National Synchrotron Light Source II





# X-ray Photoelectron Spectroscopy (XPS)

### Ira Waluyo

IOS (23-ID-2) Lead Beamline Scientist NSLS-II, Brookhaven National Laboratory iwaluyo@bnl.gov



## X-ray photoelectron spectroscopy (XPS) was featured in all of these research



ACS Publications

XPS is a powerful and versatile technique for studying the chemical and electronic state of surfaces as well as understanding phenomena and processes occurring on surfaces

40,057 Materials Science Multidisciplinary	16,498 Physics Condensed Matter	10,162 Engineering Chemical	5,557 Metallurgy Metallurgical Engineering	4,368 Environmental Sciences		4,283 Er Elec Electro		4,158 Engir Enviror	4,158 Engineering Environmental	
34,062 Chemistry Physical	15,705 Chemistry Multidisciplinary	8,376 Electrochemistry	3,408 Chemistry Analytica		2,548 Phys		2,522 Materials		2,304 Materials	
	13,571 Nanoscience Nanotechnology	5,956 Polymer Science	3,302 Chemistry Applied		Atom Molecu Chemica	Atomic Science Molecular Ceramics chemical		Science Biomaterials		
27,240 Physics Applied	11,741 Materials Science Coatings Films	5,923 Energy Fuels	2,785 Chemistry Inorganic Nuclear		1,755 Optics 1,706 Instru	55 tics 06 Instruments		1,341 Physics Multidisc	1,196 Gre Sus Scient Techno	

Webofscience.com search result from 2003-2023

Nati

#### What will you learn today?











## Part 1

## **Basic Principles of XPS and**

## Instrumentation

#### A little history: From the photoelectric effect...

1887 – Heinrich Hertz observed sparks when UV light hits metal electrodes.

**1899** – J. J. Thomson discovered that UV light causes particles to be emitted. The particles were the same as the ones found in cathode rays (i.e. electrons).

**1902** – Philipp Lenard found that the energy of the emitted electrons depend on the color (i.e. frequency) of the light, not intensity.



**1905** – Albert Einstein explained this phenomenon. Light is composed of discrete packets of energy called "quanta" (later "photons") – Nobel Prize in Physics (1921).

#### ...to Modern XPS





- Kai Siegbahn developed an instrument for analyzing emitted photoelectrons and used it for chemical analysis
- The technique was called Electron Spectroscopy for Chemical Analysis (ESCA)
- Awarded Nobel Prize in Physics in 1981



- X-ray photon ionizes an atom and causes the emission of a photoelectron.
- Kinetic energy of the photoelectrons depends on:
  - Energy of the incident X-ray photons
  - Binding energy of the electron
  - Work function of the sample (in practice we use the work function of the spectrometer)

### Why is XPS Surface Sensitive?



- Inelastic mean free path (IMFP): how far can an electron travel before losing energy
- IMFP depends on electron energy and density of the material
- The "Universal Curve" shows the typical IMFP for electrons in a solid
- Typical IMFP ( $\lambda$ ) is 5-20 Å for soft X-ray excitation

#### **Sampling Depth**



$$I = I_0 e^{-d/\lambda \cos \theta}$$

- Assuming normal takeoff angle ( $\theta = 0, \cos \theta = 1$ )
- For  $d = 3\lambda$ ,  $I = \sim 0.05 I_0$
- This means 95% of electrons come from depth of  $3\lambda$
- Typical sampling depth = 1-5 nm



- Photons: 100's nm for soft X-ray (<2000 eV)
- But you can only get electrons from the first top 1-5 nm
- This makes XPS highly surface sensitive



#### **Basic Instrumentation**



Three basic requirements:

- 1. We need an X-ray source
- 2. We need to be able to detect the electrons
- 3. We need the appropriate experimental environment

#### **Basic Instrumentation**



Three basic requirements:

