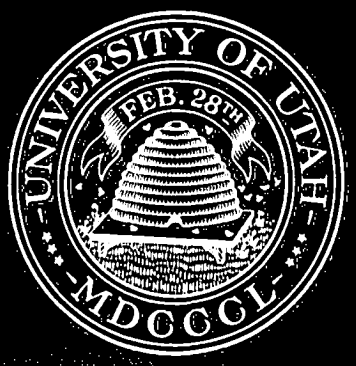
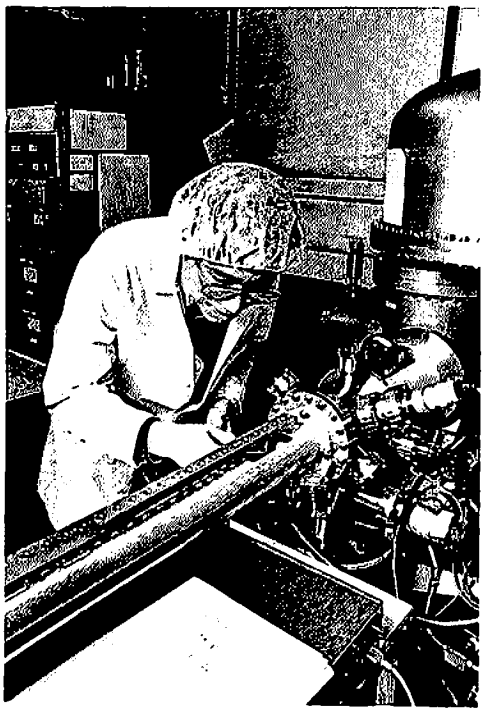


Surface Analysis Laboratory

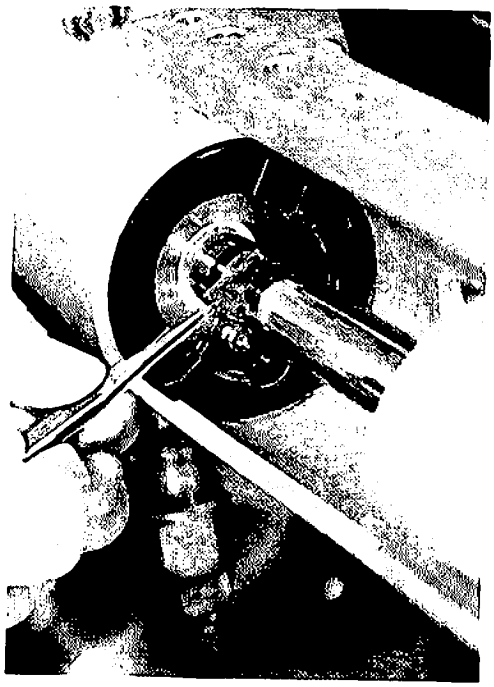
Chemical and Physical Characterization of Solid Surfaces and Films for Practical Problem Solving, Basic and Applied Research

The University of Utah
College of Engineering
"A Commitment to Innovation"

ESCA: The Most Quantitative Analysis



View of HP X-ray photoelectron spectrometer.



Detail of sample mounting geometry.

ESCA (Electron Spectroscopy for Chemical Analysis) is a non-destructive surface analysis technique that provides total elemental analysis (except for H or He) of a solid surface or film. The technique samples the top 20-50 Å of the surface (5 to 10 atomic monolayers) and possesses a sensitivity of 0.1 to 1 atom percent, depending on the element, with ca. 10% accuracy. It will analyze all classes of material including non-conductors. It also provides chemical bonding information, and it can be used for surface quality control determinations.

Applications for ESCA Analysis

- Detection of or tracing of surface contaminants. By judicious selection of samples, contaminants can often be traced to their source and then eliminated.
- Analysis of both sides of a bond failure. Since delaminations are often due to surface contaminants, this can be useful in determining which side of a bond failed and if a contaminant such as a fluorocarbon or silicone was involved.
- Measurement of oxide film thickness on metals. ESCA analysis can distinguish the metal oxide from the elemental form beneath.
- Elemental analysis of powders or wear residues. A quick wide scan analysis can suggest the residue source; e.g., a fluorocarbon seal, hydrocarbon lubricant, or a metallic part.
- Determination of the degree of oxidation of polymers, carbon fibers, etc. — where it counts, the surface region.
- Detection of surface segregation of mold release agents, plasticizers, additives, or alloy components such as Be in Cu foil.
- Determination of film thickness in the 0-150 Å range where layer compositions are distinguishable.

About the Instrument

The HP 5950B electron spectrometer is equipped with a monochromatic X-ray source and is coupled to an HP 9845 computer for data manipulation and storage. Accessories include an argon etch gun, an inert gas glove box, variable angle sample probe, and a cold temperature probe.

Example of ESCA Analysis

Surface treatments of biomaterials

Much biomedical research involves surface modification of polymers to enhance biocompatibility. ESCA can be used to monitor surface treatments. Figures 1 and 2 below illustrate the use of high resolution narrow scans to visualize chemical changes in the surface of contact lenses and silica, respectively.

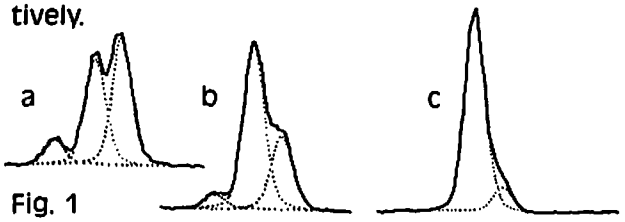


Fig. 1

Curves a, b, and c in figure 1 represent carbon spectra for varying degrees of treatment from untreated to fully PEO-coated PHEMA contact lenses, respectively. Note the increase of the ether component in the treated lens with respect to both the ester and alkyl carbon peaks as the PHEMA becomes masked by the coating.

