Hard X-Ray Photoemission for Materials Science at the LBNL Advanced Light Source—Update of June, 2019

- We have in 2013 completed installing our upgraded Multi-Technique Spectrometer/Diffractometer on Beamline 9.3.1 (Slides 3-7), together with a five-axis variable-temperature sample manipulator (Slides 8-11(a)), other mechanical upgrades to improve spectrometer rotation and temporarily provide a sixth tilt axis of sample rotation (Slide 12(b)) and to improve angular resolution (Slide 13). This bend-magnet beamline provides photons between about 2.3 keV and 5.5 keV (slide 2). This permitted doing materials-science based hard x-ray photoemission (HXPS, HAXPES) for the first time at the ALS, and this effort represented only the second possibility for broad-based materials-related HXPS in the U.S., together with beamline X24A at NSLS, also situated at a bend magnet. For more on the 9.3.1 beamline, see Slide 3 and W. Stolte at: http://ssg.als.lbl.gov/ssgbeamlines/beamline9-3-1 . Allowing for ring current and topoff, the ~10^{11} measured fluxes from 9.3.1 are comparable to those from both X24A and the HIKE beamline at BESSY (still a very successful bend-magnet HXPS facility).

- This beamline became unusable due to instability and difficulty of alignment in 2013-14, and is now being upgraded with a new monochromator, scheduled to be completed in summer 2019.

- Looking back before the shutdown, to the first hard x-ray photoemission results that follow (Slides 4-6 and 14-22), the spectrometer contribution to linewidth should be 0.20 eV. The 9.3.1 beamline contribution derives from a resolving power $E/\Delta E$ that is approximately constant at ~6,800 over its full range, which has now been verified by us in photoemission, and will thus yield for two of the energies used here 0.42 eV at 2842 and 0.66 eV at 4500 eV. The spectrometer should thus be contributing only a small amount to linewidth for both photon energies. These resolution values are thus over the full range better than typical monochromatized laboratory XPS systems using Al Kα at 1487 eV, and can be compared to optimum practical working resolutions of 0.20-0.30 eV in our prior measurements at the best undulator beamlines in the world (SPring-8, DESY-Petra III, Diamond, Soleil). See last slide for a partial list of prior publications based on HXPS from 9.3.1 and other facilities.

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- A non-monochromatized Al Kα or MgKα x-ray source is also available in the system (Slide 7) for lower-energy measurements at 1487 eV or 1254 eV, with the usual ultimate resolutions of ca. 0.9 and 0.8 eV, respectively.

- The hard x-ray measurements involve much greater information depths than typical XPS at 1.5 keV, by ca 1.6x at 2842 eV and 2.3x at 4500 eV. As another comparison to typical UPS or ARPES measurements between 20 and 150 eV, the information depths in the hard x-ray measurements will be greater by ca. 9-30x at 2842 eV and 13-50x at 4500 eV. Thus, surface effects are minimized and bulk or buried-layer properties are more directly accessible.

- The Berkeley system is also fully automated for angle scanning, making use of the very versatile Bostwick software, with first standing-wave rocking curves in multilayer samples now obtained (Slides), and the system has also demonstrated its ability to do hard x-ray angle-resolved photoemission (HARPES—See Gray et al., Nature Materials 10, 759–764 (2011) –Slides 18-20), including a sixth temporary tilt axis that has been demonstrated in core-level photoelectron diffraction and angle-resolved photoemission (Slides 18-20).

- Being able to rotate the spectrometer in-plane by ca. 50 degrees (see configuration in Slides 5 and 6) also permits doing variable surface sensitivity angle-resolved XPS (ARXPS) with a fixed grazing x-ray incidence angle at which intensities are a maximum, or doing HARPES with fixed photon-sample relationship. Other hard x-ray photoemission systems suffer severe losses in intensity when going away from grazing incidence to do ARXPS (see e.g. Slide 16, where this does not occur).

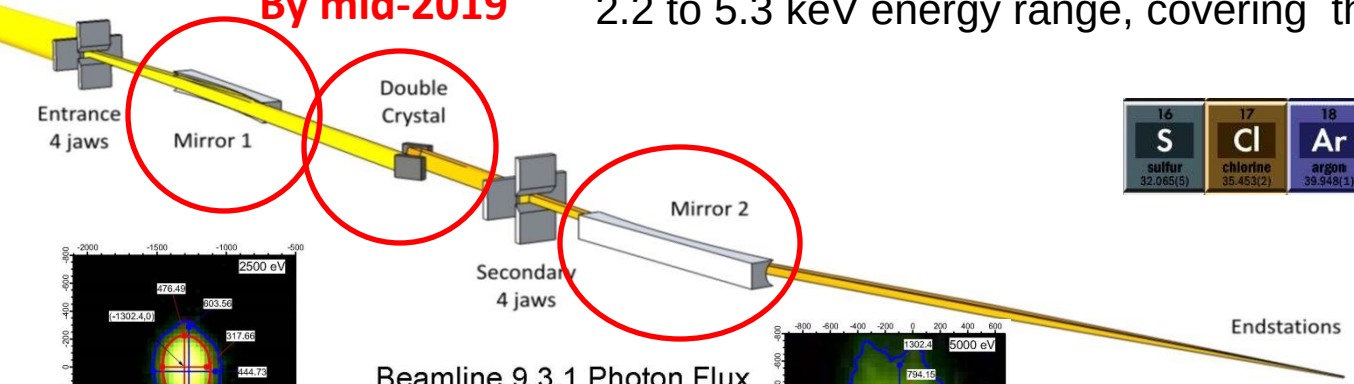
- Improvements anticipated for 2019 and beyond are:
  - Pepper-design true 6-axis variable-temp. manipulator (Figure 11(b)), including new x,y,z stage
  - In situ reflectivity measurement
  - Motorized analyzer rotation in plane

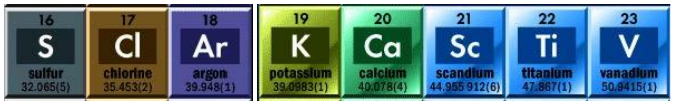
Beamline 9.3.1 : Description Prior to Problems and Upgrades over 2013-2019

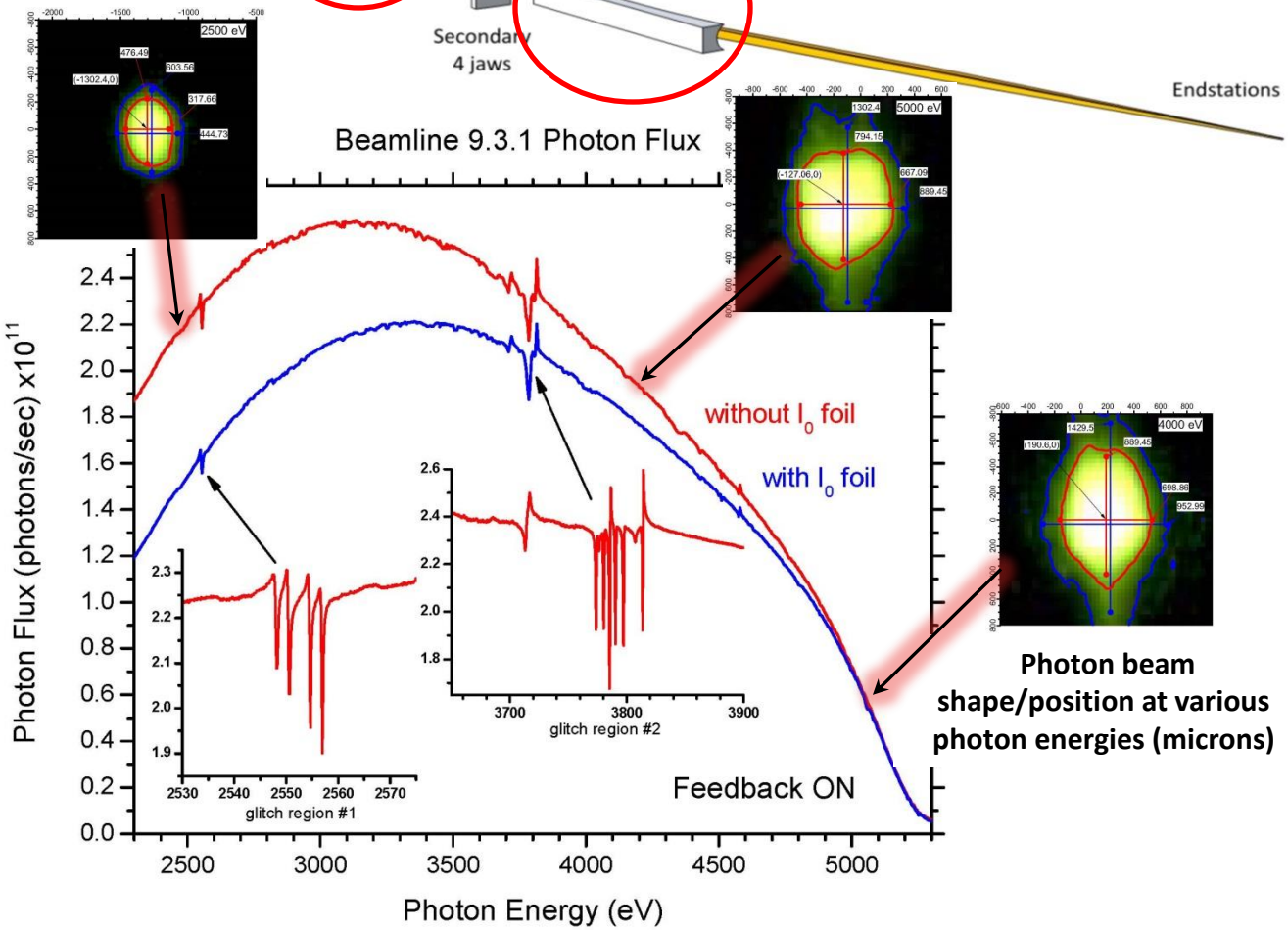
Storage ring

Being replaced, By mid-2019

Beamline 9.3.1 is a double Si (1,1,1) crystal monochromator with a 2.2 to 5.3 keV energy range, covering the K-edges of :





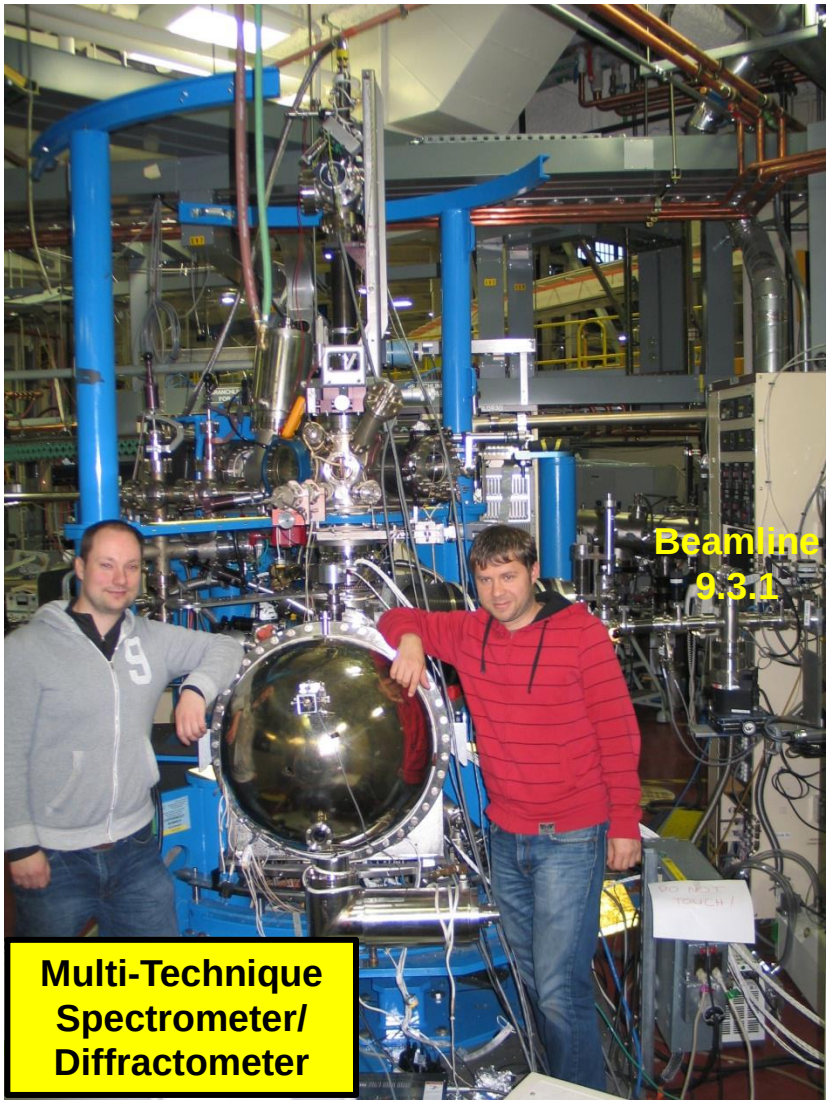


Photon beam shape/position at various photon energies (microns)

