Poster abstracts

Sponsor presentations
HAXPES on buried interfaces in the graphite negative electrode of a Li-ion battery

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Abstract
Graphite is often used as the negative electrode material in commercial Li-ion batteries. In the first insertion of lithium-ions into the graphite a Solid Electrolyte Interphase (SEI) is formed. The SEI protects the electrode from unwanted side reactions, e.g. co-intercalation of solvent molecules; it also leads to irreversible loss of battery capacity. The SEI composition is complex, including organic and inorganic compounds, depending on the chemistry in the electrolyte. We have earlier shown that especially the anion in the lithium salt in the electrolyte influences the SEI thermal stability. There is a debate if there is a depth gradient in the SEI and discussion about the “real” chemical content. The ambiguities in literature show the difficulty to characterize these thin layers (in the range 5-20 nm). All published studies are based on ex situ studies done in post mortem by ripping the battery apart and depth profiles have mainly been obtained using Ar⁺ etching. We show how the combination of soft X-ray PES and HAXPES can give more reliable depth profiling results. We show, for the first time, that we can “see” the interface between the SEI and the lithium intercalated graphite. Depth profiling of the C1s, F1s, O1s, P2p peak are seen in the Figure.

Keywords: 01 P
Hard X-ray Photoemission and Energy Research: Applications of PES and XAS

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Abstract
Fuel performance issues will play an important role in both light water reactor (LWR) sustainability and in the design of fuel forms for new reactor designs. The need to develop fuels that are more robust under accident scenarios is even more important after the fuel failure observed at the Fukushima Dai-ichi Nuclear Power Plant. Both fuel development and performance modeling require a better scientific understanding of irradiation effects in fuels. The physicochemical behavior of TRISO fuel pellets and compacts under irradiation has not been well studied. One of the reasons for this is that it has been very difficult to study irradiated fuels with modern scientific techniques. Irradiations that affect the entire volume of the sample and can be studied with bulk probes tend to result in high activity samples. Irradiations that don’t activate the sample have historically only damaged a fraction of the sample which makes the study of the sample behavior difficult. Irradiation with low energy protons and high energy ions does lead to radiation damage that can be used to infer the damage effects of neutron irradiation. There are two advantages of ion irradiation. First, damaged samples that have not been activated and that can be studied anywhere are created. Second, high total doses can be achieved in reasonable time frames. The major disadvantage is that the damage layer can be less than a micron thick. Thin damage layers can be difficult to study with modern probes. By using electrons with tailored kinetic energy as a probe [Terry and Sickafus, unpublished], it is possible to limit the information depth in the ion irradiated sample to the region below the surface layer but above the cutoff of radiation damage. This effect minimizes the surface contribution and as long as the escape depth is not too large, it is possible to stay within the region damaged by the ion beam. This requirement is met by monitoring electrons with kinetic energy in the range of 2 to 12 keV, high kinetic energy. Both the local atomic structure of the damaged layer and the electronic structure can be determined using X-ray Absorption Spectroscopy (XAS) and Photoelectron Spectroscopy (PES), respectively. The changes in local structure and chemical state can be compared with models of radiation damage to improve the understanding of fuels and structural materials at high dose. The first measurements using the High Kinetic Energy instrument will be on ion-irradiated UO₂ thin films to study the Soret effect; the reaction chemistry of Pd with ion-irradiated SiC to understand Pd attack and failure in TRISO fuels; and to study the changes in local atomic structure in ion-irradiated Fe structural materials at high dose. These experiments will commence in August 2013.

Keywords: 02 P
HAXPES of silicon negative electrodes in Li-ion batteries cycled with different electrolytes

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Abstract
HAXPES has shown to be very useful for depth profiling through electrode/electrolyte interfaces in Li-ion batteries. In this presentation, the focus will be on silicon which is expected to be the next generation negative electrode material in a commercial battery. There are, however, many obstacles that need to be handled before we see this material in, for example, our mobile phones. One obstacle is the volume expansion (400%) occurring during lithium alloying. This leads to cracking of the electrodes and loss of particle contact which means fast battery fading during cycling. One solution is to improve the different binder materials used to attach the particles to the current collector allowing for a good electronic conductivity between the particles. Another obstacle is the reactivity between different electrolytes and the silicon particles. We will show how the anion in the lithium salt leads to different reactions in the surface of the silicon particle and how this influence battery performance. We will show how HAXPES can give information on where in the surface the reactions between the electrolyte and the silicon surface take place during charge and discharge. We will show how the surface chemistry will evolve during long-term cycling. In Figure 1 the electrochemical cycling of a silicon electrode using the salts LiPF₆ (Fig.1a) and LiFSI (Fig.1 b) give very different result in terms of cycling stability. LiPF₆ is today used in commercial batteries but it is easily hydrolyzed leading to formation of HF. LiFSI (LiN(SO₂F)₂) is a new salt not leading to formation of HF.

Figure 1. The Si2p signal of silicon electrode cycled with LiPF₆ (a) and LiFSI (b) in a Li-ion battery.

Keywords: 03 P
Pronounced effects of interchannel coupling in high-energy photoionization

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Abstract
The 3d core level photoemission of metallic Ag and In was measured over a wide energy range including the 2p ionization thresholds. The intensities (photoionization cross sections) of the 3d\textsubscript{5/2} and 3d\textsubscript{3/2} lines were observed to modulate significantly with photon energy, both absolute and relative, and is most pronounced in the vicinity of the 2p thresholds, i.e., at photon energies about an order of magnitude above the 3d thresholds. Calculations based on the relativistic-random-phase approximation show that this effect is due to interchannel coupling of the 3d photoionization channels with the 2p channels affecting the cross section over a wide range of energies. It is argued that this is a general phenomenon in high-energy photoionization throughout the periodic table.

Keywords: 04 P
Near L-edge photoionization in Xenon: Study of multiple Auger decay, lifetime interference and PCI effect

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Abstract
We present preliminary results of x-ray photoemission and absorption experiments collected during the commissioning of our newly operational HArd X-ray Photo Electron Spectroscopy (HAXPES) setup located on the GALAXIES beamline at SOLEIL [1, 2]. The photon energy provided by this beamline covers the 2.4 - 12 keV range, and electrons of kinetic energy up to 12 keV can be analyzed with state-of-the-art resolution. The HAXPES station consists of a UHV analysis chamber designed for investigating both solid samples and gases. The new gas cell has liquid nitrogen cooling facility for reducing the Doppler broadening.
To exploit the newly accessible wide photon energy range combined with low bandwidth and high resolution of the spectrometer, we investigated the resonant Auger decay [3] of Xenon following L-edges photoexcitation. Near the L- edges there are two distinct processes such as the normal Auger process and the spectator Auger process that lead to ejection of electrons. Just above threshold, the normal Auger lines are distorted and shifted due to postcollision interaction (PCI) between escaping photo- and Auger electrons. Just below threshold, narrow spectator or resonant lines are observed, associated with the electron excitation to Rydberg orbitals. Under resonant Raman condition (photon band width << core-hole lifetime width), it is likely to resolve various higher Rydberg transitions. The photon bandwidth provided by GALAXIES beamline, Synchrotron SOLEIL is 1eV and the spectrometer resolution is about 0.5eV which are much narrower than the L₃ hole lifetime width (2.8eV). This allows us to record the Auger electron spectra following excitation of L₃, L₂ and L₁ edges in Xenon under resonant Raman condition. The processes near threshold are investigated in detail. We successfully resolved the 3d²5d, 3d²6d and the 3d²7d final states which were not resolved yet due to resolution issues. We have found the signature of lifetime interferences in the cross-section of 3d²6d. We also studied the PCI effect [4] in Auger decay processes

