X-ray Photoelectron Spectroscopy

Diagram showing energy levels and transition of electrons.
XPS – ESCA
XPS instrument at UIO?

Kratos Axis Ultra – new in 2007
Introduction

X-ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy works by irradiating a sample material with mono energetic soft x-rays causing electrons to be ejected.

Identification of the elements in the sample can be made directly from the kinetic energies of these ejected photoelectrons.

The relative concentrations of elements can be determined from the photoelectron intensities.
XPS - ESCA

• Surface sensitive analysis technique based on photoelectric effect. Depth of analysis ~4-40 nm.
• All elements except Hydrogen.
• Wide range of materials: Polymers, Ceramics, metals …. (vacuum compatible)
• Applications: corrosion, catalysis, thin films, surface coatings, segregation …
• Gives information on chemical composition and chemical state.
History

- 1887 Heinrich Hertz / 1888 Wilhelm Hallwaches illuminating metal surfaces resulted in electronic emission
- 1900 Max Planck - black body radiation
- 1905 Einstein - light is quantized
  \[ E = h \nu \]
- 1950 Kai Siegbahn 1981: Nobel Prize studied photoejection of electrons with x-rays
- 1950: Instrumentation 1960: Chemical Applications
Photoelectric effect

1887 Heinrich Hertz / 1888 Wilhelm Hallwaches
1921 Albert Einstein - Nobel Prize
1981 Kai Siegbahn - Nobel Prize
Most commonly observed phenomena with light can be explained by waves. But the photoelectric effect suggested a particle nature for light.
1. The electrons were emitted immediately - no time lag!

2. Increasing the intensity of the light increased the number of photoelectrons, but not their maximum kinetic energy!

3. Red light will not cause the ejection of electrons, no matter what the intensity!

4. A weak violet light will eject only a few electrons, but their maximum kinetic energies are greater than those for intense light of longer wavelengths!
Bohr’s atomic model and Binding energy

In the Bohr model of the atom, electrons travel in defined circular orbits around the nucleus. The orbits are labeled by an integer, the quantum number $n$. Electrons can jump from one orbit to another by emitting or absorbing energy. The inset shows an electron jumping from orbit $n=3$ to orbit $n=2$, emitting a photon of red light with an energy of 1.89 eV.

\[ E = \frac{-13.58Z^2}{n^2} \text{ eV} \]
Basic equations:

The relationship governing the interaction of a photon with a core level is:

\[ KE = h\nu - BE - \Phi \]

- **KE** = kinetic energy of ejected photoelectron
- **\(h\nu\)** = characteristic energy of X-ray photon
- **BE** = binding energy of the atomic orbital
- from which the electron originates.
- **\(\Phi\)** = spectrometer work function
Surface Sensitivity
Ionization

Ground State

XPS (one hole final state)

UPS (one hole final state)
XPS example: Gold

Gold XPS wide scan spectrum

<table>
<thead>
<tr>
<th>Photoelectron Peaks</th>
<th>4s</th>
<th>4p(_{1/2})</th>
<th>4p(_{3/2})</th>
<th>4d(_{3/2})</th>
<th>4d(_{5/2})</th>
<th>5s</th>
<th>4f(_{5/2})</th>
<th>4f(_{7/2})</th>
<th>5f(_{1/2})</th>
<th>5p(_{3/2})</th>
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<tbody>
<tr>
<td>Binding energies</td>
<td>763</td>
<td>643</td>
<td>547</td>
<td>353</td>
<td>335</td>
<td>110</td>
<td>88</td>
<td>84</td>
<td>74</td>
<td>57</td>
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</table>

<table>
<thead>
<tr>
<th>Auger Peaks</th>
<th>N(<em>{57})O(</em>{45})O(_{45})</th>
<th>N(<em>{5})N(</em>{6})N(_{67})</th>
<th>N(<em>{4})N(</em>{6})N(_{67})</th>
<th>N(<em>{5})N(</em>{67})V</th>
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<tbody>
<tr>
<td>Binding Energies</td>
<td>1416</td>
<td>1342</td>
<td>1324</td>
<td>1247</td>
</tr>
</tbody>
</table>
XPS example: Gold

Au 3d	extsubscript{5/2} electrons have a binding energy of 2206 eV therefore they are not ejected by Al Kα X-rays of energy 1486.6 eV.
Relaxation - Auger

Auger (CVV) (two hole final state)

Auger (C_1 C_2 C_2) (two hole final state)

