EAgLE – WP 4.1 and 4.2 -XPS at IF-PAN

Introduction

X-Ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA) is a widely used technique to investigate the chemical composition of surfaces. The technique provides elemental and chemical information on surface layers or thin film structures is of value in many industrial applications including: semiconductor, dielectric materials, electronics, catalysis, corrosion, polymer surface modification, adhesion, etc.

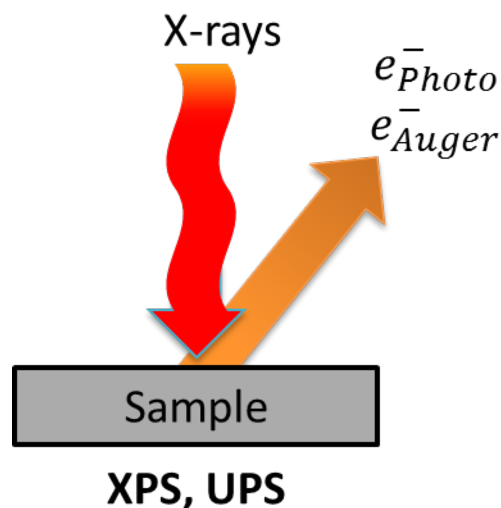


Figure : Work principle of XPS, UPS

As part of the EAgLE project a modern high resolution X-ray and UV photoelectron spectrometer based on VG Scienta R3000 XPS/UPS/ARPES analyzer has been purchased. The configuration of the spectrometer allows not only making standard XPS and UPS analysis, but permits also performing imaging, angle-resolved XPS, depth profiling, REELS and ISS analysis. Furthermore the instrument is equipped with a preparation chamber which includes several preparation and characterization techniques such as LEED, high pressure reactor, electron beam evaporator with quartz micro balance, mechanical and ion cleaning of the sample, sample cleavage, …

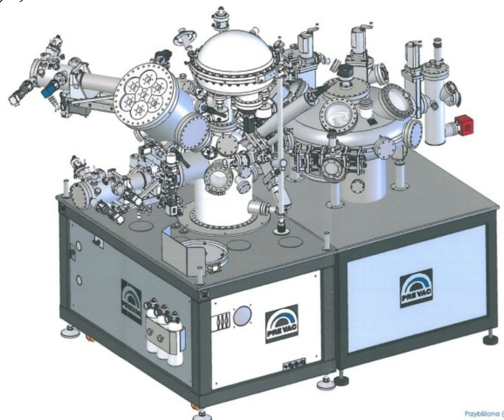


Figure : 3d representation of the instrument

This customized scientific instrument is composed of three (ultra-) high vacuum chambers:

1) Analysis Chamber (XPS, UPS, REELS, ISS)
2) Preparation Chamber (LEED, high pressure, evaporation, crystals cleaving device, surface scraping, sputtering)
3) Distribution Chamber including sample storage and high pressure reactor.

This equipment will be installed in February 2015 in a renovated laboratory room at the IF-PAN, building IX, rooms 11 and 12.

