HAXPES on the beamline ID32 at the ESRF and its application to buried interfaces in exchange bias heterostructures

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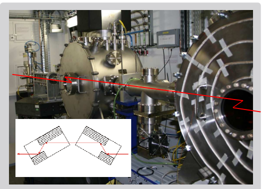
Science at the beamline ID32

- Surface X-ray diffraction: determination of surface structure, electronic properties and solid/electrolyte interfaces. In-situ electrochemistry
- X-ray standing waves (combined with XPS, SXRD): site specific XPS, imaging of surface structures and adsorbates
- Surface spectroscopy - SEXAFS; high energy resolution available for resonant scattering and absorption spectroscopy
- HAXPES

Beamline setup

- windowless beamline, 3 undulators (1 to ~35 keV)
- fixed-exit, liquid nitrogen cooled high-precision double crystal monochromator; with Si(111) crystals: photon energies 2.1 to 30 keV, energy resolution $\Delta E/E = 1.3\cdot10^{-4}$
- flux $> 10^{+13}$ $mm^{-2}s^{-1}$
- 2nd set of multilayer crystals installed for possible experiments below 2 keV
- main mono 2nd crystal piezo control: for vertical beam position feedback (diffraction experiments)
- flat mirror (Ni, Pd and SiO_2 coating) for higher harmonics rejection
- parabolic Be compound refractive lenses (2:1 focusing) → beam-size at sample 30 x 300 µm²
- Si Fresnel zone plates (30:1 focusing) → beam-size at sample 4 x 15 µm² at certain energies

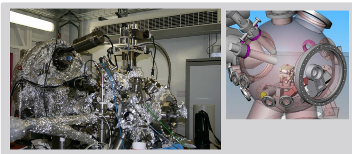


High energy resolution: post-monochromator

- high energy resolution at variable energy
- two axes, presently with Si(111), Si(220) and Si(311) channel-cut crystals
- dispersive arrangement (+/-, -/+) for high energy resolution down to $\Delta E/E = 10^{-6}$
- backscattering condition (fixed energies) to match photon beam divergence
- asymmetrically cut crystals for flexible energies (in preparation)

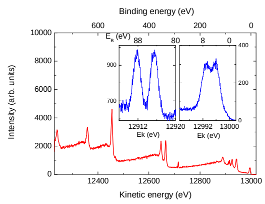
HAXPES

- Phoibos225 electron analyzer by SPECS:
  - for electron energies up to 15 keV
  - 2D delay-line detector (or CCD): excellent signal-to-noise ratio
  - different modes of operation
    - UPS, XPS and HAXPES
    - angular or spatial resolution in the non-dispersive direction
    - scanning or imaging mode
  - ultra high energy resolution (specifications: in UPS < 1meV, in HAXPES <15 meV)
- HAXPES chamber to be equipped with
  - 4-circle kappa goniometer
  - preparation chamber (early 2010)
  - He flow cryostat (summer 2009)

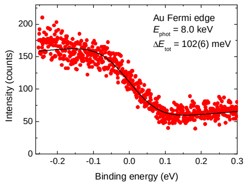


HAXPES commissioning (May 2008 – May 2009)

- HV power supply operational up to 15 kV
- data acquisition up to 13 kV successful

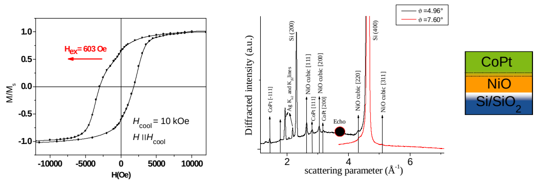


- energy resolution tests:
  - at $E = 8.0$ keV, using Si(444) post-monochromator (one axis only): $\Delta E_{phot} \sim 40$ meV, at room temperature
  - $\Delta E_{tot} = 102(6)$ meV → $\Delta E_{analyzer} < 30$ meV



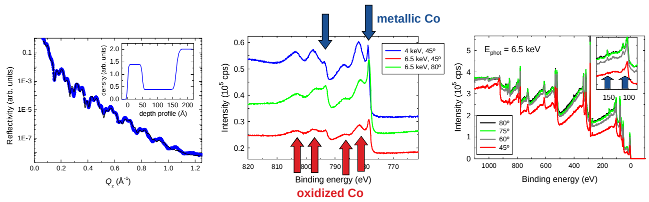
Exchange bias

- exchange coupling at the interface between a ferromagnetic (FM) and an antiferromagnetic (AFM) material
- shift of the hysteresis loop (exchange bias field, HE) is observed when the sample is cooled in a magnetic field through the Néel temperature: induced unidirectional anisotropy that arises from the direct exchange coupling at the FM/AFM interface
- the exchange coupling usually leads also to an increase in coercivity (Hc), as found in both nanoparticles and bilayer EB systems
- wide technological applications (spin-valve systems, GMR read heads, tunneling devices)
- complexity of the interface microstructure
- factors: interface roughness, thickness of both FM and AFM phases, interface stress etc.
- control of growth mechanism
- detailed characterization of the interfacial region



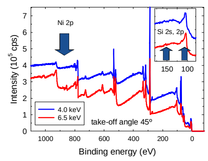
Exchange bias bilayers

- fcc-CoPt(FM)/NiO (AFM) fabricated by PLD on Si(100) substrate (+ native oxide)
- different thickness of NiO (10- 50 nm) + 5 nm of CoPt: ablation of a composite Co and Pt target
- laboratory characterization: EDXD and SQUID measurement
- X-ray reflectivity measurements: example - sample with 11 nm NiO + 5 nm of CoPt: roughness (SiO2/NiO) = 0.3 nm, roughness (NiO/CoPt) = 0.2 nm



Depth profiling using HAXPES

- probing depth increases with increasing photon energy: Ni 2p and Si 2s, 2p states discernible
- probing depth increases with increasing take-off angle
- Co 2p states: lower CoO signal (vs. metallic Co corresponding to Co bound to Pt) with increasing probing depth (both increasing E_{phot}, increasing take-off angle): CoO present mostly on the bilayer surface



Summary

- HAXPES operational at the beamline ID32 at the ESRF
- high energy resolution available at variable photon energy
- enlarged HAXPES possibilities in future - low temperature, X-ray standing wave technique, in-situ sample preparation and characterization, remote control operation, in-situ electrochemistry
- depth profiling of heterostructures of ~10 - 15 nm possible at energies above 5 keV: quantitative analysis difficult in heterostructures with (possibly) repeating chemical composition
- combination of HAXPES with reflectivity measurements desirable for valid structural model

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