E_{kin} = (E_3 - E_1) - (E_{vac} - E_3)
E_{kin} = (E_2 - E_1) - (E_{vac} - E_2)

Initial State
Spin-Orbit Coupling

Orbital = s
l = 0
s = +/- 1/2
ls = 1/2
Spin-Orbit Coupling

Orbital = p

l = 1
s = ±1/2
ls = 1/2, 3/2
Spin-orbital splitting
Peak Notations

L-S Coupling ($j = l \pm s$)

\[ e^\pm \]

\[ s = \frac{1}{2} \]
\[ j = l + \frac{1}{2} \]
\[ s = -\frac{1}{2} \]
\[ j = l - \frac{1}{2} \]

\[ n \]
\[ \ell = 0 \ldots S \]
\[ 1 \ldots p \]
\[ 2 \ldots d \]
\[ 3 \ldots f \]

\[ 2p_{3/2} \]

\[ j = \ell - s \]
\[ j = \ell + s \]

\[ l = 1 \quad p \]
\[ p_{1/2} \quad p_{3/2} \]
\[ s = -1/2 \quad s = +1/2 \]

Area ratio
1 : 2

\[ l = 2 \quad d \]
\[ d_{3/2} \quad d_{5/2} \]
\[ s = -1/2 \quad s = +1/2 \]

Area ratio
2 : 3

\[ l = 3 \quad f \]
\[ f_{5/2} \quad f_{7/2} \]
\[ s = -1/2 \quad s = +1/2 \]

Area ratio
3 : 4
Electronic structure and spectroscopy

**Na 1s**

*Mg Kα* from NaCl

**Na 2p**

*Mg Kα* from NaCl pellet etched

Vacuum Level

- **VB**
- **2p** (BE = 31 eV)
- **2s** (BE = 64 eV)
- **1s** (BE = 1072 eV)
## Binding energies?

**Sodium**

### Electron binding energies

<table>
<thead>
<tr>
<th>Label</th>
<th>Orbital</th>
<th>eV [literature reference]</th>
</tr>
</thead>
<tbody>
<tr>
<td>K</td>
<td>1s</td>
<td>1070.8 [3]</td>
</tr>
<tr>
<td>(\text{L}_I)</td>
<td>2s</td>
<td>63.5 [3]</td>
</tr>
<tr>
<td>(\text{L}_{\text{II}})</td>
<td>2p(_{1/2})</td>
<td>30.4 [3]</td>
</tr>
<tr>
<td>(\text{L}_{\text{III}})</td>
<td>2p(_{3/2})</td>
<td>30.5 [2]</td>
</tr>
</tbody>
</table>
Calculate binding energies

According to Bohr's atomic model electrons bound to a positively charged nucleus has a discrete set of allowed energies:

\[ E = \frac{-13.58Z^2}{n^2} \text{ eV} \]

\( Z=\text{atomic number} \), \( n=\text{orbit number} \)

The binding energy \( EB \) of such an electron equals the positive value of this equation (e.g. \(-E\))

**A:** Using Bohr's model - calculate the binding energies for 1s and 2s levels in atoms with \( Z=11,20,30 \) and 40.

**B:** Do you think Bohr's theory will:

1) serve as a good approximation for calculating binding energies?
2) overestimate or underestimate the binding energies?
XPS spectra of Palladium

$BE = h\nu - KE - \Phi$
Instrumentation

Surface analysis by XPS requires irradiating a solid in an Ultra-high Vacuum (UHV) chamber with monoenergetic soft X-rays and analysing the energies of the emitted electrons.
Instrument

1. Chambers
2. Vacuum and Pumps
3. Sources
4. Monochromator
5. Analysers

Specimen handling
Planning experiments
Dual anode X-ray source

Fig. 3. Double-anode X-ray source.

The two components of the Kα spectrum (K_{α1} + K_{α2}) which comprise the Kα spectrum of Al. [Spectrum from Siegbahn et al., 1967.]
Survey scan: Most elements have major photoelectron peaks below 1100 eV, so a range from 1100 - 0 eV is usually sufficient.