- **The optical design includes two identical, but oppositely deflecting, toroidal mirrors positioned symmetrically before and after the monochromator. This approach yields two benefits: high resolution by providing parallel x-rays for diffraction by the Si crystals, and a small beam spot by means of 1:1 focusing of the storage ring source with a minimum of aberrations.**
- **The beamline delivers approximately 2 x 10^{11} photons/s over most of its photon energy range, with a resolving power of ~6800 over the entire range.**
- **The minimum beam size is better than 500 x 1000 microns, and the usual position stability is less than ±200 microns. Beam motion in scanning energy over the full range is very small, in the ±5 micron range for vertical and ± 10 micron range for horizontal.**

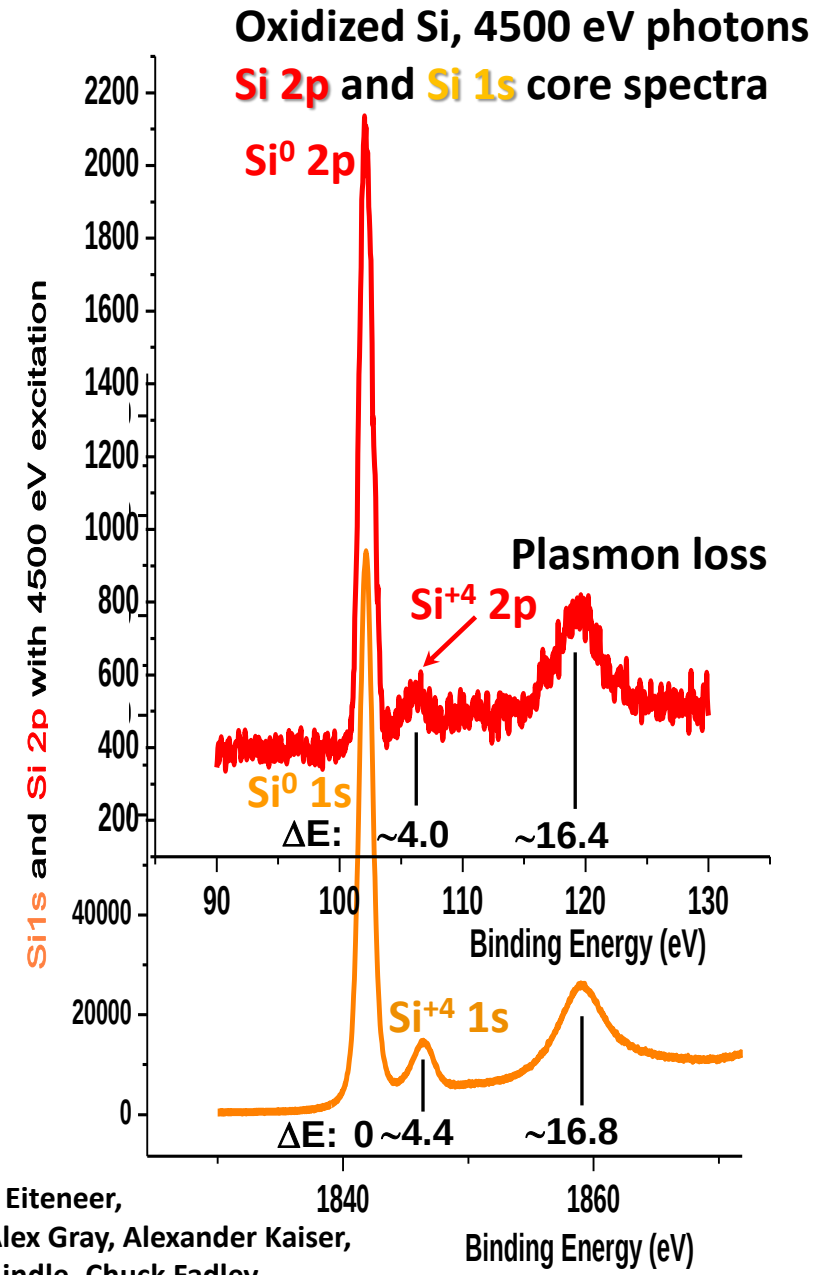
W. Stolte, ALS, http://ssg.als.lbl.gov/ssgbeamlines/beamline9-3-1

Hard X-Ray Photoemission at the LBNL Advanced Light Source—First Results, March, 2012



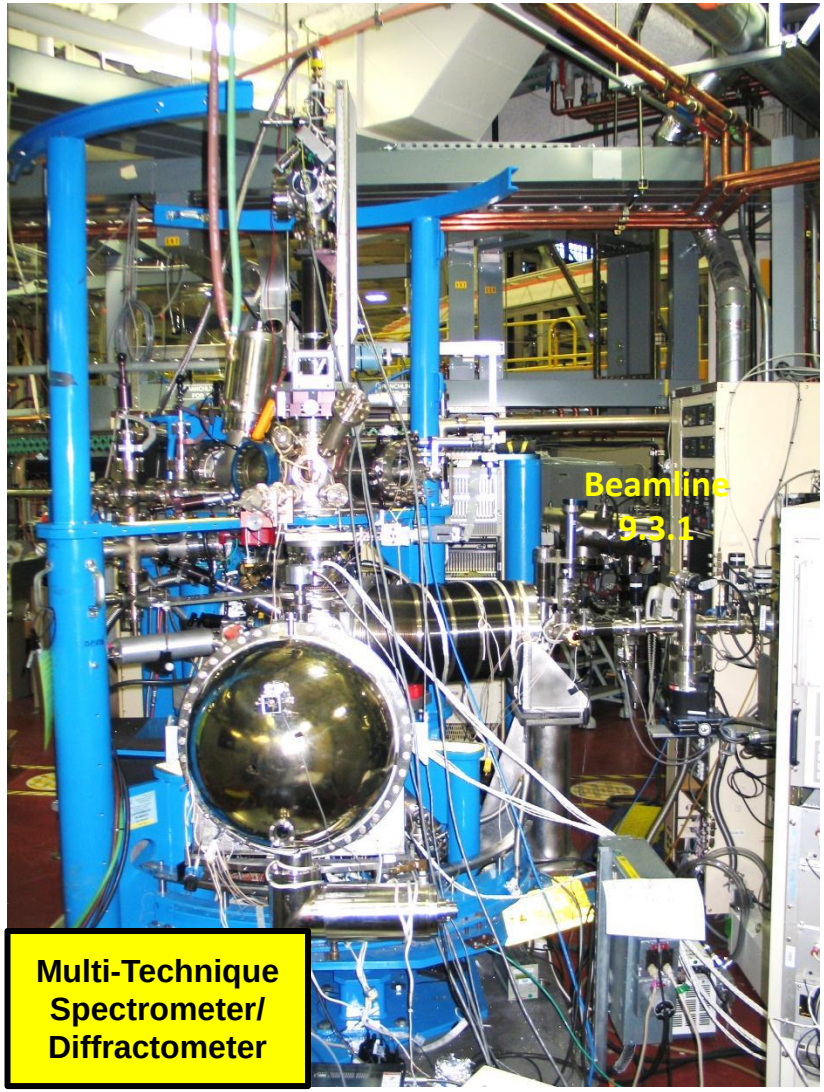
Multi-Technique Spectrometer/ Diffractometer

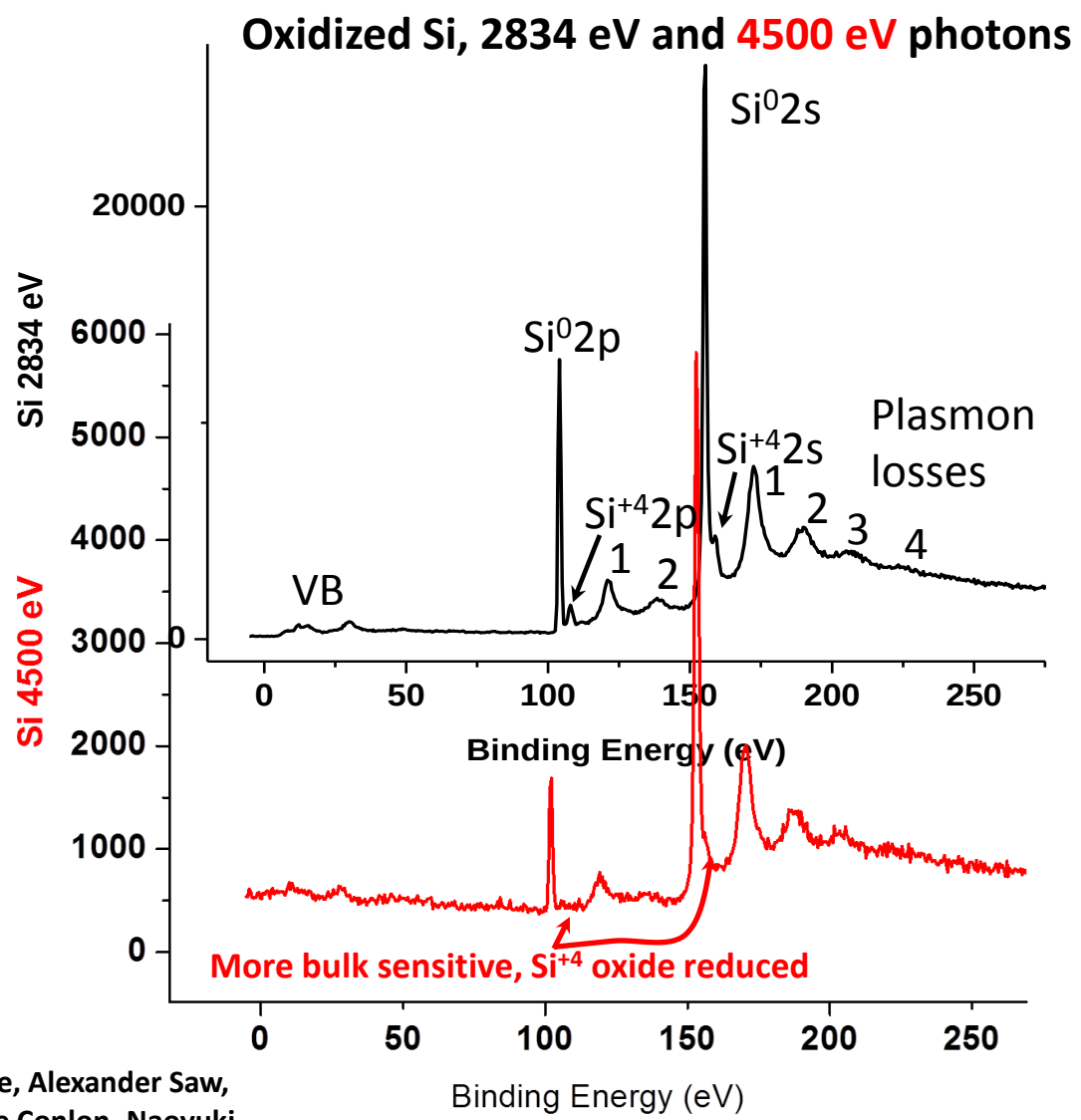
Gunnar Palsson Slavomir Nemsak



with Mark West, Wayne Stolte, Alexander Saw, Aru Rattachanata, Daria Eiteneer, Aaron Bostwick, Catherine Conlon, Naoyuki Maejima, Armela Perona, Alex Gray, Alexander Kaiser, Giuseppina Conti, John Thomson, David Hemer, Zahid Hussain, Dennis Lindle. Chuck Fadley