1. We need an X-ray source

2. We need to be able to detect the electrons

3. We need the appropriate experimental environment

### X-ray Source – X-ray anode ("lab source")

LINE WIDTH (EV)

0.7



thermofisher.com

ANODE

Ma

specs-group.com

Pros:

- Cheaper than building a synchrotron
- Commonly available in research labs and shared university facilities

#### Cons:

- Fixed energy various consequences
- Lower flux
- Poorer resolution

	6						
	Al	Κα	1486.8	0.85			
	Zr	Lα	2042.4	1.6			
	Ag	Lα	2984.3	2.6			

RADIATION

Κα

ENERGY (EV)

1253.6

thermofisher.com

#### X-ray Source - Synchrotron



#### Pros:

- Tunable energy, usually 100-2000 eV for soft XPS, 2000-6000 eV for HAXPES
- Higher flux
- Higher resolution
- Beamtime is free

#### Cons:

- Not too many facilities available
- Beamtime can be competitive (you need to do good science!)





#### **Basic Instrumentation**



Three basic requirements:

1. We need an X-ray source

2. We need to be able to detect the electrons

3. We need the appropriate experimental environment

#### **Detecting Electrons**



The most common electron spectrometer: Hemispherical electron analyzer



www.specs-group.com

#### **Hemispherical Electron Analyzer**



- Electrostatic lenses focus electron to the detector
- Electron kinetic energy is adjusted to the same pass energy  $(E_p)$
- To improve resolution, you need to:
  - Use lower pass energy
  - Decrease slit width

National Synchrotron Light Source II

$$\Delta E = E_p \left( \frac{W}{2R} + \frac{\alpha^2}{2} \right)$$

Greczynski and Hultman, J. Appl. Phys. 132, 011101 (2022) Stevie and Donley, J. Vac. Sci. Technol. A 38, 063204 (2020)

#### **Basic Instrumentation**



Three basic requirements:

1. We need an X-ray source

2. We need to be able to detect the electrons

3. We need the appropriate experimental environment

#### **Sample Environment**

- Electrons can't travel very far in a gas environment (short mean free path)
- A 200 eV electron can only travel 1 mm in 1 Torr gas (atmospheric pressure is 760 Torr)
- Need to minimize collision between electrons and gas molecules
- XPS needs ultra-high vacuum chamber (10<sup>-8</sup>-10<sup>-10</sup> Torr)
- Sample surface also needs to be kept clean from surface contamination



vacgen.com

#### **Ambient Pressure XPS**

XPS needs vacuum, but vacuum is not real life!



UHV surface science (<10<sup>-9</sup> Torr)



Ambient pressure XPS



Industry (> 760 Torr)

The first AP-XPS instrument was built by Kai Siegbahn's group in the 1970's (H. Siegbahn and K. Siegbahn, J. Electron. Spectrosc. Relat. Phenom. 2, 319 (1973))

Applications in:

- Heterogeneous catalysis
- Electrochemistry
- Energy storage
- Corrosion science

- Quantum information science
- Environmental science
- Atmospheric chemistry
- and many more...



National Synchrotron Light Source II

PUMP

#### **AP-XPS Instrumentation**



AP-XPS endstation at IOS beamline, NSLS-II

- By differentially pumping the electron analyzer, the pressure in the chamber can be raised while maintaining UHV in the analyzer
- Electrostatic focusing lenses needed to focus electrons to the analyzer

### **AP-XPS Experimental Setup**









- A small aperture is needed to restrict the flow of gases into the analyzer
- Aperture diameter ~100 um to 500 um (smaller aperture  $\rightarrow$  higher pressure)
- Sample needs to be close to aperture to minimize scattering by gases
- Small beam size is important!
- Chamber and beamline are separated by 100 nm thick  $Si_3N_4$  window National Synchrotron Light Source II