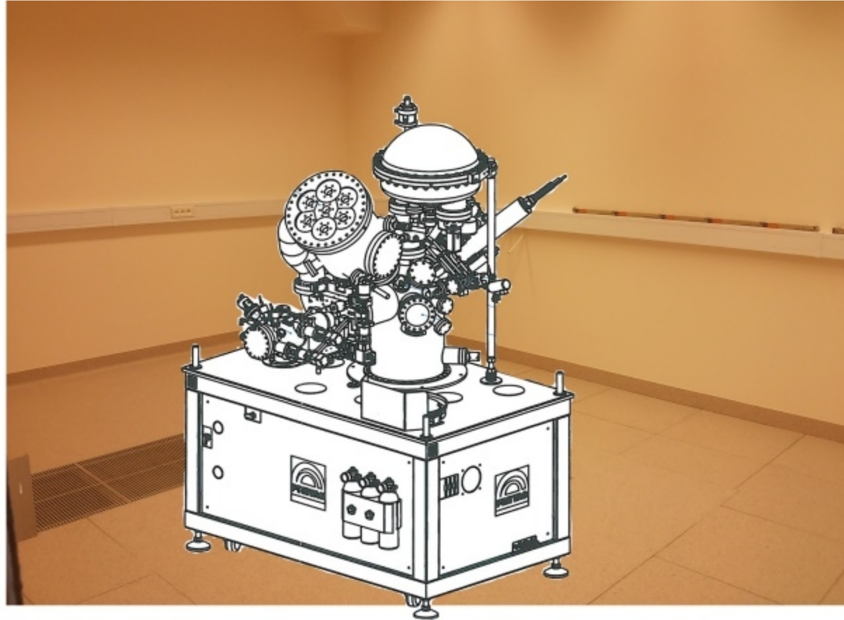


Figure : Representation of the XPS instrument in the renovated laboratory

Analysis Chamber

Specification of the technique and instrument

- Elemental Detection: Li to U (no H, He)
- Detection Limit >0.5%-100%
- Spatial Resolution: <10µm
- Information depth: ~10nm (2 nm in AR-XPS), up to 10µm by ion erosion)
- Basis pressure : $<10^{-9}$mbar
- Sources:
  - X-ray gun non-monochromated Al-Kα and Mg-Kα source
  - X-ray gun monochromated Al-Kα source
  - UV (He-I, He-II) source
- Analyzer:
  - Pass Energy: 2-200eV
  - Resolution : <0.5eV
- Charge Compensation for insulators
- Ion Gun for Depth Profiling (Ar^+ 0.15-5keV)
- Sample:
  - Temperature: 90K-1500K
  - maximal later size: <20 mm
  - maximal thickness: <10 mm

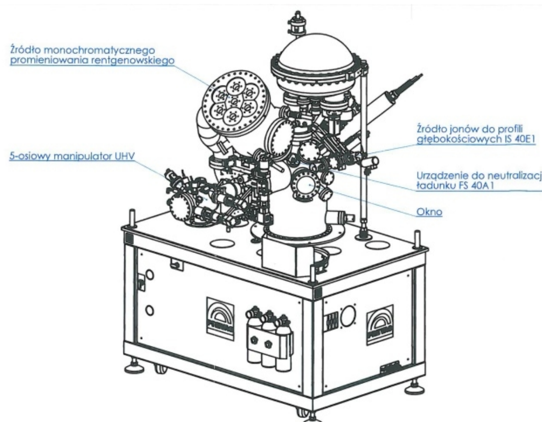


Figure : Details of the analysis chamber

Characterization by XPS/UPS

Description

Xray Photoelectron Spectroscopy (XPS), also referred to as Electron Spectroscopy for Chemical Analysis (ESCA), irradiates the sample surface with a low energy Xray (Al – 1486 eV or Mg – 1253 eV). The Xrays excite the bounded (core and valence) electrons of the sample atoms. These electrons will be emitted from the parent atom as a photoelectron if their binding energy is lower than the X-ray energy. From the binding energy and intensity of a photoelectron peak, the elemental identity, chemical state (chemical bonds), and quantity of an element can be determined.

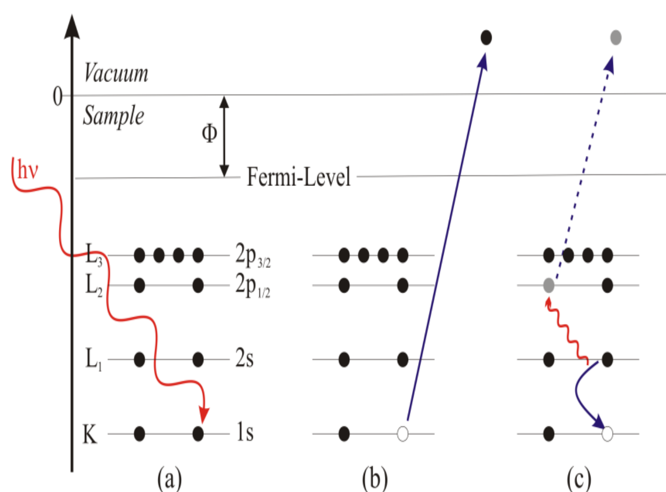


Figure : Principle of photoelectron emission

This analysis technique is surface sensitive as most of the detected photoelectrons originate from atoms near the sample surface (1-10 nm).