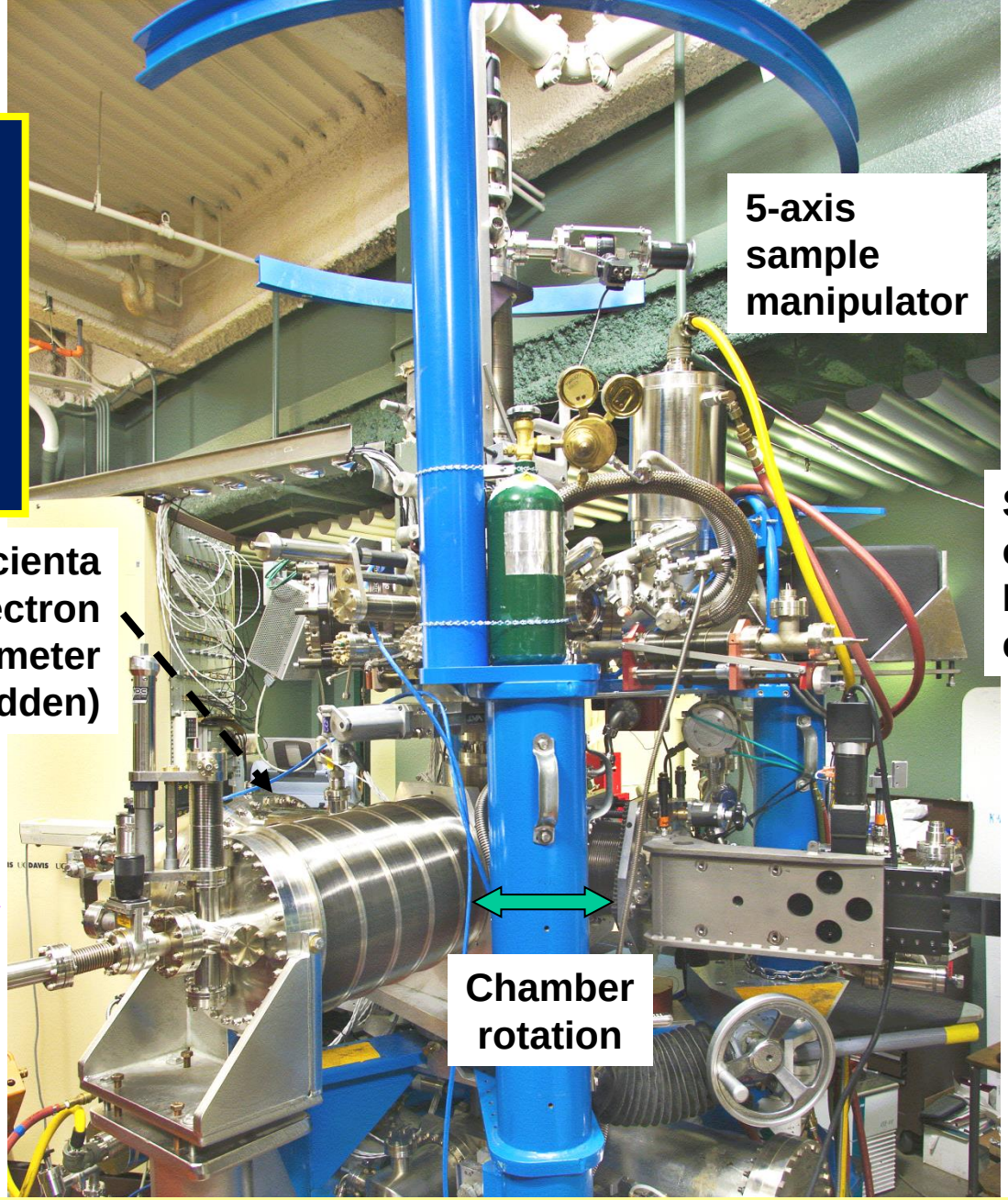
Hard X-Ray Photoemission at the LBNL Advanced Light Source—First Results, March, 2012





Slavomir Nemsak, Gunnar Palsson , Mark West, Wayne Stolte, Alexander Saw, Aru Rattachanata, Daria Eiteneer, Aaron Bostwick, Catherine Conlon, Naoyuki Maejima, Armela Perona, Alex Gray, Alexander Kaiser, Giuseppina Conti, John Thomson, David Hemer, Zahid Hussain, Dennis Lindle. Chuck Fadley

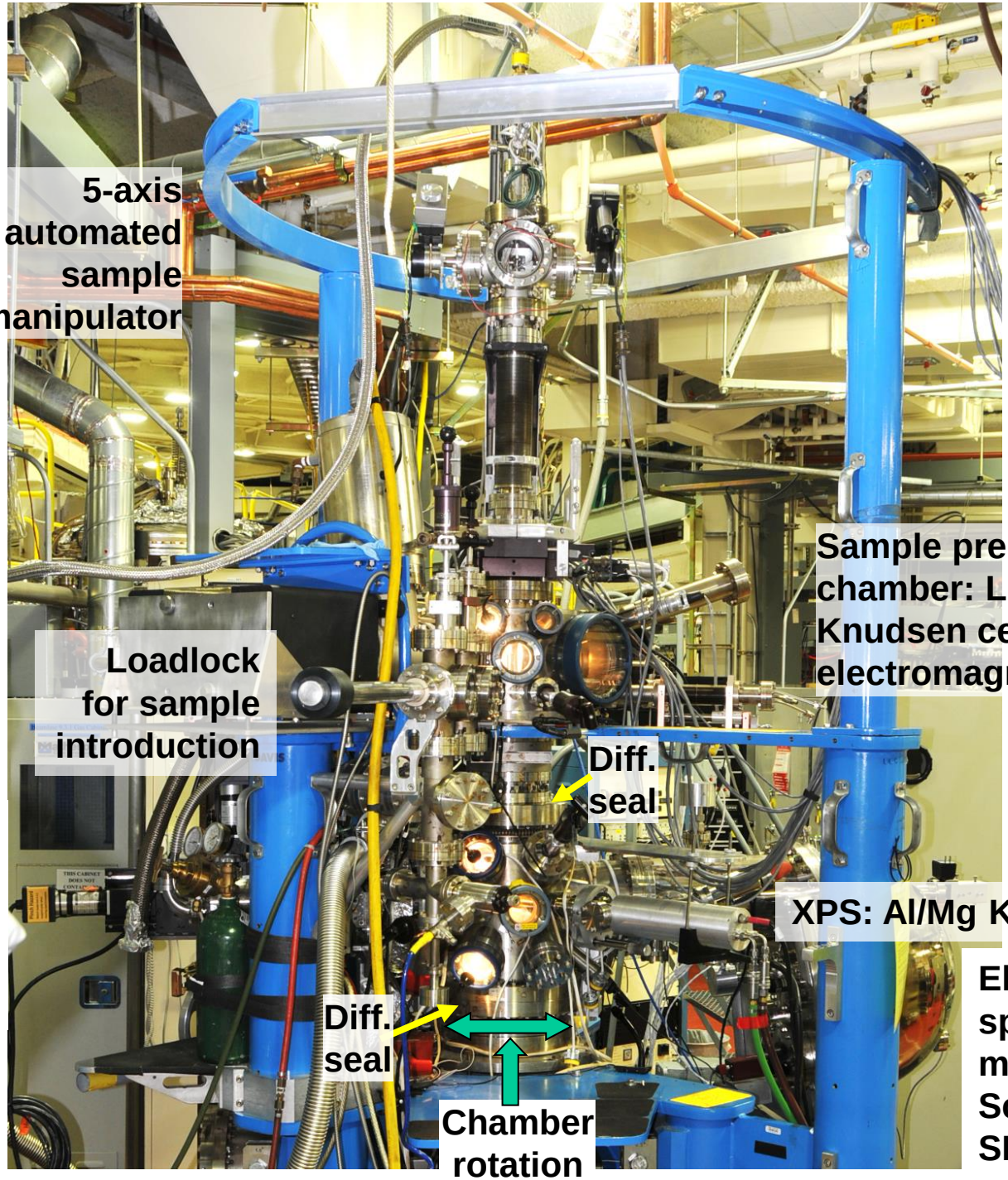
Hard X-ray Photoemission at the Advanced Light Source: The Multi-Technique Spectrometer/ Diffractometer (MTSD)



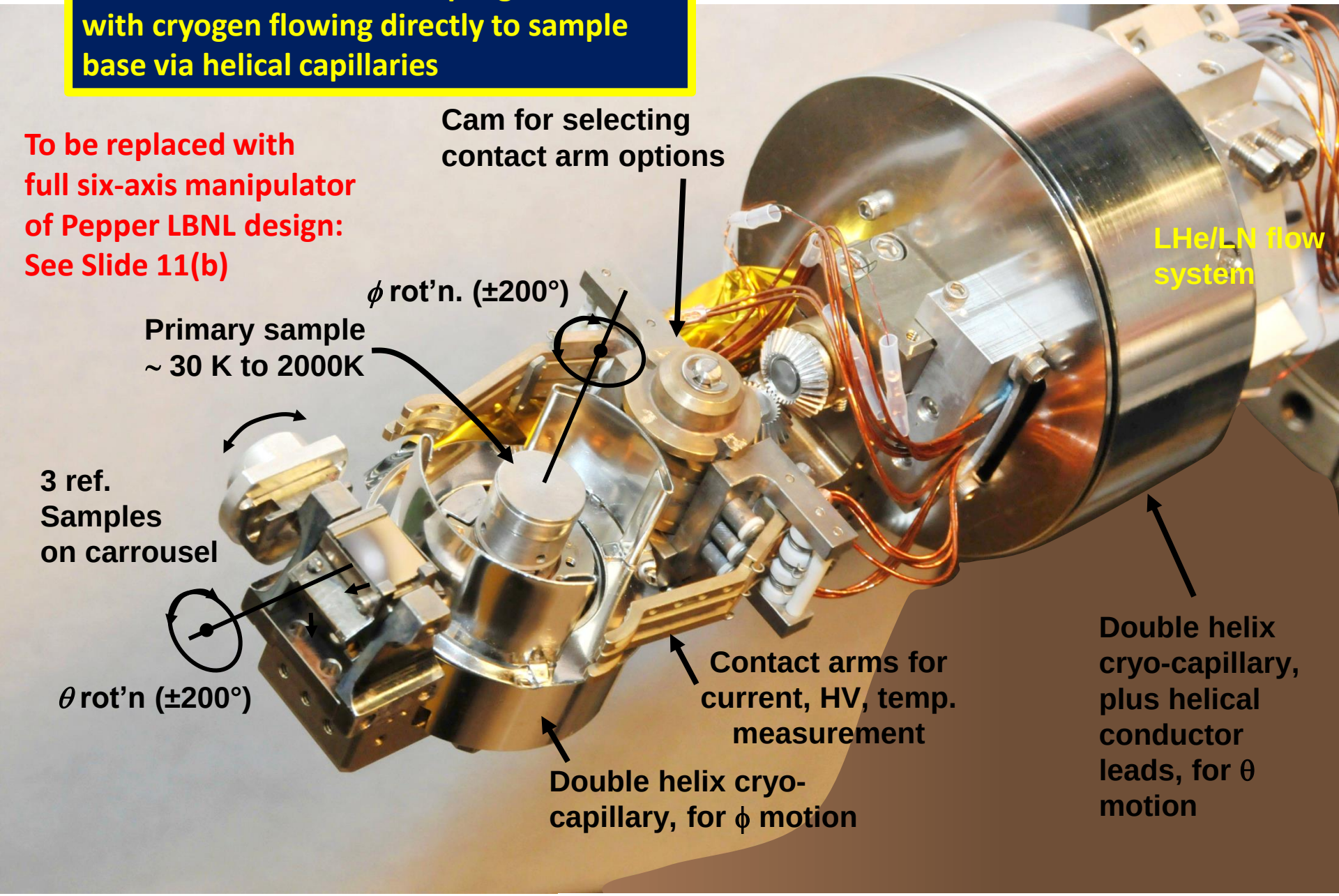
ALS
BL 9.3.1
$h\nu$ = 2-5 keV

Permits using all relevant spectroscopies on a single sample:
XPS (incl. Al and Mg Kα), HXPS, XPD; XAS (e^- or photon detection), soft XES/RIXS

Hard X-ray Photoemission at the Advanced Light Source: The Multi-Technique Spectrometer/ Diffractometer (MTSD)