References
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Keywords: 05 P
Modifying the Energy Levels in Hole Conducting Molecules for Dye-sensitized Solar Cells

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Abstract

Dye-sensitized solar cells are a promising alternative for conversion of solar energy to electricity. Important for the function of the solar cell is the relation between the energy levels in the different materials in the device. We show how direct measurements of the energy level alignment can be performed using HAXPES. In focus of the study are hole conducting molecules and how dopants or other additives can modify the energy level alignment. In particular, the hole-conducting molecule spiro-OMeTAD was studied with and without addition of a Cobalt complex. Different Co-complexes with different functional groups giving a variation in redox potential was used to follow the doping behaviour of the hole-conducting molecule. The Co-complex was mixed with the hole conductor in solution and thin films were spin-coated onto conducting glass. The system was studied with hard X-rays to monitor the bulk of the molecular film. The results show a significant change in energy levels of the hole-conductor (spiro-OMeTAD) towards lower binding energy when the dopants are added. The changes suggest that there is a redox reaction taking place when adding the dopant resulting in a partially oxidized hole-conductor and that the energy level shift is dependent on the redox potential of the Co-complex.


Keywords: 06 P
HAXPEEM: Photoelectron Spectromicroscopy with Hard X-Ray Excitation

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Abstract

Hard x-ray photoemission spectroscopy (HAXPES) as a tool for bulk sensitive photoemission [1] lacks lateral spectroscopic information needed for inhomogeneous or laterally structured samples. To extend this technique, the NanoESCA [2] photoelectron microscope, up to now used for imaging XPS, k-space imaging or workfunction mapping, has been upgraded to be used for hard x-ray photoemission electron microscopy (HAXPEEM) with kinetic energy up to 10\textup{keV} [3]. For the high kinetic energy the extraction voltage has been raised to 24\textup{keV} to enable an easy sample biasing scheme. Image stacks or spectra can be acquired continuously from threshold photoemission towards 10\textup{keV}. A simple model predicts the change of magnification with kinetic energy, that is confirmed by simulations. During an energy scan the focusing of the objective lens is maintained automatically by using look-up tables and focusing parameters obtained from threshold imaging. First results were obtained during a beamtime at PETRA III [3]. The figure (a) shows a checkerboard-patterned Au/Si sample imaged with Au\textup{4f}\textsuperscript{7/2} core level electrons at 6.43\textup{keV} kinetic energy. The image proofs the sub-micron resolution even at very high energy and for long acquisition time. Bulk sensitivity is demonstrated at the Si\textup{2p} peak. Whereas the spectrum taken at h\nu=4.9\textup{keV} (Fig.(b)) shows predominantly the elemental silicon signal and a shifted oxide peak, consistent with a native oxide layer of 10\textup{Å}, the spectrum at h\nu=400\textup{eV} acquired with the same sample and instrument at Elettra [4] is dominated by a mixture of different oxidation states of silicon, probably resulting from the non-prepared micro-strutured surface.

![Image of Au/Si sample](image_url)

Figure: (a) Image of the Au/Si sample using Au\textup{4f}\textsuperscript{7/2} electrons at 6.43\textup{keV}. The image acquisition time was 2 h. (b) Si \textup{2p} spectra for hard (h\nu=4.9\textup{keV}, taken at PETRAIII, beamline P09 and soft x-ray excitation (h\nu=400\textup{eV}, taken at the Elettra NanoESCA-beamline).


Keywords: 07 P
HIKE: the HAXPES Facility at BESSY II

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Abstract

In the last decade hard x-ray high kinetic energy photoelectron spectroscopy (HAXPES) has lead to a break-through in the field of photoemission due to its non destructive way of investigating the bulk electronic properties of materials and in particular buried interfaces in layered systems. Nanolayered systems are the keystones of current and future devices. The electronic, magnetic and magneto-transport properties of these structures are determined not only by the characteristics of each layer, but also by their interfaces.

The HIKE facility at the Berliner synchrotron light source BESSY II successfully combines the bending magnet source of the KMC-1 beamline [1] with a new generation electron spectrometer optimized for high kinetic energy electrons. In the present contribution several experiments performed at the HIKE HAXPES user facility [2] will be detailed with emphasis on the performance and abilities of the technique.

References


Keywords: 08 P
Beamlines for the Energy Material in Situ Lab (EMIL) at BESSY II

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Abstract
The Energy Material in Situ Lab (EMIL) at BESSY II will become a worldwide unique research laboratory on materials, processes and devices for sustainable energy production and storage. Its light source for X-ray analytics will be the most complex beamline built at BESSY II so far. An elliptical APPLE II undulator for soft X-rays and a planar cryogenic in-vacuum undulator for X-rays up to 10 keV serve up to four endstations, which operate with both beams simultaneously in a common focus or use alternatively either the soft or the hard X-ray radiation. Two beamlines with twenty optical elements disperse and distribute the radiation: The hard X-ray beamline consists of a plane grating monochromator for the first harmonic of the cryogenic undulator (700-2000 eV) and a double crystal monochromator for the energy range up to 10 keV. The radiation of the soft X-ray beamline (70-2200 eV) is dispersed by another plane grating monochromator operating in collimated light. Switching mirror units are used to feed four endstations:

- the Solar Energy Material In-Situ Spectroscopy at the Synchrotron (SISSY) for HAXPES, XES, XAS, XRF
- catalysis research for sustainable energy supply (CAT) for ambient pressure XPS at kinetic electron energies up to 8 keV
- a multi-purpose beamport for additional experiments such as HXPEEM
- an additional multilayer mirror in the hard X-ray beamline is used to provide high photon flux of broad bandwidth to a fourth experimental station dedicated to e.g. non-resonant ambient pressure XES (PINK)

These endstations will be set up in a separate laboratory with a total square footage of over 2000 m² outside the current BESSY II building. A pointing stability of 20 μm for the common focus of both 60 m long beamlines has to be realized.

EMIL will go into user operation in mid-2015.

Keywords: 09 P
E-line project of SSRF

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Abstract
A wide energy range (90eV ~ 10keV) ambient pressure XPS beamline (E-Line), which will be focused on the energy materials analyzing, is proposed at the Phase II project of Shanghai Synchrotron Radiation Facility (SSRF). X-ray absorption spectroscopy (XAS) technique is also included in this project. The electrical and atomic structures of the energy materials can be clear clarified by the insitu analysis of combination XAS and XES techniques.

Keywords: 10 P
Development of single-shot CCD-based data acquisition system for time-resolved hard x-ray photoelectron spectroscopy using x-ray free electron laser source

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Abstract
The recent advent of free electron laser (FEL) sources from the extreme ultraviolet to hard x rays is expected to revolutionize studies based on coherence and dynamical properties of materials. Especially, x-ray FEL (XFEL) is expected to explore the spectroscopy for the ultrafast dynamics in the intrinsic electronic structure of condensed matter. Thus, in order to advance time-resolved hard x-ray photoelectron spectroscopy (TR-HAXPES) for the study of transient electronic structure of materials at an XFEL facility, such as SACLA [1] in Japan, we have recently developed a single-shot CCD-based data acquisition (DAQ) system combined with a high-resolution hemispherical electron energy analyzer [2]. The system can be controlled by an external trigger signal for single-shot pump-probe experiments. The basic performance of the system has been demonstrated with an offline test, followed by an online core-level spectroscopy of Au 4f photoelectrons with "shot-to-shot image" and "shot-to-shot sweep" modes at soft x-ray undulator beamline BL17SU of SPring-8. The function for correcting the shot-to-shot intensity fluctuations of the exciting photon beam, an important requirement for the TR-HAXPES experiments at FEL sources, has been successfully tested. In the conference, we will show the details of the DAQ system together with the results of the performance test. We also hope to present a preliminary result of the TR-HAXPES experiment, which is scheduled for the middle of April 2013, at the Conference.