## Part 2

## **Understanding XPS Spectra**

#### What do you get from the analyzer?



If you change the energy of the X-ray, the electron kinetic energy also changes

#### **Convert to binding energy**



Electron binding energy is fixed at any photon energy

**Convert to binding energy** 

 $E_b = hv - E_k - \phi$ C<u>u 2p</u> Spectra from IOS (23-ID-2) beamline, NSLS-II Intensity (arb. units) Cu 3p 850 840 Cu 3s VB hv = 1110 eVhv = 710 eV1000 800 600 200 400 0

Binding Energy (eV)

Select your scan regions, scan at smaller step size, and take multiple scans to improve signal-to-noise ratio





Binding Energy (eV)



 $E_{k} = hv - E_{b} - \phi$ 

- XPS leaves a core hole
- An electron from a higher energy orbital fills the hole
- The released energy results in the emission of another electron
- Auger kinetic energy is not photon energy dependent



#### **Identifying Auger Peaks**



#### **Identifying Auger Peaks**





Doublets (two peaks) are always observed for p, d, f orbitals



National Synchrotron Light Source II

Spectra from IOS (23-ID-2) beamline, NSLS-II

 $l = \text{orbital angular momentum quantum number}}$  (0 = s, 1 = p, 2 = d, 3 = f)  $p_{1/2}$   $j = \text{total angular momentum quantum number}}$  j = |l + s| n = principal quantum number (1, 2, 3, ...)s: l = 0, no split

S = spin quantum number (-1/2 or +1/2)





- Spin orbit splitting helps us identify peaks
- Energy splitting is always the same for the same core level of the same element
- Peak area ratio is determined by degeneracy 2j+1
  - $> p_{3/2}:p_{1/2} = 2:1$

► 
$$d_{5/2}: d_{3/2} = 3:2$$

$$> f_{7/2}:f_{5/2} = 4:3$$

#### **Photoionization Cross-section**

- The probability of a photon ionizing the atom and causing the emission of an electron
- It varies for different elements
- It varies for different core levels of the same element
- It varies with photon energy for the same core level of the same element
- Need a synchrotron to tune energy to optimize cross-section



https://vuo.elettra.eu/services/elements/WebElements.html National Synchrotron Light Source II

#### **Photoionization Cross-section**


# What information can you get from XPS?

### Elemental specificity

What elements do you have in the sample?

Chemical sensitivity

What is the chemical environment of each element in the sample?

Depth profiling

Where are the elements in the sample?

Quantitative analysis

How much is there of each element in the sample?

# What information can you get from XPS?

## Elemental specificity

What elements do you have in the sample?

Chemical sensitivity

What is the chemical environment of each element in the sample?

Depth profiling

Where are the elements in the sample?

Quantitative analysis

How much is there of each element in the sample?

# **Elemental Specificity**



American Chemical Society

www.acs.org/outreach

#### Each element has a unique set of electron binding energies

National Synchrotron Light Source II

# **Electron Binding Energy Table**

Element	K 1s	L <sub>1</sub> 2s	L <sub>2</sub> 2p <sub>1/2</sub>	L <sub>3</sub> 2p <sub>3/2</sub>	M <sub>1</sub> 3s	M <sub>2</sub> 3p <sub>1/2</sub>	M <sub>3</sub> 3p <sub>3/2</sub>	M4 3d <sub>3/2</sub>	M5 3d5/2	N <sub>1</sub> 4s	N <sub>2</sub> 4p <sub>1/2</sub>	N <sub>3</sub> 4p <sub>3/2</sub>
1 H	13.6											
2 He	24.6*											
3 Li	54.7*											
4 Be	111.5*											
5 B	188*											
6 C	284.2*											
7 N	409.9*	37.3*										
8 O	543.1*	41.6*										
9 F	696.7*											
10 Ne	870.2*	48.5*	21.7*	21.6*								
11 Na	1070.8†	63.5†	30.65	30.81								
12 Mg	1303.0†	88.7	49.78	49.50								
13 Al	1559.6	117.8	72.95	72.55								
14 Si	1839	149.7*b	99.82	99.42						1.8NL/P08-490	8m 2	
15 P	2145.5	189*	136*	135*					Center for	X-Ray Optics		
16 S	2472	230.9	163.6*	162.5*					Advanced	Light Source		
17 Cl	2822.4	270*	202*	200*					X-RAY		Δ	
18 Ar	3205.9*	326.3*	250.6†	248.4*	29.3*	15.9*	15.7*					
19 K	3608.4*	378.6*	297.3*	294.6*	34.8*	18.3*	18.3*		<b>BOO</b>	KLET		
20 Ca	4038.5*	438.4†	349.7†	346.2†	44.3 †	25.4†	25.4†		Albert Thompson	Ingolf Lindau		
21 Sc	4492	498.0*	403.6*	398.7*	51.1*	28.3*	28.3*		David Attwood Eric Gußikson	Fiero Pianetta Arthur Robins	on	
22 Ti	4966	560.9†	460.2†	453.8†	58.7†	32.6†	32.6†		Malcoim Howells Kwang Je Kim	James Scoffeld James Underv	VOOd	