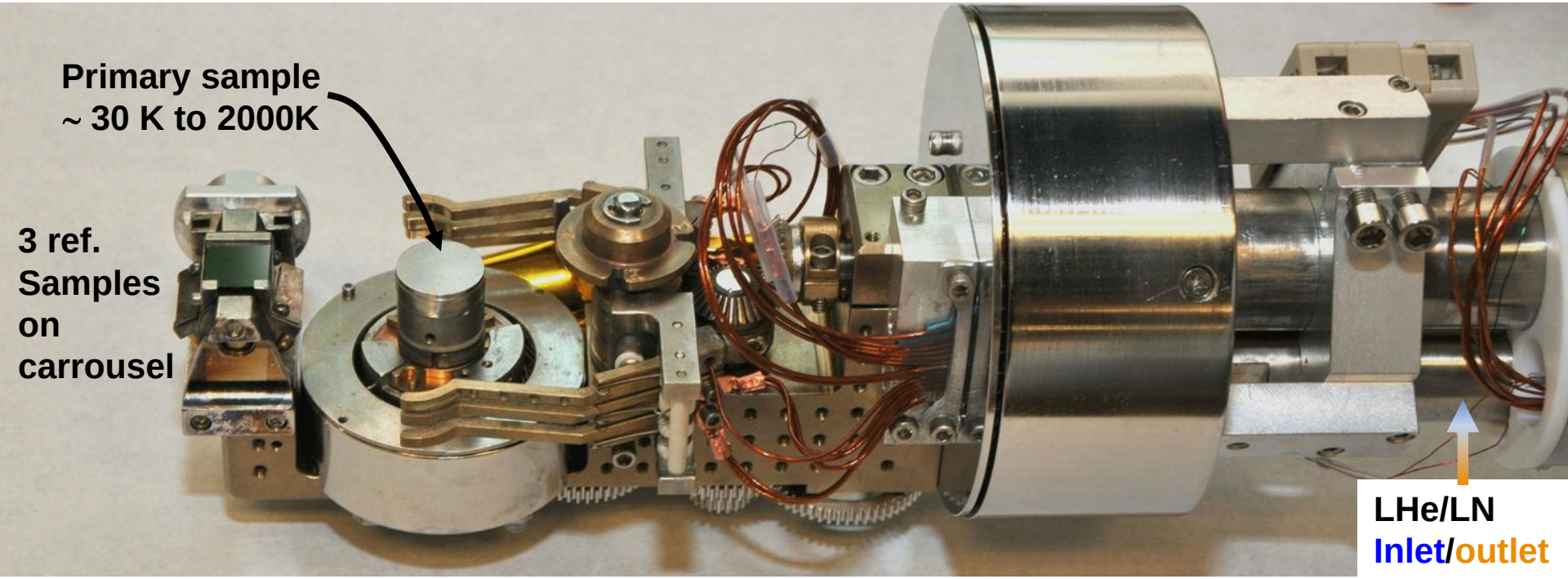


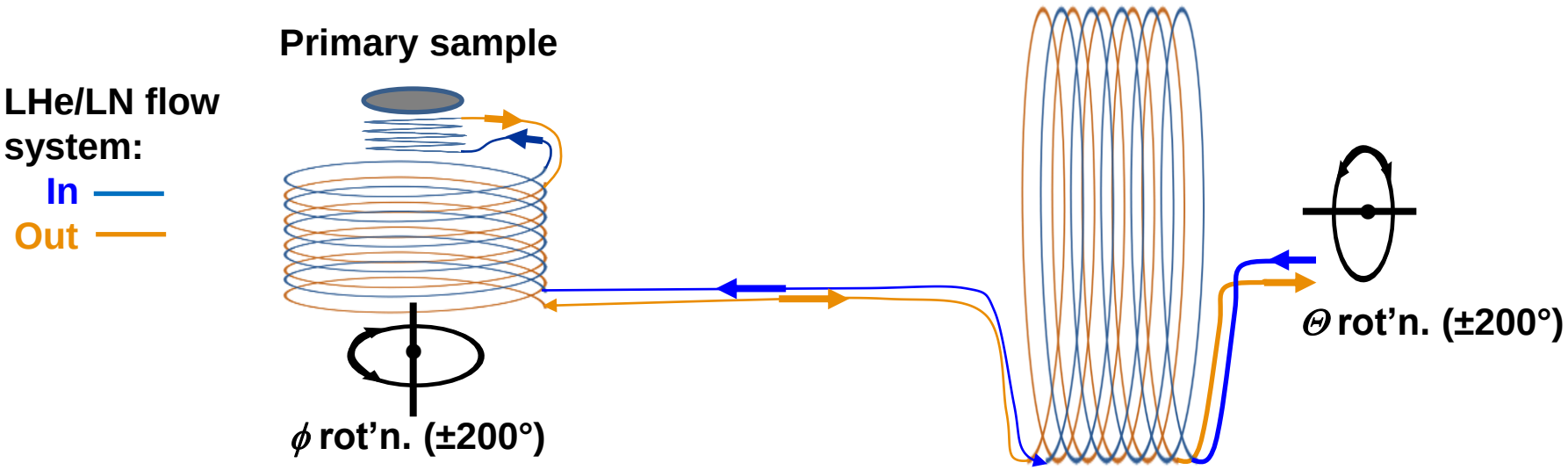
Custom-built five-axis sample goniometer with cryogen flowing directly to sample base via helical capillaries



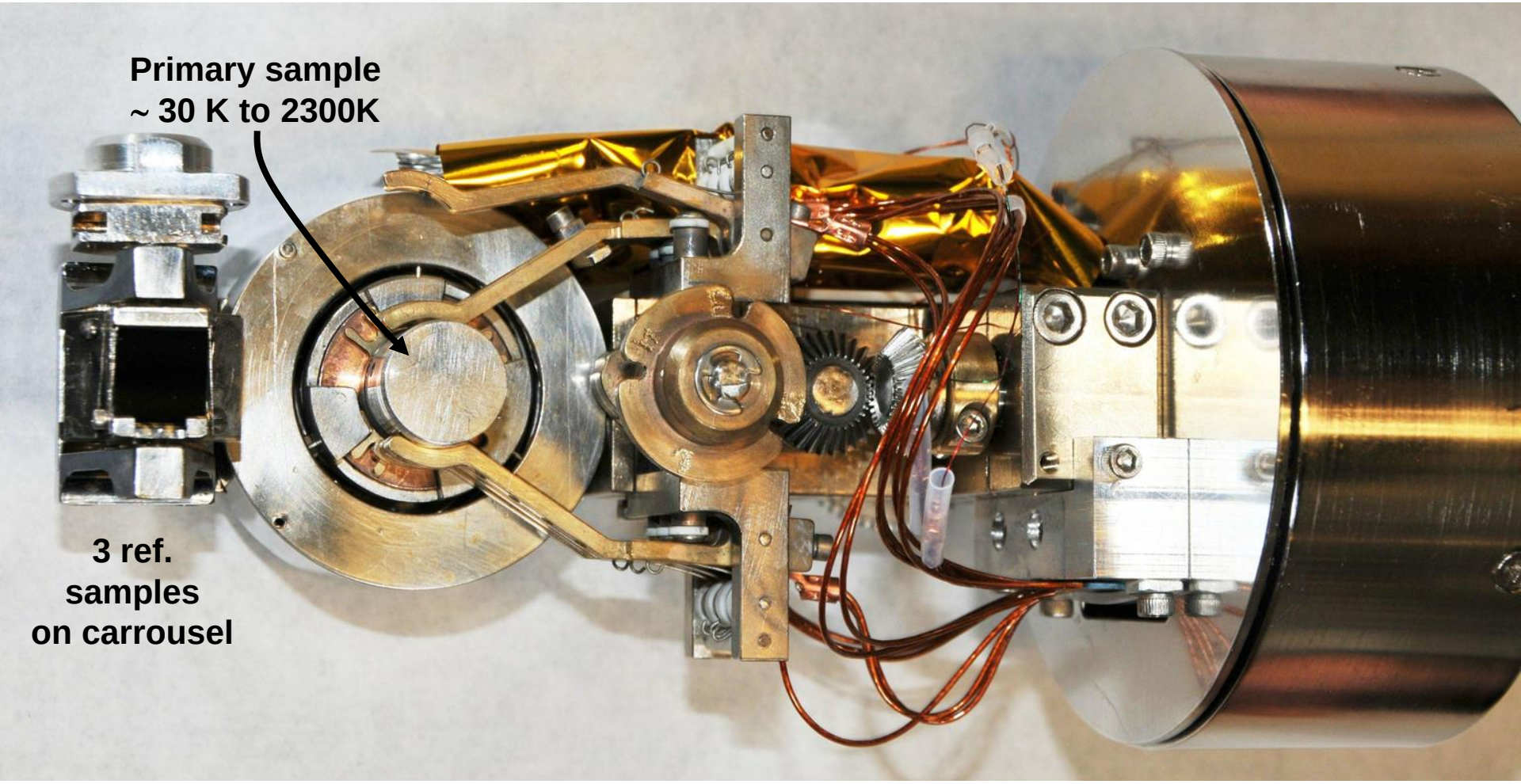
S. Nemsak, M. West, J. Pepper, …

Custom-built five-axis sample goniometer with cryogen flowing directly to sample base



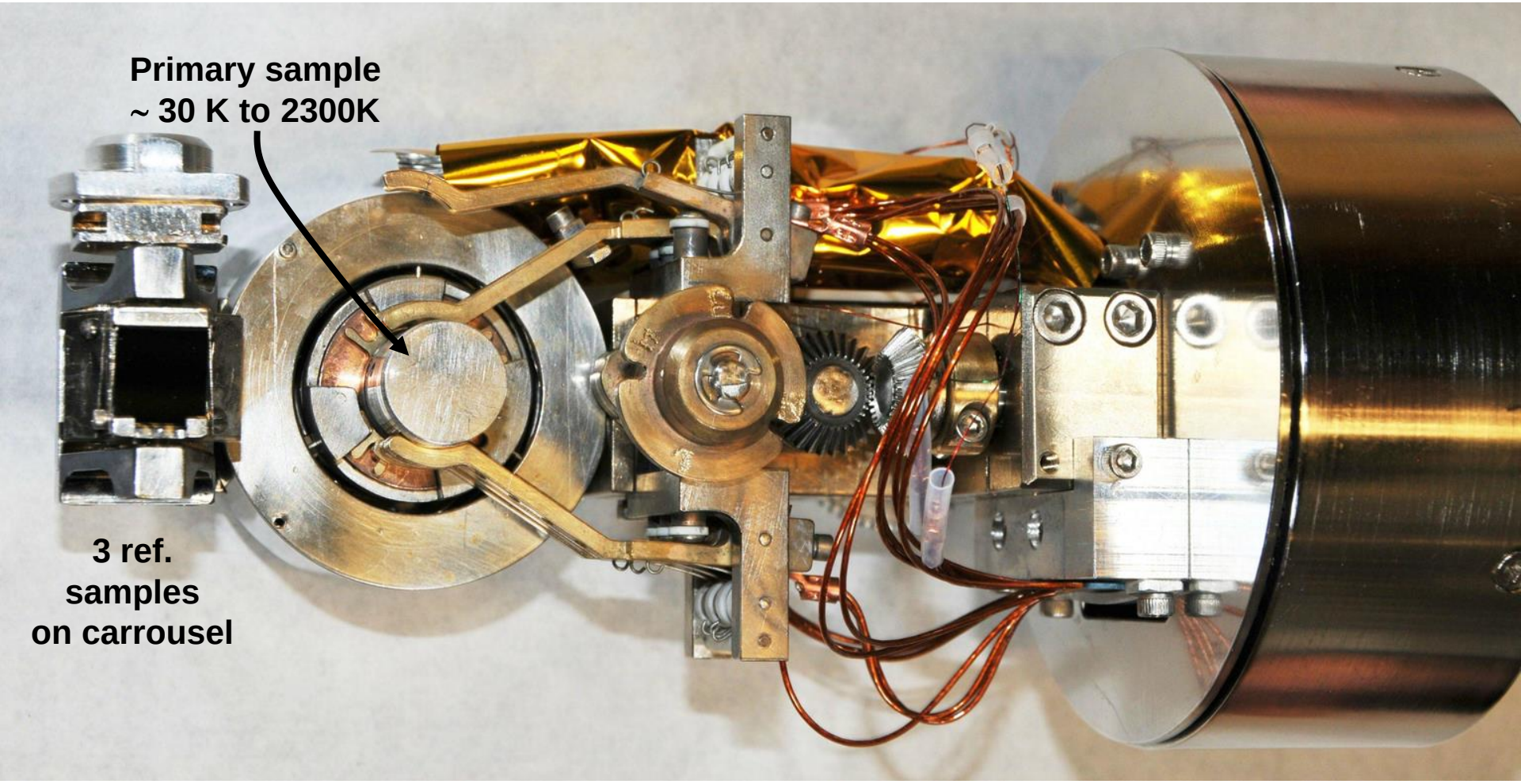


Custom-built two-axis sample goniometer with cryogen flowing directly to sample base



