Opportunities for Energy Science with Soft X-rays from the ALS

Zahid Hussain
Advanced Light Source
Lawrence Berkeley National Laboratory

Outline

- Shirley Group Synchrotron Radiation Research:
  - Science and Instrumentation
    - Time of flight Angle-Resolved Parabolic Mirror Analyzer for studying Photoelectron Diffraction @ SSRL
    - Tender x-rays beamline (1keV-5keV): Jumbo
- Instrumentation @ALS (inspired by early research by Shirley):
    - Time-of-Flight spin-ARPES & Imaging ARPES
    - Use Inspired Energy Science with ARPES and Ambient Pressure XPS

Understanding complex phenomena require sharper and sharper tools

Early Days (1980-82) of Photoemission

ELSEVIER

Nuclear Instruments and Methods in Physics Research

Volume 195, Issues 1–2, 1 April 1982, Pages 115–131

Performance and application of a double crystal monochromator in the energy region 800 < hν < 4500 eV

(Jumbo)

Z. Hussain[a, b, *], E. Umbach[a, b, **], D.A. Shirley[a, b]

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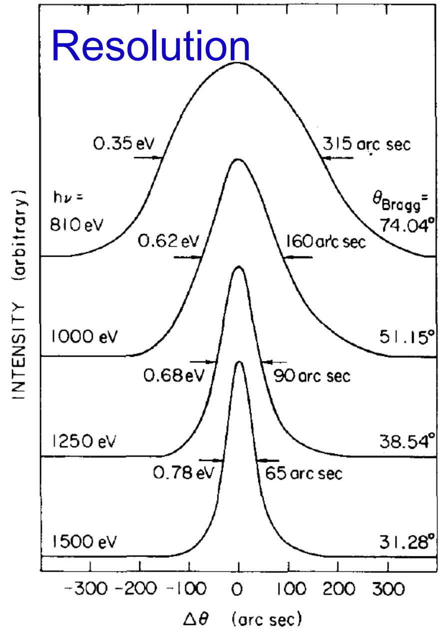
[b] Department of Chemistry, University of California, Berkeley, California 94720, U.S.A.

J. Stöhr[***], J. Feldhaus[†]

Stanford Synchrotron Radiation Laboratory, Stanford University, Stanford, California 94305, U.S.A.

http://dx.doi.org/10.1016/0029-554X(82)90766-2, How to Cite or Link Using DOI

Permissions & Reprints



PHYSICAL REVIEW B VOLUME 25, NUMBER 2 15 JANUARY 1982

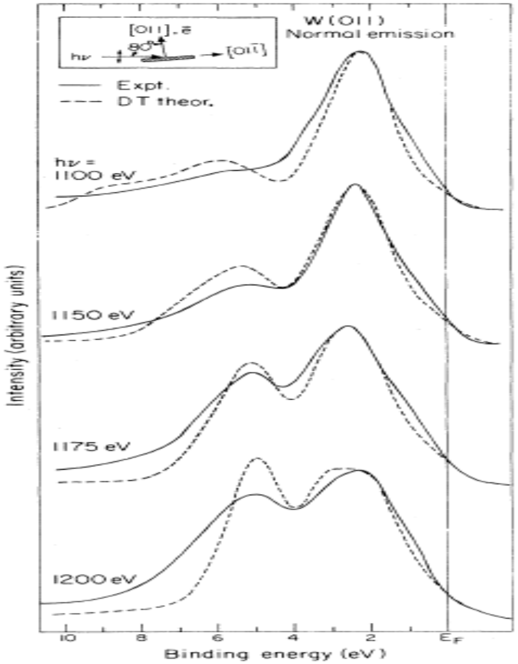
Angle-resolved photoemission study of the valence bands of W(011) in the photon energy range 1100–1250 eV: Observation of strong direct transitions and phonon effects

Z. Hussain, E. Umbach,* J. J. Barton, J. G. Tobin, and D. A. Shirley

Materials and Molecular Research Division, Lawrence Berkeley Laboratory, and Department of Chemistry, University of California, Berkeley, California 94720

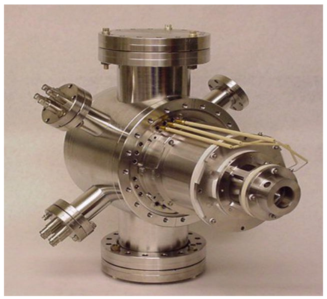
(Received 3 August 1981)

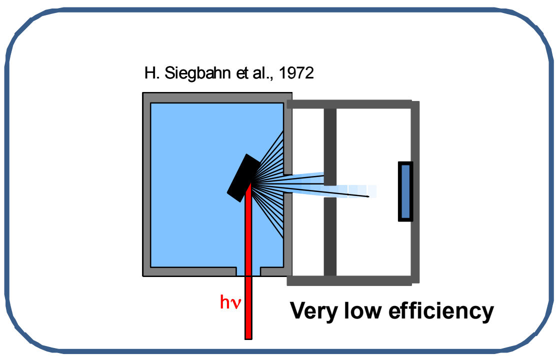
ARPES of Valence Band



ALS Ambient Pressures XPS Systems

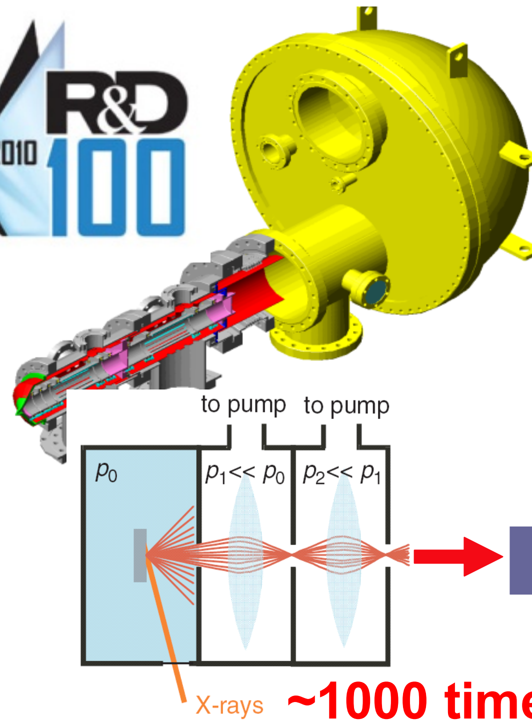
1st Gen:Proto type





2000: Differentially-pumped electrostatic transfer lens allows operation at p ~ 5 torr (equilibrium vapor pressure of water at 0 °C)

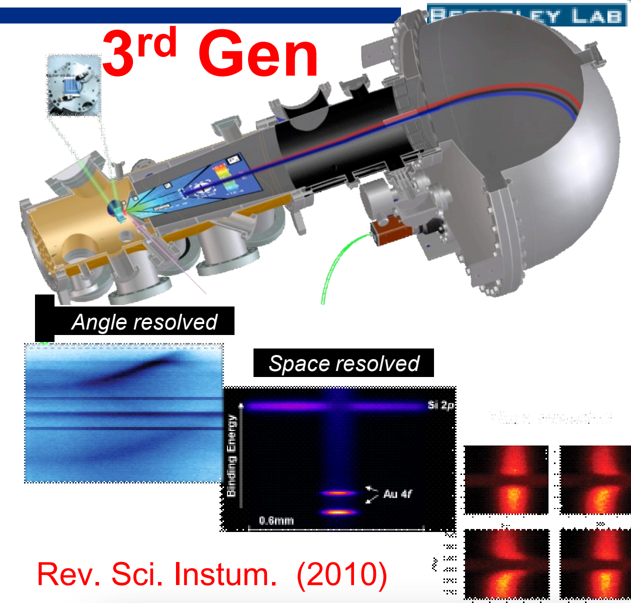
2nd Gen



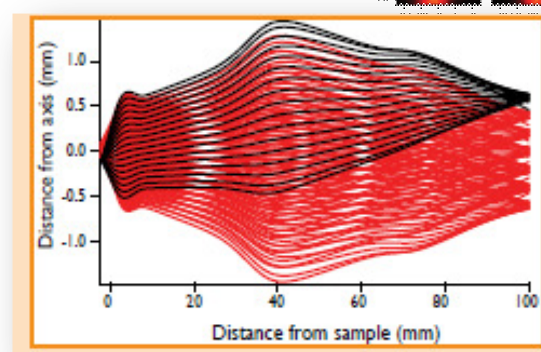
~1000 times

2005: The first commercial system from Specs. Installed at ALS and BESSY

3rd Gen



Rev. Sci. Instum. (2010)

