20 ppb			

SAMPLE REQUIREMENTS

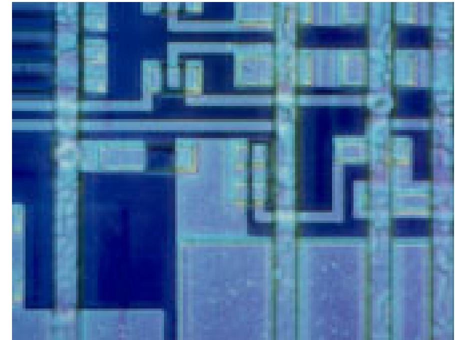
Samples must be immersible in a solution that is compatible with the LC system, or the components of interest must be extractable from the sample. Components must cause a change in the conductivity of the solution to be detected.

LIGHT MICROSCOPY

DESCRIPTION OF TECHNIQUE

Light microscopy in materials analysis generally refers to reflected light microscopy. In this method, light is directed vertically through the microscope objective and reflected back through the objective to an eyepiece, view screen, or camera. Transmitted light is occasionally used for transparent and translucent materials. For some low-magnification work (stereo microscopy), external, oblique illumination can be reflected off the sample into the objective.

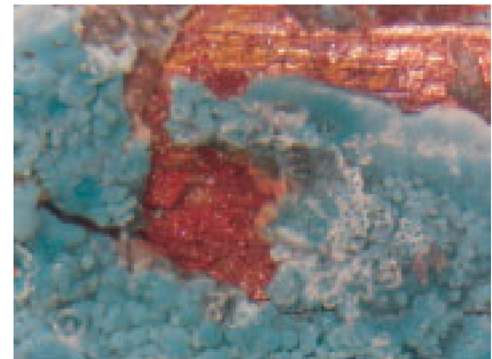
Magnification of the sample image is obtained by light refraction through a combination of objective lenses and eyepieces. The minimum feature resolution is approximately 0.2 µm. However, smaller features - as small as about 0.05 µm - can be detected by image contrast enhancement with polarized light, interference contrast, and dark field illuminations. The resulting images can be recorded either on traditional films or as digital files for computer display, analysis, and storage.



Interference Contrast Image of IC

ANALYTICAL INFORMATION

Bright Field Light Microscopy - This method produces true color images at magnifications up to approximately 2000X. The sample surface is uniformly illuminated by incident light rays directed perpendicularly to the sample surface. Light reflected back toward the objective lens is collected and focused on the eyepieces to form the observed image.
Surfaces that are reflective and perpendicular to the light rays appear bright. Alternatively, nonreflective or oblique features reflect less light and appear darker.



Corrosion on the Inside of a Copper Pipe

Polarized Light - This method produces enhanced contrast for features that have anisotropic refractive properties. Two polarizing lenses are inserted into the optical path - one in the incoming illumination and one between the sample and eyepieces. When these lenses are rotated 90° to one another, the "crossed polarizers" result in the subtraction of a portion of the light spectrum by destructive interference. Contrast is obtained between sample features that have different reflective properties. Many metals, including beryllium, zirconium and titanium, are anisotropic and exhibit grain contrast with polarized light illumination. Polarized light enhances contrast for many polymer samples and shows variations of internal stress in some clear polymers.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

Differential Interference Contrast after Nomarski (DIC) - This method produces a 3-dimensional image by creating brightness contrast on very minor topographical changes. DIC utilizes crossed polarizers as described for polarized light. A double quartz prism is also inserted into the light path to split the incident light into two separate paths. This results in two slightly shifted images of the sample on the viewing plane, which produces contrast between features with different heights and topographic orientations. The analyzer can be adjusted to obtain various degrees of interference to enhance selected features or create contrast colors in the image.

Darkfield - Enhanced contrast from subtle topographic features is produced with this method. An occluding disk is placed in the light path, blocking the direct vertical illumination. Peripheral rays in the illumination are reflected in such a way that light reaches the sample at oblique angles. The absence of incident vertical rays results in bright reflectance only from oblique features, such as ridges, pits, scratches and particles. Thus, subtle features that might be completely invisible in bright field microscopy are readily observed with this method.

Quantitation - Microscope magnification is calibrated against reference standards. Lateral feature dimensions can be measured to an accuracy greater than 0.5 µm. Computer analysis of digitally-acquired images can measure area and volume fractions, particle sizes, grain size, and other features.

TYPICAL APPLICATIONS

- Small sample inspection
- Metal microstructure evaluation
- Small feature measurements
- Fracture mode identification
- Corrosion failure inspection
- Surface contamination evaluation

Metallurgical Light Microscope

SAMPLE REQUIREMENTS

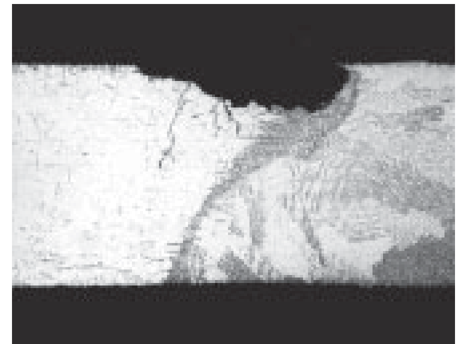
Sample size, shape, and condition requirements depend on the configuration of the microscope. Low-magnification stereo microscopes are small and have a long focal length (up to 5 in.), so these can be set up to examine even relatively large samples. Some portable field microscopes can be fixtured directly to large structures. For magnifications of 100X and above, microscopes are not usually amenable to portable use (except specialized field units). Samples for typical high-resolution light microscopes are limited to a few pounds in weight, and examination is limited to readily accessible flat surfaces due to the small depth of field at higher magnifications.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

METALLOGRAPHIC STUDY

DESCRIPTION OF TECHNIQUE

Metallographic study, or metallography, is the imaging of topographical or microstructural features on prepared surfaces of materials. The structures studied by metallography are indicative of the properties and performance of materials studied.



Stress Corrosion Cracking at SS Weld

In this technique, planar surfaces are prepared to obtain a polished finish. Chemical or other etching methods are often used to delineate macrostructure and microstructure features. Once prepared, samples are examined by the unaided eye, light microscopy, and/or electron microscopy. (See sections on Light Microscopy and Scanning Electron Microscopy.)

Samples for microstructure evaluation are typically encapsulated in a plastic mount for handling during sample preparation. Large samples or samples for macrostructure evaluation can be prepared without mounting. Sample preparation consists of grinding and then polishing using successively finer abrasives to obtain the desired surface finish. For microstructure examination, a mirror finish is needed, but a finely-ground finish is adequate for macrostructure evaluation. Etchants are specially formulated for the specific sample material and evaluation objectives.

Sampling for metallography can be a random section to evaluate representative bulk properties or a section in a specific location to characterize localized material conditions. For example, a section through the facture initiation site is often made to assist with a component failure analysis. For microelectronic components, precision metallographic methods can obtain sections though specific wire bonds, solder pads, or even individual components on an integrated circuit device.

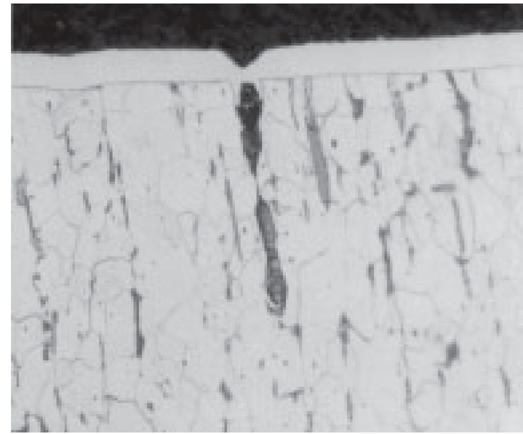
Microstructure of Welded Titanium

ANALYTICAL INFORMATION

Macrostructure Evaluation - Deep chemical etching is used to characterize large-scale variations in material composition, structure, density, etc. This method is useful for evaluation of welds, brazes, forgings, and polymer-matrix composites for configuration, defects, and structure.

Microstructure Evaluation - Characteristic features provide information about composition, phase distribution, mechanical and physical properties, thermo-mechanical process history, and defects. In failure analysis, the morphology of corrosion or cracks can be characteristic of the failure mode.

Quantitative Metallography - Observed features can be analyzed to obtain measurements of microscopic characteristics, including grain size, phase volume fractions, and linear dimensions. Measurements are made manually or by computerized semi-automated methods on digitally-acquired images.