11a

Custom-built two-axis sample goniometer with cryogen flowing directly to sample base

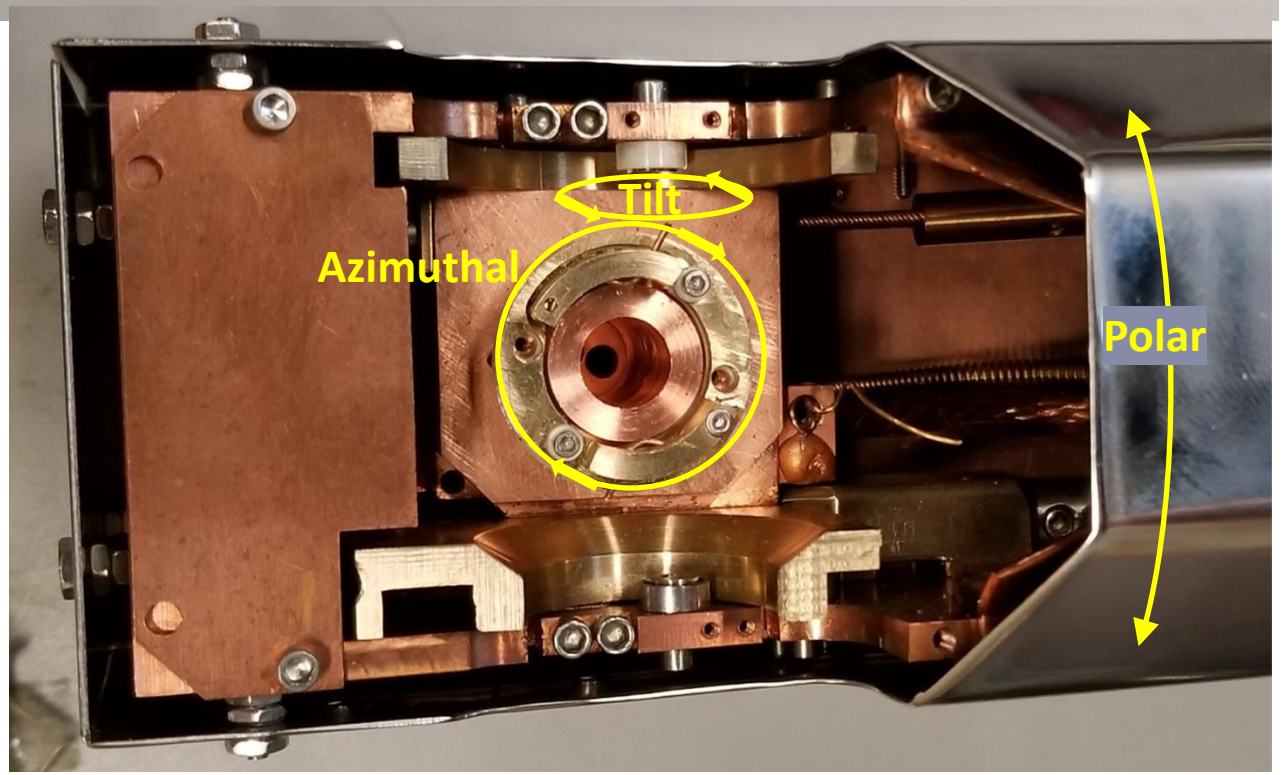


Major Pepper-manipulator upgrade: Overall and closeup view of sample end, with stainless heat shield. Rotations indicated. High precision 6-axis, ultralow T

11b

Low temp. standard sample holder to <10K

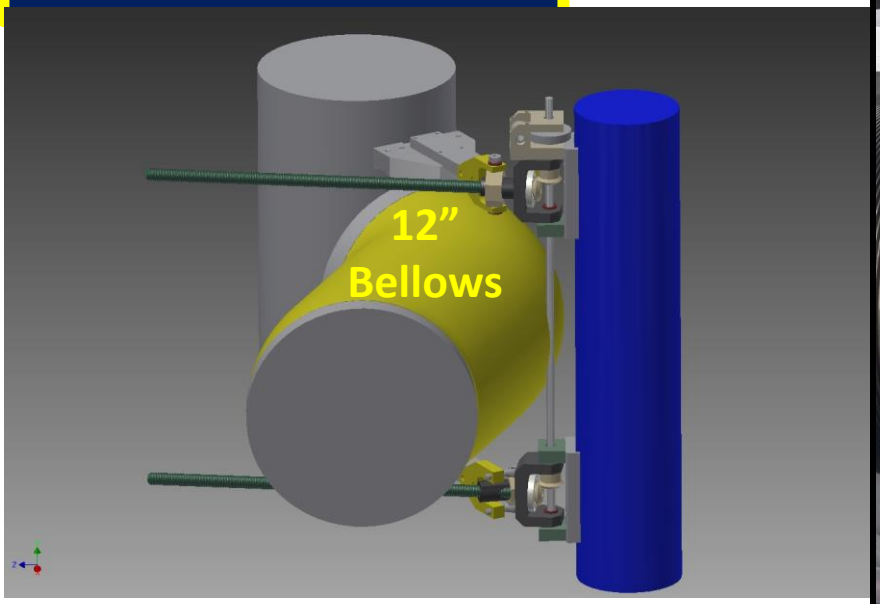
Plus high-temp. holder to ca. 1000K (To be added)



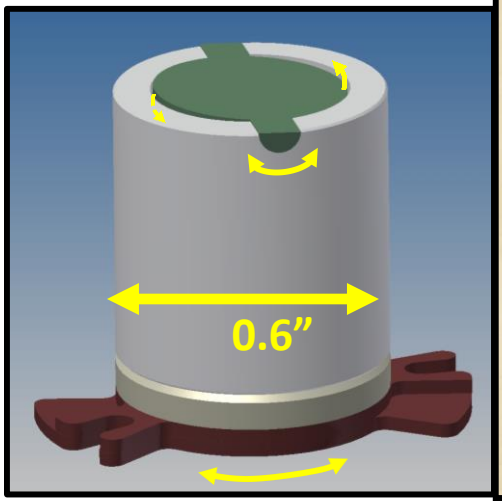
12(a)

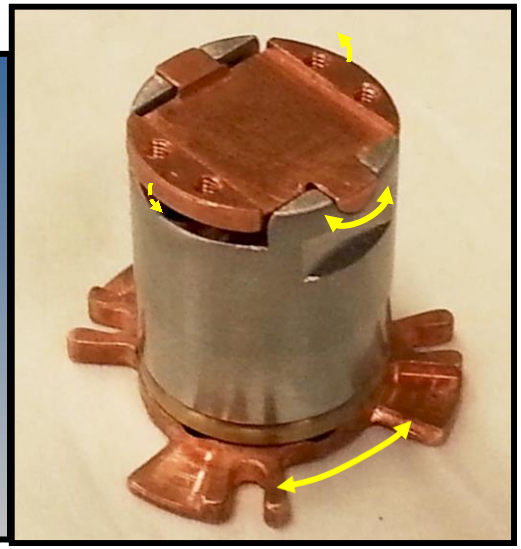
Other prior upgrades

- Non-torquing rotation mechanism for the main analysis chamber—to be automated



12(b)