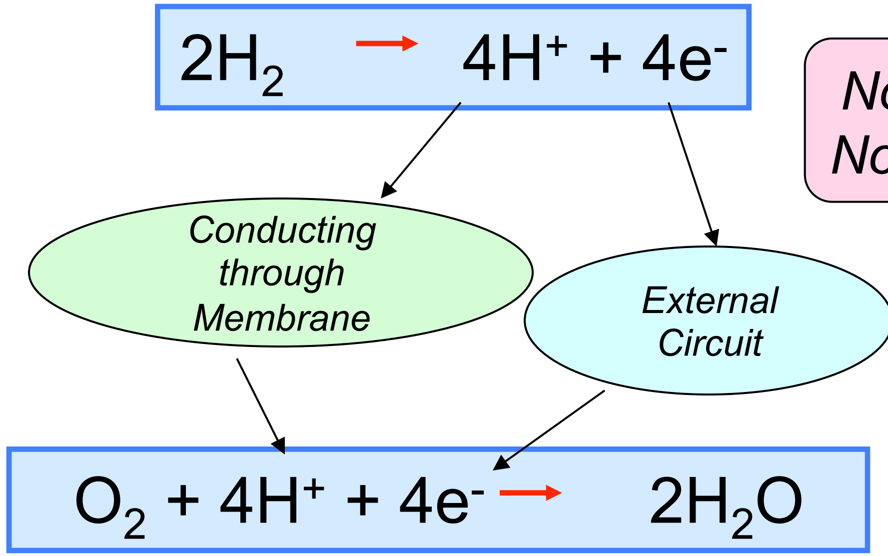


~2010/2012: Scienta/ALS Collaboration

with Imaging @ BL9.3.2 tender x-rays(BL 9.3.1)

Fuel Cell:

Anode : Hydrogen oxidation
Hydrogen gas = Hydrogen Ions + Electrons



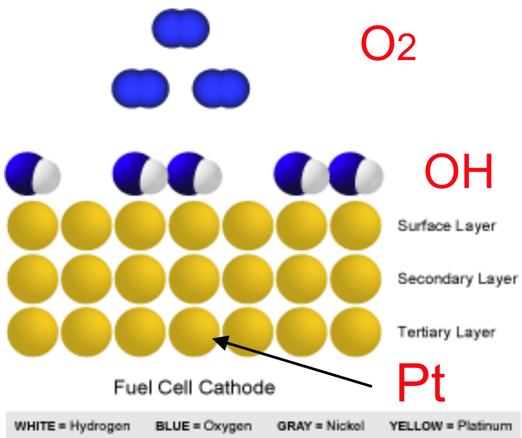
Not too weak!
Not too strong!

Cathode : Oxygen reduction
Oxygen gas + Protons + Electrons = Water

In both cathode and anode, Pt based catalysts are applied to increase the rate of each chemical reactions. Need better material than presently used Pt.

Cathode : the performance of polymer electrolyte membrane fuel cells is limited by the slow rate of O_2 reduction (ORR) at Cathode, ~5 orders of magnitude slower than H_2 oxidation at Anode

80% of All Important Chemical Reactions Take Place on Interfaces



NEWS OF THE WEEK

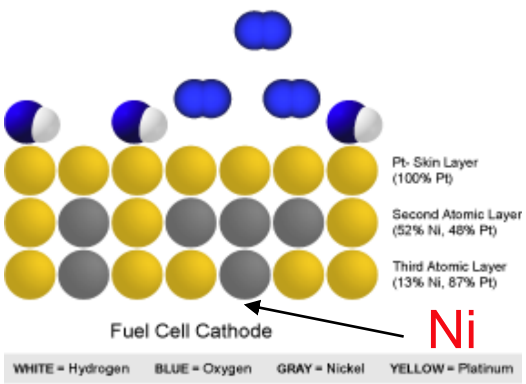
Science AAAS

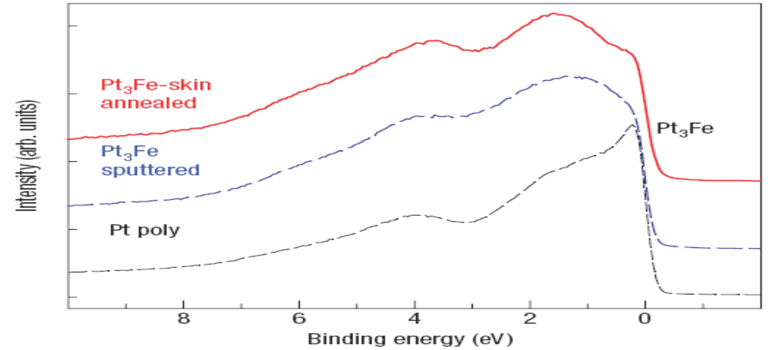
CHEMISTRY

Science 315 Jan 2007

Platinum in Fuel Cells Gets a Helping Hand

The discovery of a unique ***platinum-nickel alloy*** represents a breakthrough in catalyst research: **it is *90 times more active* than state-of-the-art platinum catalysts currently used.**





BL 9.3.2

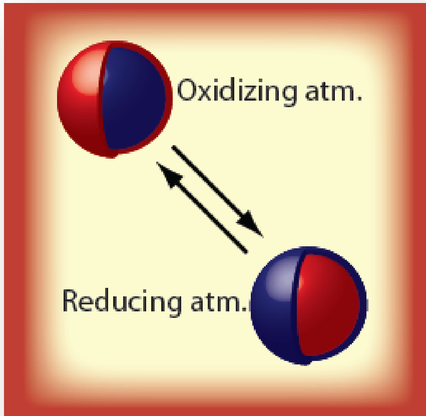
Top: In standard Pt catalysts absorption of oxygen on the surface is hindered by the binding of other molecules, such as OH.

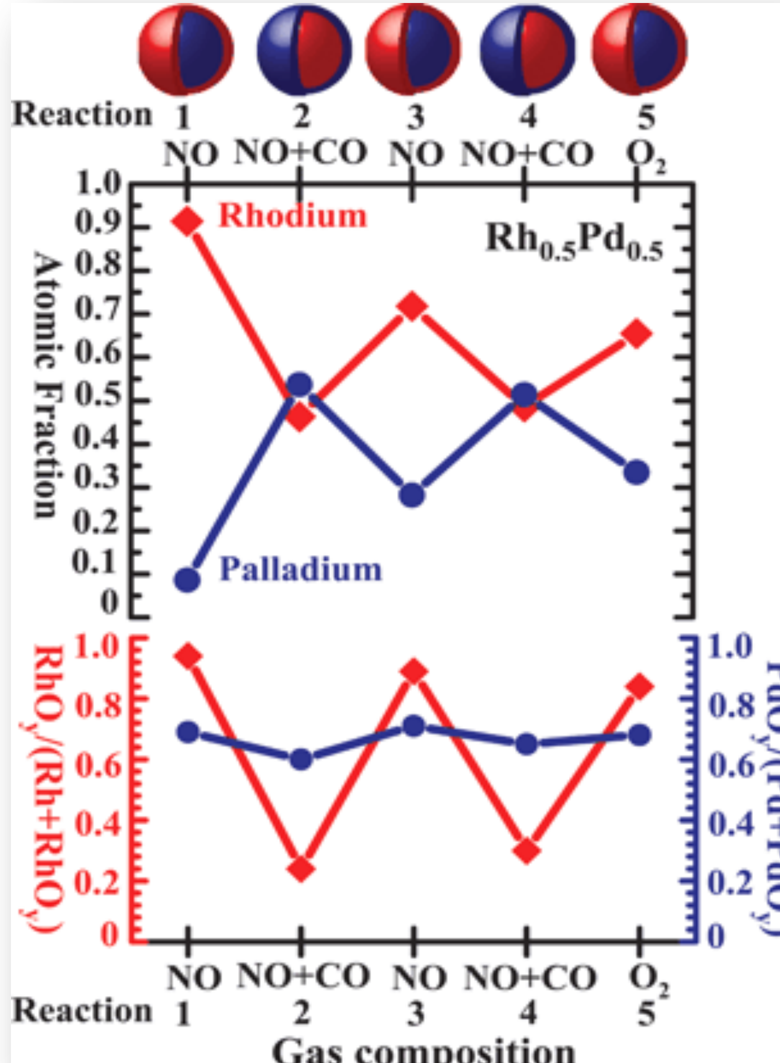
Bottom: In the new material The nickel atoms change the surface properties such that OH cannot bind as well, leaving room for oxygen.

Research team includes: Argonne and Berkeley National Labs (Phil Ross et al), U. South Carolina.

nature materials | VOL 6 | MARCH 2007 |

Reaction-Driven Restructuring of Bimetallic Nanoparticle Catalysts





Restructuring of $Rh_{0.5}Pd_{0.5}$ nanoparticles

Right top: Evolution of Rh ($Rh^0 + Rh^{2y+}$) and Pd ($Pd^0 + Pd^{2y+}$) atomic fractions in the $Rh_{0.5}Pd_{0.5}$ at 300 °C under oxidizing conditions (100 mtorr NO or O_2) and catalytic conditions (100 mtorr NO and 100 mtorr CO).