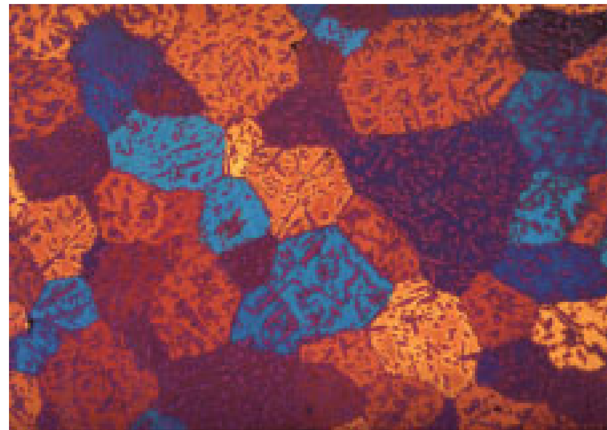


Plating Defect Caused by Chemical Attack Before Plating at Nonmetallic Inclusion

Field Metallography - Metallographic examination can be performed in situ for large components or on structures in the field. The selected areas of the sample surface are polished using portable tools. The prepared surface can be examined directly with a portable light microscope. Alternatively, the surface can be replicated with an acetate tape or castable polymer for examination by light microscopy or electron microscopy in the laboratory.

TYPICAL APPLICATIONS

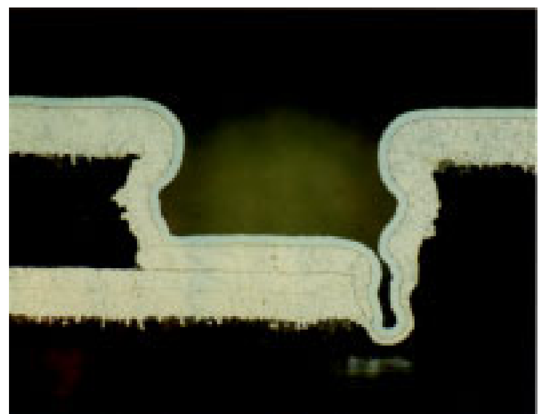
- Metal alloy heat treatment verification
- Coating thickness measurement
- Weld or braze joint evaluation
- Case hardening depth determination
- Corrosion resistance evaluation
- Failure analysis
- Microscopic defects in IC devices
- In situ evaluation of thermo-mechanical degradation



Manganese Bronze Microstructure

SAMPLE REQUIREMENTS

Most samples are sectioned and encapsulated in a metallographic mount to facilitate preparation. The mount sizes range from about 1 in. (25 mm) to 3 in. (75 mm) in diameter. Sections up to approximately 8 in. (200 mm) across can be prepared in the laboratory without mounting. Localized areas on large samples or those that cannot be cut are prepared in situ and evaluated using a field microscope or replicas of the prepared surface.



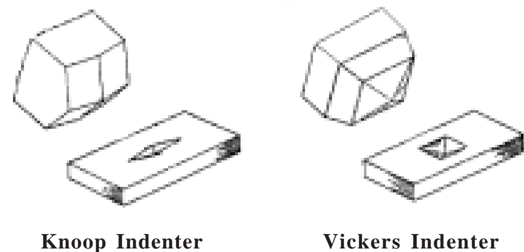
Mis-registration in Multilayered Circuit Board

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

MICROINDENTATION HARDNESS TESTING

DESCRIPTION OF TECHNIQUE

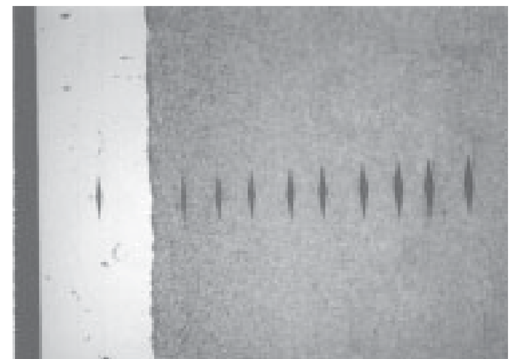
Microindentation hardness testing (or microhardness testing) is a method for measuring the hardness of a material on a microscopic scale. A precision diamond indenter is impressed into the material at loads from a few grams to 1 kilogram. The impression length, measured microscopically, and the test load are used to calculate a hardness value. The hardness values obtained are useful indicators of a material's properties and expected service behavior. Conversions from microindentation hardness values to tensile strength and other hardness scales (e.g., Rockwell) are available for many metals and alloys.



Knoop Indenter Vickers Indenter

The indentations are typically made using either a square-based pyramid indenter (Vickers hardness scale) or an elongated, rhombohedral-shaped indenter (Knoop hardness scale). The tester applies the selected test load using dead weights. The length of the hardness impressions are precisely measured with a light microscope using either a filar eyepiece or a video image and computer software. A hardness number is then calculated using the test load, the impression length, and a shape factor for the indenter type used for the test.

ANALYTICAL INFORMATION



Knoop Microindentation Hardness Survey for Chromium-Plated, Case-Hardened Steel

Bulk Hardness - Randomly-located impressions measure the representative bulk hardness value of a relatively homogeneous material.

Localized Hardness - Impressions are made at specific sites located using the light microscope to determine the hardness at discrete features or phases in the sample. Hardness can be measured for features less than 0.1 mm across.

Hardness Survey - A series of hardness impressions are made along a line from a surface or a specific point in the sample to systematically measure the hardness variation within a sample.

Thin Coatings - The hardness of coatings as thin as a few microns can be determined by measuring directly on the coated surface of a sample. Coating thickness must be known to assess accuracy of these measurements.

TYPICAL APPLICATIONS

- Bulk hardness of small or thin samples
- Heat treated steel case depth evaluation
- Decarburization in steels
- Evaluation of welds
- Hardness of thin coatings
- Evaluation of machinability

SAMPLE REQUIREMENTS

Most microindentation hardness testing is performed on samples that have been metallographically mounted and polished. These samples are usually no larger than about 1 in. (25 mm) by 1 in. (25 mm) by 1/2 in. (12 mm) thick. Larger samples can be tested with special fixturing. Thin, flat samples, such as sheet material, can be tested without mounting or preparation if the surface finish is suitable.

The ideal surface finish is a high-quality metallographic polish. Where polishing is not feasible, the surface finish must be sufficiently smooth and reflective to clearly resolve the microscopic hardness impression with the measuring microscope. The specific finish requirement depends on the material and test load.

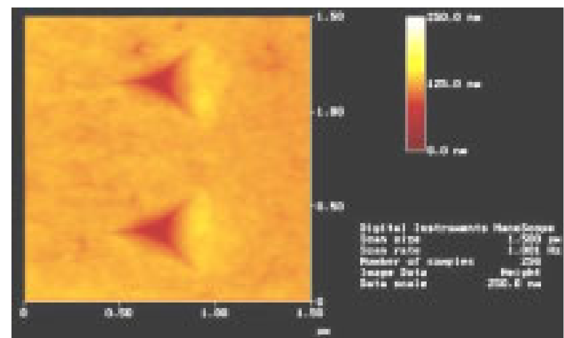
Microhardness Tester

NANOINDENTATION HARDNESS TESTING

DESCRIPTION OF TECHNIQUE

Nanoindenting is a new method to characterize material mechanical properties on a very small scale. Features less than 100 nm across, as well as thin films less than 5 nm thick, can be evaluated. Test methods include indentation for comparative and quantitative hardness determination and scratching for evaluation of wear resistance and thin film adhesion.

Nanoindenting is performed in conjunction with atomic force microscopy (AFM). The area for testing is located by AFM imaging, and indentations and scratching marks are imaged by AFM after testing. A three-sided, pyramid-shaped diamond probe tip is typically used to indent, scratch and image the sample.



AFM Images of Two Impressions in a Polymer

For indentation, the probe is forced into the surface at a selected rate and to a selected maximum force. In scratching, the probe is dragged across the sample surface. The force, rate, length and angle of the scratch is controlled.

Imaging is performed in situ using the probe in intermittent contact (tapping mode) AFM. The depth of the indentation is measured from the AFM image to evaluate hardness. A force-displacement curve obtained during indentation also provides indications of the sample material's mechanical and physical properties.

ANALYTICAL INFORMATION

Nanoindentation - Indentation forces ranging from 1µN to 100 mN can be made to measure material hardness. Indentation depth or area is inversely proportional to hardness. Force displacement curves obtained during the indentation process indicate hardness and elastic modulus properties.

Scratching - Patterns scribed in the sample surface show the potential for spalling or delamination of thin films.