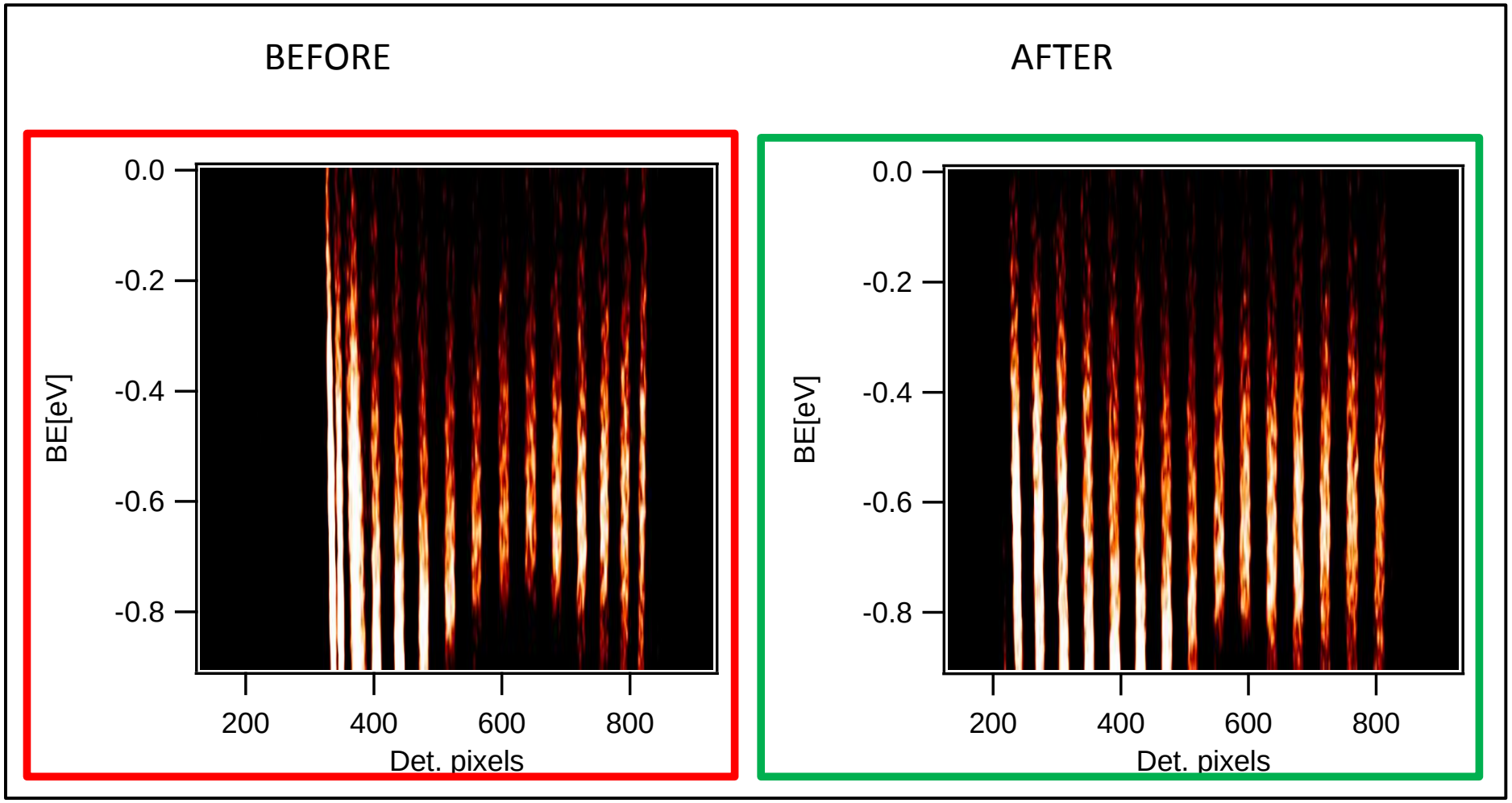




- Sample tilt mechanism 5-5-axis →temporary 6-axis sample holder

M. West, S. Nemsak, J. Pepper, …

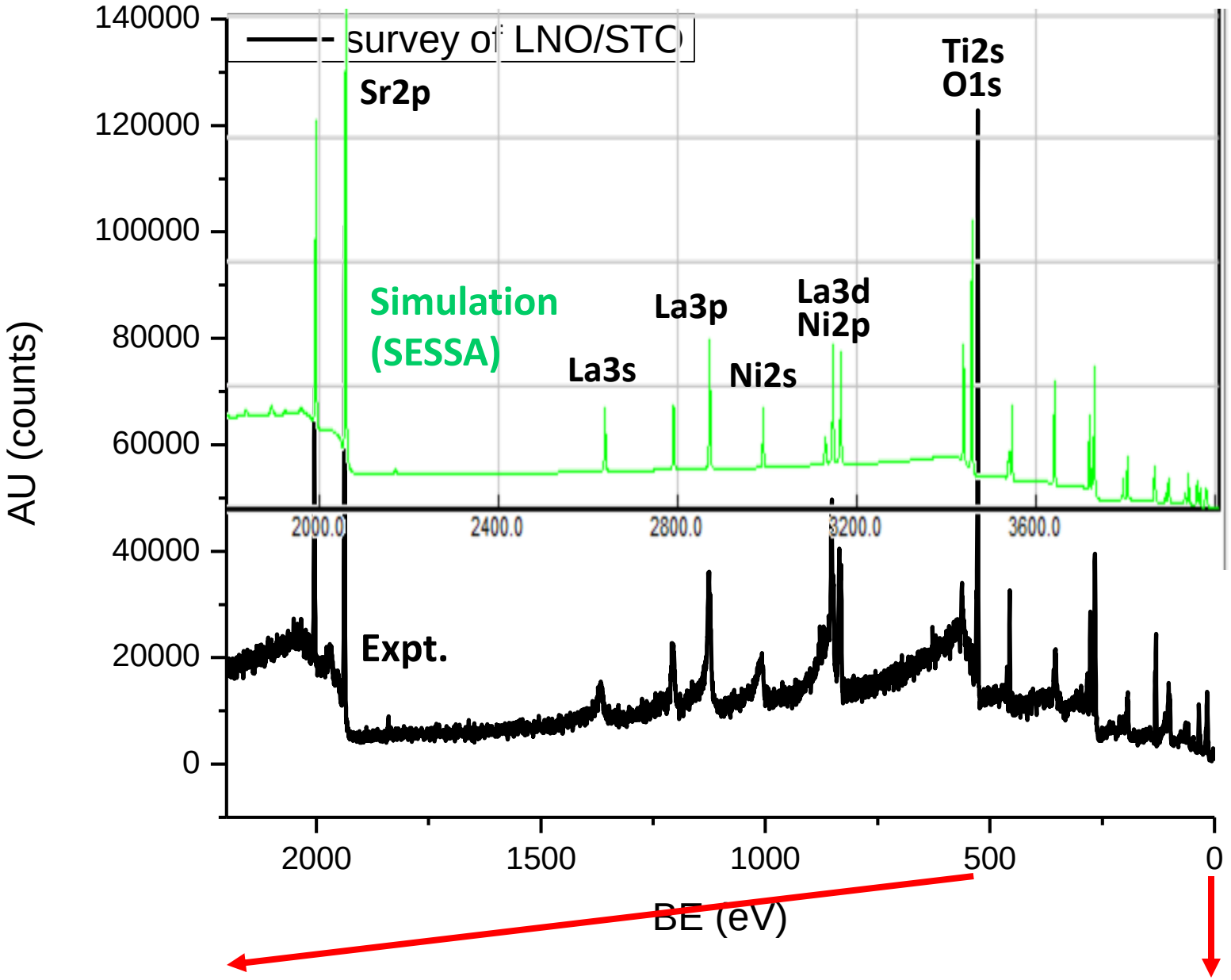
Recalibration of the angular resolution for hard x-ray photoelectron diffraction or HARPES measurements



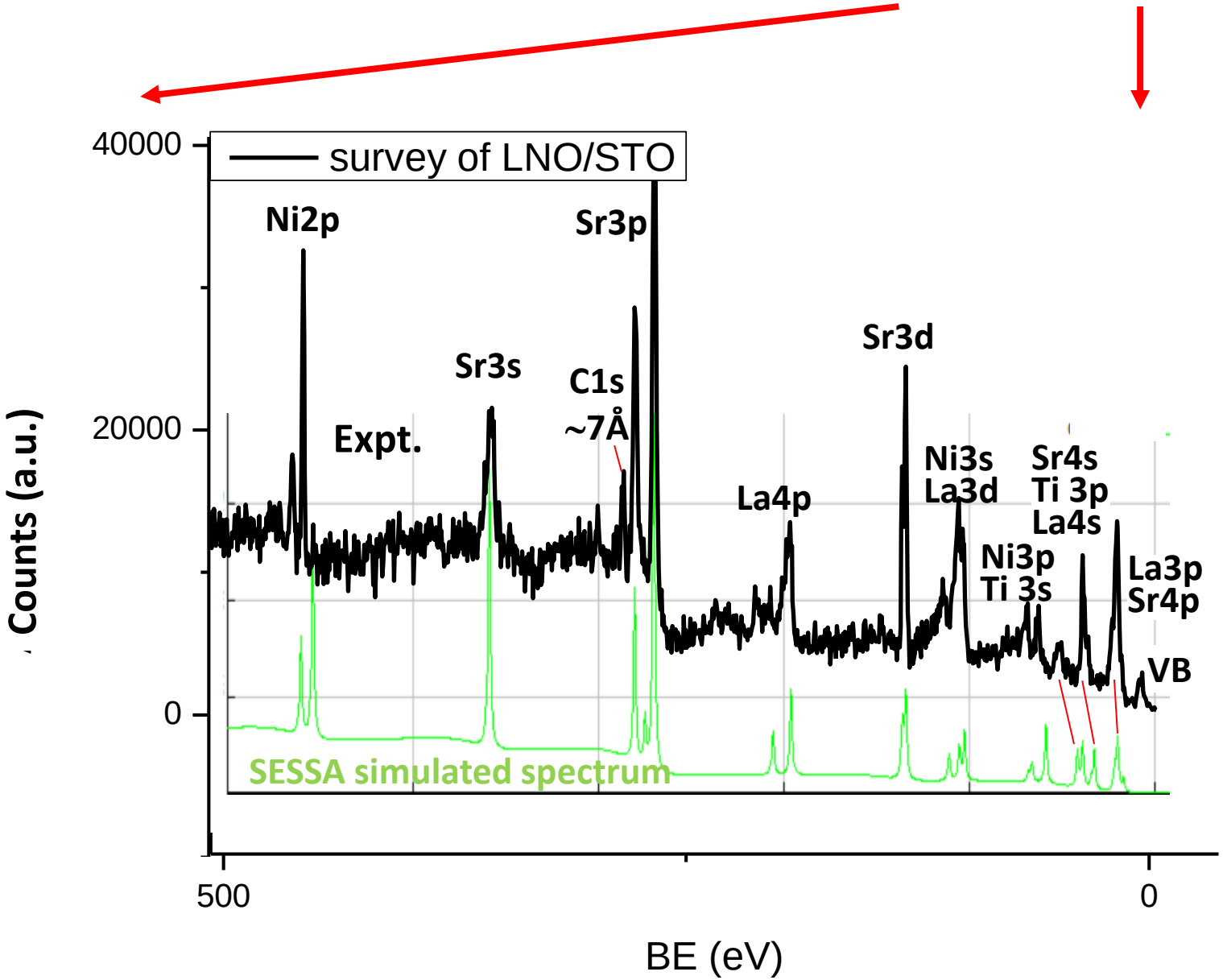
Resolution: 0.03-0.04 degrees over 2.5-5.5 keV

S. Nemsak, P. Karlsson (VGScienta)

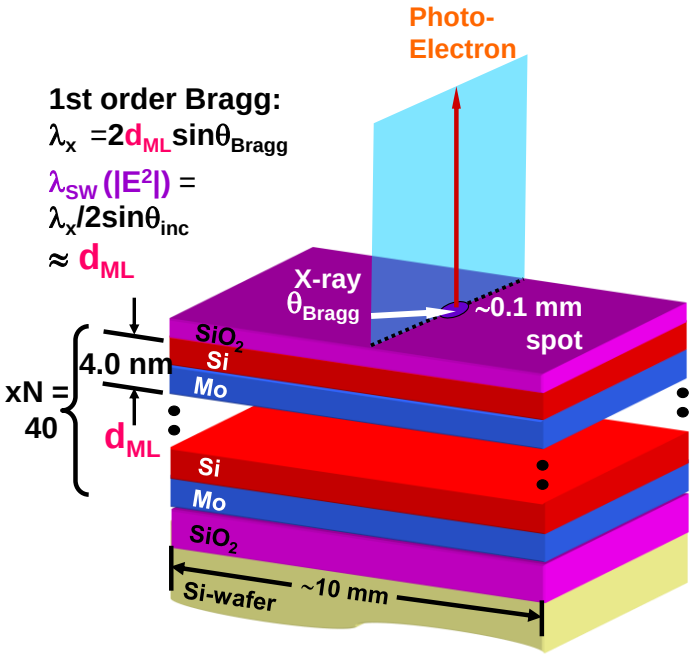
First survey from 1.4 nm LNO on STO, hν = 4000 eV



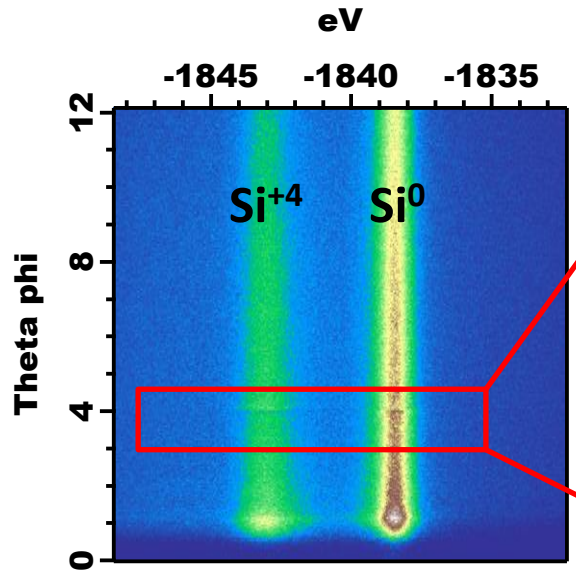
First survey from 1.4 nm LNO on STO, hν = 4000 eV



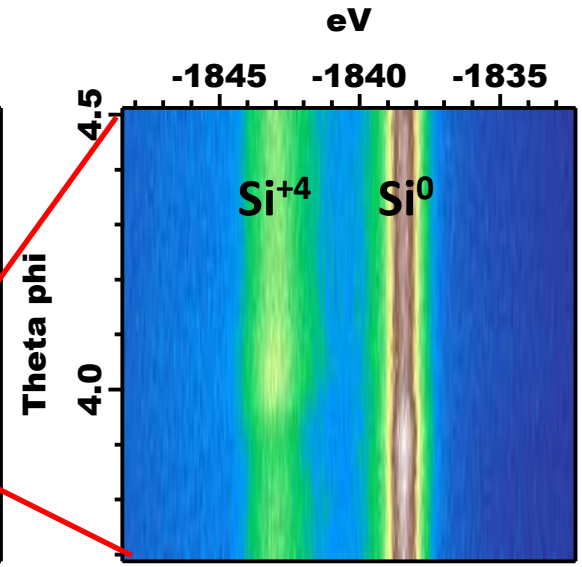
First standing-wave rocking curves from a test Si/Mo multilayer: photon energy 2300 eV