References :

Keywords: 11 P
New opportunities for HAXPES at PETRA III

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Abstract

At PETRA III, DESY's new 3rd generation hard X-ray source, a dedicated HAXPES instrument is being operated at undulator beamline P09 since 2010. This beamline is shared by three different experimental setups leaving only limited beamtime for the steadily growing and very productive HAXPES community.

It is therefore planned to relocate the HAXPES activities to a new beamline as part of the PETRA III extension project where ten new beamlines will be built at the storage ring. The new HAXPES instrument at the planned X-ray spectroscopy undulator beamline P22 will use a new SPECS 225HV spectrometer with a newly developed +-30 deg wide-angle lens system, improved high-voltage supplies and a larger diameter 2D delayline detector. This instrument will already be tested at the current beamline P09 in late spring of 2013.

The X-ray optical system of the new beamline will be similar to the present one, employing an stability-improved primary cryo-cooled double-crystal monochromator with Si(111) and Si(311) crystal pairs, Si channel-cut post monochromators and a diamond phase-retarder for generating variable linear and circular X-ray polarization. Using an intermediate focus by primary mirrors, a demagnifying KB-mirror system will produce a micrometer-sized focal spot on the sample.

The new and somewhat larger UHV analysis chamber will be equipped with supplementary analytical tools (e.g. REELS) and a 5-axis manipulator with an integrated LHe cryostat. Special emphasis will be on control software development to remotely access all data acquisition functions of the electron spectrometer to allow flexible accumulation of spectra as function of beamline and/or sample parameters.

Selected scientific results obtained at the current P09 HAXPES station will also be presented.

Keywords: 12 P
Study of Interface for MIS structures by HAXPES under Biases

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Abstract

The electronic state of insulator/semiconductor interface affects the properties of metal-insulator-semiconductor (MIS) devices. The shifts of the band energy under biases strongly depend on the interface state in the band gap. The interface state density can be measured by the capacitance-voltage (C-V) technique. This technique works well for the Si-based structure. However, in the case of the wide band gap semiconductors, the interface states around mid-gap can not be detected by the C-V measurement at room temperature, because of the slow emission rate of the electrons trapped around the mid-gap.

The interface state density can be also obtained from XPS under biases [1]. The effect of the emission rate of the trapped electrons is negligible, so the interface states around mid-gap can be detected. We studied the interface state density in the band gap by HAXPES under biases. We evaluated the validity of this technique using well-defined structure of SiO₂/Si(111) and SiO₂/Si(001) MIS structures.

The n-type Si(111) and Si(001) wafers (1-10 Ωcm⁻¹) were used for the samples. The 10nm SiO₂ films were grown on the Si substrates by the pyrogenic oxidation. The Al electrodes were deposited on the front- and back-side of the substrates with the thickness of approximately 10nm and 300nm, respectively. The front-side electrodes were electrically connected to the ground, and the biases were applied to the back-side electrodes. HAXPES measurements were carried out at SPring8 BL46XU using photon energy of 8keV.

The Al₁s and Si₁s peaks were observed depending on biases. The Al₁s for the front-side electrodes connecting to the ground were not shifted by applying the biases. The Si₁s for the substrates were shifted depending on the bias. These results indicate that the biases are reliably applied to the MIS samples. The energy shift of the Si₁s for the SiO₂ is smaller than the Si₁s for the substrate, which are caused by the potential slope in the SiO₂ layer. The flat band voltage can be estimated from the spectral shapes of the Si₁s for the SiO₂. The quasi-Fermi energy is obtained from the flat band voltage. The shifts of the Si₁s for the substrate include the energy drop induced by the change of the occupation of the interface states by applying the biases. As a results, the interface state density in the band gap for the Si(111) and Si(001) were in the range of 1x10⁻¹⁰ - 1x10⁻¹¹ cm⁻²eV⁻¹.


Keywords: 13 P
Understanding the effect of sputter damage by using HAXPES when studying the bulk of W-S thin films

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Abstract
Thin films deposited by magnetron sputtering from a WS₂ target have been studied by HAXPES. The W-S films were analysed as-deposited, and after sputter cleaning of the surface by Ar + ion bombardment. Spectra from deep core levels W 3d and S 1s were studied and used for interpreting the more complex W 4f and S 2p core levels. The as-deposited films were found to consist of sulphide(WSₓ), oxide (WO₃), and a mixed oxysulfide (WO₇Sₓ) of tungsten. It is shown that the sputter cleaning process not only removes the outermost oxidized material, but that the ion bombardment also leads to preferential sputtering of sulphur and the formation of metallic tungsten. While necessary to study the film beneath the oxidized surface, sputter cleaning thus alters the stoichiometry of the material as well as the chemical bonding. The results are of strong interest for the interpretation of soft X-ray photoelectron spectroscopy of WS₂ and WS₂-based materials, as they demonstrate that the component at low binding energies in the tungsten core-level spectra is an effect of sputter damage.

Keywords: 14 P
Angle-resolved Hard X-ray Photoemission Study of Nb Hydride Formation in 10-GPa Supercritical Water

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Abstract

Nb hydride formation in high pressure 10-GPa supercritical water [1] has been investigated by angle-resolved micro-beam hard x-ray photoelectron spectroscopy. The photoelectron measurements were performed at room temperature by using a recently developed input electron lens with wide angle acceptance of 64 degrees and an 8-keV x-ray beam focused to a size of 0.8-μm vertical and 2.6-μm horizontal widths by a Kirkpatrick and Baez optics at beamline BL47XU of SPring-8. Nb hydride specimens were synthesized from Nb foils of 0.1 x 0.1 x 0.025 mm³ in typical size in a diamond anvil cell filled with H₂O or D₂O under a high pressure of 7~10 GPa and infrared-laser heating at 1500~3000 K.

Comparison of spectra of the Nb 2p and 4d core-levels and valence-band of Nb hydride specimens with those of starting Nb foils and commercially available Nb oxides shows that (i) for the Nb hydride specimens, a new peak attributed to the Nb hydride appears in the slightly high binding energy side of the metallic Nb components in the core-level spectra; (ii) Nb hydride specimens are covered with Nb₂O₅ oxide, even though little oxides are observed by x-ray diffraction patterns; and (iii) the Nb hydride components increase with the depth from the surface, while the oxides components decrease. Thus Nb hydrides are formed inside the specimen, irrespective to the well-known high oxidation ability of supercritical water. One probable process is as follows; hydrogen atoms produced on decomposition of water at Nb surfaces rapidly penetrate deep inside the specimen to form the hydride because of the high hydrogen permeability of Nb [2], while a part of produced oxygen form a passive layer of Nb oxide, which may act as a barrier to keep the hydrogen in the specimen on recovery to the atmospheric pressure as well as resistance to further oxidation, and the residual oxygen is dissolved into the surrounding water or in the form of H₂O₂.

