

INTRODUCTION

X-ray Photoelectron Spectroscopy (XPS) is a surface analytical technique that is used to provide information about the elemental and chemical composition of the top 10 nm of a surface. It can determine the surface concentration of elements present, chemical state information of the detected elements such as distinguishing between SiO_2 and elemental Si; and oxidation states of metals (e.g. Ni^{+2} vs. Ni^0). It is widely used to characterise organic polymer materials as well. Both Kratos AXIS 165 and the latest Kratos AXIS Ultra DLD X-ray photoelectron spectrometers available at MSSSI are high performing instruments with combined capabilities of conventional spectroscopy, imaging, small spot spectroscopy, Auger electron spectroscopy and in-situ heating/cooling facilities. Sputter depth profiling of thin organic layers are now possible with the state of the art Argon Gas Cluster ion source which can also operate in monoatomic mode for sputtering inorganic materials.

Figure 1: Kratos AXIS Ultra DLD X-ray Photoelectron Spectrometer

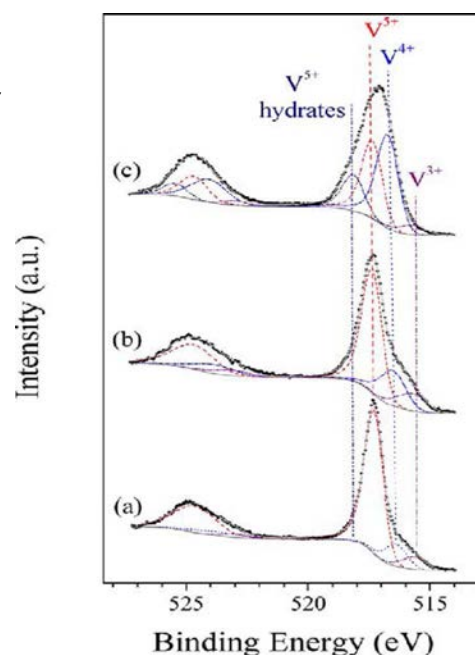


Figure 2: Vanadium 2p core level spectra obtained from (a) commercial V_2O_5 as is, (b) V_2O_5 refluxed in a 90:10 alcohol mixture of 2-methyl propan-1-ol and benzyl alcohol for 4 hours at a temperature of 108°C and (c) V_2O_5 refluxed as in (b) and then refluxed in the presence of 85% ortho-phosphoric acid for 1 hour at 90°C

TECHNICAL SPECIFICATIONS

- Depth of analysis: 3 - 10 nm
- Elements detected: all elements except H and He
- Detection limit: 0.1 atomic %
- Chemical imaging with spatial resolution of $3\mu\text{m}$
- Small spot spectroscopy with analysis area of $15\mu\text{m}$
- High power monochromatic Al X-ray source
- Dual Mg/Al X-ray source
- Sample charge neutraliser for semiconducting and insulating materials
- Ar Gas Cluster ion source for depth profiling organic materials
- Monoatomic Ar ion gun for depth profiling inorganic materials
- Schottky field emission electron gun with 100 nm beam diameter for Auger Electron spectroscopy, Secondary Electron Microscopy and Scanning Auger Microscopy
- In situ sample heating and cooling (-150°C to $+600^\circ\text{C}$)
- Crystal cleaver for fracturing/cleaving samples under UHV for subsequent analysis
- High temperature Gas Reaction Cell for sample treatment in vacuum conditions prior to analysis
- Sample compatibility with UHV environment



KEY FEATURES OF THE XPS TECHNIQUES ARE:

- Quantitative elemental surface analysis
- Chemical state surface analysis
- Depth profiling
- Chemical imaging
- Suitable for conductors and insulators
- Angle resolved XPS
- Auger Electron spectroscopy, SEM and Auger Scanning Microscopy

EXAMPLES OF WORK DONE IN MSSI:

- Identifying contaminants and stains on materials
- Determining chemical composition of powders and unknown materials
- Determining surface chemical composition of biomedical components such as stents, catheters, etc.
- Identifying the degree of surface oxidation/segregation in metal/alloy samples
- Surface characterisation of bi/tri metallic catalysts
- Surface characterisation of vanadium phosphorous pentaoxide catalysts (Figure 2)
- Examining changes in functional groups in polymers/ composites before and after processing
- Fe mapping for possible contamination in an artificial joint
- Examining oxygen spectra in vacuum cleaved bioglass for influence of added network modifiers/formers
- Chemistry of organic surfactant capped nanorods and nanoparticles undergoing various treatments
- Compositional analysis of mud, clay and ash minerals
- Depth profiling of NiTi alloy to characterise surface oxide layer
- Oscillatory anodic film growth on III-V compound semiconductors
- Molecular interconnect for nanotechnology
- Surface analysis of electro grafted polymers on carbon fibre

Figure 3: (a) plasma deposited TiAlN after oxidation in air at >900 °C (b) Ti 2p image background subtracted (c) Ti 2p line scan from image (d) Ti 2p, Fe 2p and Cr 2p images showing Fe and Cr segregation to the surface