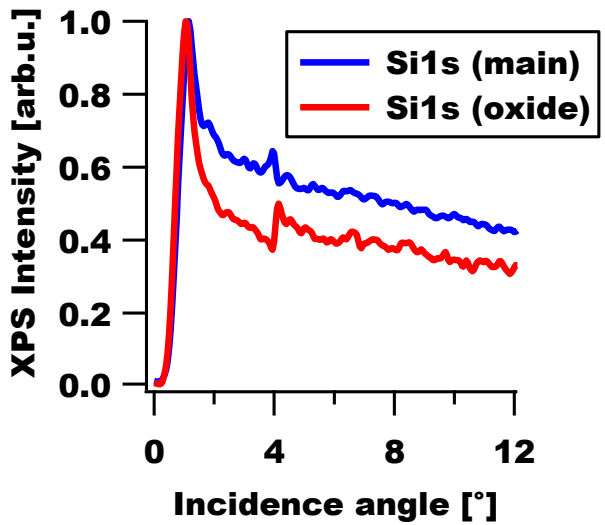


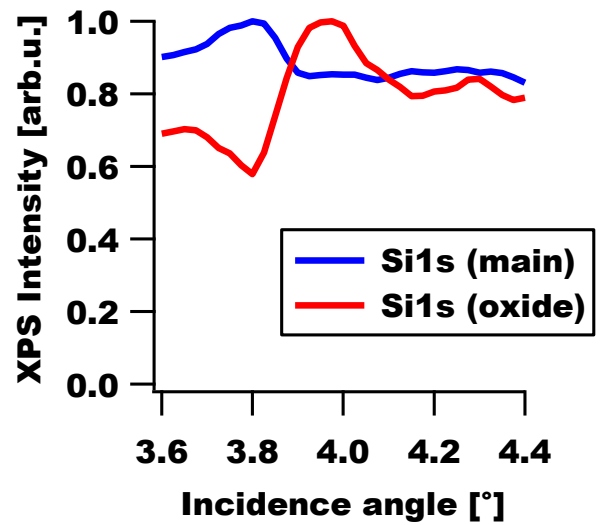
(a) Si 1s



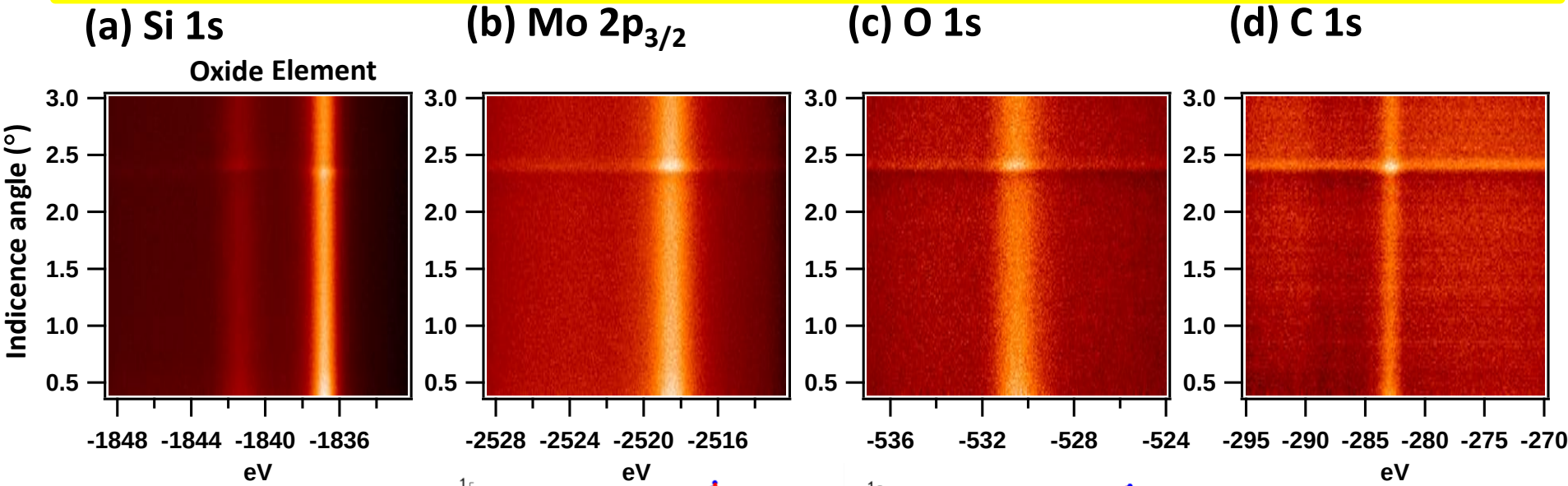
(b) Si 1s-zoom

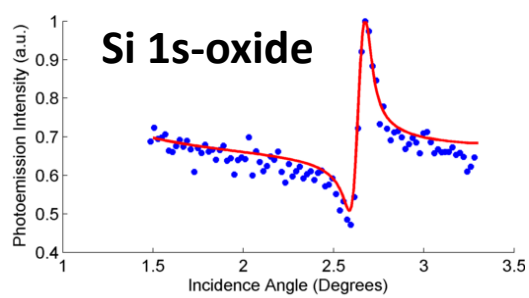


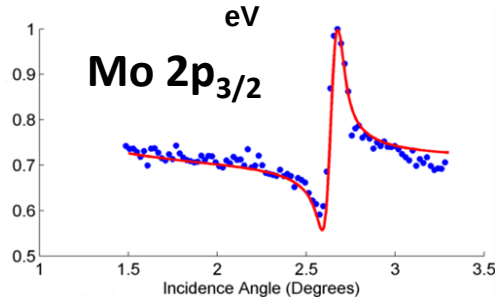


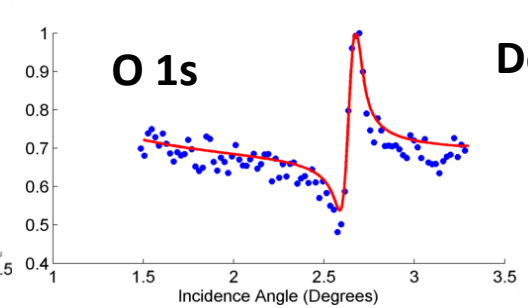


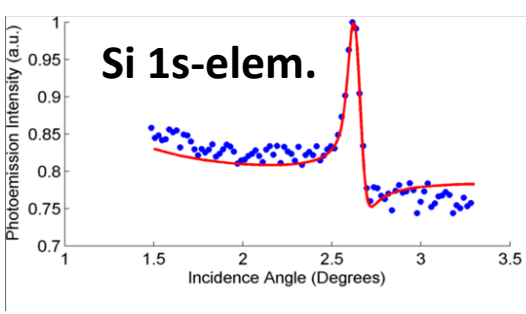
First standing-wave rocking curves analysis for a Si/Mo multilayer: photon energy 4000 eV-expt. and x-ray optical modeling (YXRO)





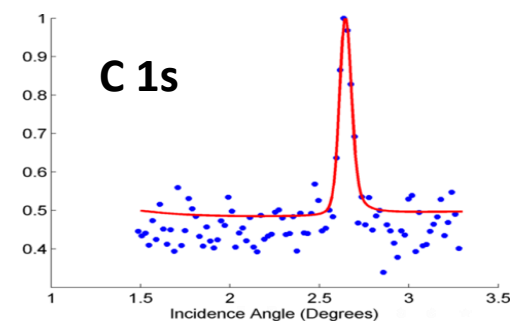






Core-level rocking curves:
.... Expt. —— Theory

G. Conti, S.Nemšák, H. Fang, C. Conlon, G. K. Pálsson, C. Battaglia, A. Javey, C.S. Fadley, Appl. Phys. Lett. Materials 6, 058101 (2018)