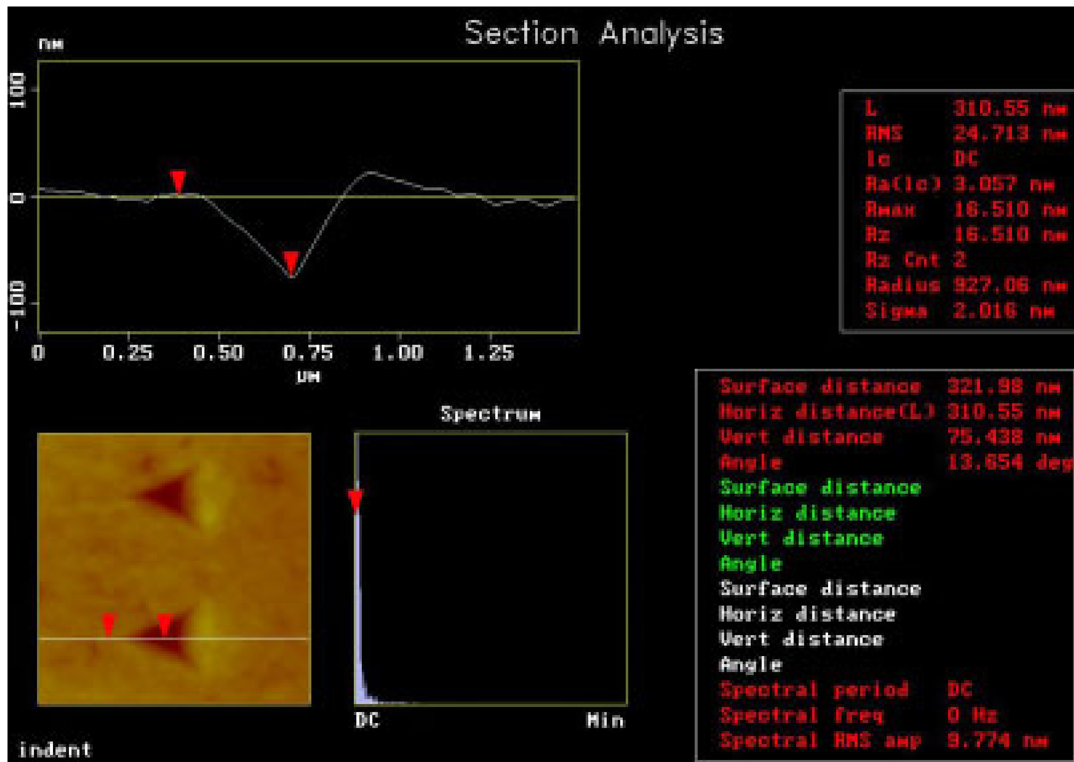
Wear Testing - The diamond probe tip is repeatedly scanned over the same sample surface area at a selected force. Wear durability is measured by the material lost from the tested surface. Depth of material loss is measured by AFM imaging after testing.

TYPICAL APPLICATIONS

- Hardness measurements for submicron-size features
- Thin film adhesion evaluation
- Coating wear durability evaluation
- Elastic modulus comparisons for thin films

SAMPLE REQUIREMENTS

Samples up to 8 in. (200 mm) across and 1 in. (25 mm) thick can be fixtured and tested in some instruments. Larger samples can be fixtured for access to limited surface areas. The sample surface must have a smooth finish for uniform indentation and to allow AFM imaging for indentation depth. The required finish depends on the material and the test force.



AFM Section Analysis for Nanoindentation Hardness

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

QUANTITATIVE CHEMICAL ANALYSIS

DESCRIPTION OF TECHNIQUES

Quantitative chemical analysis is performed to accurately determine the concentration of elements in the material comprising a given sample. A variety of analysis techniques are used for metals and alloys to determine the alloy composition of raw materials to verify conformance to a specification or to identify the alloy used to make a specific component. Quantitative analysis methods are also used occasionally for evaluation of foreign material contaminants in special cases for failure analysis or investigation of product manufacturing or handling problems.

Quantitative chemical analysis may be performed by one or more complimentary techniques, commonly including spark optical emission spectroscopy (Spark-OES), inductively-coupled plasma spectroscopy optical emission spectroscopy (ICP-OES), x-ray fluorescence spectroscopy (XRF), wet chemical analyses, combustion methods, and inert gas fusion(IG).The specific technique chosen will depend on the type of sample, quantity of material available for analysis, desired result, and cost constraints. In most cases, the applicable analysis techniques can detect parts-per-million concentrations or better.

Most of these techniques are destructive to the original sample. XRF can be performed nondestructively and Spark-OES can be performed with only minimal surface damage if the specimen size configuration allow the part to fit into the instrument without cutting. For the remaining methods discussed here, a small specimen is removed from the sample and is consumed in the analysis.

Prior to the widespread availability of analytical instruments, chemical analyses were performed by dissolving the sample and performing a specific chemical reaction with a standardized reagent for each element of interest. These ‘wet chemistry’ techniques are typically labor intensive and time consuming, and sometimes less accurate than the current instrumental methods.

Wet Chemistry - These methods include gravimetric and titrimetric techniques. An example of a gravimetric technique is the precipitation of chloride ion with silver to form a silver chloride precipitate which is dried and weighed to determine the chloride concentration in the original sample solution. Titrimetric procedures are typically based on acid-base reactions or complexing agents for metal ions. Since wet chemical analyses are now less common for the analysis of metals and similar inorganic materials, the remainder of this section will focus on the instrumental methods of analysis.

Spark-OES - Spark optical emission spectroscopy is a technique used for direct analysis of solid metal samples. The specimen is prepared by grinding to obtain a uniform, clean, flat area about 1 to 2 cm across. The prepared sample is placed in the spark-OES instrument and flooded with argon. A rapid series of high energy sparks are created across the argon-filled gap between an electrode (cathode) and the prepared sample’s surface (acting as the anode). The sparks first ionize the argon, creating a conductive plasma. Secondly, the sparks melt, evaporate, and excite the sample elements at the spark point of impact. When the excited atoms in the plasma relax (de-excite) to a lower energy state, they emit light at characteristic wavelengths for each element. The intensities of these

emissions at the characteristic wavelengths are detected, measured, and compared to intensities for known standards to provide quantitative results. The total duration of the sparking is only a few milliseconds. Prior to actual measurements, the sample surface may be subjected to high power discharges to melt the surface and create a more homogeneous material.

XRF - X-ray fluorescence spectroscopy is a technique that can be used for direct analysis of solid metal samples, thin metal films, petroleum products, cement, coal, and various other materials. XRF is a fast technique and is non-destructive to the sample. It is frequently used for analyses performed in the field and for industrial quality control.

An x-ray tube is used to irradiate the sample with a primary beam of x-rays. Some of the impinging primary x-rays are absorbed by the sample elements in a process known as the photoelectric effect. The photoelectric effect occurs when all the energy of a primary x-ray is absorbed by an electron in an atom's innermost electron shell. This causes excitation and ejection of the absorbing electron (photoejection). The electron vacancies caused by the photoelectric effect are filled by electrons from higher energy states, and x-rays are emitted (fluorescence) to balance the energy difference between the electron states. The x-ray energy is characteristic of the element from which it was emitted.

The fluorescence x-rays are collimated and directed to an x-ray detector. The energy of each x-ray and number of x-rays at each energy are recorded. The x-ray intensities (counts) at each energy are compared to values for known standards for quantitatively analysis of the unknown specimen.

ICP-OES - Inductively coupled plasma-optical emission spectroscopy is a technique for analyzing the concentration of metallic elements in solid and liquid samples. Like spark-OES, ICP-OES uses the optical emission principles of exited atoms to determine the elemental concentration. However, for ICP-OES, solid samples are dissolved (digested) in an appropriate solvent (typically acid) to produce a solution for analysis. The resulting sample solution (or an original liquid solution for analysis) is often diluted in water to obtain a final specimen suitable for analysis.

The ICP-OES instrument uses argon gas flowing through a torch consisting of three concentric quartz tubes. A copper coil circumscribing the top of the torch is connected to a radio frequency (RF) generator. The use of the copper coil with the RF power is called inductive coupling.

When the RF power is applied in the copper coil, an alternating current occurs within the coil. The oscillation of the alternating current causes electric and magnetic fields at the end of the torch. A spark applied to the argon gas causes some electrons to be stripped from the argon atoms. The electrons are caught and accelerated by the RF generated electric/magnetic field. The high energy free electrons collide with other atoms, stripping off more electrons in a chain reaction, resulting in a plasma of electrons, ions, and atoms. This is known as an inductively coupled plasma (ICP) discharge. This ICP discharge is maintained as the RF energy is continually transferred to the plasma by the copper coil.