The present results demonstrate that the angle-resolved micro-beam hard x-ray photoelectron spectroscopy is a powerful tool for characterizing the depth profiles of the chemical species in small specimens such as newly synthesized in the diamond anvil cell.

References


Keywords: 15 P
Electronic and optical properties of phenyl-passivated Si nanoclusters

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Abstract

Semiconductor nanoclusters are attracting much interest from the viewpoints of both optoelectronic devices and fundamental physics. For example, Si nanoclusters exhibit the intense photoluminescence (PL) in the visible region but the origin of photoluminescence of Si nanostructure is still unknown, because such optical properties depend on the sample preparations such as morphology, oxide on the surface, passivation and so on. Therefore, in order to investigate the size-dependent optical properties of Si nanoclusters in detail, it is important to prepare Si nanoclusters which have inert and well-defined surface and elucidate their optical and electronic properties. In this study, we have prepared various-sized Si nanoclusters that have well-defined surface and carried out the optical measurement, photoemission spectroscopy, and X-ray absorption. The size-selected phenyl-passivated Si nanoclusters without oxygen contamination have been prepared in the solution route. The intense PL observed in Si nanoclusters, in which the band gap energies increase up to approximately 4 eV. Furthermore, monotonic shifts of PL as a function of excitation energy and the corresponding PLE as a function of emission energy were observed. The hard X-ray photoemission study was conducted at Spring-8 BL15. From the valence-band photoelectron measurements using a hard X-ray synchrotron radiation, the size-dependent valence-band-maximum energies were observed. From the X-ray absorption measurements in the Si L_{2,3}-edge regions at the Saga University beamline BL13 in the Saga Light Source (Saga-LS), conduction-band-minimum energies were elucidated. The obtained band-gap energy from the photoelectron and X-ray absorption studies were consistent with the band-gap energy that observed at PL spectra. Therefore, it is concluded that the present PL originates from the electron-hole pair recombination between the modified conduction-band (LUMO) and valence-band (HOMO) due to the intrinsic quantum size effect. Furthermore, we have carried out the time-resolved photoluminescence study. From time-resolved PL, two kinds of exponential PL decay components with fast and slow lifetime were observed. The identical decays with slow lifetime will be derived from the surface states of phenyl-passivated Si nanoclusters. The size-dependent zero-phonon assisted optical transitions by quantum size effects occur at a high rate of sub-nanosecond timescale.

Keywords: 16 P
Electronic structure of L10 ordered FePt thin film

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Abstract

The L1₀ ordered FePt alloy has attracted much attention in recent years due to large uniaxial magnetocrystalline anisotropy (MCA) [1]. Intensive theoretical studies [2] have been done to investigate the electronic structure and large perpendicular MCA, while the electronic structure of L1₀ FePt has not been studied experimentally. We have performed hard X-ray photoemission spectroscopy (HAXPES) to investigate the electronic structure of the L1₀ ordered FePt thin film in comparison with the disordered FePt thin film. The L1₀ ordered FePt thin film was fabricated by sputtering [3]. The HAXPES measurements were performed at BL15XU [4] of SPring-8. The Fe 2p core-level HAXPES spectra for the ordered and disordered FePt films showed the doublet structure in the 2p₃/₂ main peak region. This result suggests the strong electron correlation in the Fe 3d states and the importance of the hybridization between the Fe 3d and Pt 5d states. The valence band HAXPES showed the main peak structure around 2 eV and a broad structure in the region between the Fermi level and 8 eV. The main peak structure was considered to be the Pt 5d_yz and 5d_zx orbitals, which hybridized with the Fe 3d_y and 3d_z orbitals. The detailed analysis by the configuration interaction theory based on the cluster model and first-principles calculation will be presented.


Keywords: 17 P
Construction of hard x-ray photoemission spectroscopy data base with photon energy of around 8keV at BL46XU of SPring-8: 4d and 5d elements

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Abstract
High-energy, high-resolution hard x-ray photoelectron spectroscopy (HAXPES, total energy resolution of ~0.25eV with hv~8keV at room temperature) has attracted great interests for investigating electronic structure of bulk materials, Nano scale buried layers and their interfaces, since the relative contribution of signal from the surface region is reduced1,2, due to the large escape depth of photoelectrons with high kinetic energy. The advances in x-ray undulator technology at third generation synchrotron light sources enable the delivery of unprecedented high photon flux3,4, which can well compensate the suppression of photoionization cross section (PICS) and analyser transmission to make HAXPES experiment accessible. Recently, it was widely used in the industrial applications such as high K electronic device, catalyst, batteries, and so on. Usually, the industrial users need some standard spectra as well as the parameters of those core level spectra for comparison. Unfortunately, the standard spectra for lab XPS some time is insufficient, due to the change of PICS against excitation photon energy. Additionally, the Auger spectra move out of the regions where they appear in lab XPS, owing to the change of kinetic energy of photoelectrons. The demands push us to construct spectra data base of HAXPES at the industrial application beam line III -- BL46XU of Spring-8, where the most industrial proposals will be performed for HAXPES, since no such kind of data base can be referred to so far due to the recent development of HAXPES.

This presentation focuses on the spectra of part of 4d and 5d transition metals (Zr, Nb, Mo, Ru, Rh, Pd, Ag, Hf, Ta, W, Pt and Au) with monochromatic photon energy of around 8keV (Si (444) channel cut). Possible core levels and valence band are investigated. The background and peaks are fitted by thinking about inelastic mean free path (IMFP) of photoelectrons. In order to evaluate PICS and the surface induced effects at different electron kinetic energies, the intensity of core levels and valence band is used. As a result, valence bands are compared with the calculated ones by considering PICS and total energy resolution.

REFERENCES:

Keywords: 18 P
Vibration-mode selection by site-selected photoelectron-induced recoil effect in CO₂

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Abstract
Photoelectron ejected from molecular core carries energy, momentum and angular momentum and results in complicated changes of molecular dynamics. The recoil molecular dynamics evidently depends on position of the core ionized atom and direction of transferred momentum in the molecular frame.

To investigate the photoelectron-induced recoil dynamics we pass from atomic displacements \( x \) and momenta \( p \) to normal vibrational mass-dependent coordinates \( X \) and momenta \( P \), respectively, by using the matrix \( A: x = AX \) and \( p = A^{-1}P \). It is shown that the mean recoil energy of any type corresponding to site- and direction-selected photoelectron ejection can be calculated as

\[
E_i = \frac{p_0^2}{2} = \frac{1}{2} \left| A_{ij} \right|^2,
\]

where \( p_0 \) is the photoelectron momentum in the laboratory frame of reference, \( i \) indicates the normal mode, \( j \) corresponds to the photoelectron ejection direction.

The matrix approach is applied to examine the recoil dynamics of core-ionized CO₂ molecule. Our main emphasis is put on C and O 1s \( \Sigma \rightarrow \Sigma \) and \( \Sigma \rightarrow \Pi \) transitions when the fast photoelectrons are mainly ejected parallel and perpendicular to the molecular axis, respectively. Analysis of the corresponding \( A \)-matrix results in the following conclusions on the CO₂ photoelectron-induced dynamics:

- O 1s \( \Sigma \rightarrow \Pi \) transition produces rotational recoil with \( E_{\text{rot}} = \frac{K_e m_e}{2M} \);
- (C and O) 1s \( \Sigma \rightarrow \Pi \) transitions produce the bending mode excitation and the mean recoil energies are \( E_{\text{bend}}(O,\Pi) = (K_e m_e/\mu)(m/2M) \) and \( E_{\text{bend}}(C,\Pi) = (K_e m_e/\mu)(2M/m) \);
- C 1s \( \Sigma \rightarrow \Sigma \) transition produces only asymmetric mode with \( E_{\text{asym}}(C,\Sigma) = (K_e m_e/\mu)(2M/m) \);
- O 1s \( \Sigma \rightarrow \Sigma \) transition produces both symmetric and asymmetric modes with ratio of the recoil energies equal to \( E_{\text{asym}}/E_{\text{sym}} = m/\mu = 3/11 \approx 0.27 \).