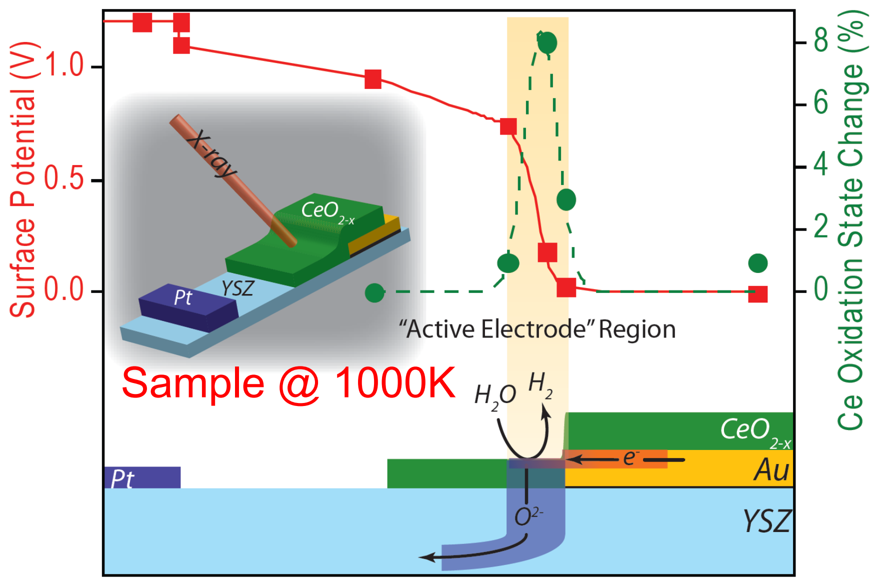
Right bottom: Evolution of the fraction of the oxidized Rh (left y axis) and Pd atoms (right y axis) under the same reaction conditions.

F. Tao and Gabor Somorjai et al., Science 322, 932 (2008).

Somorjai, " *APXPS justifies the existence of the ALS*"

Solid Oxide Fuel Cells

Current ALS Research/Operando



Ambient pressure XPS:

- reveals the behavior of ***mixed ionic-electronic conducting electrode*** materials
- crucial for high-performance SOFCs

- Width of the electrochemically active region has been directly mapped *operando* for the first time
- ***combined surface potential and surface chemistry*** sensitive measurements

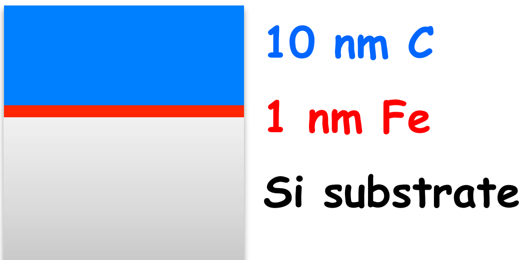
Development of a new class of low-temp high-efficiency SOFC requires:

- **spectro-nanoscopy imaging of interfaces and ultrafast probing**
- **fsec – fundamental oxidation chemistry at electrodes**
- **nsec to msec – evolution of material properties during operation**

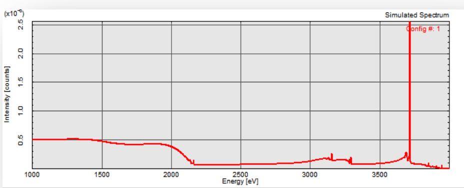
C. Zhang, H. Bluhm, Z. Liu, Z. Hussain et al. *Nature Materials* (2010).

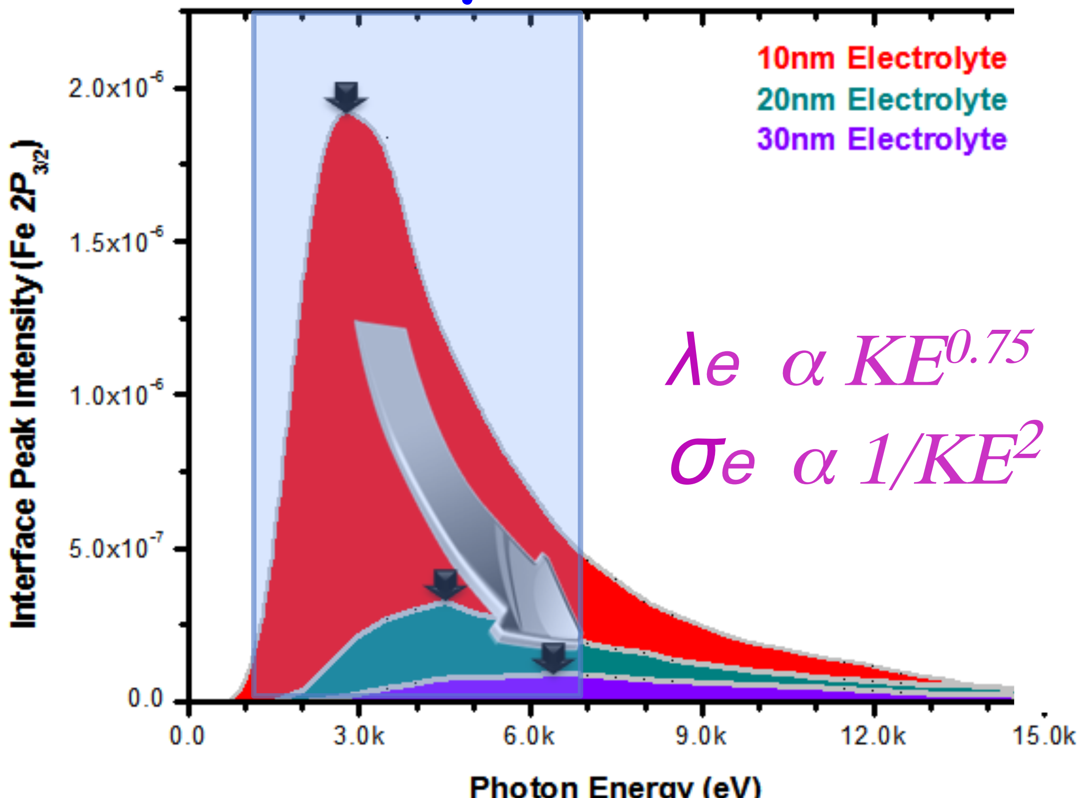
…

Probing Solid-Liquid Interface Region with Tender X-rays



Simulated with SESSA



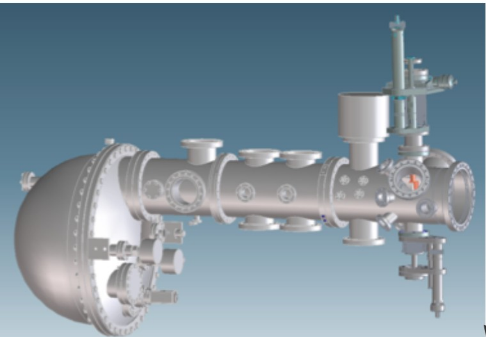


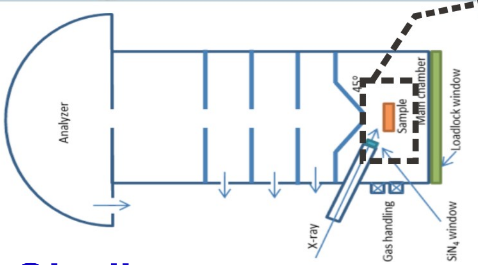
1. Model cells (Science), tools (Spectroscopists), and photon energy (Facility) are intertwined.
2. More challenging when the electrolyte layer is thicker than 30nm.

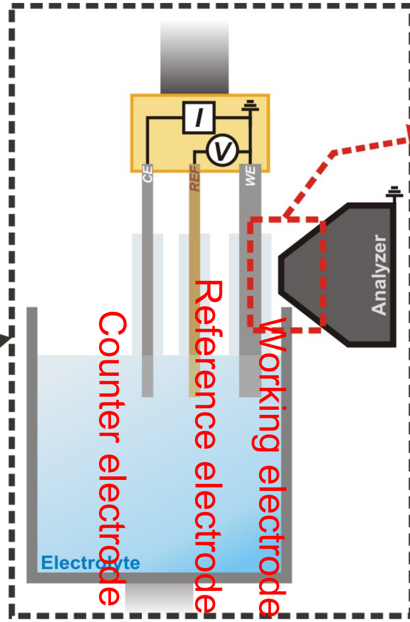
Beamline 9.3.1, 2.5 KeV - 5.0 KeV
Scienta HiPP 2 Analyzer , up to 7 KeV and 250 Torr

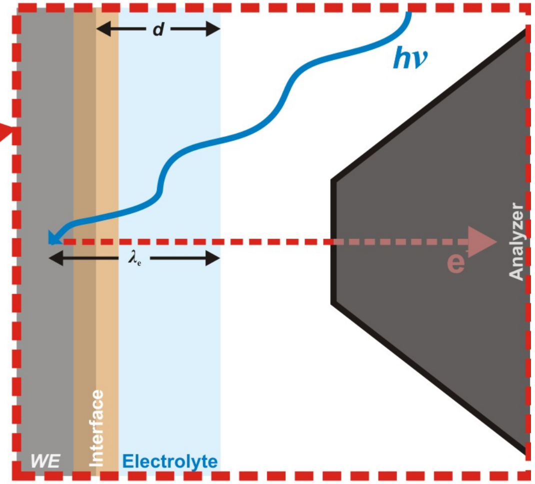
Study Liquid-Solid Interface with AP-XPS Endstation and "Tender" X-ray

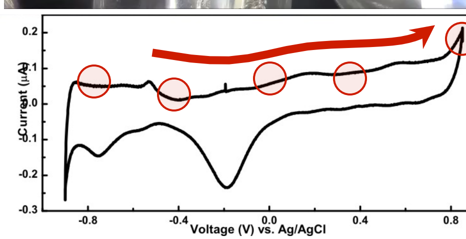
Z. Liu, S. Axnanda, E.J. Crumlin, W. Stolte, Z. Hussain (ALS), P.N. Ross (LBL-BATT)