The liquid samples are nebulized into an aerosol and introduced into the center of the plasma. The plasma excites the sample atoms, which subsequently relax to a lower energy state by emitting light at elementally characteristic wavelengths. The intensities of these characteristic wavelengths are detected, measured, and compared to intensities for known standards to provide quantitative results.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

Combustion Methods - High temperature combustion is used to determine carbon and sulfur content in a variety of materials, both organic and inorganic. The sample is accurately weighed and placed in a ceramic crucible or combustion boat, often along with combustion accelerators. The crucible is placed in a high-temperature furnace which is then flooded with oxygen. The furnace is heated to 1370 - 1425°C, causing the combustion of the carbon and sulfur in the sample to form CO, CO_2, and SO_2. The gases are separated and analyzed by infrared absorption or thermal conductivity detectors. A heated catalyst is used to convert the CO to CO_2 prior to detection.

The infrared absorption detector measures the absorption of the infrared wavelengths characteristic to CO_2 and SO_2.The amount of infrared absorption at these wavelengths is correlated to a quantitative content based on standards and the weight of the original specimen.

The thermal conductivity detectors monitor the thermal conductivity of the carrier gas. As the evolved gases pass the detector, changes in the thermal conductivity correspond to a change in the gas (e.g. from the inert carrier gas to hydrogen) and the amount of evolved gas present. These changes correspond to the amount of CO_2 and SO_2 generated and indicate the amount of sulfur or carbon in the original specimen.

IG - Inert gas fusion is a quantitative analytical technique for determining the concentrations of nitrogen, oxygen, and hydrogen in ferrous and nonferrous materials. The sample is accurately weighed and placed in a pure graphite crucible in a fusion furnace with an inert gas atmosphere. The crucible is heated to 2000 - 3000°C, resulting in the sample fusing to a molten state. The hydrogen and nitrogen gases dissociate from the molten material and are carried away from the fusion chamber as H_2 and N_2. The oxygen released from the material bonds with carbon (from the graphite crucible) to form CO or CO_2 and is carried away.

An inert carrier gas flushes the gases evolved from the sample out of the fusion chamber. The fusion gases are separated and carried to the detector. The individual concentrations for the evolved gases are detected by infrared absorption (for CO and CO_2 only) or thermal conductive techniques (N_2, H_2, CO, and CO_2) as described for Combustion Methods above.

ANALYTICAL INFORMATION

Spark-OES - The intensities of the characteristic wavelengths of emitted photons are measured and compared to intensities for known standards to provide quantitative results. All metallic elements plus carbon, sulfur, and phosphorus can be detected, with analysis for most performed simultaneously. The minimum detection limits are in the parts-per-million range.

XRF - The energy of each x-ray and the number of x-rays for each energy are measured. Elements from beryllium to uranium can be detected. The minimum detection limits are typically in the parts-per-million range. Because thecharacteristic x-ray intensity will vary with the thickness of films on a dissimilar substrate, the thickness of thin films can also be measured by XRF.

ICP-OES - The intensities of the characteristic wavelengths are measured and compared to intensities for known standards to provide quantitative results. All metallic elements can be detected, with analysis for most performed simultaneously. The minimum detection limits are typically parts-per-million to parts-per-billion for the dissolved samples. Since specimens for this technique are solutions, standards suitable for most material types can be easily prepared.

Combustion methods - Quantitative results are obtained for carbon and sulfur contents in metals, inorganics, and organics. Lower detection limits for carbon range from 0.1 to 10 parts per-million with upper detection limits of 2.5 - 3.5 %. Lower detection limits for sulfur range from 0.1 to 50 parts per-million with upper detection limits of 0.2 - 2.5 %.

IG - Quantitative results for most metals and alloys can be obtained in the parts-per-million to parts-per-billion range for nitrogen, hydrogen, and oxygen.

TYPICAL APPLICATIONS

- Alloy identification for ferrous and non-ferrous materials
- Industrial alloy verification for quality control
- Mineral and Cement composition
- Sulfur, chlorine, lead, etc.,determination in petroleum products
- Additives to polymers
- Trace metals in alloys, water, or solutions
- Contamination of water or solutions

SAMPLE REQUIREMENTS

Spark -OES - The sample must be a conductive metallic solid with a minimum diameter of 5 mm or larger, depending on the instrument.

XRF - The samples may be solids, liquids or powders. Samples often require little or no preparation prior to analysis. Qualitative analysis may use samples as small as 1 mm across. Quantitative analysis may require a larger sample, up to 30 mm in diameter.

ICP-OES - The samples may be solid or in a solution. A few grams of a solid sample are typically needed for digestion and dilution. For samples in solution, at least several milliliters may be required for dilution.

Combustion methods - One gram or less of a solid, chips, or powder sample is typically required. Samples should not be contaminated with sulfur or carbon prior to analysis.

IG - One gram of material is required for nitrogen or oxygen determination. Samples may be solids, chips, or powders. Hydrogen determination generally requires two grams of a solid sample.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

ROCKWELL HARDNESS TESTING

DESCRIPTION OF TECHNIQUE

Rockwell hardness testing is a general method for measuring the bulk hardness of metallic and polymer materials. Although hardness testing does not give a direct measurement of any performance properties, hardness of a material correlates directly with its strength, wear resistance, and other properties. Hardness testing is widely used for material evaluation because of its simplicity and low cost relative to direct measurement of many properties. Specifically, conversion charts from Rockwell hardness to tensile strength are available for some structural alloys, including steel and aluminum.

Rockwell hardness testing is an indentation testing method. The indenter is either a conical diamond (brale) or a hard steel ball. Different indenter ball diameters from 1/16 to 1/2 in. are used depending on the test scale.

To start the test, the indenter is "set" into the sample at a prescribed minor load. A major load is then applied and held for a set time period. The force on the indenter is then decreased back to the minor load. The Rockwell hardness number is calculated from the depth of permanent deformation of the indenter into the sample, i.e. the difference in indenter position before and after application of the major load. The minor and major loads can be applied using dead weights or springs. The indenter position is measured using an analog dial indicator or an electronic device with digital readout.

The various indenter types combined with a range of test loads form a matrix of Rockwell hardness scales that are applicable to a wide variety of materials. Each Rockwell hardness scale is identified by a letter designation indicative of the indenter type and the major and minor loads used for the test. The Rockwell hardness number is expressed as a combination of the measured numerical hardness value and the scale letter preceded by the letters, HR. For example, a hardness value of 80 on the Rockwell A scale is reported as 80 HRA.

TYPICAL APPLICATIONS OF ROCKWELL TEST SCALES

SCALE	APPLICATIONS
A	Cemented carbides, thin steels, shallow case-hardened steels. Only scale that is continuous over a wide range of material hardnesses.
B	Aluminum, copper, soft steels, and malleable iron.
C	Hardened steels, hard irons, deep case-hardened steels, titanium.
D	Thin steels, medium case-hardened steels, and pearlitic malleable iron.
E	Cast iron, aluminum, magnesium, and bearing metals.
F	Annealed coppers and thin, soft sheet metal.
G	Phosphor bronze, beryllium copper, and malleable irons.
H	Aluminum, zinc, and lead
K,L,M, P,R,S,V	Bearing metals and other very soft or thin materials.
N	Same materials as for HRA, HRC, and HRD, but thinner gauge or case depths.
T	Same materials as for HRB, HRF, and HRG, but for thinner gauge.
W,X,Y	Bearing materials, plasma spray coatings.

ANALYTICAL INFORMATION

Regular Rockwell Hardness Testing - Measures the bulk hardness of the material. There are separate scales for ferrous metals, nonferrous metals, and plastics. Common Rockwell hardness scales include A, B,C and F for metals and M and R for polymers.

Superficial Rockwell Hardness Testing - A more surface-sensitive measurement of hardness than regular Rockwell scales. This technique is useful for testing thin samples, samples with hardness gradients at the surface, and small areas. Superficial Rockwell hardness scales are N and T for metals and W, X and Y for nonmetallic materials and soft coatings.

TYPICAL APPLICATIONS

- Quality control for metal heat treatment
- Incoming material inspection
- Weld evaluations in steels and other alloys
- Grade verification for hard plastics
- Failure analysis

SAMPLE REQUIREMENTS

Testing is typically performed on flat or cylindrical samples. Cutting and/or machining are often required to obtain suitable test specimens from complex-shaped components. Smooth parallel surfaces, free of coatings, scale and gross contamination, are required for testing. The specific finish requirements depend on the material and test scale.