**Table 1-1.** Electron binding energies, in electron volts, for the elements in their natural forms,

https://xdb.lbl.gov/Section1/Table 1-1.pdf From X-ray Data Booklet: <u>https://xdb.lbl.gov</u> National Synchrotron Light Source II

	1.8Nk/P08-490 8m 3
Center for 3	K-Ray Optics
31	nd
Advanced i	ight source
V_DAV	DATA .
<b>N-KAT</b>	UAIA
PAA	
Albert Thompson	Ingoil Lindau
Alber! Thompson David Attwood	Ingoil Lindau Fiero Pianetta
Albert Thompson David Attwood Eric Gußikson	Ingolf Lindau Fiero Pianetta Arthur Robinson
Alber! Thompson David Attwood Eric Gußikson Malcolm Howells	Ingolf Lindau Fiero Planetta Arthur Robinson James Scofield
Albert Thompson David Attwood Eric Gullikson Malcoim Howells Kwarig-Je Kim	Ingolf Lindau Piero Planetta Arthur Robinson James Scofield James Underwood
Albert Thompson David Attwood Eric Gußikson Malcolin Howells Kwang-Je Kim Janos Kirz	Ingolf Lindau Piero Pianette Arthur Robinson James Scofield James Underwood Douglas Vaughan
Alber! Thompson David Attwood Eric Gullikson Malcoim Howells Kwarig-Je Kim Janos Kirz Jeffrey Kortright	Ingoil Lindau Fiero Pianetta Arthur Robinson James Scofield James Underwood Douglas Vaughan Owyn Williams
Albert Thompson David Attwood Eric Gullikson Malcoim Howells Kwang-Je Kim Janos Kirz Jeffrey Kortright Hermar	Ingolf Lindau Fiero Pianetta Arthur Robinson James Scofield James Underwood Douglas Vaughan Gwyn Williams I Winick
Albert Thompson David Attwood Eric Gutlikson Malcolm Howells Kwang-Je Kim Janga Kirz Jeffrey Kortright Hermar Janga	Ingolf Lindau Piero Pianetta Arthur Robinson James Scoffeld James Underwood Douglas Vaughan Gwyn Withiams i Winick y 2001
Albert Thompson David Attwood Eric Gullikson Malcolm Howells Kwang-Je Kim Jang Kirz Jeffrey Kortright Hermar Janeau Janeau	Ingolf Lindau Piero Pianetta Arthur Robinson James Scofield James Underwood Douglas Vaughan Gwyn Wifiliams i Winick y 2001 National Japonavy
Albert Thompson David Attwood Eric Gulfikson Malcoim Howells Kwarg.Jo Kim Janos Kirz Jeffrey Kortright Hermar Janeau Janeau Janeau	Ingolf Lindau Piero Pianetta Arthur Robinson James Scoffeld James Underwood Douglas Vaughan Gwyn Withiams I Winick y 2001 Nitional Lancasary of Castrian