Here \( m_e \) is non-relativistic electron mass, \( K_e \) is photoelectron kinetic energy in the laboratory frame of reference, \( m \) and \( M \) are carbon and oxygen atomic masses, respectively, \( \mu \) is the total molecular mass. The case of arbitrary ejection direction is considered as well.

Keywords: 19 P
FREE-ELECTRON FINAL-STATE CALCULATIONS FOR THE INTERPRETATION OF HARD X-RAY ANGLE-RESOLVED PHOTOEMISSION

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Abstract

A simple two-step numerical procedure has been developed in order to interpret the electron band dispersions measured by angle-resolved photoemission at energies where free-electron final states are a good approximation. Our procedure has been used in the evaluation of the k-resolved data published in Refs. [1-4]. The useful photon energy range is from above approx. 50 eV up until several keV, while for much higher energies the modulations due to band dispersions are smeared out by the phonon effects in the experimental data.

In the first step the k-point position in the Brillouin zone is calculated taking into account the vector momentums of the emitted electron and incoming photon. A macro written in Matlab allows full 3D geometry by means of matrix rotations and can be automatized for predicting the trajectory in the Brillouin zone or the Fermi or constant energy surfaces.

In the second step the set of k-points calculated by macro is plugged into the DFT code which calculates the ground state electron eigenvalues at these points. It can be done by virtually any code, but we found the WIEN2k (www.wien2k.at) in particular useful due to its convenient interface, no need to use pseudopotentials, and excellent online support.

We will present results of the free-electron final state calculations for various materials at various photon energies from VUV up to hard x-rays, and compare them to the experimental results and one-step model photoemission calculations.


Keywords: 20 P
Evidence for Kondo resonance in semi-metallic CeCuAs$_2$

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Abstract

Introduction: We study the electronic structure of CeCuAs$_2$, which is known to show an anomalous negative coefficient of resistivity between 45 mK - 300 K, while the thermopower measurements measured between 4.2 K – 300 K show typical metallic behavior seen in Kondo systems without a semiconducting gap[1].

Materials and Methods: We have carried out hard x-ray photoelectron spectroscopy (HAXPES) of the core levels and valence band of CeCuAs$_2$ at BL29XU, SPring-8. We also carried out Ce 3d-4f x-ray absorption spectroscopy (XAS) and resonant-PES at BL17SU, SPring-8. The obtained spectra were analyzed to determine the role of Kondo screening on the electronic structure of CeCuAs$_2$.

Results: The Ce 3d core level PES measured with HAXPES at an incident photon energy of ~ 8 keV and at T = 25 K, shows the spin-orbit split Ce 3d$_{5/2}$ and 3d$_{3/2}$ peaks with clear evidence of the f$_0$, f$_1$ and f$_2$ features which are typical of Kondo metals. The measured Ce 3d-4f XAS at T = 25 K shows the main peak due to transitions from initial state $i$ to $f^2$, while a weak feature due to the $i$ to $f^1$ transition is also observed, indicating evidence of Kondo screening in CeCuAs$_2$. The spectral feature assignments have been confirmed by single-impurity Anderson model calculations. Resonant-PES measured at various energies across the Ce 3d-4f show a giant Ce 4f$^1$ resonance just below the Fermi level, while the Ce 4f$^0$ feature is observed at 2.5 V binding energy.

Conclusions: The HAXPES and Ce 3d-4f resonant PES results confirm the Kondo resonance in semi-metallic CeCuAs$_2$.

Reference:


Keywords: 21 P
Segregation and oxidation in Fe-Cr and Fe-Cr-Al alloys

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Abstract
The fundamental physical difference between Fe and Cr plays a decisive role in the thermodynamics of Fe-Cr based alloys. Chromium in bulk affects also surface properties: within 8–23 at. % Cr in bulk the Cr containing surface has lower surface energy than the pure Fe surface [1] and adding 10 at. % Cr to Fe-Al, the Al concentration can be dropped without weakening the protective alumina surface scale [2]. In the present work some effects of substitutional Cr in Fe and Fe-Al are investigated using Hard X-ray Photoelectron Spectroscopy (HAXPES) and computer simulations. Diffusion and segregation in Fe-Cr and initial oxidation of Fe-Al and Fe-Cr-Al were investigated as a function of heating time and oxidation rate at HIKE end station at KMC-1 beamline, Bessy. In Fe/Cr double layer sample the gradual Cr diffusion to the surface through the Fe layer was investigated with annealing time. The observed fast oxidation well demonstrates the difficulties that relate to the studies of the initial oxidation highlighting the importance of non-destructive depth profiling provided by HAXPES. Results show that with annealing time the amount of Cr at the surface (oxidized Cr) increases faster than the amount of Cr in the Fe-layer indicating that there is an upper limit of dissolved Cr in bulk Fe. This is in agreement with previous first-principles calculations [3] and with the present MC-MD calculations for Fe/Cr interface. Segregation and oxidation of Al in Fe-Al and Fe-Cr-Al alloys were also investigated using HAXPES. The the oxide thickness, chemical state of Al and formation of Al rich layer close to the surface were be studied in a detailed way by comparing the intensities of different components of Al 1s spectra observed with different photon energies. The segregation of Al to the surface of Fe-Cr-Al is stronger than that of Fe-Al showing that Cr enhances the surface segregation of Al, i.e. Cr improves the high-temperature oxidation resistance of Fe-Al alloys.


Keywords: 22 P
HAXPES study of La0.7Sr0.3MnO3 thin films on (100)- and (110)- oriented SrTiO3 substrates

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Abstract
A renewed surge of interest in the manganese perovskite oxides of R₁₋ₓAₓMnO₃ (where R is a rare earth element and A is Ca, Sr, Ba etc.) in the 1990s started with the discovery of the phenomena of colossal magnetoresistance [1]. The nature of magnetism at surfaces and interfaces has been a fundamental issue. However, experiments probing the magnetization at the surface and interfaces of highly spin-polarized materials suggest that surface magnetization is suppressed compared to the bulk. Among different manganites, La₀.₇Sr₀.₃MnO₃ (LSMO) is due to its 100% spin polarized conducting band a promising candidate for spintronic applications. In order to study, how the transport and magnetic properties of epitaxial LSMO films depend on the lattice mismatch between the substrate and the film, the electronic structure of LSMO samples on different substrate have been explored in this work by Hard X-ray PhotoElectron Spectroscopy (HAXPES). Here the results of the HAXPES measurements for two La₀.₇Sr₀.₃MnO₃ samples, that are grown on (100)- and (110)- oriented SrTiO₃ single crystal substrates are presented. The photoelectron spectra of Mn 3p Mn 3s, Mn 2p, O 1s, La 3d core levels and valence band together with the survey spectra were recorded. Two different photon energies, 2500 eV and 7000 eV, have been used to collect the photoelectron spectra. All experiments were carried at beamline KMC-1, Bessy II, Helmholtz-Zentrum Berlin [2]. The analysis of the experimental data shows that the dependence on the STO substrate orientation causes the unit cell distortions of LSMO films and deformations of the MnO₆ octahedral, which affect the hopping of electrons along Mn³⁺ - O²⁻ - Mn⁴⁺ chains. Due to the out-of-plane or in-plane changes in the Mn-O-Mn bond angle and bond length of the LSMO films on (001)- and (110)- oriented STO substrates, there exists induced higher Mn³⁺ concentration in the first sample, respectively. The strain engineering to control the magnetic and magnetotransport properties of LSMO films depending on the orientation of the underlying SrTiO₃ substrate provides an additional tuning parameter for the functional characteristics.