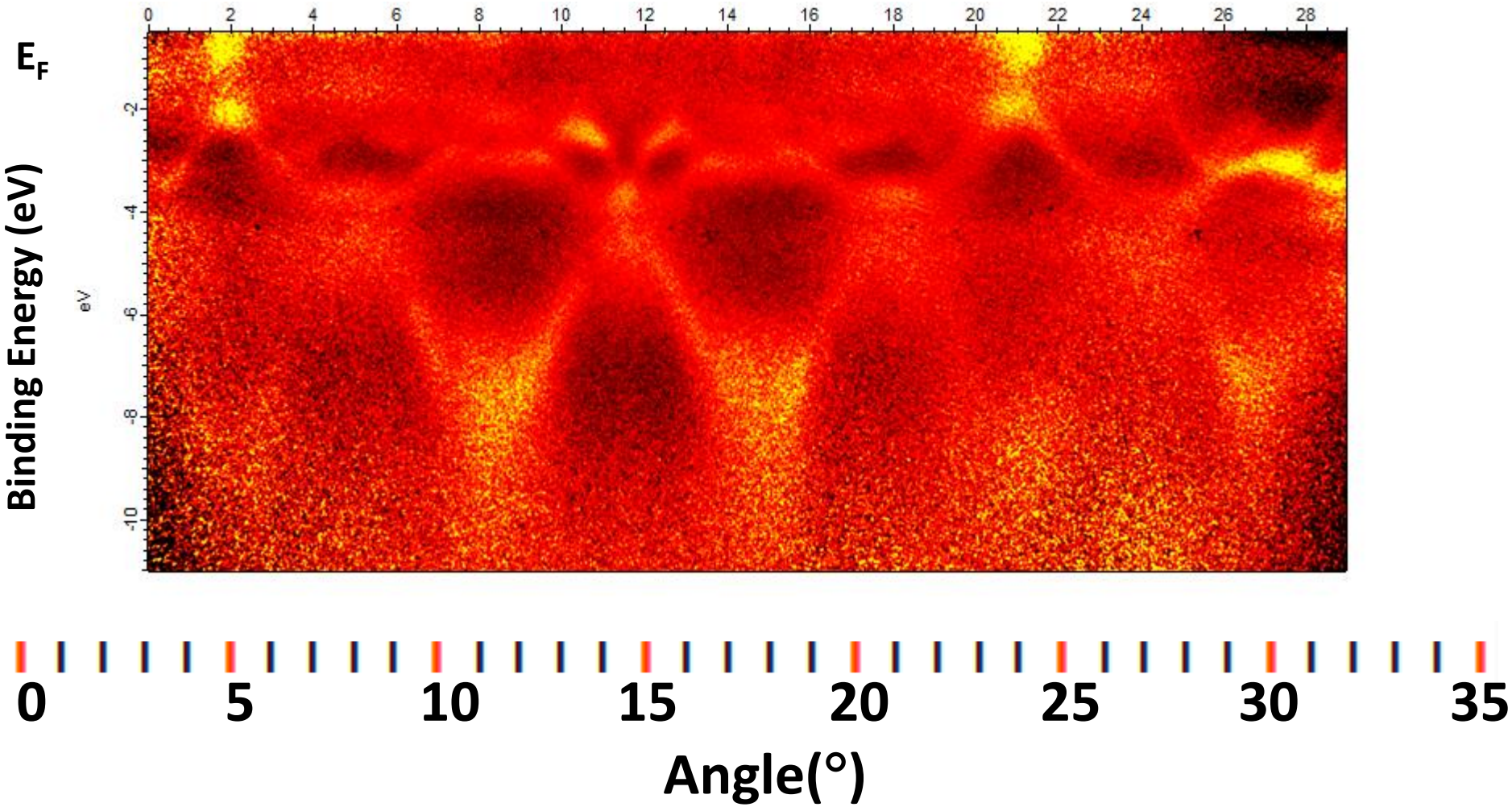
Derived structure

(Å)	Best Fit
SiO2	13.2
Si	20
MoSi2	6
Mo	4
MoSi2	8
Si	16.4

80x

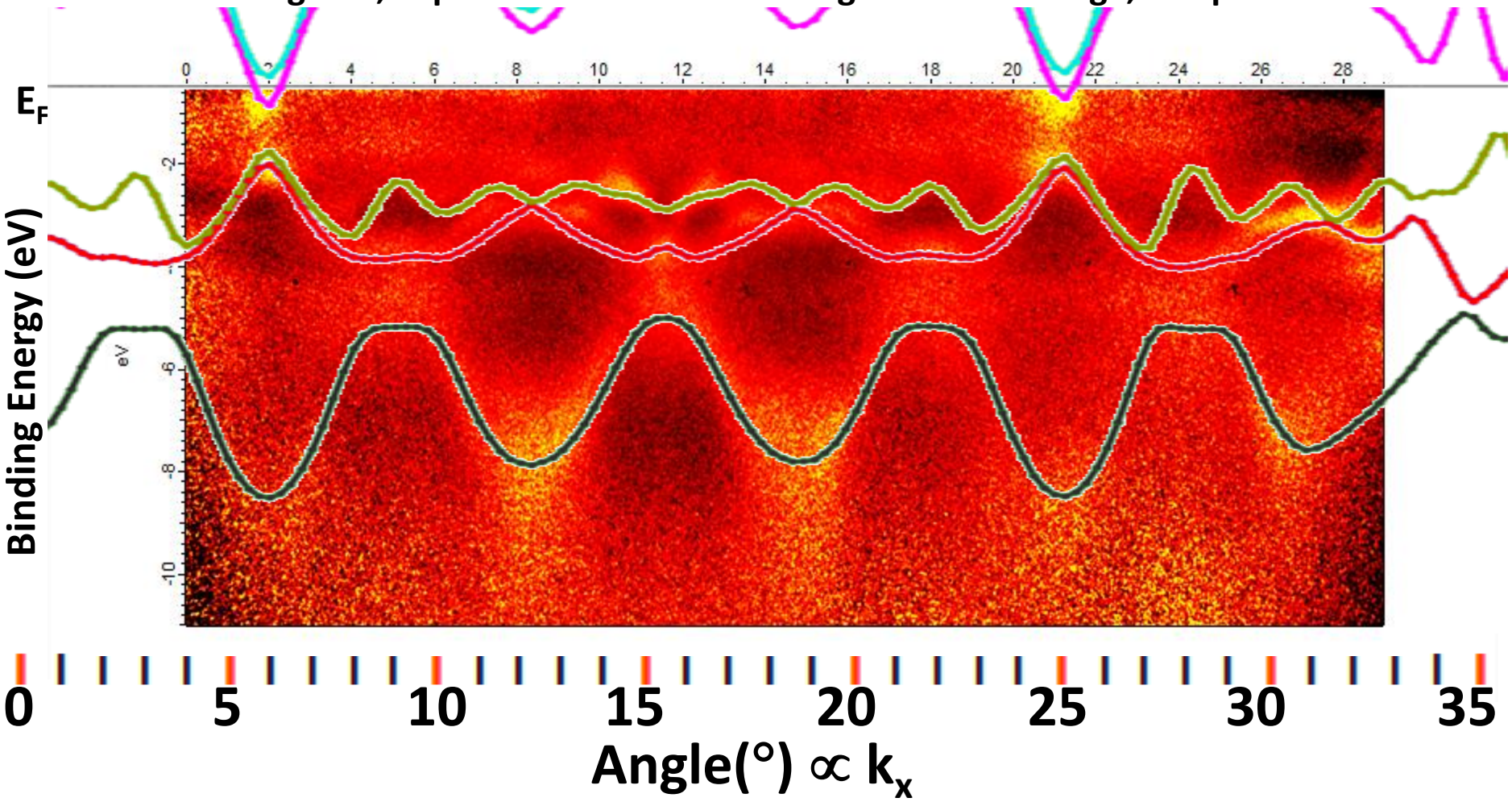
First hard x-ray angle-resolved photoemission (HARPES) from test-case W(110): photon energy 2500 eV, T ≈ 90K: expt.-multiple stitched images (Nemsak)

Incidence angle 2°; Expt'l. electron emission angle over 29° range, sample tilt 1.25°

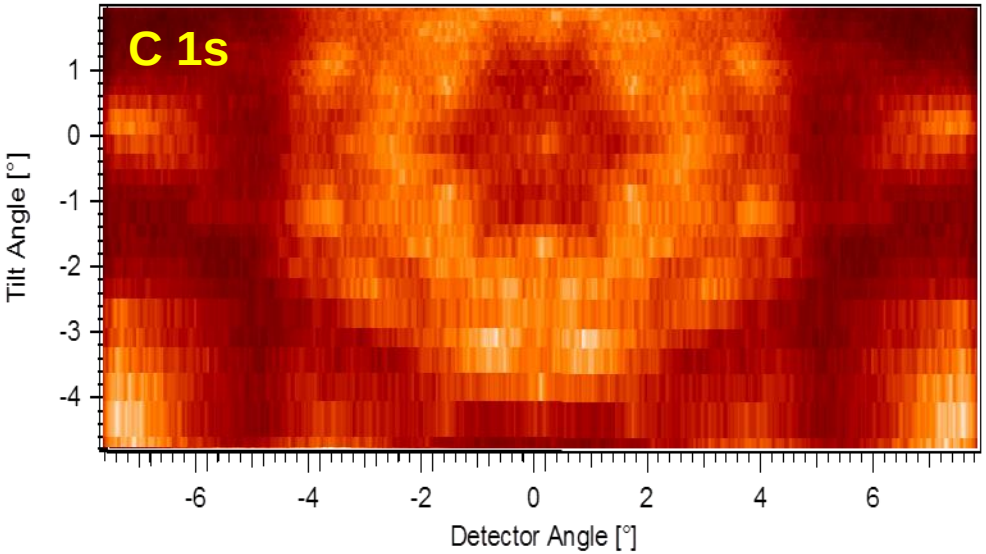


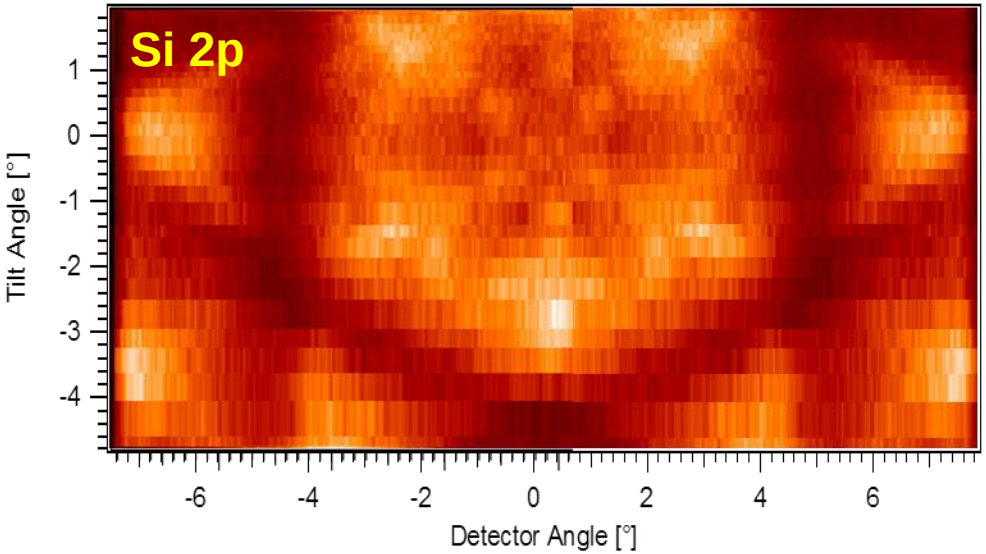
First hard x-ray angle-resolved photoemission (HARPES) from test-case W(110): photon energy 2500 eV, T ≈ 90K: expt.-multiple stitched images (Nemsak) versus free-electron final-state calculations (Plucinski)

Incidence angle 2°; Expt'l. electron emission angle over 29° range, sample tilt 1.25°



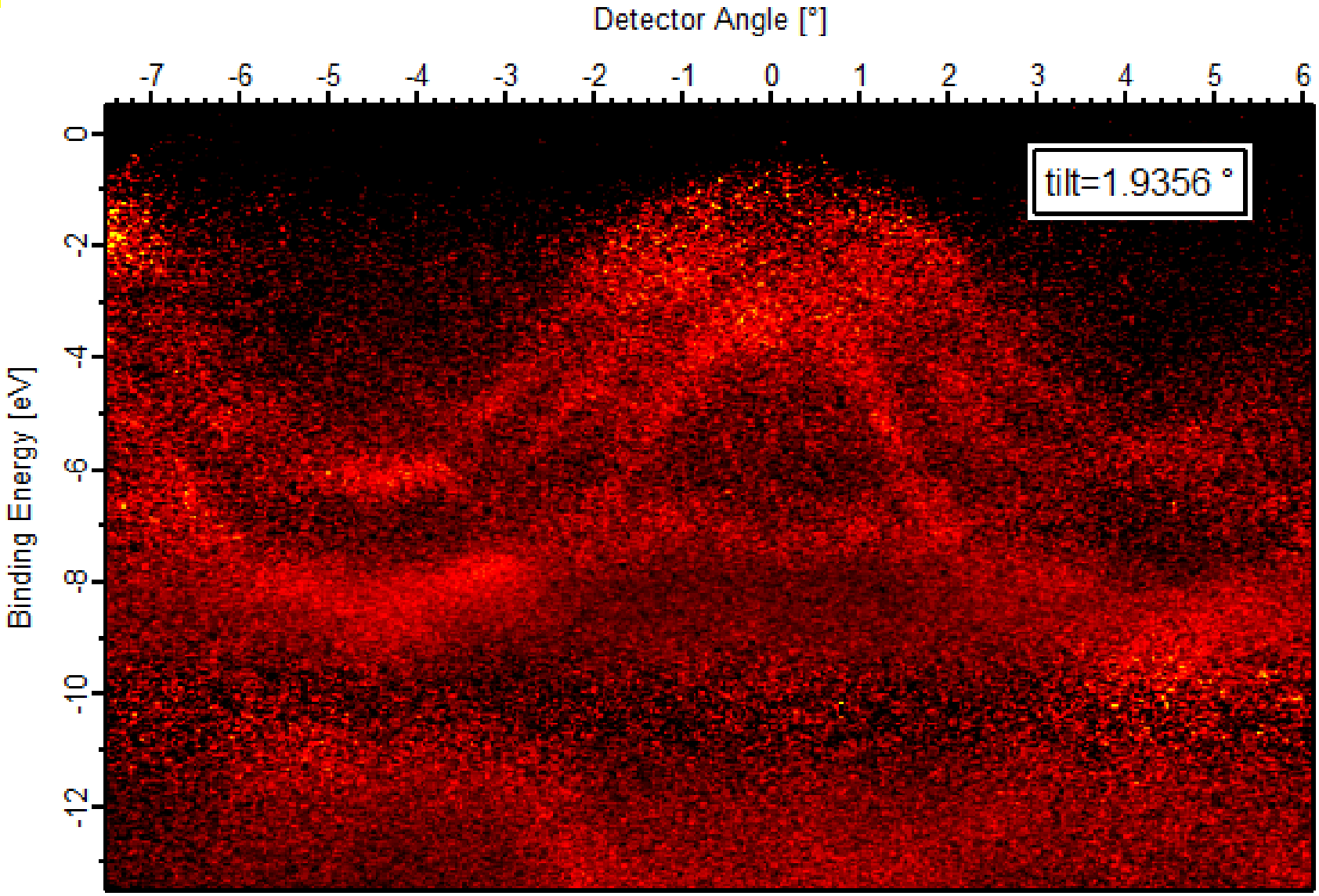
First 2D hard x-ray photoelectron diffraction (HXPD) making use of the new temporary tilt mechanism: SiC(0001)- hν = 3100 eV





S. Nemsak, M. West, C. Conlon, A. Rattachanatta, A. Keqi et al.

First 2D hard x-ray angle-resolved photoemission (HARPES) making use of the new tilt mechanism: SiC(0001)- hν = 3100 eV



Acknowledgements

Recent and current support is from the *Department of Energy*, Lawrence Berkeley National Laboratory, Materials Sciences Division, via the Magnetic Materials Program involving P.J. Fischer, F. Hellman, and J.B. Kortright, as well as the LBNL Center for X-Ray Optics; from the LBNL Laboratory Directed Research and Development Fund; from the *Department of Energy*, Office of Basic Energy Sciences, Materials Sciences and Engineering Division, Scattering and Instrumentation Sciences Program; from the *Army Research Office*, via a Multi-University Research Initiative entitled "Emergent Properties of Mott Interfaces" (http://mottmuri.mrl.ucsb.edu/index.html); from the *Peter Grünberg Institute* of the Jülich Research Center, PGI-6 Electronic Properties (group of C.M. Schneider); and from the *Labex APTCOM Project of the Triangle de Physique*, Paris.

Wayne Stolte and other Beamline 9.3.1 support has also been supported through the University of Nevada Las Vegas, by National Science Foundation Grant No. PHY-05-55699 and by the ALS.

Background references to prior group papers on hard x-ray photoemission systems at ALS 9.3.1 and other facilities before and during its shutdown

1) "X-Ray Photoelectron Spectroscopy and Diffraction in The Hard X-Ray Regime: Fundamental Considerations and Future Possibilities", C. S. Fadley, Nuclear Instruments and Methods A 547, 24-41 (2005), review in special issue edited by J. Zegenhagen and C. Kunz –overview.
2) "Bulk electronic properties of the bilayered manganite La1.2Sr1.8Mn2O7 from hard-x-ray photoemission", F. Offi, P. Torelli, M. Sacchi, P. Lacovig, A. Fondacaro, G. Paolicelli, S. Huotari, G. Monaco, C.S. Fadley, J.F. Mitchell, G. Stefani, and G. Panaccione, Phys. Rev. B 75, 014422 (2007) --ESRF.
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