Fig. 2

In an oxidized form of Si such as quartz or glass, the Si peak is seen several volts higher than for polymeric forms. The curve to the left shows the Si signal from untreated quartz while the one to the right is from the same surface treated with an alkylating agent. The new peak showing is from the silane Si of the agent.

Surface changes due to heat treatments

A diode constructed from layers of In, P, Al, Ag exhibits different electrical properties depending on heat treatments.

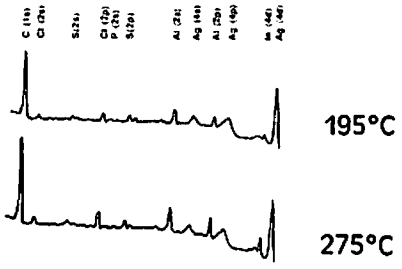


Fig. 3

ESCA wide scan data show increases in Al with temperature suggesting movement of the Al through the Ag overlayer. Oxidized P is also seen migrating to the surface.

Heat treatment to 900°C under argon will transform polyimide sheet material into a conductive amorphous film.

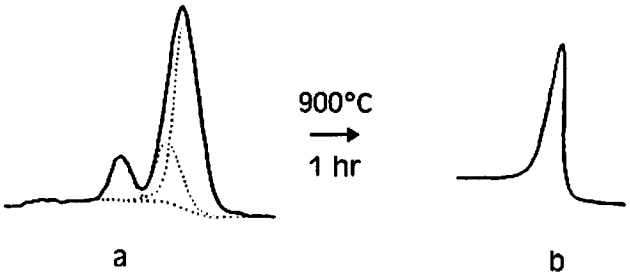


Fig. 4

Narrow scans of carbon reflect the transition. Native polyimide (curve a) has well defined components, which are missing after heat treatment, (curve b, typical of elemental carbon).

Angular Resolved ESCA

The surface sensitivity of ESCA can be varied between near 10 to 100 Å by rotating the sample relative to the electron analyzer. Computer analysis of sets of spectra taken at different angles can be used to estimate fractional surface coverage and/or film thicknesses. Shown below are a set of theoretical curves relating film/bulk signal ratios to rotation angle. Figure 6 depicts the distribution of the chi square statistic for a range of thickness and coverage values in an experiment of lysozyme adsorption onto glass.

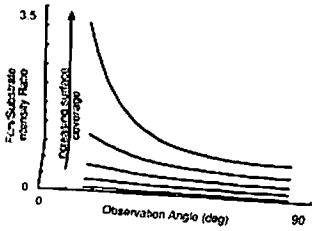


Fig. 5

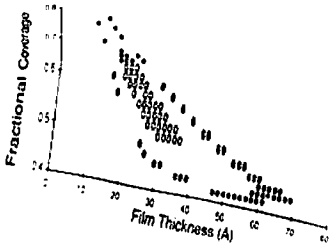


Fig. 6

Surface Analysis Laboratory

Other Available Techniques

Contact Angle / Surface Tension
Zeta Potential / Streaming Potential
Scanning Electron Microscopy
Energy Dispersive X-ray Analysis
Raman Spectroscopy
Surface Infrared Spectroscopy
Ellipsometry

Staff

J. D. Andrade
Professor, Departments of Materials Science and Engineering, Bioengineering

Paul Dryden
Manager, Surface Analysis Laboratory

For Further Information:

Paul Dryden
Manager, Surface Analysis Laboratory
2480 Merrill Engineering Building
University of Utah
Salt Lake City, Utah 84112
(801) 581-7730 or (801) 581-3391

University of Utah
Surface Analysis Laboratory
2480 Merrill Engineering Building
University of Utah
Salt Lake City, UT 84112
801-581-7730

OFF CAMPUS RATE SHEET
ANALYSIS BY X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

General Instrument Time: $100/hr.

Typical analysis times and costs

Type of Spectrum	Elapsed Time	Estimated Cost
Wide scan -- detects all elements except for H and He. Good for unknown surfaces or if contamination is suspected	45 minutes	$75
Narrow scan of 20 eV window. One spectrum per element of interest. Shows chemical shift information for that element.	15-30 minutes, depending on concentration of element in the surface.	$25-$50 per element or spectrum.

Data Analysis and Report Preparation.

There is no charge for routine report preparation, which includes peak identification and tables of estimated surface elemental compositions and of binding energies of components resolved from narrow scan spectra. More complex data reduction operations or literature review, including outside consultation, will be billed at $100/hr plus expenses.

Sample Preparation

XPS analyzes the top 100 Å of the sample. The sample must not be hygroscopic or volatile, since it must be stable at 10^{-9} torr vacuum. Samples must be capable of being mounted within a volume of 1 mm by 9 mm by 11 mm. We will mount powders and cut thin foils, paper, etc, which can easily be cut by scissors or scalpel. We cannot cut thick, hard metals, or brittle materials like glass. These types of materials should be submitted of the proper size for direct mounting.

When packaging samples for shipment, care must be taken to prevent contamination of the surface of interest by fingerprints or contact with the shipping container. Double stick tape may be used to attach the samples to the container, if they may be readily removed. If the sample is packaged loose (say wrapped in a clean, lint-free cloth), clearly indicate the surface of interest.

For further information, contact Paul Dryden at 801-581-7730.