Keywords: 23 P
Electronic structure of SrTiO3 and TiO2 studied by hard x-ray and soft x-ray induced electron spectroscopy

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Abstract
We study the electronic structure of La-doped SrTiO3 (STO) and TiO2 using hard x-ray photoelectron spectroscopy (HAXPES) and Auger electron spectroscopy (AES). We have carried out HAXPES of the core levels and valence band at BL19LXU of SPring-8. The titanium KLL, KLM, KMM and LMM Auger spectra were also measured to estimate the two-hole correlation energy in the final state. The Auger spectra have been compared with the theoretical spectra calculated based on the cluster model. We also carried out Ti 2p-3d x-ray absorption spectroscopy (XAS) and resonant-AES at the soft x-ray undulator beamline BL17SU of SPring-8. The obtained spectra can be consistently described based on cluster model calculations. Figure 1 shows the Ti normal LMM Auger spectrum excited by 471.09 eV soft x-ray beam. The details will be discussed at the Conference.

Fig.1 The Ti normal LMM Auger spectrum together with the cluster model calculations.

Keywords: 24 P
Influence of water adsorbates on the electronic properties of LaAlO3/SrTiO3 heterostructures studied by hard and soft x-ray photoelectron spectroscopy

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Abstract

Oxide heterostructures display many interesting phenomena, one example being the formation of a two-dimensional electron system (2DES) at the LaAlO\textsubscript{3}/SrTiO\textsubscript{3} (LAO/STO) interface at a critical thickness of 4 unit cells (uc) and beyond of the polar LAO overlayer \cite{1,2}. The origin of this high-mobility 2DES is still subject of intense research activities both in theory and experiment. One promising explanation is the electronic reconstruction scenario, in which charge is transferred from the surface to the interface to avoid the divergence of the electric potential gradient across the polar LAO film. In this context it has recently been shown that polar adsorbates can enhance the conductivity of the 2DES \cite{3}. Therefore, beside their electrostatic influence it was discussed that surface defects/adsorbates can also act as a charge reservoir \cite{4}. To examine the impact of surface adsorbates on the 2DES we performed in situ photoelectron spectroscopy on samples with a 6uc thick LAO film exposed to water vapour between $10^{3}$ and $10^{6}$ Langmuir. Indeed, we observe an increase of charge carriers at titanium sites in response to the adsorbed water in photoemission. Additionally, we performed depth profiling by using hard x-rays and varying the detection angle, indicating that the additional charge carriers are located at the interface. We discuss this increase in the charge carrier concentration in terms of the electronic reconstruction scenario and a possible protonation and hydroxylation of the sample surface.

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\cite{2} Thiel et al., Science 313, 1942 (2006).
\cite{3} Xie et al., Nature Comm. 2, 494 (2011).

Keywords: 25 P
Electronic structure studies on conducting polypyrrole polymer nanotubes showing switching transitions

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Abstract

Conducting polypyrrole polymer nanotubes (PPY) is a classical model system for disordered strongly correlated materials showing intriguing switching transitions from low to high conductivity states. This switching behaviour can be tuned by incorporating gold nanoclusters to form composite nanotubes (AuPPY). Here we present core level electronic structure studies on PPY and AuPPY nanotubes with different diameter using hard X-ray photoemission spectroscopy. The spectroscopic data provide information on the role of diameter as well as metal cluster incorporation for a modification of the electronic structure of this important class of nanotubes. Based on these results we explain the observed electrical transport behavior of these nanotubes as the interplay of disorder and carrier concentration.

Keywords: 26 P
Abstract

Antiperovskite manganese nitrides $\text{Mn}_3\text{XN}$ (A=Ni, Zn, Ga, etc.) show a sudden contraction on heating at certain temperature ranges, which is called as a thermal negative expansion [1, 2]. The magnitude of the contraction is as much as $10^{-3}$ order in $\Delta L/L$. It has been reported that the sudden contraction on heating is smoothed by a partial substitution of X atoms [2], which attracts an intense interest in applicationwise as an alternative for invar alloys. This contraction is considered to be a magnetovolume effect, since it is accompanied by the deformation of $\Gamma^5g$-type antiferromagnetic order, while the crystal structure remains cubic during the transition [1].

In order to discuss the mechanism of the thermal negative expansion in $\text{Mn}_3\text{XN}$, we have performed a hard x-ray photoemission (HAXPES) experiment on polycrystal $\text{Mn}_3\text{NiN}$, whose Néel temperature is 266 K [1]. We used linearly-polarized photons of 8 keV as an excitation source. Using a diamond phase retarder, we utilized the polarization vector of incident photons to be s-polarization and/or p-polarization to the sample surface. Due to the cross section of photoionization process in hard x-ray region, it is expected that the emission from $s$ orbitals is dominant in p-polarization settings, while that from $d$ orbitals is dominant in s-polarization case. The clean surface was obtained in-situ cleaving in an ultrahigh vacuum of $\sim 1 \times 10^{-7}$ Pa.

As a result, we have successfully observed the temperature dependence of valence band HAXPES spectra in $\text{Mn}_3\text{NiN}$, that is, the spectral weight at 2-6 binding energy shifts to 8-10 eV on cooling below $T_N$ at p-polarization. By comparing the polarization dependence of HAXPES spectra with the results of the first principles band structure calculations using Akai-KKR, we have concluded that the observed change in the spectra mainly reflects the signal from $s$ electrons in transition metals. The first principles calculations, where the noncolinear magnetic structures in $\text{Mn}_3\text{NiN}$ are taking into account, predict the change in the partial density of states of 4$s$ electrons through the magnetic phase transition, consistent with our HAXPES experiment.


Keywords: 27 P
High-temperature electronic transitions revealed by HAXPES

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Abstract

Strongly correlated transition metal oxides show a wide variety and richness in their physical properties. Most photoelectron spectroscopy measurements on these materials are performed at low or room temperature. Going to high temperatures allows to access disordered phases and switch off various inter-site spin, orbital or charge correlations. However, the compounds tend to lose oxygen in the region close to the surface. This makes it difficult, if not impossible, to do experiments with surface-sensitive soft X-ray PES. Due to the increased probing depth of HAXPES the oxygen deficient surface regions become much less important. This allows to perform measurements at up to about 750K.

We present temperature dependent valence band spectra of several transition metal oxides with direct spectroscopic observation of phase transitions. By making use of the polarization dependence of the asymmetry parameter in the photoionization cross section we are able to obtain spectra which are almost free from distortions by strongly enhanced 4s signals.