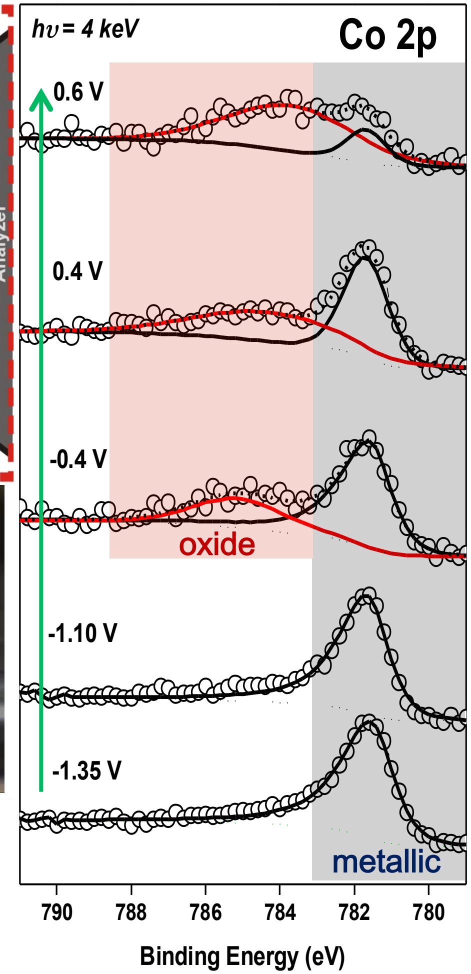








CV of Pt in 6 M KF



Co in 0.1 M KOH
Ref. electrode: Ag/AgCl
Counter electrode : Pt

Challenges:

Identifying the surface species and changes to the solid-liquid interface

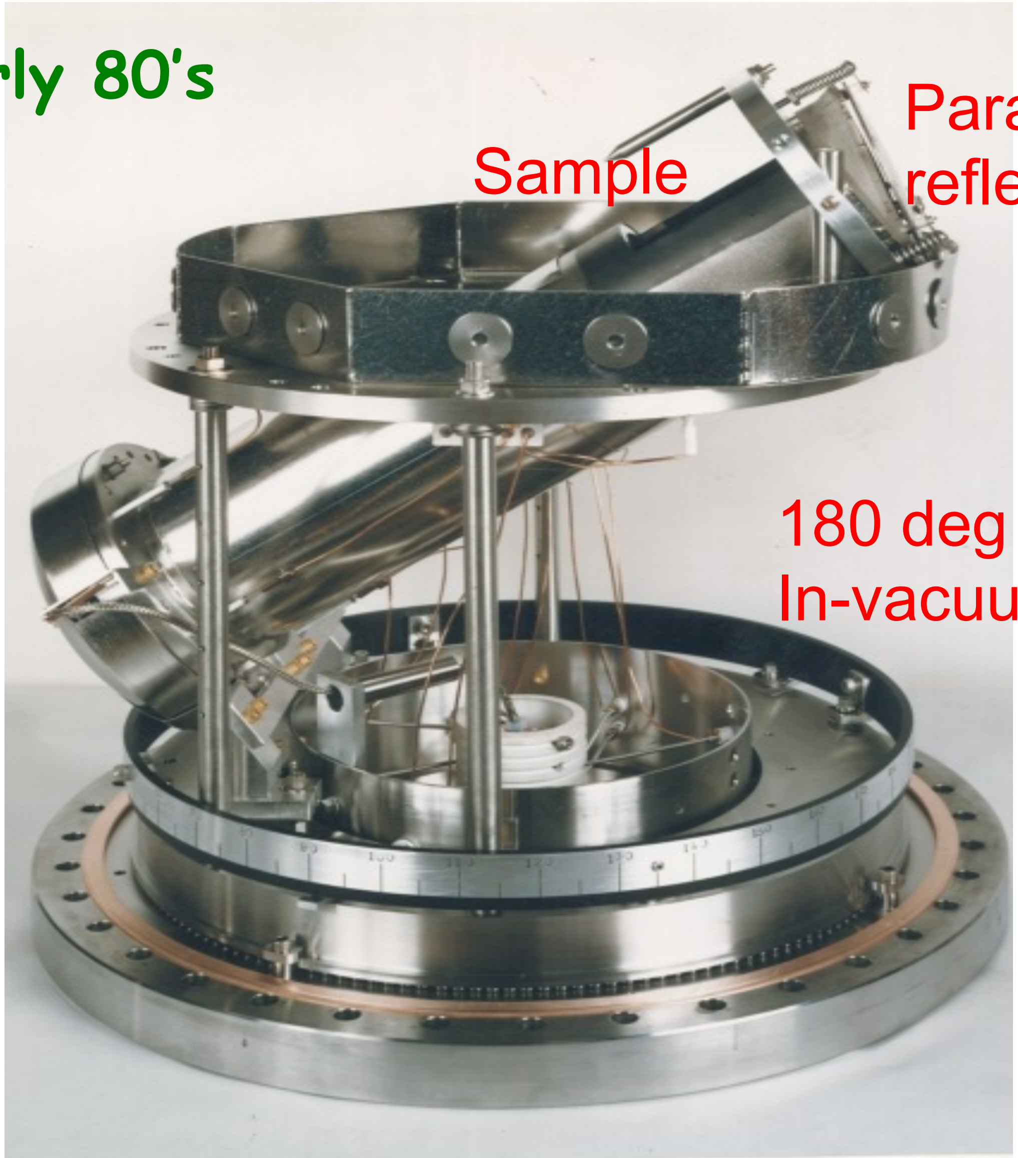
The Important First Step:

First Ever *In operando* APXPS using "Tender" X-ray can simultaneously observe direct changes to both the electrode (solid) surface and electrolyte (liquid)

