Commissioning of the Solid/Liquid Analysis System SoLiAS

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The SoLiAS experimental station is a versatile preparation system dedicated to the investigation of solid/liquid interfaces with synchrotron radiation induced photoelectron spectroscopy SPS. During 2002 the system was installed at BESSY and successfully commissioned at beamlines TGM 7 and U49/2. With the SPECS Phoibos 150 analyser an overall resolution below 200 meV is reached (Fig.1). The Phoibos 150 is retractable allowing free rotation of a VG ADES 500 ARUPS analyser installed in addition. In order to adapt the station to various beamlines it can be inclined horizontally versus its frame by 7°.

The UHV analysis of solid/liquid interfaces may be performed with SoLiAS by three complementary routes:

i) synthesis of a model electrolyte in vacuo by coadsorption of solvent and redox species onto LN2 cooled sample surfaces,

ii) emersion of a specimen from a liquid electrolyte and transfer to vacuum under inert (N2) atmosphere,

iii) creation of frozen electrolytes by cooling emersed samples before pumping to UHV conditions.

Interface engineering by means of electrochemical processes is of great technological interest. For the analysis of solid/solid interfaces SoLiAS is equipped with

iv) vacuum preparation like heat treatment, sputtering, or PVD/CVD of contact materials.

Thus SoLiAS allows by emersion experiments ii) to analyse surfaces after technologically relevant wet preparation processes like etching [1,2], electrochemical oxidation and reduction [3], or electrodeposition of contact materials [4]. While wet chemical processes may be complex reaction sequences and ii) allows for the analysis of a final situation only, emersion experiments may be complemented by adsorption experiments i). By adsorption the interaction of a defined single electrolyte species with a surface may be investigated [5], by coadsorption of solvent and redox species the cooperative interaction with a specimen is accessible and may provide insight into intermediate reaction steps [1]. Furthermore the interaction of the solvent and an electrolyte species may be investigated by coadsorbing

![Fig.1: Uncorrected width of Ag Fermi edge at RT measured with the Phoibos 150 analyser at beamline U49/2 with pinhole 1mm x 1mm. 1st harmonic red, 3rd harmonic blue.](image-url)
electrolyte constituents on inert surfaces [6]. Cooling of the sample before evacuation iii) allows to analyse soluble and volatile reaction products. In addition iii) allows to measure the occupied electron states of real (albeit) frozen electrolytes. Not only the impact of wet chemical processes on interface formation may be analyzed by UHV deposition iv), but also a comparison of wet chemically produced interfaces to vacuum deposited interfaces may be drawn [7]. The unique combination of integrated preparation options of SoLiAS is complemented by a fast entry load lock permitting high flexibility in sample usage.

While the data of the cited references were taken at different experimental systems with one of i)-iv) preparation facilities temporarily in use at a beamtime, all four are now permanently available at the same time. The presented data of fig. 1 to 5 were actually taken with SoLiAS, exemplifying its versatility. LN$_2$ cooled transfer between adsorption chamber and analysis chamber is demonstrated by H$_2$O adsorption onto a vacuum cleaved WSe$_2$ single crystal. Fig.2 shows the valence band emissions of orbitals 1b$_2$, 3a$_1$, and 1b$_1$, the fingerprint of molecularly adsorbed water. The spectra were taken with a photon energy of hv=30 eV at beamline TGM7.

Fig.2
Valence band spectra of H$_2$O adsorption on a LN$_2$ cooled WSe$_2$ crystal. The emissions 1b$_2$, 3a$_1$ and 1b$_1$ indicate molecular adsorption. Spectra taken at beamline TGM7.

SPS on emersed samples is demonstrated by etching off the natural oxide of a GaAs (100) wafer surface in a solution of 3% HCl in water. The As and Ga 3d core level spectra of Fig.3a and b were taken at the U49/2 with highest surface sensitivity. The etched surface is covered with a layer of elementary As and As-H. Some remnants of intermediate AsCl and GaCl reaction products are found which can be rinsed off by deionized water as we have shown before.

Fig.3 a,b
As and Ga 3d core levels of GaAs (100) native oxide and etched in 3% HCl in H$_2$O. Spectra taken at beamline U49/2.
Electrochemistry on defined vacuum-prepared surfaces immersed and emersed in clean N\textsubscript{2} atmosphere without contact to ambient air is demonstrated by a GaAs (100) surface vacuum cleaned by sputter-anneal cycles. Detailed comparison of the surface composition after contact to the solvents water or isopropanol is shown. In January 2003 also the freezing in of volatile reaction products by cooling of an emersed electrode was demonstrated.

Fig. 4 a,b
As and Ga 3d core level of sputter cleaned GaAs (100), and this surface after contact to liquid H\textsubscript{2}O or isopropanol.

XPS analysis of wet chemical etching of GaAs by Br\textsubscript{2}/H\textsubscript{2}O: Comparison of emersion and model experiments.

Synchrotron-Induced Photoemission of emersed GaAs Electrodes after Electrochemical Etching in Br\textsubscript{2}/H\textsubscript{2}O Solutions.

Synchrotron-Induced Photoemission of GaAs Electrodes after Electrochemical Treatment in Aqueous Electrolytes.
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Institute of Materials Science TU-Darmstadt
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Pulse Plating of Pt on n-GaAs (100) Wafer Surfaces: Synchrotron Induced Photoelectron Spectroscopy and XPS of Wet Fabrication Processes

H\textsubscript{2}O adsorption on the layered chalcogenide semiconductors WSe\textsubscript{2}, InSe, and GaSe

A Photoemission Study of the Solvent-Solvent Interaction: Stepwise Solvation of Na\textsuperscript{+} by Coadsorbed H\textsubscript{2}O on a Semiconductor Surface Induced by the Synchrotron Photoemission Source.

Electronic Properties of Chemically Etched CdTe Thin Films: Role of Te for Back Contact Formation