Keywords: 28 P
Band offsets of Lanthanum Oxide on Gallium Nitride as measured by photoelectron spectroscopy

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Abstract
Preparation of high quality gate oxides on wide bandgap semiconductors remains an important challenge toward the realization of efficient high performance devices. For GaN electronics there are a limited number of compatible oxides that have a sufficiently large bandgap to minimize electrical leakage. In this work, reactive molecular beam epitaxy was used to deposit La₂O₃ films on (0001)-oriented GaN substrates. The film structure was characterized using reflection high-energy electron and X-ray diffraction. Phase transformations were observed for La₂O₃ film thicknesses greater than 10nm. The attenuation of the substrate photoelectron peaks using monochromatic Al K-alpha X-rays was significant for a 3 nm La₂O₃ film, while a 6 nm La₂O₃ film completely blocked the signal from the underlying GaN. HAXPES, performed at beamline X24A at the National Synchrotron Light Source at Brookhaven National Labs, could be used to probe through the lanthanide oxide layer. The valence band offset on hexagonally symmetric films was found to be 0.63 ± 0.04 eV at the La₂O₃ /GaN interface.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-AC04-94AL85000.

Keywords: 29 P
HAXPES measurement of changes in energy level alignment in efficient solid state dye-sensitized solar cells upon addition of 4-tert-butyl pyridine for increased photovoltage

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Abstract

4-tert butyl pyridine (t-BP) is usually used in dye-sensitized solar cells (DSCs) to increase the photovoltage. The energy level alignment at the interface in solid state DSCs was measured using hard X-ray photoelectron spectroscopy (HAXPES). Using this technique it was possible to measure through the active interface in the complete solar cell device and thereby determine the energy of the core electronic levels corresponding to the electron conductor titanium dioxide (Ti2p) and the hole conductor spiro-OMeTAD (N1s). Comparing the different samples, a clear trend was observed in the energy difference between the Ti2p and N1s levels, showing a larger energy difference when more t-BP was added to the spiro-OMeTAD. The results therefore directly show that the energy level matching between the spiro-OMeTAD and the titanium dioxide is changed, and that the energy levels of titanium dioxide are shifted further away from the spiro-OMeTAD energy levels when t-BP is added. This clearly shows that the increased voltage in the solar cells is a result of changes in the relative position of the TiO₂ and spiro-OMeTAD energy levels, and explains therefore the improved device efficiency.

Keywords: 30 P
Photoion recoil effect on 1s^{-1} photoelectron line as a probe of adsorbate-substrate interaction

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Abstract
Photoelectron leaving an ionized system carries energy, momentum and angular momentum and results in changes of nuclear and electronic motion of the system. Translational, rotational and vibrational recoiled motion of the system arises. Photoelectron-induced photoion recoil effect on a monolayer on substrate is investigated in the work. Structure and properties of atomically sharp interfaces between sp²-bonded materials and metallic surfaces are intensively studied nowadays in order to utilize their peculiar characteristics in electronics and nanotechnology. The most prominent examples are graphene and monolayer boron nitride (h-BN) grown on surfaces of transition metals (TM). High kinetic energy (HIKE) photoelectron spectroscopy is suggested to be applied to study the interaction between monolayer adsorbate and transition metal substrate.

Quasi-molecular recoil (QMR) model is developed: the ionized atom of the monolayer and neighbouring atom(s) in the substrate are supposed to form a quasi-molecule. The interaction potential is approximated by the Morse potential, as the interaction is determined by covalent bonds of 2p_{z}(\pi) and nd_{z^2}(\pi) orbitals of monolayer and substrate atoms, respectively.

The model is firstly applied to analysis of Highly Ordered Pyrolitic Graphite (HOPG) carbon 1s photoelectron spectra measured at photon energy 7940 eV [1]. The extracted effective mass within the QMR approach is equal to 11.6 amu. Good agreement between model and experimental spectra is observed.

The QMR model is applied to construct C 1s photoelectron spectra of graphene/TM and h-BN/TM for different photoelectron kinetic energies (100 eV, 4 keV, 8 keV). The cases of weak (low vibration quantum) and strong (high vibration quantum) bond are considered. The results demonstrate sensitivity of the QMR approach to such a characteristic of the interaction potential as vibration quantum.

References

Keywords: 31 Pl
HAXPES characterization of planar arrays of Fe-rich nanocrystals embedded in a GaN matrix

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Abstract

Hard x-ray high kinetic energy photoelectron spectroscopy, (HAXPES), covers an excitation energy range of ca. 2 keV to 12 keV. This technique offers an enhanced probing depth, reduced surface sensitivity and allows the investigation of bulk structures, buried nano-layers and interfaces in a non-destructive manner. In our present contribution we used HAXPES to investigate single planar arrays of Ga\textsubscript{x}Fe\textsubscript{4-x}N magnetic nanocrystals (NCs) embedded in GaN and fabricated in an epitaxial process \cite{1}. By changing the growth parameters, the crystallographic phase and chemical composition of the NCs can be selected. In view of the different magnetic properties of the various NC phases, applications for room-temperature ferromagnetic as well as antiferromagnetic spintronic devices are foreseen \cite{2}. The arrangement of the Fe-rich NCs in a single planar array has been previously made evident through high-resolution transmission electron microscopy measurements and the layers have been characterized via x-ray diffraction and SQUID magnetometry \cite{3}.

The Fe-rich NCs are covered with a 60 nm thick GaN capping layer, which makes it impossible to study the electronic and chemical properties without the use of HAXPES, which we now did at beamline KMC-1 at the BESSY II synchrotron radiation facility in Berlin. We focused our attention primarily on the chemical properties of the Fe-rich NCs in order to determine the mode of bonding with the GaN matrix. Therefore, the core levels of the pure GaN matrix and their modification in proximity of the Fe-rich NCs confined layer were characterized. We measured the Ga 2p, N 1s and Fe 1s core levels using different excitation energies ranging from 2 keV to 9.5 keV in order to get information from different depths. Subsequently, we studied the valence band density of states in the GaN matrix and in the region where the Fe-rich NCs are confined in order to identify the different contributions coming from the pure GaN and from the Fe-rich NCs. Particular care was dedicated to determine the recoil effect in the studied systems \cite{4-6}.


Keywords: 32 P
Study of (Ga,Mn)N layers by hard x-ray high kinetic energy photoelectron spectroscopy

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Abstract

Dilute magnetic semiconductors (DMSs) have lately attracted great attention as a field of research since they are promising candidates, e.g., for magneto-optical devices that combine the functionalities of semiconducting materials with the information storage capabilities of magnetic elements. To introduce the desired magnetic properties, the semiconducting material is magnetically doped with e.g., Mn, so that a fraction of the host crystal sites is substituted by magnetic ions. To gain information on the surface and bulk electronic structure of the magnetic dopant Mn diluted in a GaN matrix, we employed hard x-ray high kinetic energy photoelectron spectroscopy (HIKE-PES or HAXPES), which offers a probing depth in excess of 20 nm, less surface sensitivity, and allows the investigation of bulk structures, buried nanolayers and interfaces in a non-destructive manner. Dilute Ga₁₋ₓMnₓN layers with a thickness of about 200 nm were grown by metalorganic vapor phase epitaxy (with Mn cation concentrations up to 3.4%) on substrates consisting of 2 µm GaN layers on c-plane sapphire [1]. Both pure GaN and dilute Ga₁₋ₓMnₓN layers were characterized by HAXPES at the HIKE end-station of the KMC-1 beamline at the BESSY II synchrotron radiation facility in Berlin. We focused primarily on the chemical properties of the Ga₁₋ₓMnₓN layers in order to determine the Mn bonding with the GaN host matrix. Core levels of the pure GaN layers are compared with core levels of the dilute Ga₁₋ₓMnₓN layers. The Ga 2p, N 1s, Mn 2p, and Mn 1s core levels were measured using excitation energies ranging from 2 keV to 9.5 keV to study the chemical environment of Mn at the surface and in the bulk of the Ga₁₋ₓMnₓN layers. In addition, we characterized the valence band density of states of the GaN and the Ga₁₋ₓMnₓN layers in order to identify the different contributions stemming from the pure GaN and the Mn dopant. The HAXPES recoil effect [2-4] was carefully considered in the data analysis.


