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AMICUS / ESCA 3400

Quality XPS instrumentation for routine analysis

The AMICUS, also known as the ESCA 3400 in Japan, has been developed from proven technology and incorporates the latest in digital control electronics producing an easy to use, flexible system suitable for all levels of expertise.

Application

The high efficiency energy dispersive analyser of the AMICUS means that a high sample throughput is easily obtained.

Depth profiling TiN/SiO2 Overview

Overview

Thin coatings are increasingly being used in many industries to improve surface properties of materials, such as increased resistance to friction and wear or increasing the hardness and strength. Hard coatings like TiN are particularly useful in the coating of machine tools in extending the working life of the tool, where the properties of the thin film depend on the thickness and the adhesion between the coating and the substrate. This information can be gained using X-ray photoelectron spectroscopy (XPS) which provides qualitative and quantitative information on the elements present in complex materials and whilst providing chemical state information from these elements.

The Use of AMICUS

AMICUS, with its high energy X-ray source, efficient electron transfer lens, electron energy analyser and high speed ion etching, is ideally suited to elemental depth profiling. The AMICUS instrument was used to determine the depth dependent concentration profile through a thin TiN/SiO2 double layer film. To ensure good interface resolution between layers the X-ray photoelectron spectra were recorded from a small, 3mm2 sample which was rotated during sputtering to ensure an even etch crater. Figure 1 shows the data acquisition manager of the fully automated VISION control software. Complex data collection procedures are performed by means of a simple flowchart which may be saved and recalled for future analysis of similar samples. The use of the integrated VISION control software means that the analysis/sputter routine may be left to generate a complete concentration depth profile dataset unsupervised.

Probing the Chemical Nature of TiN

A full concentration profile for the constituent elements of the TiN/SiO2 layers on a Si substrate is shown in Figure 2. It is immediately apparent from the concentration profile that the TiN shows a sharp boundary with the SiO2 substrate at 40nm The second interface is observed between SiO2 and Si at 150nm, with no degradation in the interface resolution.

The great advantage of XPS over other techniques is that chemical state information is also available from the spectra, allowing a chemical state concentration depth profile for the elements to be generated. Changes in the surface layer as a function of depth from the original surface are easily observed. Figure 3(a) shows the Ti 2p region after introduction of the sample into AMICUS, with Figure 3(b) showing the same region after a single sputter cycle. The Ti 2p peak is spin-orbit split into two peaks, separated by approximately 6eV, with each of these peaks showing further structure, reflecting different Ti chemical states. The Ti 2p peaks are deconvoluted into three component peaks corresponding to Ti in titanium monoxide (TiO), titanium nitride (TiN) and titanium dioxide (TiO_2), with a shift to higher binding energy relative to metallic Ti 2p quoted in the literature at 454eV binding energy.

The X-ray photoelectron spectrum of the uppermost TiN layer shows that Ti is in fact predominantly present as TiO_2 and, as shown in the concentration depth profile, the titanium oxide species persist throughout the TiN film. The oxidation of titanium will decrease the hardness of the thin film and suggests that the deposition of the TiN film should be performed in a controlled, inert atmosphere.

Similar chemical state information is also contained in the Si 2p spectra shown in Figure 4, where the spectra are shown as a function of depth from the surface. At the surface, where the TiN overlayer is expected to completely attenuate the Si 2p signal it is noted that there is still some signal at 99eV binding energy. This is thought to be due to the Si substrate exposed during sample preparation, as it remains constant through the TiN and SiO_2 layers. After removal of the TiN overlayer, the Si 2p signal from the SiO_2 layer is observed at 103.5eV binding energy. Further removal of surface material exposed the Si substrate, characterized by the elemental Si 2p photoemission peak at 99.3eV.

Summary

X-ray photoelectron spectroscopy using the AMICUS instrument is an invaluable tool for the characterization of thin film materials. The fully automated operation using VISION control software allows repetitive depth profiling to be completed with ease.
The key features of AMICUS demonstrated are:

- Concentration depth profiling,
- Unsupervised, fully computer controlled data collection using VISION software,
- Chemical state determination of surface atoms.

Specification

AMICUS XPS has been designed with a compact footprint enabling it to be used for a wide variety of applications ranging from routine laboratory use to quality control and assurance in production environments. Compact yet high performance design has been accomplished through the incorporation of the successful "Dupont" type analyser and newly optimised digital electronics.

A simple, rapid sample introduction system is integrated with a fully automated multi sample carousel system enabling a wide range of applications to be addressed, including high throughput completely unattended routine operation. The standardised, highly user friendly XPS acquisition and data processing software package enables both simple and complex studies to be carried out in a totally automated fashion. The ability to set up and store previously defined data acquisition and processing parameters ensures that reproducible procedures are maintained, an important criterion in quality control environments.

Options

An abbreviated instrument specification.

VACUUM SYSTEM

- Sample analysis chamber (SAC)
- 150l/s turbomolecular pump (base pressure $5x10^{-7}$Pa)

SAMPLE INTRODUCTION CHAMBER

- 50l/s turbomolecular pump
- 10 sample introduction chamber (optional)

SYSTEM BAKING

- integrated timer controlled heating tapes

SAMPLE HANDLING

- 10 sample carousel
- fully software controlled

SAMPLE DIMENSIONS

- ≤10mm diameter
- ≤ 5mm thick

X-RAY SOURCE

- conical formed magnesium target
- 300W max power 12kV 25mA
- Dual Mg/Al anodes (optional)

ELECTRON ENERGY ANALYSER

- Low pass/high pass filter
- selectable pass energy, 25, 75, and 150eV
- single channeltron detector

ION ETCHING SYSTEM

- Standard ion etching source

  0.5, 1, 1.5, and 2kV accelerating voltage
  Etch rate 5-100 Angstrom/min (material dependent)
  sample rotation during etching

- Kaufman high speed etching source (option)

  0-1000V accelearting potential (continuously variable)
  Etch rate 50-800 Angstrom/min (material dependent)