1) J. Electr. Spectrosc. Rel Phenom., 190, 84-92, 2013; 2) *In preparation*

Time of Flight Parabolic Mirror Analyzer

Shirley Group: early 80's



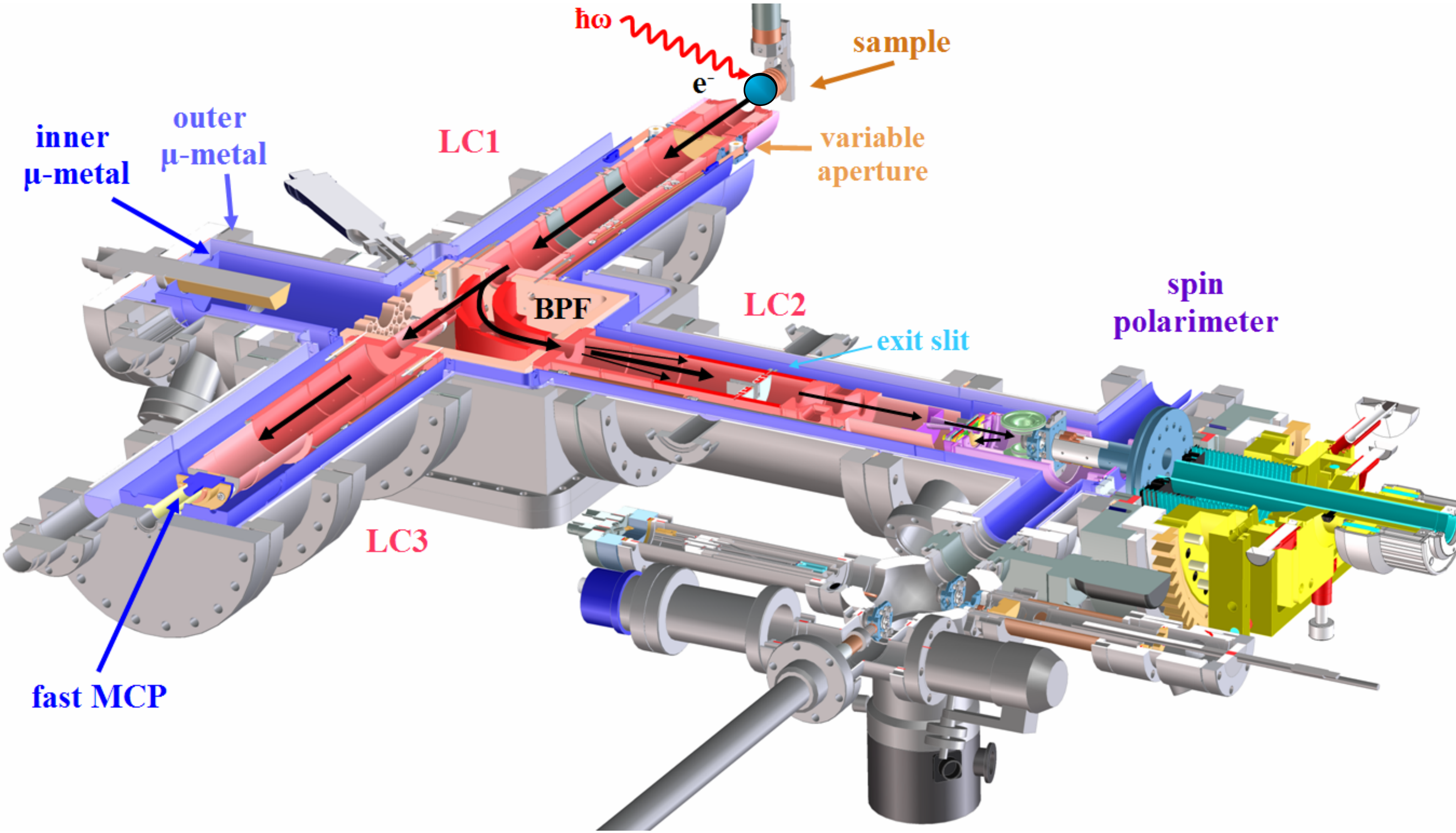
Sample

Paraboloididal reflector

180 deg
In-vacuum rotation

Position sensitive
Detector:
100 mm diameter

The spin-TOF spectrometer



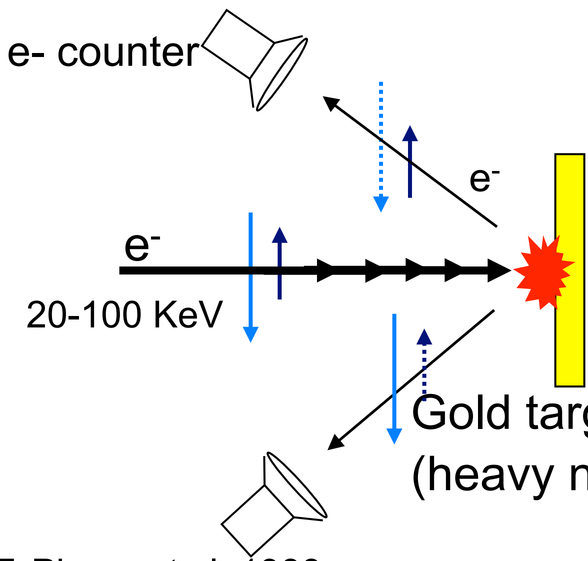
2013 Claus Halbach Award

Spin detection (two schemes)

Mott Detector

Spin-orbit interaction

Hint = L · S



D.T. Pierce et al. 1988 +….

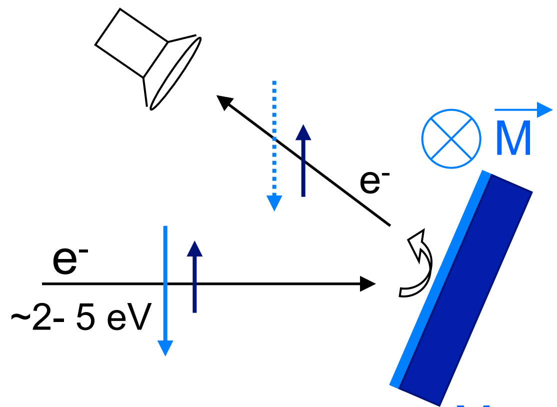
FOM ≤ 10^{-4}

x 100

Exchange scattering interaction

Reflectivity contains a term:

α P · M



R. Bertacco et al. 2001

(e.g.,Co/W(110))

FOM ~ 10^{-2}

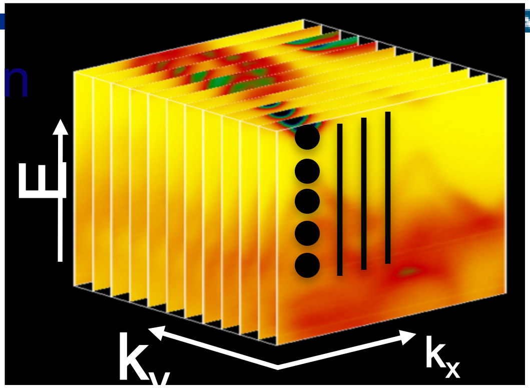
Exchange scattering spin detection with TOF analyzer
>1000 times more efficient than Mott detector with Scienta analyzer

Graf, Schmid, Jozwiak, Hussain, Lanzara et al, PRB, 71, 144429 (2005) &RSI(2009)

Spin resolution & efficiency

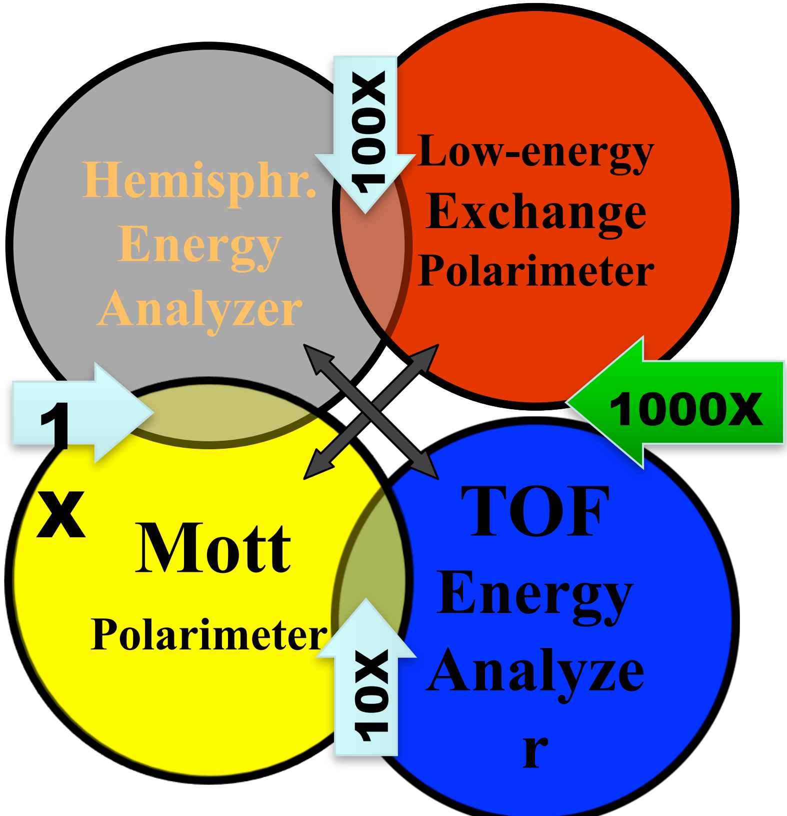
"Standard" ARPES : high speed 2D data acquisition

Efficiency ⟷ resolution, scope



Spin analysis:(1) low efficiency (FOMs ~ 10^{-4})

(2) "single" channel



Hemispherical Energy Analyzer
+ Mott scattering polarimeter

D.-J. Huang, *et al.*, Rev. Sci. Instrum. (1993) (U. Texas)
A.V. Fedorov, *et al.*, J. El. Spectr. Rel. Phen. (1998) (BNL)
G. Ghiringhelli, *et al.*, Rev. Sci. Instrum. (1999) (ESRF)
M. Hoesch, *et al.*, J. El. Spectr. Rel. Phen. (2002) (SLS)

Hemispherical Energy Analyzer
+ Low Energy Exchange scattering polarimeter

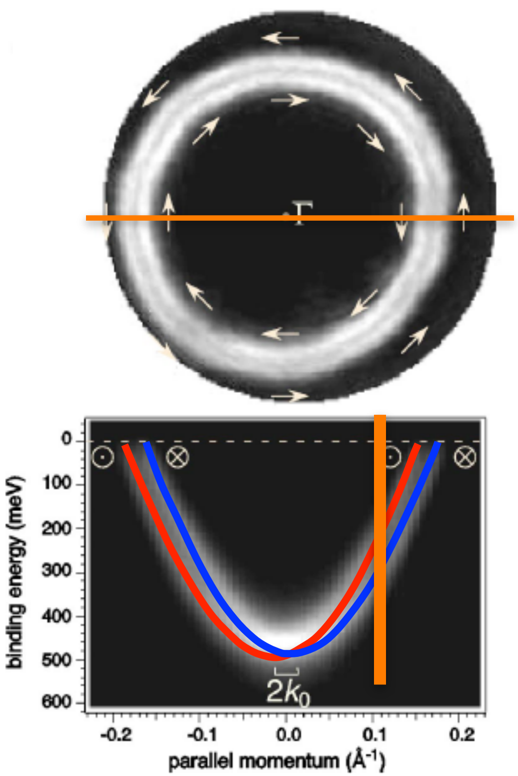
F.U. Hillebrecht, *et al.*, Rev. Sci. Instrum. (2002) (Düsseldorf)
R. Bertacco, *et al.*, Rev. Sci. Instrum. (2002) (Milan)
T. Okuda, *et al.*, Rev. Sci. Instrum. (2008) (SRL, Tokyo)
A. Winkelmann, *et al.*, Rev. Sci. Instrum. (2008) (Max-Planck)

Time-of-Flight Analyzer
+ Mott scattering polarimeter

N. Müller, *et al.*, J. El. Spectr. Rel. Phen. (1995) (BESSY)
G. Snell, *et al.*, Rev. Sci. Instrum. (2000) (U. W. Mich., ALS)
C.M. Cacho, *et al.*, Rev. Sci. Instrum. (2009) (Trieste)