#### National Synchrotron Light Source II



Nickel-Chromium alloy – corrosion resistant material





Spectra from IOS (23-ID-2) beamline, NSLS-II

# **Overlaps with Auger Peaks**



Binding Energy (eV)

Overlapping Auger peaks can be shifted away by changing photon energy National Synchrotron Light Source II

# Things are not always that simple...

- Some XPS peaks of different elements may still overlap partially or completely
- You need to look at other peaks to identify the element
- Peak fitting is important for separating peaks from different elements





NSLS-II

# What information can you get from XPS?

Elemental specificity

What elements do you have in the sample?

Chemical sensitivity

What is the chemical environment of each element in the sample?

Depth profiling

Where are the elements in the sample?

Quantitative analysis

How much is there of each element in the sample?

# **Sensitivity to Chemical Environment**



- Binding energy of an electron is affected by the chemical environment of the atom
- Energy shift can be up to 10 eV
- This makes XPS a powerful tool for chemical analysis
- You can tell the chemical bonding of the probed atom
- Bonding to a more electronegative atom → shift to higher binding energy

K. Siegbahn, J. Electr. Spectrosc. Relat. Phenom., 5, 3 (1974)

# **Core Level Shift due to Oxidation State**

• In general, higher oxidation state causes a shift to higher binding energy







But... alkali metals behave the opposite way



# Sometimes it's not that clear...



Spectra from IOS (23-ID-2) beamline, NSLS-II National Synchrotron Light Source II

# **Surface Core Level Shift**

Surface atoms of a metal can have distinct binding energy from the bulk atoms



Gustafson et al. Phys. Rev. Let. 91, 056102 (2003) National Synchrotron Light Source II



Simonovis et al. J. Phys. Chem. C 126, 7870 (2022)

# **Distinguishing Different Adsorption Sites**

National Synchrotron Light Source II



Toyoshima et al. Phys. Chem. Chem. Phys. 16, 23564 (2014)

# What information can you get from XPS?

- Elemental specificity
  - What elements do you have in the sample?
- Chemical sensitivity

What is the chemical environment of each element in the sample?

Depth profiling

Where are the elements in the sample?

Quantitative analysis

How much is there of each element in the sample?

# **Depth Profiling**

You can tune the sampling depth of XPS (non-destructively) in two ways:



1. Change the photon energy  $\rightarrow$  lower E<sub>k</sub> means lower IMFP and more surface sensitive

You can also depth-profile by sputtering (i.e. remove materials layer by layer) but that is destructive



 Change emission angle → more grazing emission is more surface sensitive



# **Depth Profiling Example**



# What information can you get from XPS?

- Elemental specificity
  - What elements do you have in the sample?
- Chemical sensitivity

What is the chemical environment of each element in the sample?

Depth profiling

Where are the elements in the sample?

Quantitative analysis

How much is there of each element in the sample?

# **Quantitative Analysis**

- $I_x = J N_x \lambda \sigma K$
- $I_x$  = peak intensity of element x
- J = photon flux
- $N_x$  = concentration of element x in the sample
- $\lambda$  = inelastic mean free path
- $\sigma$  = photoionization cross-section
- *K* = instrumentation factors



National Synchrotron Light Source II

# Other things to consider...

- Shake up/shake off
- Multiplet splitting
- Plasmon loss
- Charging

# Shake up and Shake off



shake up

Extra "satellite" peaks appear on the lower kinetic energy (higher binding energy side)

shake off





Khalakhan et al. J. Elect. Spectrosc. Relat. Phenom. 246, 147027 (2021) <sup>56</sup>

# **Multiplet Splitting**

- Observed for compounds that have unpaired electrons in the valence band
- Unpaired core electron interacts with unpaired valence electron, creating multiple possible final states