Samples 6 in. (150 mm) thick or larger can be accommodated. The minimum sample size depends on the sample hardness and test scale. Cylindrical samples as small as 1/8 in. (3 mm) in diameter, and thin sheets 0.006 in. (150 μm) thick, are the minimum size for testing.

Rockwell Hardness Tester

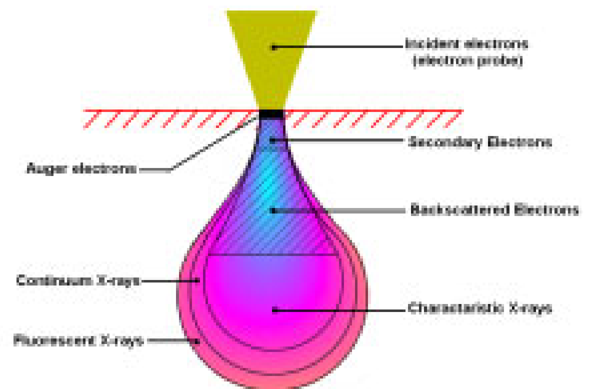
Rockwell Hardness Test Scales		
Scale Symbol	**Penetrator**	**Load kg**
A	Brale	60
B	1/16-in. Ball	100
C	Brale	150
D	Brale	100
E	1/8-in. Ball	100
F	1/16-in. Ball	60
G	1/16-in. Ball	150
H	1/8-in. Ball	60
K	1/8-in. Ball	150
L	1/4-in. Ball	60
M	1/4-in. Ball	100
P	1/4-in. Ball	150
R	1/2-in. Ball	60
S	1/2-in. Ball	100
V	1/2-in. Ball	150
Superficial Tester Scales		
15N, 30N, 45N	N Brale	15, 30, 45
15T, 30T, 45T	1/16-in. Ball	15, 30, 45
15W, 30W, 45W	1/8-in. Ball	15, 30, 45
15X, 30X, 45X	1/4-in. Ball	15, 30, 45
15Y, 30Y, 45Y	1/2-in. Ball	15, 30, 45

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

SCANNING ELECTRON MICROSCOPY

DESCRIPTION OF TECHNIQUE

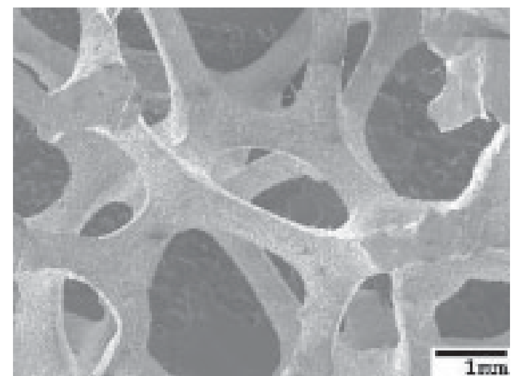
Scanning electron microscopy (SEM) is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include much higher magnification (>100,000X) and greater depth of field up to 100 times that of light microscopy. Qualitative and quantitative chemical analysis information is also obtained using an energy dispersive x-ray spectrometer (EDS) with the SEM. (See Handbook section on EDS analysis.)



Electron Beam Interaction Diagram

The SEM generates a beam of incident electrons in an electron column above the sample chamber. The electrons are produced by a thermal emission source, such as a heated tungsten filament, or by a field emission cathode. The energy of the incident electrons can be as low as 100 eV or as high as 30 keV depending on the evaluation objectives. The electrons are focused into a small beam by a series of electromagnetic lenses in the SEM column. Scanning coils near the end of the column direct and position the focused beam onto the sample surface. The electron beam is scanned in a raster pattern over the surface for imaging. The beam can also be focused at a single point or scanned along a line for x-ray analysis. The beam can be focused to a final probe diameter as small as about 10 Å.

The incident electrons cause electrons to be emitted from the sample due to elastic and inelastic scattering events within the sample's surface and near-surface material. High-energy electrons that are ejected by an elastic collision of an incident electron, typically with a sample atom's nucleus, are referred to as backscattered electrons. The energy of backscattered electrons will be comparable to that of the incident electrons. Emitted lower-energy electrons resulting from inelastic scattering are called secondary electrons. Secondary electrons can be formed by collisions with the nucleus where substantial energy loss occurs or by the ejection of loosely bound electrons from the sample atoms. The energy of secondary electrons is typically 50 eV or less.



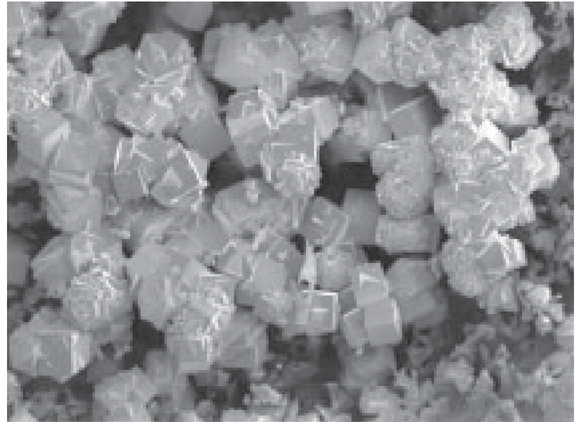
SEM Image of Metal Foam Structure

To create an SEM image, the incident electron beam is scanned in a raster pattern across the sample's surface. The

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

emitted electrons are detected for each position in the scanned area by an electron detector. The intensity of the emitted electron signal is displayed as brightness on a cathode ray tube (CRT). By sychromizing the CRT scan to that of the scan of the incident electron beam, the CRT display represents the morphology of the sample surface area scanned by the beam. Magnification of the CRT image is the ratio of the image display size to the sample area scanned by the electron beam.

Two electron detector types are predominantly used for SEM imaging. Scintillator type detectors (Everhart-Thornley) are used for secondary electron imaging. This detector is charged with a positive voltage to attract electrons to the detector for improved signal to noise ratio. Detectors for backscattered electrons can be scintillator types or a solid-state detector.

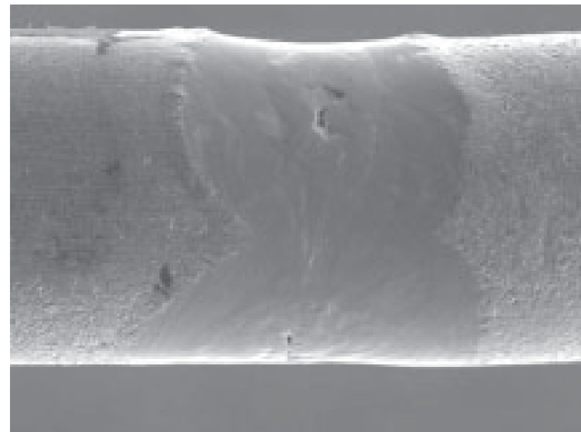


Corrosion Product on Inside of Copper Tubing

The SEM column and sample chamber are at a moderate vacuum to allow the electrons to travel freely from the electron beam source to the sample and then to the detectors. High-resolution imaging is done with the chamber at higher vacuum, typically from 10^{-5} to 10^{-7} Torr. Imaging of nonconductive, volatile, and vacuum-sensitive samples can be performed at higher pressures.

ANALYTICAL INFORMATION

Secondary Electron Imaging - This mode provides high-resolution imaging of fine surface morphology. Inelastic electron scattering caused by the interaction between the sample's electrons and the incident electrons results in the emission of low-energy electrons from near the sample's surface. The topography of surface features influences the number of electrons that reach the secondary electron detector from any point on the scanned surface. This local variation in electron intensity creates the image contrast that reveals the surface morphology. The secondary electron image resolution for an ideal sample is about 3.5 nm for a tungsten-filament electron source SEM or 1.5 nm for field emission SEM.