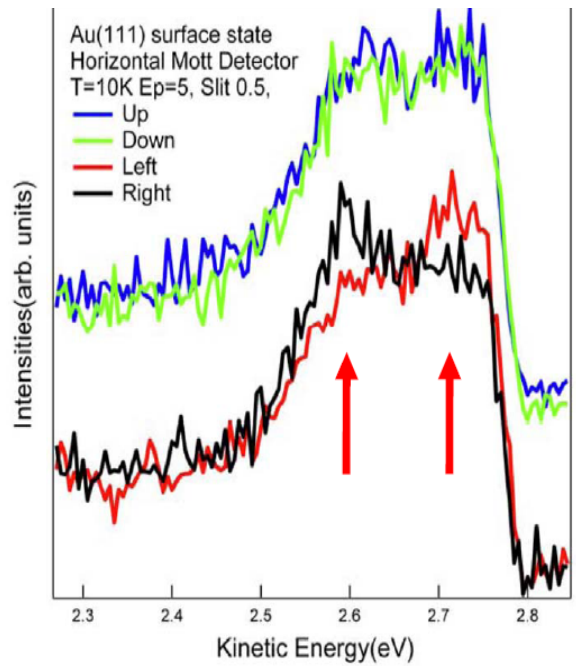
Spin-ARPES: Dispersive Analyzer vs TOF

Rashba-split Au (111) SS



M. Hoesch, *et al.*, PRB (2004)

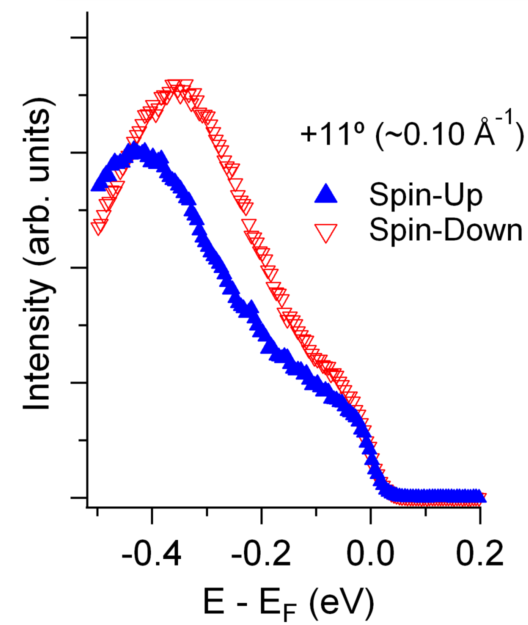
7 eV laser, 80MHz
Scienta R4000 + Mott system



50 minutes (2012)

Xingjiang Zhou, Inst. of Phys., CAS

6 eV laser, 3MHz
spin-TOF

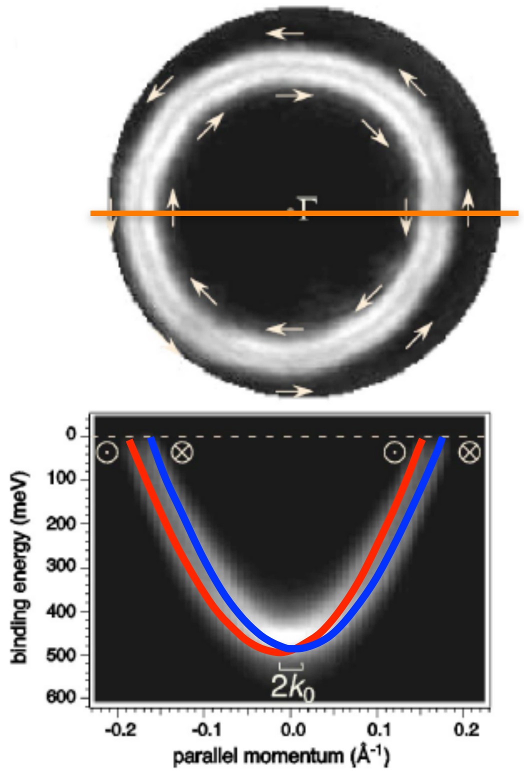


3 minutes!

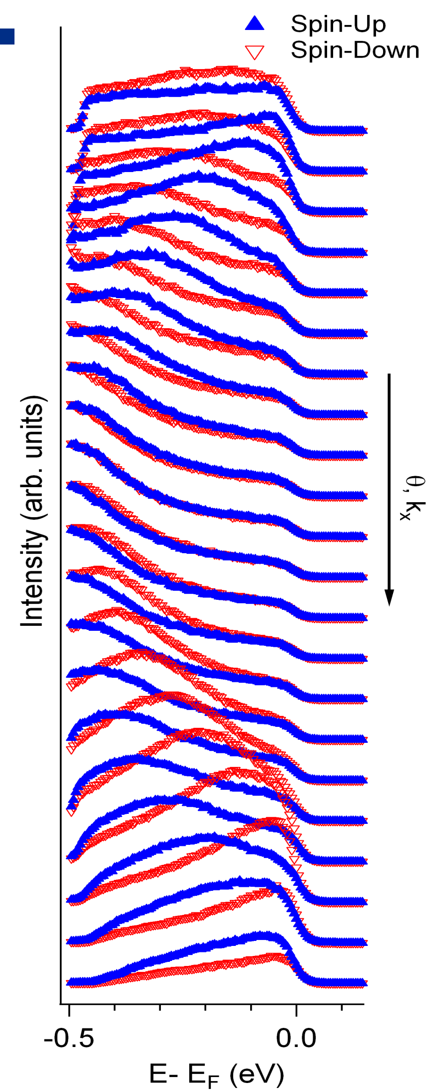
Energy resolution =10 meV

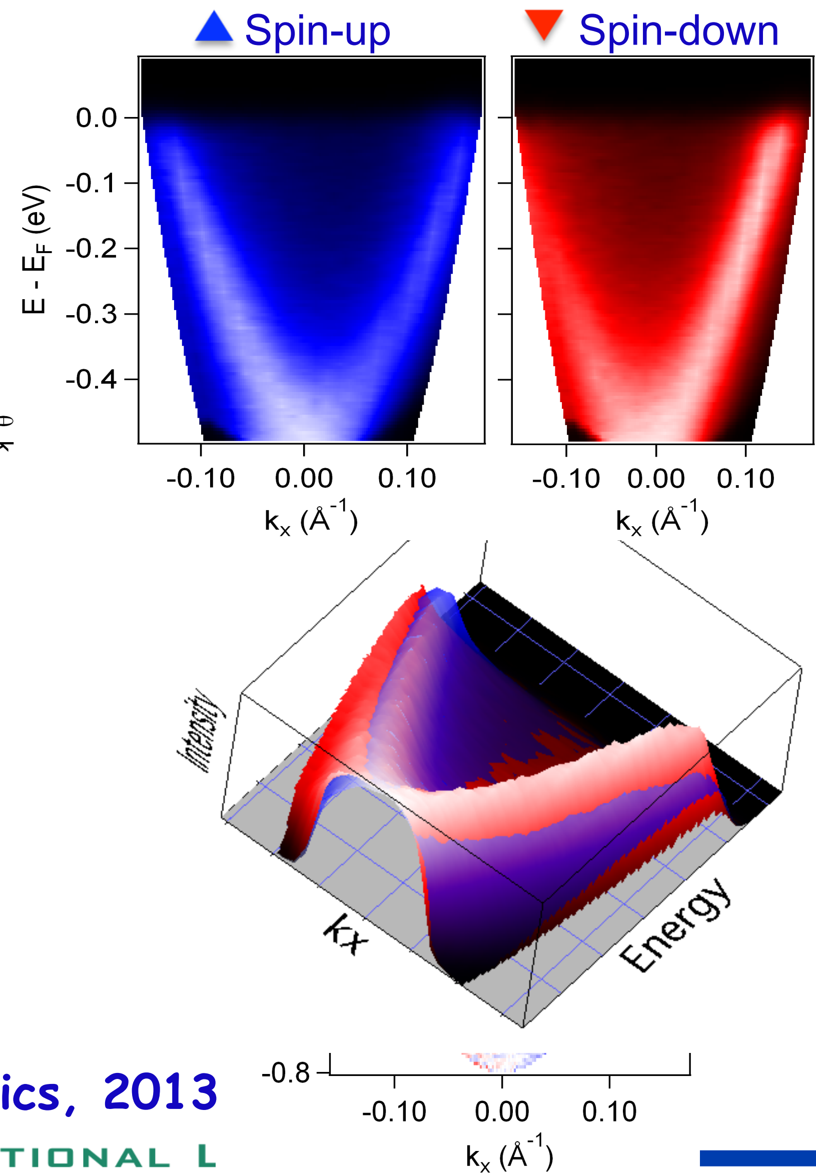
Spin-resolution + laser source

Rashba-split Au (111) SS



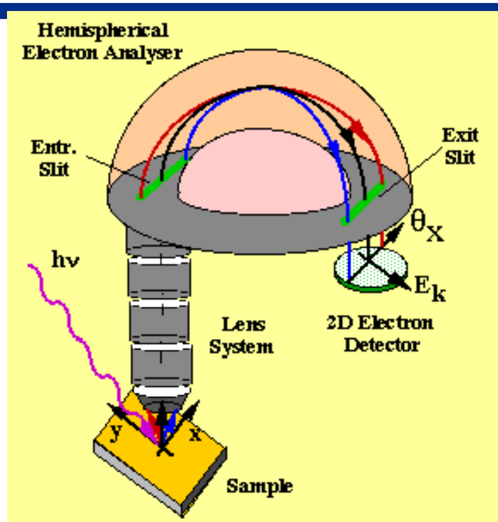
M. Hoesch, *et al.*, PRB (2004)