#### 57

# **Plasmon Loss**

- Usually observed for metallic surfaces
- Outgoing photoelectron excites collective oscillations of electrons in the conduction band
- Photoelectron suffers energy loss and extra peaks appear at low KE/high BE at discrete energy values
- Can be strong for AI, which can interfere with other peaks in the region



Spectra from IOS (23-ID-2) beamline, NSLS-II

# **Charging and Charge Compensation**

- Photoionization process causes positive charge on the surface
- For grounded electrically conductive samples, the charge is automatically neutralized
- For semi-conductive and insulating samples, the positive charge accumulates, photoelectron KE decreases
- Effect: shift to higher BE, peak broadening, double peaks, sometimes no peaks at all
- Charge compensation: add electrons back to the sample (use an electron flood gun, or with the presence of ambient gases)





J. W. Niemantsverdriet, Spectroscopy in Catalysis: An Introduction, 3<sup>rd</sup>. ed. Wiley (2007)



Spectra from IOS (23-ID-2) beamline, NSLS-II

National Synchrotron Light Source II

# Part 3

# **Recent Research Examples**

National Synchrotron Light Source II



# The Effect of Aging in Air









National Synchrotron Light Source in

Clark et al. Nanoscale, 9, 6056-6067 (2017)

# **Passivating Effect of Cd**



# **Single-Atom Catalysis**



Metal	Price per oz
Pt	\$978
Pd	\$1,440
Rh	\$10,100
Ni	\$0.70
Cu	\$0.28

www.dailymetalprice.com (updated March 2023)

National Synchrotron Light Source II

- Precious metals (e.g. Pt, Pd, Rh) are highly active but expensive catalysts
- Single-atom catalyst approach: minimize the amount of active but expensive catalyst (e.g. Pt) by supporting it on a cheaper and more abundant material
- Reduces cost and optimize catalytic efficiency



PtCu single atom alloy (SAA)  $\rightarrow$  < 5% Pt forms isolated Pt atoms alloyed in the top layer of Cu metal



64

Hannagan et al. Chem. Rev. 120, 12044 (2020)

# **CO Adsorption on PtCu Single-Atom Alloy**



National Synchrotron Light Source II

Simonovis et al. J. Phys. Chem. C 122, 4488 (2018)

# **Dynamic Surface State of PtCu SAA**

- AP-XPS can track changes in the surface and near surface composition
- Heating in vacuum causes loss of active surface Pt
- Pt moves to subsurface and bulk of Cu
- Heating in CO causes surface segregation of Pt back up to the surface
- Initial surface composition is recovered



Simonovis et al. J. Phys. Chem. C 122, 4488 (2018)

National Synchrotron Light Source II

# Watching Catalyst in Action (CRS Group, BNL Chemistry)

- Methane oxidation to methanol is a difficult process that requires high temperatures (600 K)
- A new catalyst was developed for low temperature methane oxidation promoted by water
- The catalyst: CeO<sub>2</sub>/Cu<sub>2</sub>O/Cu(111)
- The reactants:  $CH_4 + O_2 + H_2O$
- How does this catalyst work?
- AP-XPS is a powerful tool for identifying reaction intermediates

Zuo et al. J. Am. Chem. Soc. 138, 13810 (2016) https://www.bnl.gov/newsroom/news.php?a=26694



# **Converting Methane to Methanol**





Liu et al. Science, 368, 513 (2020)

National Synchrotron Light Source II

# **Converting Methane to Methanol**



Zuo et al. J. Am. Chem. Soc. 138, 13810 (2016) Liu et al. Science, 368, 513 (2020)

#### **DVANCED** Metal exsolution from perovskites - Yildiz group (MIT)

Raw image



Fe









Bulk exsolution (cross section)

Nanocomposite

- Metals can exsolve from bulk perovskite under high temperature requiremended on dition, forming stable nanoparticles on the surface
- Synthesis method for highly stable, oxide-supported metal nanoparticles with applications in energy conversion and storage technologies
- Metal nanoparticles are uniformly distributed and resistant to agglomeration



e Wang et al. Chem. Mater. 33, 5021 (2021), Adv. Funct. Mater. 32, 2108005 (2022), Chem. Mater. 34, 5138 (2022) → ←