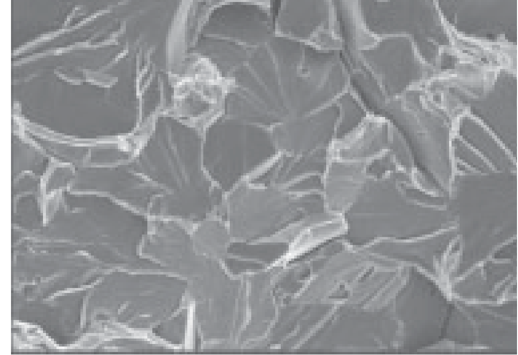


Laser Welded Wire

Backscatter Electron Imaging - This mode provides image contrast as a function of elemental composition, as well as, surface topography. Backscattered electrons are produced by the elastic interactions between the sample and the incident electron beam. These high-energy electrons can

escape from much deeper than secondary electrons, so surface topography is not as accurately resolved as for secondary electron imaging. The production effeciency for backscattered electrons is proportional to the sample material's mean atomic number, which results in image contrast as a function of composition, i.e., higher atomic number material appears brighter than low atomic number material in a backscattered electron image. The optimum resolution for backscattered electron imaging is about 5.5 nm.

Variable Pressure SEM - Traditionally, SEM has required an electrically-conductive sample or continuous conductive surface film to allow incident electrons to be conducted away from the sample surface to ground. If electrons accumulate on a nonconductive surface, the charge buildup causes a divergence of the electron beam and degrades the SEM image. In variable-pressure SEM, some air is allowed into the sample chamber, and the interaction between the electron beam and the air molecules creates a cloud of positive ions around the electron beam. These ions will neutralize the negative charge from electrons collecting on the surface of a nonconductive material. SEM imaging can be performed on a nonconductive sample when the chamber pressure is maintained at a level where most of the electrons reach the sample surface, but there are enough gas molecules to ionize and neutralize charging. Variable pressure SEM is also valuable for examination of samples that are not compatible with high vacuum.



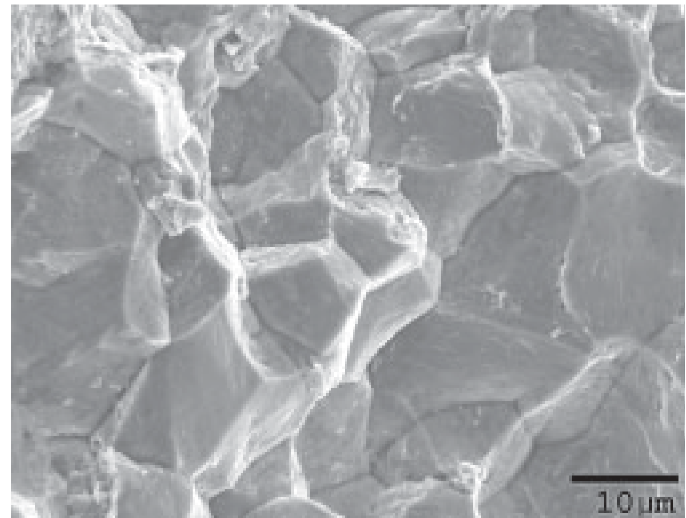
Cleavage Fracture in Steel

Quantitation - Image magnification is calibrated against a reference standard. Lateral feature dimensions can be readily quantified to an accuracy of less than 0.1 µm. Computer analysis of images can quantify area or volume fractions and particle shapes and sizes.

Data Formats - Images can be recorded on Polaroid instant film, low-cost video prints, videotape, or as bitmap (.bmp), tagged-image (.tif), or other computer file formats.

TYPICAL APPLICATIONS

- Microscopic feature measurement
- Fracture characterization
- Microstructure studies
- Thin coating evaluations
- Surface contamination examination
- IC failure analysis

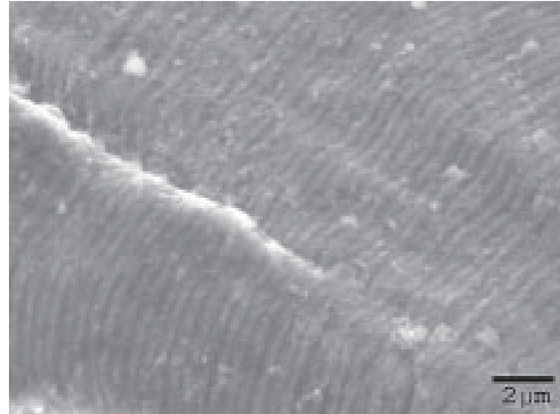


Intergranular Fracture in Steel

 Copyright © 2001 by Materials Evaluation and Engineering, Inc.

SAMPLE REQUIREMENTS

In a large-chamber SEM, samples up to 8 in. (200 mm) in diameter can be readily accommodated. Larger samples, up to 12 in. (300 mm) across can be loaded with limited stage movement. Sample height is typically limited to ~2 in. (50 mm). Backscattered electron imaging can be performed on conductive or nonconductive samples. For secondary electron imaging, samples must be electrically conductive. Nonconductive materials can be evaporatively coated with a thin film of carbon, gold or other conductive material to obtain conductivity without significantly affecting observed surface morphology.



Fatigue Fracture in Aluminum

Samples must be compatible with at least a moderate vacuum. For high-resolution secondary electron imaging, the sample environment is at a pressure of 1 x 10^{-5} Torr or less. The pressure can be adjusted up to about 2 Torr for vacuum sensitive samples.

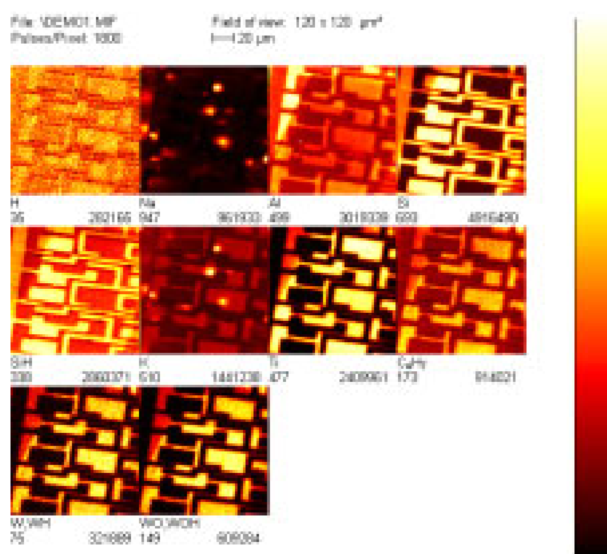
SECONDARY ION MASS SPECTROMETRY

DESCRIPTION OF TECHNIQUE

Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is an analytical technique used to obtain elemental and molecular chemical data about surfaces (static SIMS), and detect parts per billion (ppb) concentrations of impurities in semiconductors and metals (dynamic SIMS). All elements, including hydrogen, are detectable by SIMS.

In ToF-SIMS analysis, the sample is placed in an ultrahigh vacuum environment where primary ions bombard the sample and sputter atoms, molecules, and molecular fragments from the sample surface. The mass of the ejected particles (i.e., secondary ions) are analyzed via time-of-flight mass spectrometry.

In the ToF analyzer, ejected ions are accelerated into the analyzer with a common energy, but at different velocities depending on the particle mass. Due to the differences in these velocities, smaller ions move through the analyzer faster than the larger ions. The mass of the secondary ions are determined by their travel time through the analyzer. SIMS is a surface-sensitive analysis method, since only the secondary ions generated in the outermost 10 to 20 Å region of a sample surface can overcome the surface binding energy and escape the sample surface for detection and analysis.



ToF-SIMS Positive Secondary Ion Map

ANALYTICAL INFORMATION

Mass Spectrum - SIMS analysis identifies the elemental and ion composition of the uppermost 10 to 20 Å of the analyzed surface from positive and negative mass spectra. The high resolution of the ToF analyzer can distinguish species whose masses differ by only a few millimass units.

Depth Profile - During SIMS analysis, the sample surface is slowly sputtered away. Continuous analysis obtains composition information as a function of depth. Depth resolution of a few angstroms is possible. High-sensitivity mass spectra can be recorded or reconstructed at any depth of the profile.

 Copyright © 2001 by Materials Evaluation and Engineering, Inc.

Secondary Ion Mapping - A SIMS map measures the lateral distribution of elements and molecules on the sample's surface. To obtain a SIMS map, a highly focused primary ion beam is scanned in a raster pattern across the sample surface, and the secondary ions are analyzed at specific points on a grid pattern over the selected surface area. Image brightness at each point is a function of the relative concentration of the mapped element or molecule. Lateral resolution is less than 0.1 µm for elements and about 0.5 µm for large molecules.

TYPICAL APPLICATIONS

- Identifying lubricants on magnetic hard discs
- Measuring dopant distributions in semiconductors
- Profiling thickness of insulating films on glass
- Mapping elemental and molecular patterned surfaces
- Identifying compounds in thin organic films
- Determining the extent of crosslinking in polymers

SAMPLE REQUIREMENTS

Sample size cannot exceed 3.5 in. (85 mm) in any lateral direction. Height should not exceed 0.8 in (20 mm). Sample must be compatible with ultra-high vacuum (>$1x10^{-9}$ Torr).