Al anode

Mg anode
# Alternative X-ray sources

## TABLE 1
Some X-ray lines of use in photoelectron spectroscopy

<table>
<thead>
<tr>
<th>Line</th>
<th>Be K</th>
<th>Y M\textsuperscript{(\beta)}</th>
<th>Zr M\textsuperscript{(\beta)}</th>
<th>Nb M\textsuperscript{(\alpha)}</th>
<th>Mo M\textsuperscript{(\alpha)}</th>
<th>Ru M\textsuperscript{(\beta)}</th>
<th>Rh M\textsuperscript{(\beta)}</th>
<th>C K</th>
<th>Ti L\textsuperscript{(\beta)}</th>
<th>Ti L\textsuperscript{(\alpha)}</th>
<th>O K</th>
<th>Cr La</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy (eV)</td>
<td>108.9</td>
<td>132.3</td>
<td>151.4</td>
<td>171.4</td>
<td>192.3</td>
<td>236.9</td>
<td>260.1</td>
<td>278</td>
<td>395.3</td>
<td>452.2</td>
<td>524.9</td>
<td>572.8</td>
</tr>
<tr>
<td>Width (eV)</td>
<td>5.0</td>
<td>0.47</td>
<td>0.77</td>
<td>1.21</td>
<td>1.53</td>
<td>2.49</td>
<td>4.0</td>
<td>6</td>
<td>3</td>
<td>3</td>
<td>4</td>
<td>3</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Line</th>
<th>Ne Ka</th>
<th>Ni L\textsuperscript{(\alpha)}</th>
<th>Cu L\textsuperscript{(\alpha)}</th>
<th>Zn L\textsuperscript{(\alpha)}</th>
<th>Na Ka</th>
<th>Mg Ka</th>
<th>Al Ka</th>
<th>Zr La</th>
<th>Ti Ka</th>
<th>Cr Ka</th>
<th>Cu Ka</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy (eV)</td>
<td>849</td>
<td>851.5</td>
<td>929.7</td>
<td>1011.7</td>
<td>1041.0</td>
<td>1253.6</td>
<td>1486.6</td>
<td>2042</td>
<td>4510</td>
<td>5417</td>
<td>8048</td>
</tr>
<tr>
<td>Width (eV)</td>
<td>0.3</td>
<td>2.5</td>
<td>3.8</td>
<td>2.0</td>
<td>0.42</td>
<td>0.7</td>
<td>0.85</td>
<td>1.7</td>
<td>2.0</td>
<td>2.1</td>
<td>2.6</td>
</tr>
</tbody>
</table>
Monochromator

\[ N\lambda = 2d \sin \Theta \]
For Al K\(\alpha\) \(\lambda = 8.3\) Å

1010 planes in quartz:
d = 4.25 Å
Monochromated X-rays

![Graphs showing Si 2p subshells for Al Monochromator and Mg Anode with Clean Si.](image-url)
Flooded or focused X-ray beam
CHA with standard input lens
To resolve a 1000 eV electron to +/- 0.5 eV would require an analyser with $w=1\text{mm}$ and $R=1.2\text{ meters}$.
Pass energies and transfer lens

It is convenient to retard the energy of the incoming electrons to a lower (and constant) energy before passing through the detector.
Pass Energy and Resolution
Pass Energy

![Graphs showing binding energy and intensity for different pass energies (10 eV, 20 eV, 40 eV, 80 eV).]
Pure Au after Ar⁺ sputtering

Pass energy = 80 eV

Pass energy = 20 eV
Ion Etching

- Photons in
- Electrons out
- Rastered ion beam
- Analysis area
Sputter depth profile craters in Multilayer Films using Ar+ ions with and without sample rotation
## Chemical shift

<table>
<thead>
<tr>
<th>Functional Group</th>
<th>Binding Energy (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>hydrocarbon C-H, C-C</td>
<td>285.0</td>
</tr>
<tr>
<td>amine C-N</td>
<td>286.0</td>
</tr>
<tr>
<td>alcohol, ether C-O-H, C-O-C</td>
<td>286.5</td>
</tr>
<tr>
<td>Cl bound to C C-Cl</td>
<td>286.5</td>
</tr>
<tr>
<td>F bound to C C-F</td>
<td>287.8</td>
</tr>
<tr>
<td>carbonyl C=O</td>
<td>288.0</td>
</tr>
</tbody>
</table>

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### XPS of Poly(ethylene terephthalate)

- **Atom %**
  - C: 71
  - O: 29

- **% of C 1s**
  - CH: 63%
  - C-O: 20%
  - O=C-O: 17%

---

Quantitative elemental information

Chemical state information.
Chemical shift

Ti 2p
Mg Kα

Ti foil etched

Ti + TiOx

463.5

2p1 459.5

457.6

2p3 453.7

468 466 464 462 460 458 456 454 452 450

Binding Energy /eV
Aluminium foil  XPS
Aluminium foil XPS
Inelastic Background

![Diagram of inelastic background with energy losses and mean photoelectron binding energy.]