AFM

# **Mechanism of Fe exsolution**





- Exsolution of Fe nanoparticles from La<sub>0.6</sub>Sr<sub>0.4</sub>FeO<sub>3</sub> (LSF) was studied in operando with combined AP-XPS and AP-XAS
- AP-XPS reveals a two step process for Fe exsolution: O vacancy formation, followed by metallic Fe precipitation



National Synchrotron Light Source II

Wang et al. Chem. Mater. 33, 5021 (2021), Adv. Funct. Mater. 32, 2108005 (2022), Chem. Mater. 34, 5138 (2022)

# **Tuning Fe exsolution**





- Fe exsolution can be controlled by tuning lattice strain
- Different substrates with different strains result in different concentration, density, and size of exsolved Fe

National Synchrotron Light Source II

Wang et al. Chem. Mater. 33, 5021 (2021), Adv. Funct. Mater. 32, 2108005 (2022), Chem. Mater. 34, 5138 (2022)
# SCIENCE NEWS Cycling of Exsolved LSF



National Synchrotron Light Source II

Wang et al. Chem. Mater. 33, 5021 (2021), Adv. Funct. Mater. 32, 2108005 (2022), Chem. Mater. 34, 5138 (2022)

# Summary

- XPS is a powerful technique for the surface characterization of materials
- It provides:
  - Elemental specificity
  - Chemical sensitivity
  - Depth profiling
  - Quantitative analysis
- Synchrotron-based XPS offers high flux, high resolution, and energy tunability
- Aspects that affect data interpretation: overlaps with Auger peaks, shake-up/off, multiplet splitting, plasmon peaks, charging
- XPS usually needs UHV conditions, but ambient pressure XPS can be performed for in situ/operando experiments

# **Useful Online Resources**

- X-ray data booklet: <u>https://xdb.lbl.gov</u>
- Photoionization cross-section: <a href="https://vuo.elettra.eu/services/elements/WebElements.html">https://vuo.elettra.eu/services/elements/WebElements.html</a>
- NIST XPS Database: <u>https://srdata.nist.gov/xps/main\_search\_menu.aspx</u>
- <u>http://www.xpsfitting.com</u>
- <u>https://xpssimplified.com/periodictable.php</u>
- <u>http://www.casaxps.com/ebooks/XPS%20AES%20Book%20new%20margins%20rev%201.2%20</u> <u>for%20web.pdf</u> – CasaXPS is a popular XPS data analysis software. Their manual has a lot of useful basic information about XPS

### AP-XPS at IOS (23-ID-2) Beamline, NSLS-II



- Pressure = UHV to 10 Torr
- Sample temperature: RT to 900°C
- Sputter gun, flood gun, metal evaporator, RGA mass spec

- Samples: single crystals, thin films, powders
- Gases: H<sub>2</sub>, O<sub>2</sub>, CO, CO<sub>2</sub>, noble gases, C1-C4 hydrocarbons, water vapor, C1-C3 alcohol vapors

https://www.bnl.gov/nsls2/beamlines/ beamline.php?r=23-ID-2

National Synchrotron Light Source II



Photos from IOS (23-ID-2) beamline, NSLS-II

### **AP-XPS at IOS Beamline, NSLS-II**





Photos from IOS (23-ID-2) beamline, NSLS-II

National Synchrotron Light Source II

### **Other AP-XPS Setups**



commercial high pressure cell from SPECS specs-group.com



custom high pressure cell at SSRL Kaya et al, Catal. Today 205, 101 (2013) National Synchrotron Light Source II



POLARIS AP-HAXPES at PETRA III, DESY Degerman et al. Synchrotron Radiat. News, 35, 11-18 (2022)