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

THERMAL ANALYSIS

DESCRIPTION OF TECHNIQUES

Thermal analysis measures physical or chemical changes in a material as a function of temperature. Two common complimentary techniques in this category are differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). These methods are typically used to determine the material properties of organic polymers as the sample is heated or cooled in a controlled manner or held isothermally for a specified time. Differential thermal analysis (DTA) is a method similar to DSC, but performed at higher temperatures for metals, minerals, ceramics, and glasses.

DSC - Differential scanning calorimetry measures heat flow to or from a sample as a function of temperature and time. A small portion of a sample is placed in an aluminum pan and heated and/or cooled in a controlled manner. A reference material (usually an empty aluminum pan) simultaneously undergoes the same programmed time/temperature routine. Calorimetric measurements are made during the heating/cooling cycle.

Two methods can be used for the calorimetric measurements. Differences in temperature between the sample and reference material can be measured as the same amount of heat energy (calories) is added to both. Or, differences in the amount of heat energy added to both are measured as the temperature for both the sample and reference are kept constant. In both cases, the heat flow and temperature of the sample are monitored in comparison to the reference material. The analysis is usually performed in an inert gas atmosphere, such as nitrogen. The amount of energy absorbed (endotherm) or evolved (exotherm) as the sample undergoes physical or chemical changes (e.g. melting, crystallization, curing) is measured in calories as a function of the temperature change. Any material reactions involving changes in heat capacity (e.g. glass transition) are also detected.

The thermal cycle for DSC typically can range from less than -50°C to 300°C or greater. The principals for differential thermal analysis (DTA) are similar to DSC, but the temperature range for DTA can reach temperatures greater than 1500°C.

TGA - Thermogravimetric analysis continuously measures the weight of a sample as a function of temperature and time. The sample is placed in a small pan connected to a microbalance and heated in a controlled manner and/or held isothermally for a specified time. The atmosphere around the sample may consist of an inert gas, such as nitrogen, or a reactive gas, such as air or oxygen. The heating program may start in an inert atmosphere and then be switched to air at a certain point to complete the analysis. Weight changes observed at specific temperatures correlate to volatilization of sample components, decomposition, oxidation/reduction reactions, or other reactions or changes. Fourier transform infrared spectroscopy (FTIR) or mass spectroscopy (MS) may be used in conjunction with TGA to analyze and identify the evolved gases from constituents volatilized from the sample at specific temperatures. (See the FTIR and GC/MS sections of this handbook for more details about these analytical techniques.)

ANALYTICAL INFORMATION

DSC - By closely monitoring the heat flow and temperature, DSC can provide abundant information regarding a polymer material including: melting temperature, heat of fusion, glass transition temperature, curing temperature, heat of reaction, thermal history, and others. DSC is ideal for studying reversible reactions of thermoplastics such as melting-crystallization points and glass transition temperature. It is also used in the study of the kinetics of thermoset curing reactions, purity, heat capacities, and the effects of additives. Similarly, DTA analysis is used for determining the temperatures for melting and solid state phase transformations in metals, minerals, and ceramics.

TGA - As the TGA instrument measures the temperature and weight of the sample, thermally activated events are recorded. These events are expressed as weight loss or weight change for a given time or temperature. They may also be expressed as a rate of weight loss. The onset temperature for the weight loss is also recorded. These data correlate to and give information about such properties as: thermal stability, moisture or solvent content, additive or filler content, oxidation or decomposition temperatures and rate. Thermal events such as melting, glass transition, and other changes are not detected because there is no change in sample mass associated with these events. Identification of the constituents driven off as evolved gases may be obtained when the TGA is used in conjunction with FTIR or mass spectroscopy.

TYPICAL APPLICATIONS

DSC (or DTA)

- Determination of melting temperature, heat of fusion, and glass transition temperatures
- Analysis of polymer blends and copolymers
- Comparison of two lots of similar polymers
- Determination of cure temperatures/times for epoxies or other thermally-cured polymers
- Reaction rate and temperature evaluation
- Determination of thermal history, e.g. annealing, etc.

TGA

- Volatile compound concentration
- Plasticizer content
- Inorganic filler content
- Polymer thermal degradation profiles
- Polymer thermal and/or oxidative stability
- Identification of volatile components or thermal degradation products (with FTIR or MS)

SAMPLE REQUIREMENTS

DSC - Typically requires six to ten milligrams of sample. Samples may solids or liquids.

TGA - Typically requires twenty to thirty milligrams of sample. Samples may be solids or liquids.

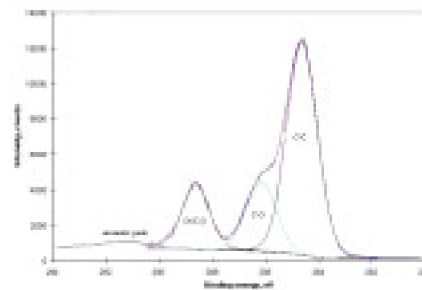
Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

X-RAY PHOTOELECTRON SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

X-Ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA), is an analysis technique used to obtain chemical information about the surfaces of solid materials. Both composition and the chemical state of surface constituents can be determined by XPS. Insulators and conductors can easily be analyzed in surface areas from a few microns to a few millimeters across.

The sample is placed in an ultrahigh vacuum environment and exposed to a low-energy, monochromatic x-ray source. The incident x-rays cause the ejection of core-level electrons from sample atoms. The energy of a photoemitted core electron is a function of its binding energy and is characteristic of the element from which it was emitted. Energy analysis of the emitted photoelectrons is the primary data used for XPS.

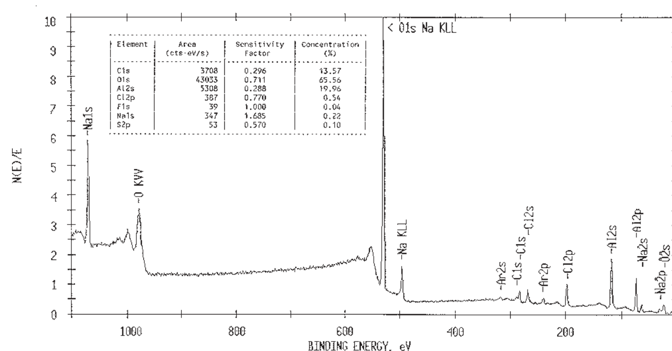


XPS Multiplex Survey for Carbon

When the core electron is ejected by the incident x-ray, an outer electron fills the core hole. The energy of this transition is balanced by the emission of an Auger electron or a characteristic x-ray. Analysis of Auger electrons can be used in XPS, in addition to emitted photoelectrons.

The photoelectrons and Auger electrons emitted from the sample are detected by an electron energy analyzer, and their energy is determined as a function of their velocity entering the detector. By counting the number of photoelectrons and Auger electrons as a function of their energy, a spectrum representing the surface composition is obtained. The energy corresponding to each peak is characteristic of an element present in the sampled volume. The area under a peak in the spectrum is a measure of the relative amount of the element represented by that peak. The peak shape and precise position indicates the chemical state for the element.

XPS is a surface sensitive technique because only those electrons generated near the surface escape and are detected. The photoelectrons of interest have relatively low kinetic energy. Due to inelastic collisions within the sample's atomic structure, photoelectrons originating more than 20 to 50 Å below the surface cannot escape with sufficient energy to be detected.



Element	Area (cts-eV/s)	Sensitivity Factor	Concentration (%)
C1s	3708	0.296	13.57
O1s	43033	0.711	65.56
Al2s	5308	0.288	19.96
Cl2p	387	0.770	0.54
F1s	39	1.000	0.04
Na1s	347	1.685	0.22
S2p	53	0.570	0.10

XPS Survey Spectrum with Surface Composition

ANALYTICAL INFORMATION

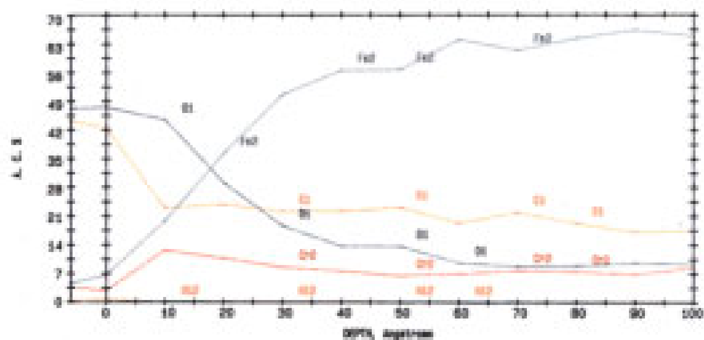
Survey Scan - Energy peaks in the survey scan identify the elemental composition of the uppermost 20 to 50 Å of the analyzed surface. All elements, except hydrogen and helium, are detected. Detection limits are approximately 0.1 atom percent for most elements.