Keywords: 33 P
Application of Laboratory Hard X-ray Photoelectron Spectrometer for Analysis of Materials Prepared by Combinatorial Synthesis

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Abstract

Combinatorial methods have been verified as powerful tools for quests of the materials for advanced devices [1-3]. One of the most versatile ways is thin film synthesis using pulse laser deposition or sputtering with multiple sources, enabling depositions of films with graded composition on the substrates. Using this method, one can prepare samples of varying compositions continuously over the area of ca. 10 mm sizes. In combination with scanning probe of ca. 100 μm size, it is possible to characterize composition dependences of sample properties over whole composition range required. Since X-ray beam size of HEARP Lab just matches to the above mentioned requirement, the combination with combinatorial synthesis is expected to open up a new opportunity. Here we introduce an example of the applications for the material research on high-quality passivation layers with controlled fixed charge for Crystalline Silicon Solar Cells[4].


Keywords: 34 P
Study of Novel Phase Change Memory Material GeCu2Te3 by Laboratory Hard X-ray Photoelectron Spectroscopy System

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Abstract

Recently, Sutou et al. has discovered that GeCu2Te3 (GCT) exhibits reversible phase change (PC) phenomena[1,2]. The most interesting point in this new class of PC materials is that it has 24 sp valence electrons per molecule (4 sp electrons/atom) thus takes a tetrahedral bonded chalcopylite related structure in the crystal phase. In the standard PC materials of GST and related compounds, average valence electron number is approximately 5/atom, corresponding to s^2p^3 valence electron configuration. The s^2 electrons form lone pair band, and p^3 electrons mainly sustain crystal bonding, resulting in the inherent instability in NaCl type cubic crystal lattice. Mechanism of reversible PC phenomena in these average 5 valence electron family have been discussed in relation with this characteristic valence electron configuration[3,4].

In case of GCT, however, tetrahedral s-p hybridized bonds are too rigid consequently not favorable for the PC. This implies that the PC in this material requires another freedom in the crystal bonding which brings flexibility in the bond configurations.

In order to investigate the problem, we have performed hard x-ray photoelectron spectroscopy (HAXPES/HXPES) using Laboratory HXPES system with Cr Ka (5.4 keV) excitation (HEARP Lab) to observe differences in valence band and core levels of GCT samples with various compositions (deviations from stoichiometry) and annealing temperatures. Among those spectra, core level spectra of Ge show distinctive changes due to deviations from stoichiometry and annealing, suggesting that Ge atoms play a key role in the PC mechanism in GCT.


Keywords: 35 P
Sponsor presentations
PHOIBOS 225 HV: High energy electron spectrometer with wide acceptance angle pre-lens

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Abstract

The PHOIBOS 225 HV is a hemispherical electron analyzer with unsurpassed transmission, energy and angular resolution. The multi-element transfer lens is optimized for ultimate energy resolution up to highest kinetic energies and can be operated in several different modes for angular or spatially resolved studies. The modular power supply concept makes it suitable for every aspect of electron spectroscopy: from laser based ARPES measurements at low kinetic energies starting at virtually zero kinetic energy, to quantitative XPS and high energy photoemission spectroscopy at electron energies of up to 15 keV. The acceptance angle can be extended to up to \( \pm 30^\circ \) with an additional pre-lens.

Several detector options with high sensitivity and dynamic range are available: 2D-CCD detector with a Peltier cooled camera, 1D and 2D-delayline detectors, and combined 2D-CCD(DLD)/SPIN detectors. Especially the DLD detectors are ideally suited for data acquisition under extreme conditions as they occur in HAXPES experiments. Here, the DLD detectors with their improved sensitivity, lateral and time resolution allow the detection of spectroscopic features with a count rates 5 counts/second or less can be detected.

Keywords: 01 SPONSOR 16.15
From HAXPES to HAXPEEM: Spectroscopy and Microscopy @ High Energies

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Abstract

Established in 1990, FOCUS is engaged since more than twenty years in the field of electron beam evaporation, electron spectroscopy, electron microscopy and newly also in the field of electron beam welding at the micro scale [1]. At the forthcoming HAXPES workshop 2013 we give an overview of our instrumentation for HAXPES electron spectroscopy and hard x-ray excited energy filtered PEEM, HAXPEEM, as well as our recent steps towards more efficient spin detection. Our HAXPES analyser HV-CSA is a high transmission analyser with a small footprint (see Figure). The analyser enables true 15keV kinetic energy detection using a dedicated 2D event counting detector [2]. Systems are currently installed at ESRF and Spring8. Selected results obtained during a beamtime at PETRA III will be highlighted. An improved version of the NanoESCA instrument [3] opened up the possibility of imaging high kinetic energy electrons. In 2011 first feasibility experiments were performed at PETRA III showing that core level electrons with kinetic energy up to 6.4keV could be imaged [4]. A new dedicated instrument will be commissioned with hard x-rays during a beamtime at PETRA III soon. Furthermore, we will describe the spin detector “FERRUM”, which is based on the exchange scattering of electrons on a magnetized and oxygen-passivated iron film [5]. This detector has a high figure of merit and very long operation time, especially well suited for experiments where highest detection efficiency is required, such as spin resolved HAXPES.

Figure: HV-CSA (left) and NanoESCA (right), as used for the HAXPEEM experiments.


Keywords: 02 SPONSOR 16.30
MB Scientific AB abstract HAXPES 2013

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Abstract
This year MB Scientific AB is celebrating the 10th anniversary of the MBS A-1 analyser. In my presentation I will show briefly our achievement of totally 13 years of experiences as reliable maker of advanced experimental analysis instruments and development ability, also our company policy.

Keywords: 03 SPONSOR 16.45
Haxpes

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Abstract
Since the early days of HAXPES, VG Scienta has been leading the instrument development in this field. Today, we are the leading supplier of high energy electron spectrometers and we continue to push borders on energy range, resolution, and transmission.

One of the challenges of HAXPES is that ionization cross sections decrease rapidly with increasing photon energy. For that reason analyser transmission is of great importance in the HAXPES regime. VG Scienta has recently released a novel analyser, denoted Scienta EW4000, that features an unprecedented parallel angular acceptance, using a novel mesh-based lens design. In addition to offer increased transmission, the large angular acceptance of this analyser enables parallel non-destructive depth profiling with unprecedented sensitivity. This new analyser will be described in the presentation.

The field of Ambient Pressure PES (APPES) is growing fast. One of the main limitations of APPES is inelastic scattering of photoelectrons. By performing APPES experiments in the HAXPES regime, the scattering is greatly reduced, since scattering cross sections reduce rapidly with increasing kinetic energy. Based on the mesh lens developments, VG Scienta has designed a new analyser, called Scienta R4000 HiPP-2, capable of recording APPES spectra in the HAXPES regime. Details will be given.

Keywords: 04 SPONSOR 17.00
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