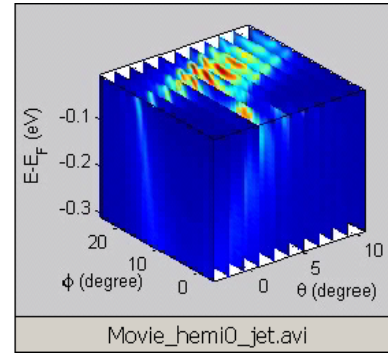
Jozwiak, Hussain, Lanzara; Nature Physics, 2013

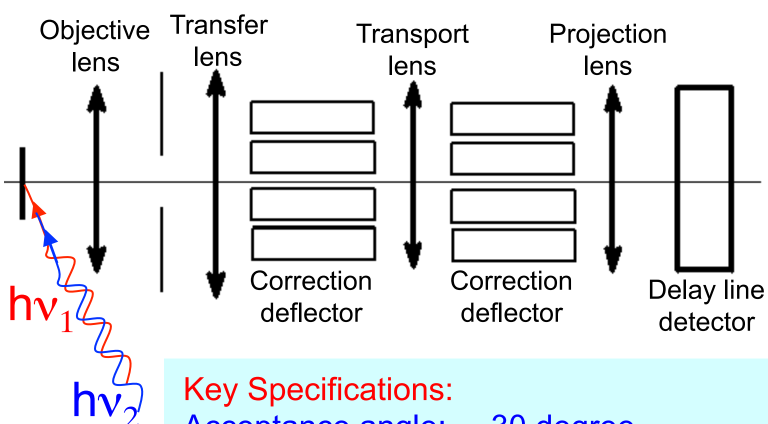
Time-Resolved Photoemission
Comparison of the Hemispherical Analyzer and the TOF Analyzer



Currently used Hemispherical Analyzer
(2D detection)

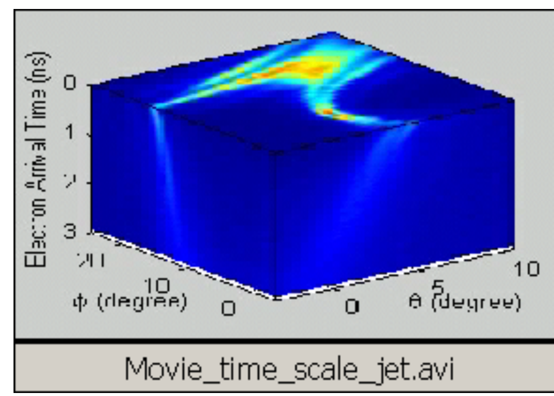
(Bi2212 Bi-layer splitting)





TOF Analyzer Proposed
(3D detection)

(Bi2212 Bi-layer splitting)

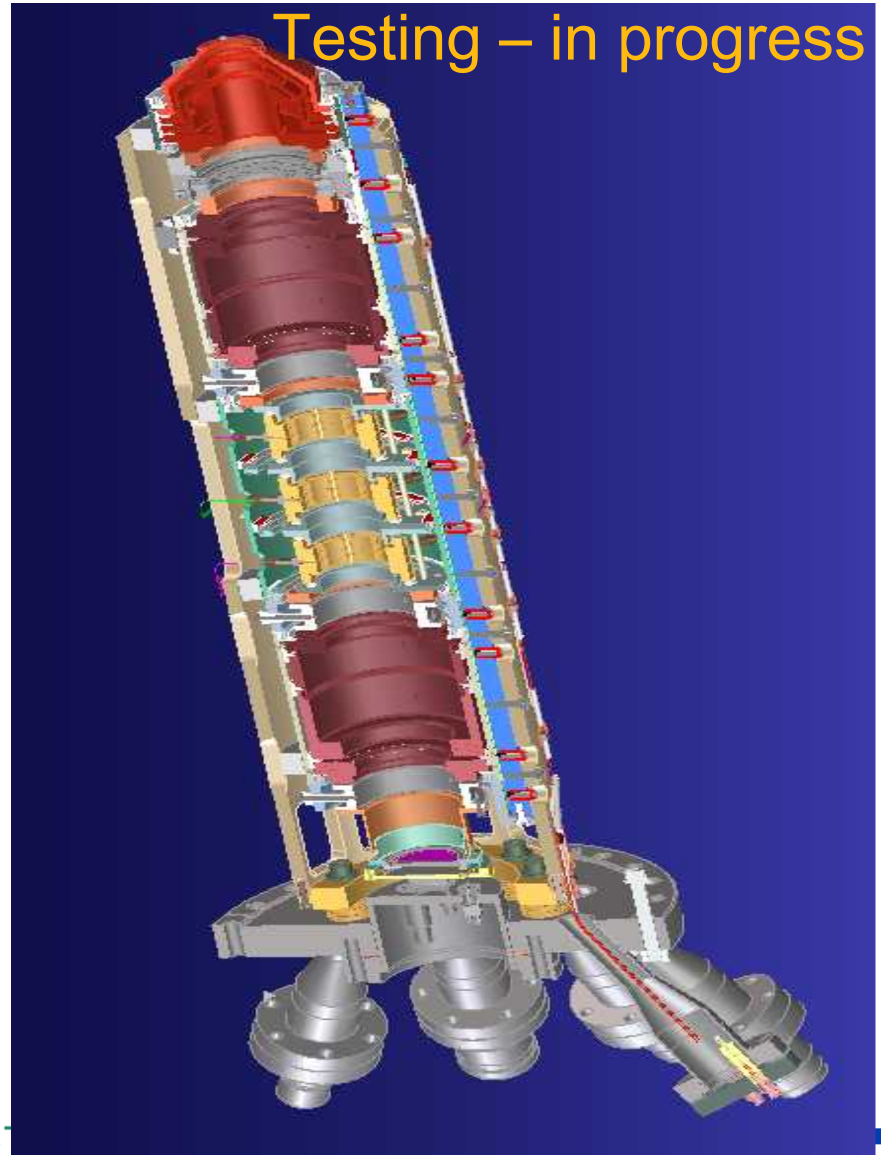


Key Specifications:
Acceptance angle: 30 degree
Energy resolution: <=2meV (5eV Pass Energy)
Angular resolution: <=0.1 degree (~2mrad)
(comparable to Scienta analyzer but 100 times faster)