High Resolution Multiplex Scan - This technique evaluates the chemical state(s) of each element through its core electron binding energies. Precise determination of binding energies are made through the use of curvefitting routines applied to the peaks in the multiplex scan. Shifts in the binding energy can result from the atom's oxidation state, chemical bonds, or crystal structure. A NIST database is available to identify binding energies with specific compounds.

Quantitation - The concentrations of the elements identified in the survey scan are determined by integrating the area under a characteristic peak for each element. Sensitivity factors are applied to the peak area values to determine the elemental concentration.

Depth Profile - The elemental composition is measured as a function of depth into the sample by alternating AES analysis with ion sputtering to remove material from the sample surface. Depth resolution of <100 Å is possible.

Mapping - The relative concentration of one or more elements is determined as a function of lateral position on the sample surface. An image is obtained where brightness indicates the element concentration.



XPS Depth Profile of Stainless Steel Surface

TYPICAL APPLICATIONS

- Analysis of thin film contamination
- Evaluation of adhesion failures
- Measurement of elemental composition of insulating materials (e.g., polymers, glasses)
- Identification of the chemical state of surface films (e.g., metal or oxide)
- Quantitative elemental depth profiling of insulators

SAMPLE REQUIREMENTS

Sample size cannot exceed 1 in. (25 mm) in any lateral direction. Height should not exceed ½ in. (12 mm). Sample must be compatible with an ultra-high vacuum environment (>10^{-9} Torr).

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

SAMPLE PRESERVATION AND HANDLING

INTRODUCTION

The importance of proper sample preservation and handling in the process of accurate materials analyses cannot be overemphasized. If samples are not cared for properly, important information may destroyed, adulterated, or obscured. At the very least, improper handling introduces a measure of uncertainty into the analytical results. Thus, a few general principles are presented here to help preserve samples and the often critical data they contain. Please note that the principles presented here are not all encompassing. If you are uncertain how to best preserve and transfer a sample, contact the analytical laboratory directly for advice and instructions.

COMMANDMENTS OF SAMPLE PRESEREVATION AND HANDLING

Don't Touch. Avoid touching the sample or area of interest with bare hands. Fingers inherently have significant amounts of organic and inorganic compounds that can contaminate the sample. Additionally, your fingers may pick up foreign material and transfer it to the sample or remove important deposits from the sample surface.

If you must use your hands to handle small samples, wear gloves. However, even gloves may transfer a certain amount of foreign material. Use clean tweezers or other handling tools for small samples.

As a rule of thumb, keep handling to a minimum, including poking, prodding, or scratching with tools or instruments. Such equipment may contaminate or destroy important material. Fracture surfaces especially prone to physical damage that can inhibit accurate analysis. Simply touching mating fracture surfaces back together after a failure will destroy microscopic fracture features that may be the key to a conclusive determination of the fracture mode.

Choose samples wisely. Select samples for analysis that are representative of what you are trying to determine, i.e., typical contamination or typical material. In many cases, a control sample of "normal" material or components may be very useful as a comparison with failed or problem components. In some cases, several specimens may need to be submitted to determine commonalities or a range of conditions.

Preserve sample integrity. Obtain samples in a way that does not influence the measurements to be made. If a sample must be cut or removed from larger piece, care must be taken not to contaminate or alter the area of interest. For example, the heat generated by flame cutting a metal sample may alter its microstructure and mechanical properties. Scraping on a hard surface with a metal instrument can produce wear debris from the instrument which is added to the component surface or to the collected surface deposits.

Submit a sample of appropriate size. Thermal analysis may require only a few milligrams. Quantitative chemical analysis may require a large surface area of several square millimeters or a few grams of material. If in doubt as to appropriate sample size, contact the analytical laboratory.

Preserve sample. Special sample handling and storage are often required to prevent potential changes in sample morphology and/or composition between time of sampling and analysis. Oxidation, evaporation, thermal degradation, or chemical interaction may occur if samples are not properly preserved.

Store samples in clean containers. This normally means new containers or those known to have been properly cleaned. Even if a previously used container appears clean, it may contain microscopic particles or liquids which could contaminate your sample and introduce uncertainty in the analytical results. If a sample must be shipped, package in such a way as to limit contamination or physical damage.

Avoid tape. Do not wrap samples or small particles in tape. Tape may leave an adhesive residue or remove critical sample constituents. Tape residue can create significant interference and uncertainty, particularly for analysis of organic compounds.

Identify and label. Clearly mark the sample containers to identify the contents. The source of the sample and, if applicable, its location within the source component should be recorded. Indicate the area of interest with a diagram rather than marking on the sample if possible. Data from the best preserved samples are meaningless if the sample and area of interest are not properly identified.

Obtain background data. Include significant background information about the sample and good instructions to the analyst with the sample. Provide a clear mandate for the analysis goals, i.e. explain why is the analysis requested. Background information about the sample that may help the analyst includes: where did it come from, what is it used for, and what has it been exposed to.

Provide control samples when possible. Submit a reference or control material(s) with the sample. A control sample will give you a baseline for comparison. If you are attempting to identify an unknown contamination, submit suspected sources of contamination along with the unknown for comparison. If an unusual condition is to be evaluated, comparison with a "normal" sample can be very useful.

Contact the analytical laboratory for specific sample preservation, handling, and shipping recommendations.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

SPECIFIC GUIDELINES FOR HANDLING FRACTURES

Fractures,even those of hard or high strength metals, are fragile and subject to mechanical and environmental damage that can destroy important microstructural features. Thus, fractures must be handled with great care from sampling through analysis.

First, a fracture surface should be preserved as soon as possible following the failure to prevent environmental attack, such as corrosion or oxidation. Ideally, the fracture and surrounding surfaces should be dried with air and stored in a dry environment. At a minimum, the fracture area should loosely covered to protect it from rain or incidental physical damage. The fracture should not be sealed in an air tight container where water could condense and corrode the fresh fracture surface. If adequate protection or storage in a dry environment is not possible, the fracture may be coated with an oil, grease, or other material that will protect the surface, but not chemically attack it. (Do not coat surface if corrosion appears to a factor in the failure.) The coating used should be easily and completely removable for the subsequent analysis.

Do not try to fit two fracture halves together or pick at fracture surface, as this will mechanically damage critical surface features. If the fracture must be removed from a larger part, make the cut far away from the fracture site. Package so as to prevent any contact with the fracture surfaces. Small bumps and dings can significantly affect the fracture morphology. Wrap each component of the failure separately.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

Handbook of Analytical Methods for Materials -- Copyright © 2001 by Materials Evaluation and Engineering, Inc.

ABOUT MATERIALS EVALUATION AND ENGINEERING, INC.

Our company name serves well as a basic description of our purpose. MEE, as we are often called, was founded in 1995 to fill a local need for an independent, high-quality materials characterization laboratory for advanced materials and critical components.

From our laboratory roots, MEE has rapidly developed into a leading failure analysis laboratory in the Upper Midwest. Referrals from satisfied customers have generated steady growth of new projects and clients. Our comprehensive online Handbook of Analysis Methods for Materials has also fostered a growing national and international clientele.

Our technical staff has experience with metals from aluminum to zinc, as well as, polymers and ceramics. Whether carbide cutting tools, soft solders, injection molded plastics, or microscopic laser welds, the MEE laboratory staff knows how to properly prepare and examine samples to obtain the optimum materials characterization data.

Failure analysis and advanced research projects are directed by registered professional engineers. These engineers specialize in the behavior of materials and how their structure affects service performance. Comprehensive reports combine reliable analytical data with a sound engineering perspective to provide practical, cost-effective solutions to our customers.

The future is bright for MEE. While continuing to focus on our strength in solving material problems, we will continue to grow in expertise and analytical capabilities. Excellent customer service and close attention to detail will remain cornerstones in all of our endeavors.

Materials Evaluation and Engineering, Inc.
13805 1st Avenue North Suite 400
Plymouth, MN, 55441

Phone: (763) 449-8870

Suggested Price: $19.99