- Energy losses
- Mean photoelectron binding energy
- Background

![XPS spectrum diagram with intensity (photoelectrons per second) vs BE (eV).]

- XPS spectrum
- Intensity (photoelectrons per second)
- BE (eV)
- 2s, 2p, 3s, 3d
Plasmon loss

Figure 12. Energy loss (plasmon) lines associated with the 2s line of aluminum (a = 15.3eV; note surface plasmon at b).
Oxidation of Titanium

Oxidation of Ti:
Titanium has a big shift. Ti-metal (Ti⁰) to TiO₂ (Ti⁴⁺).

Ti 2p-scan of Ti-metal and TiO₂
Binding energies:
metal: Ti 2p₃/₂ = 451.4 \text{ eV} \quad \text{Ti} 2p₁/₂ = 457.57 \text{ eV}
oxide: Ti 2p₃/₂ = 458.8 \text{ eV} \quad \text{Ti} 2p₁/₂ = 464.34 \text{ eV}

1. Not the same shift for the two spin doublets.
2. Metals have more asymmetric peaks than insulators/oxides.
3. Weak peak at 450.7 eV - “ghost” -
   (X-ray with higher energy = Kβ)
Density of state DOS

Degree of asymmetry proportional to DOS at $E_F$
XPS spectra of Ti2p peaks for Ti and TiO$_2$

Ti metal peak (454 eV)

TiO$_2$ (110) with some reduced states

TiO$_2$ peak (459 eV)
Depth information from XPS

For off-normal *take-off angle* $\alpha$:

$$ P = \exp \left( \frac{-d}{\lambda \cdot \sin \alpha} \right) \quad P = \frac{I}{I_0} $$

$$ d = -\ln(P) \cdot \lambda \cdot \sin \alpha $$

$$ = 3 \cdot \lambda \cdot \sin \alpha $$

$d$ decreases by a factor of 4 on going from $\alpha = 90^\circ$ (normal) to $15^\circ$ (grazing)
Depth information from Ag-anode

- **Greater analysis depth**
  The Al 2p spectra shown (Figure 3) were acquired with
  - (a) Al and
  - (b) Ag sources. Spectrum (b) is a clear illustration of the greater analysis depth of the Ag source as the element : oxide ratio is greater than that acquired with the Al source. An oxide thickness of 1.8nm indicated here was confirmed by independent angle resolved measurements.
Binding Energy Referencing

$$BE = hv - KE - \Phi_{\text{spec}} - E_{\text{ch}}$$

Where: $BE$ = Electron Binding Energy  
$KE$ = Electron Kinetic Energy  
$\Phi_{\text{spec}}$ = Spectrometer Work Function  
$E_{\text{ch}}$ = Surface Charge Energy

$E_{\text{ch}}$ can be determined by calibrating the instrument to a spectral feature.

C1s at 285.0 eV  
Au4f\textsubscript{7/2} at 84.0 eV
Binding Energy Referencing

When analysing insulating samples more care is required because of sample charging and the uncertainty in the location of the Fermi Level within the band gap.

The term Binding energy is often used without specifying the reference level.

Take care when evaluating spectra!
Charge neutralization for insulating samples

- Low-energy electrons from a cold cathode flood gun alleviates positive charging
- Low-energy source of positive ions alleviates the surrounding negative charge
Auger parameter

The Auger parameter is defined as:

\[
\text{A.P.} = \text{K.E.(Auger)} - \text{K.E.}(\text{photoelectron}) + \text{Photon Excitation Energy}
= \text{K.E.}(\text{Auger}) + \text{B.E.}(\text{photoemission})
\]

Where K.E.(Auger) and B.E.(photoemission) are normally measured for the most intense photoemission and sharpest Auger lines available.