Commissioning– in progress

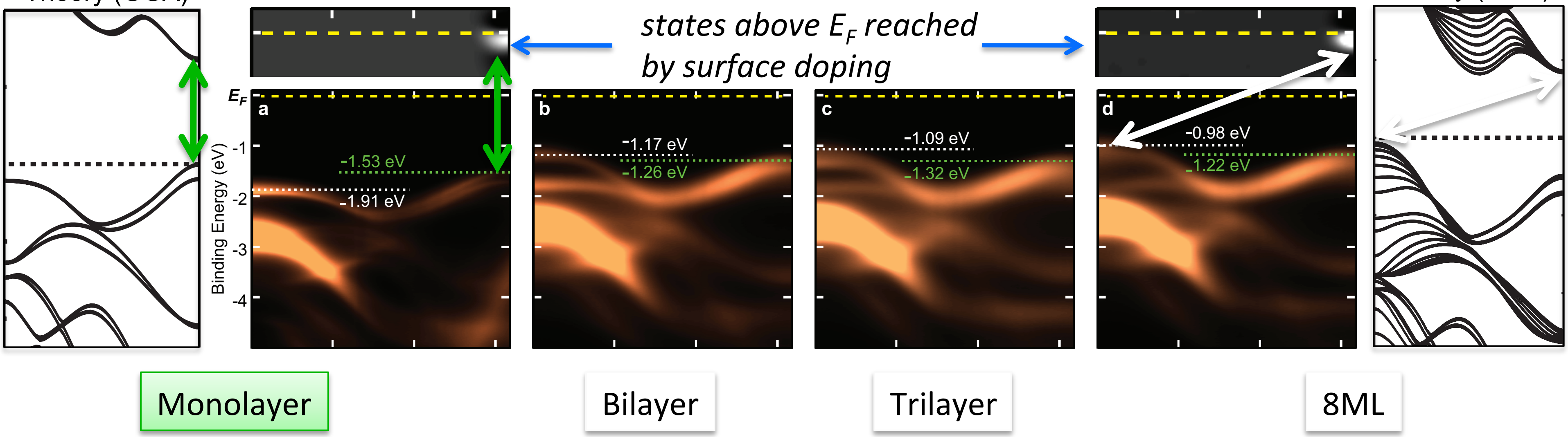
Next step: nano-ARPES with TOF analyzer

TOF 3D nano_ARPES Analyzer PEEM + ARPES



Thickness controlled band gap engineering: $MoSe_2$

In-Situ Sample Synthesis and ARPES

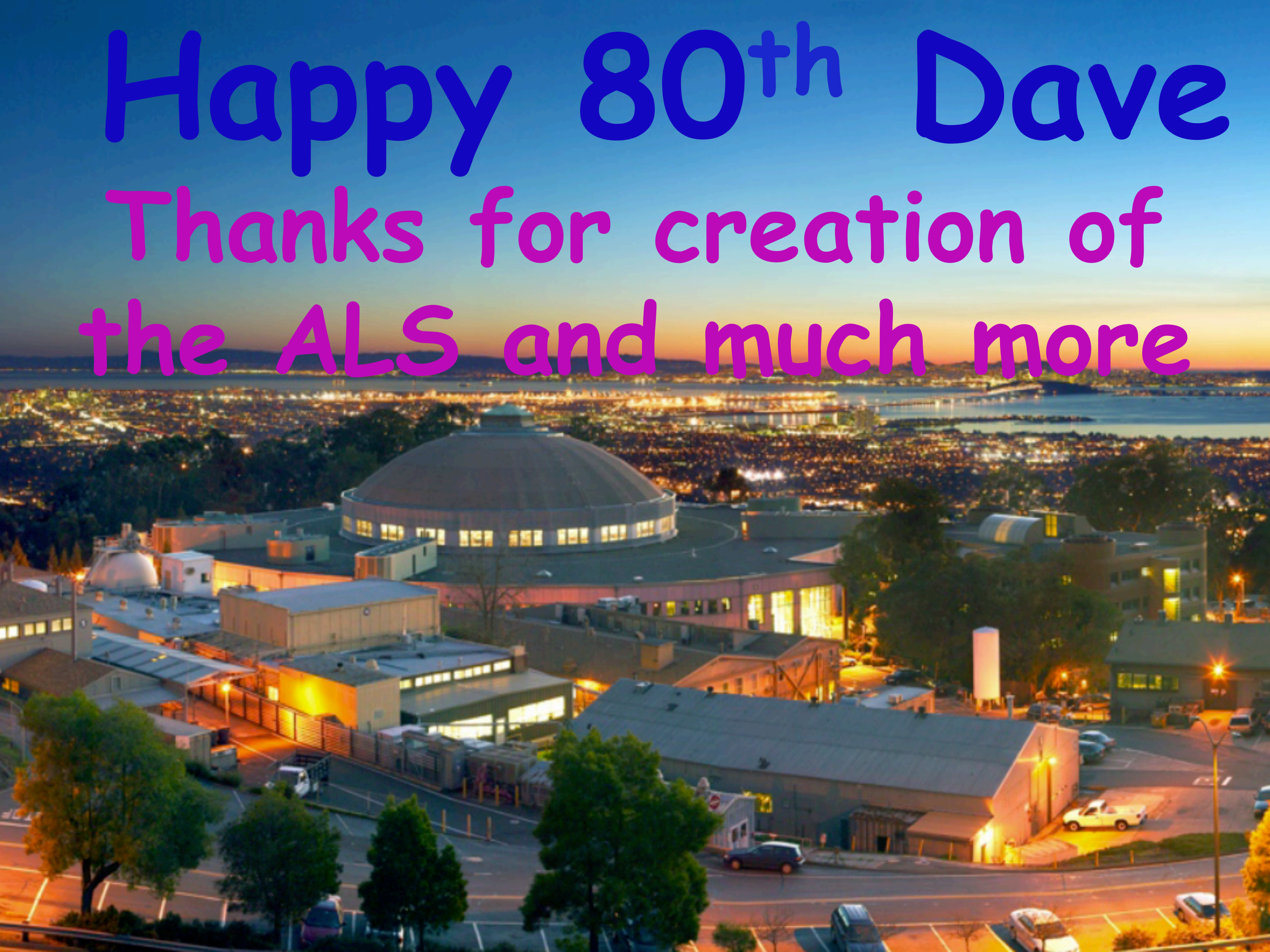


Direct band gap

Indirect band gap

- Distinct direct-indirect band gap transition from monolayer to 2+ layer
- Direct band gap semiconductor favorable for optoelectric & photonic applications
  - ultrasensitive photodetector
  - more efficient, flexible photovoltaic device
- MBE growth *and* in-situ ARPES @ BL 10.0.1

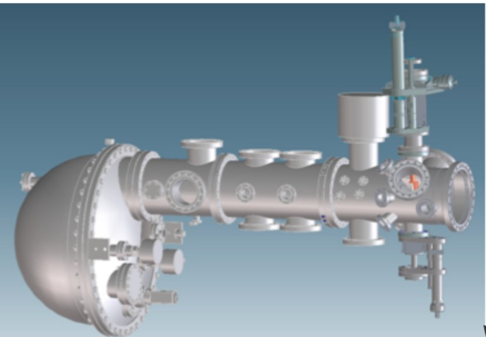
Y. Zhang, S.-K. Mo, Z. Hussain, A. Bansil, Z.-X. Shen et al., Nature Nanotechnology, 2014

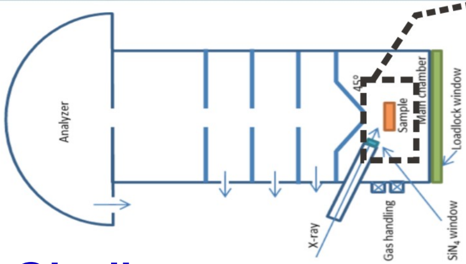

Happy 80th Dave
Thanks for creation of
the ALS and much more

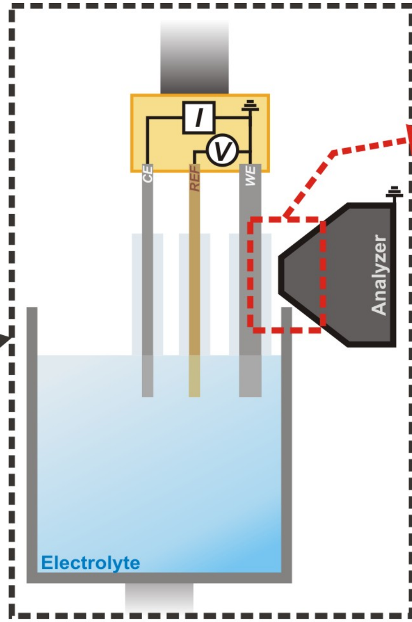

Lawrence Berkeley National Laboratory
Spectroscopy and the Structure of Matter
Symposium held in honor of David A. Shirley on the occasion of his 65th birthday
March 29, 1999

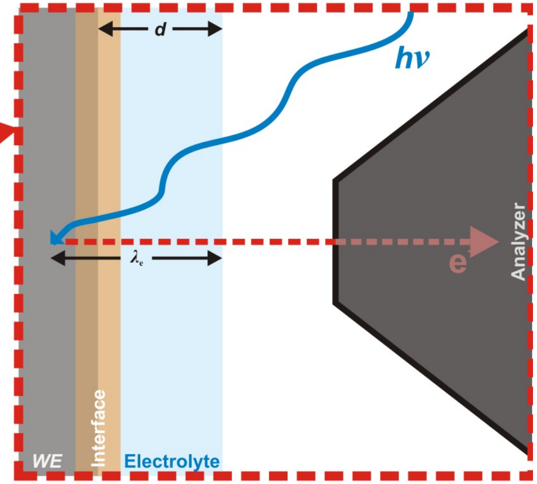
Study Liquid-Solid Interface with AP-XPS Endstation and "Tender" X-ray

Z. Liu, S. Axnanda, E.J. Crumlin, W. Stolte, Z. Hussain (ALS), P.N. Ross (LBL-BATT)







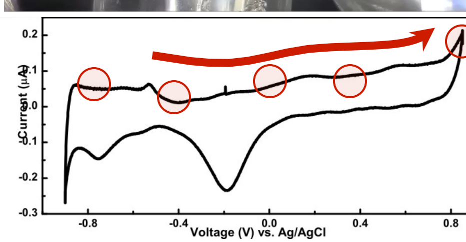


Challenges:

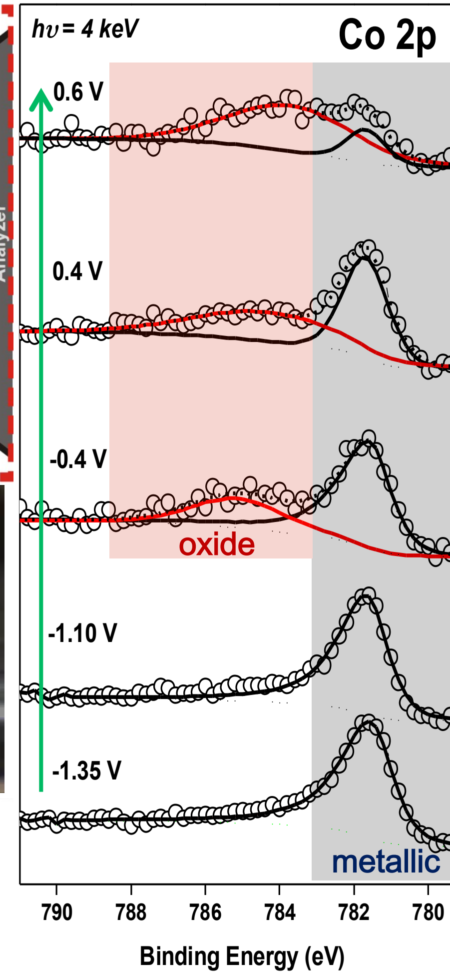
Identifying the surface species and changes to the solid-liquid interface

The Important First Step:

First Ever *In operando* APXPS using "Tender" X-ray can simultaneously observe direct changes to both the electrode (solid) surface and electrolyte (liquid)



CV of Pt in 6 M KF



Co in 0.1 M KOH
Ref. electrode: Ag/AgCl
Counter electrode : Pt

1) J. Electr. Spectrosc. Rel Phenom., 190, 84-92, 2013; 2) *In preparation*