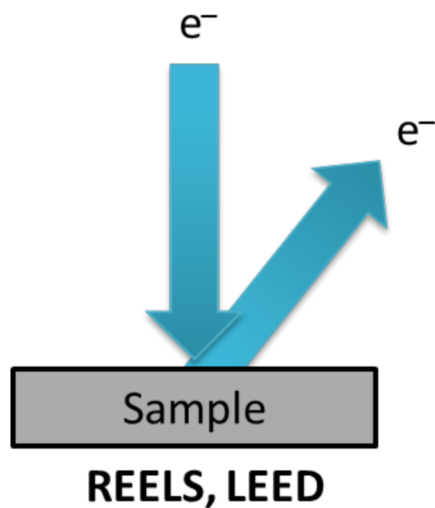
The angle-resolved XPS procedure allows determining in a non-destructive the order and thicknesses of fine multilayer (up to 10 nm) combined with the elemental distribution and chemical states of each film way.By dint of an ion gun it is possible to analyze the chemical composition deeper (>10nm) in the sample.

The use of an UV source (e.g. noble gas discharge lamp He I - 21.2 eV) instead of an X-Ray source ionize only the electrons from the outermost levels of atoms - the valence levels. The Ultraviolet photoelectron spectroscopy (UPS) allows studying the valence energy levels and chemical bonding, especially the estimation of bonding character of molecular orbitals become possible.

Applications for XPS and UPS

XPS analysis, which provides elemental and chemical information on surface layers or thin film structures, is of value in many industrial applications including: semiconductor, dielectric materials, electronics, catalysis, corrosion, polymer surface modification, adhesion, etc.
UPS is very useful as a technique to determine the electronic structure (determination of bonding, nonbonding, and/or antibonding molecular orbitals) of solids. By this way the work function of the material being analyzed can be determined which has an increasing application in characterization of organic and inorganic photovoltaics and organic LEDs.

Characterization by REELS



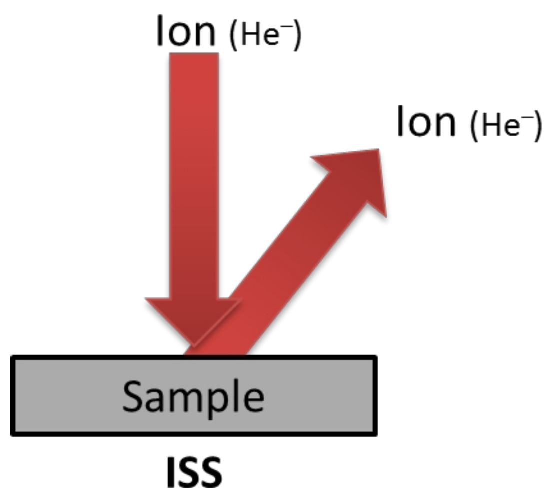
Description

By bombarding the surface with monoenergetic electrons and measuring the energy distribution of the reflected electrons, information about excitation of vibrational and plasmon states can be obtained. Furthermore a semi-quantitative determination of Hydrogen at the sample surface is possible by this technique (REELS – Reflective Electron Energy Lost Spectroscopy).

Applications for REELS

REELS not only allows to study semi-quantify Hydrogen but gives also information about the vibrational properties of adsorbents and surface phonons.

Characterization by ISS



Description

Measuring the energy distribution of scattered ions (ISS - Ion Scattering Spectroscopy, LEIS – Low Energy Ion Scattering) allows probing the elemental composition of the outermost atomic layer.

Applications for REELS

The extreme surface sensitivity of ISS and the absence of matrix effects make it possible to quantify the surface density of different elements. Also a study of intramolecular segregation processes is possible.

Preparation Chamber

Specification of the preparation chamber

The preparation chamber which is connected to the analysis system and which can be independently used, allows different kinds of sample preparation previous to the XPS/UPS analysis.
This in-situ sample treatment and characterization are possible within the preparation chamber:

- Ar-Ion cleaning

- Cleaver
- Electron beam evaporator and thickness monitor
- LEED
- Annealing up to 1500K
- Samples Storage
- High pressure reactor (up to 2 Mpa)
- Diamand knife

Characterization by LEED

Description

Low-energy electron diffraction (LEED) is a technique for the determination of the surface structure of single-crystalline materials by bombardment with low energy electrons (20–200 eV). This characterization technique is installed in the preparation chamber. The observation of the LEED diffraction pattern gives information on the symmetry of the surface structure or in case of the presence of an adsorbate can inform on the rotational alignment of the adsorbate unit cell with respect to the substrate unit cell.

Distribution Chamber

The distribution chamber allows the sample transfer between the different vacuum chambers. Also the airlock and a high vacuum sample storage chamber is connected to this distribution chamber