Its advantage as a probe of charges in screening energy is that (unlike photoemission or Auger line measurements taken separately) it is independent of energy referencing problems whilst still being measurable to a high precision. Hence small shifts in its value can be measured and interpreted.
<table>
<thead>
<tr>
<th>Compound</th>
<th>S</th>
<th>2p3/2</th>
<th>L3M45M45</th>
<th>A.P.</th>
<th>Ref.</th>
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<td>Ge</td>
<td>9</td>
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<td>76-11</td>
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<td>Ge</td>
<td>9</td>
<td>29.15</td>
<td>1145.2</td>
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<td>GeAs2</td>
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<td>GeTe</td>
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<td>GeSe3</td>
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<td>GeSe</td>
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<td>31.7</td>
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<td>GeO2</td>
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<td>Ph3GeI</td>
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<td>Ph4Ge</td>
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<td></td>
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<td>74-5</td>
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<td>1135.7</td>
<td>1169.0</td>
<td></td>
<td>82-24</td>
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<td>GeBr4(g)</td>
<td>38.95v</td>
<td>1130.32v</td>
<td>1169.27</td>
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<td>73-19</td>
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<tr>
<td>GeCl4(g)</td>
<td>39.6v</td>
<td>1129.01v</td>
<td>1168.61</td>
<td></td>
<td>73-19</td>
</tr>
<tr>
<td>GeMe4(g)</td>
<td>35.63v</td>
<td>1132.64v</td>
<td>1168.27</td>
<td></td>
<td>73-19</td>
</tr>
<tr>
<td>GeH4(g)</td>
<td>36.9v</td>
<td>1129.5v</td>
<td>1166.4</td>
<td></td>
<td>73-19</td>
</tr>
<tr>
<td>GeF4(g)</td>
<td>41.55v</td>
<td>1124.28v</td>
<td>1165.83</td>
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<td>73-19</td>
</tr>
</tbody>
</table>

- [http://www.uksaf.org/data/table.html](http://www.uksaf.org/data/table.html)
Specimen preparation and handling
Take Care – Do not touch surface
You might think that you are safe wearing gloves.

<table>
<thead>
<tr>
<th>Sample</th>
<th>C</th>
<th>N</th>
<th>O</th>
<th>Si</th>
<th>S</th>
<th>Cl</th>
<th>Ca</th>
<th>Zn</th>
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<tbody>
<tr>
<td>Glove 1</td>
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<td>1.5</td>
<td>8.6</td>
<td>1.5</td>
<td>0.5</td>
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<td>0.2</td>
<td>0.5</td>
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<td>Glove 2</td>
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<td>10</td>
<td>-</td>
<td>1.1</td>
<td>6.0</td>
<td>-</td>
<td>1.2</td>
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<tr>
<td>Glove 3</td>
<td>54</td>
<td>0.3</td>
<td>26</td>
<td>19</td>
<td>-</td>
<td>0.1</td>
<td>0.5</td>
<td>0.1</td>
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<tr>
<td>Glove 4</td>
<td>76</td>
<td>2.3</td>
<td>13.2</td>
<td>-</td>
<td>1.4</td>
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<td>1.7</td>
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<td>Glove 5</td>
<td>71</td>
<td>0.1</td>
<td>17</td>
<td>12</td>
<td>-</td>
<td>0.1</td>
<td>-</td>
<td>0.1</td>
</tr>
</tbody>
</table>
XPS depth profile of SiO$_2$ on Si

- Silicon Oxide
- Silicon (substrate)

Graph showing:
- O1s concentration
- Si2p concentration

Atomic Concentration (%) vs. Sputter Time (min)
Survey scan: Most elements have major photoelectron peaks below 1100 eV, so a range from 1100 - 0 eV is usually sufficient.
Detail Scans

For purposes of chemical state identification, for quantitative analysis of minor components and peak deconvolution or other mathematical manipulation of data.

• Scan should be wide enough to encompass the background on both sides of the region of interest - yet with small enough step size - within a reasonable time.
• Radiation-sensitive peaks should be run first.
• Sufficient Signal / noise.
• Pass Energy? ΔE same for all scans!
<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic Weight</th>
<th>Species</th>
<th>Atomic Weight</th>
<th>Species</th>
<th>Atomic Weight</th>
<th>Species</th>
<th>Atomic Weight</th>
<th>Species</th>
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<tbody>
<tr>
<td>Sr</td>
<td>278.7</td>
<td>Mg</td>
<td>301.6</td>
<td>Sr</td>
<td>382.0</td>
<td>U</td>
<td>412.7</td>
<td>Lu</td>
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<td>Os</td>
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<td>Pr</td>
<td>305.0</td>
<td>Os</td>
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<td>Ti</td>
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<td>Ta</td>
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<td>Ru</td>
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<td>Ru</td>
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<td>Mo</td>
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<td>Mo</td>
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<td>Rho</td>
<td>307.2</td>
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### Electron binding energies

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Analysis of Multi-Layer Paint Cross Section

Figure 1. Optical micrograph of multi-layer paint cross-section from the sample positioning station (1072 x 820 μm).
Probing Glass Coatings

Windows are coated with complex multi-layer thin films to meet demands:
1) Energy conservation
2) Appearance
3) Durability
Databases