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Abstract. We have performed core level photoelectron spectroscopy on a W(110) single crystal with femtosecond XUV pulses from the free-electron laser at Hamburg (FLASH). We demonstrate experimentally and through theoretical modelling that for a suitable range of photon fluences per pulse, time-resolved photoemission experiments on solid surfaces are possible. Using FLASH pulses in combination with a synchronized optical laser, we have performed femtosecond time-resolved core-level photoelectron spectroscopy and observed sideband formation on the W 4f lines indicating a cross correlation between femtosecond optical and XUV pulses.

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Contents

1. Introduction

Since the discovery of the photoelectric effect [1], photoelectron spectroscopy has evolved into a workhorse for studying the valence electronic structure, the stoichiometry and the chemical state of matter. The evolution of synchrotron radiation sources has continuously expanded the range of applicability, which presently spans from angle-resolved photoelectron spectroscopy (ARPES) for band structure determination and investigation of highly correlated electron systems [2]–[4] to high-kinetic-energy photoemission using hard x-rays to probe deep into solid matter [5, 6]. In particular, core-level photoelectron spectroscopy using x-rays (XPS, also known as electron spectroscopy for chemical analysis (ESCA)) can probe local chemical states of individual atoms embedded in molecules [7], in the bulk [8], and at interfaces [3], properties that are relevant to physics, materials science, chemical and biological applications. Here, the chemical shift in the core-level binding energy is used as a local element-specific probe, reflecting the different chemical environments surrounding the photoionized atom. Tunability of photon energy permits enhancement of the signal from selected atomic centers through the photon energy dependent atomic photoionization cross-section [9] and selection of the probing depth in matter according to the universal curve of electron mean free path [10]. Resonant photoelectron spectroscopy [11]–[13] gives insights into electron correlation and vibrational resolution in XPS allows us to map potential energy surfaces of core-excited species for free [14]–[16] and adsorbed molecules [17]–[20] and to investigate chemical reaction kinetics of complex molecular systems on surfaces [21] as a function of temperature and pressure.

The next scientific frontier is to investigate in real time ultrafast dynamics in complex matter (e.g. dilute species, surfaces and interfaces) on an atomic scale with the same degree of detail. Present day femtochemistry [22] with optical lasers is limited in acquiring atom specific and chemical state selective information. Utilizing the core-hole-clock method [23, 24], ultrafast electron delocalization [25] and molecular dissociation time constants [26, 27] have been determined at specific atomic sites. However, here, dynamic information is only accessible in the time range given by the fixed decay time of core-excited states. In order to really follow the temporal evolution at selected atomic sites, we have to develop atom specific and chemically selective x-ray photoelectron spectroscopy (or ESCA) further towards a femtosecond time resolving technique. Therefore, we need to combine femtosecond optical and x-ray pulses in pump–probe experiments, where the evolution of a system is determined as a function of the delay between optical pump and x-ray probe pulse. The time resolved pump–probe technique is well known from 2 photon photoemission spectroscopy (2PPE) based on optical lasers [28, 29] producing attosecond pulse trains [30]–[32]. In the picosecond range, synchrotron radiation based time resolved ESCA has been performed [33, 34] and using high harmonic

generation at optical lasers, even femtosecond dynamics have been investigated [35]. However, there we are restrained to some few wavelengths available from the high harmonics of the laser. Synchrotron radiation provides a wider wavelength region, but has considerably less intensity. Thus, if we want to use x-ray pulses for time resolved ESCA in a broad wavelength regime and up to high energies, we need to resort to free-electron lasers (FELs) capable of producing brilliant x-ray pulses up to kilo-electron-volt photon energies with less than 30 fs pulse duration.

In this contribution, we establish the foundation for femtosecond time-resolved ESCA (fs-ESCA) on a solid surface using the free-electron laser at Hamburg (FLASH) [36] in combination with a synchronized optical laser. Based on our experimental data for the intensity dependence of space charge effects on the shape and position of photoemission lines, we derive an expression for the range of electron numbers suitable for fs-ESCA applicable for present and future FEL sources. Applying sideband generation on a metal surface as a tool for cross correlation between the femtosecond x-ray and optical pulses, we obtain the additional temporal information needed for fs-ESCA.

2. Experiment

The experiments were performed at the plane grating monochromator beamline PG2 at FLASH [37, 38] using an UHV chamber equipped with standard surface science tools and a Scienta SES 2002 electron analyzer. A clean W(110) single crystal was prepared by annealing to 2000 °C and cleanliness was checked by x-ray photoelectron spectroscopy. FLASH pulses at 118.5 ± 0.2 eV (3rd FEL harmonic at 38.5 eV 1st FEL harmonic photon energy) impinged onto the W(110) surface at 45 ° incidence angle and at 5 Hz macropulse repetition rate in 50-microbunch mode with a microbunch separation of 1 μs. For our experiment it was crucial to determine the number of monochromatic x-ray photons impinging onto the sample on a shot-to-shot basis. For this purpose, we used a carefully calibrated MCP detector [39] which detected a fraction of the incoming photons reflected from a Au mesh (transmission 65%) behind the exit slit. The MCP detector was cross-calibrated against a facility gas monitor detector [40] taking into account beamline transmission [38], reflectivity at the mesh and quantum efficiency at the MCP.

At a focal spot size of $395 \pm 23 \times 274 \pm 14\,\mu\mathrm{m}^{-2}$ [41] and 30 fs pulse duration, intensities up to $3.23 \times 10^{10}\,\mathrm{W\,cm}^{-2}$ could be reached. The excitation bandwidth chosen was 750 meV for the peak shift investigation and 150 meV for the sideband measurement. The electron analyzer had ≈590 meV bandwidth (pass energy $E_\mathrm{p} = 500$ eV, 500 μm slit). As in our previous gas phase experiments [42, 43], the photoemission spectra were recorded on a shot-to-shot basis with a gated CCD camera synchronized to the FEL macropulse repetition rate. For optical excitation we used delayed optical pulses (800 nm) with a duration of 120–150 fs (FWHM) delivered from the optical parametric amplifier system of the FLASH facility with 1 MHz repetition rate, synchronized to the FEL. On the sample an optical excitation intensity of $4.62 \times 10^{10}\,\mathrm{W\,cm}^{-2}$ was reached.

3. Results and discussion

Our first step to establish fs-ESCA with x-ray FELs is to consider the role of Coulomb repulsion between photoelectrons created within the same fs-x-ray pulse. This space charge effect can significantly affect spectral shapes and peak positions [44]–[46] which we heavily rely

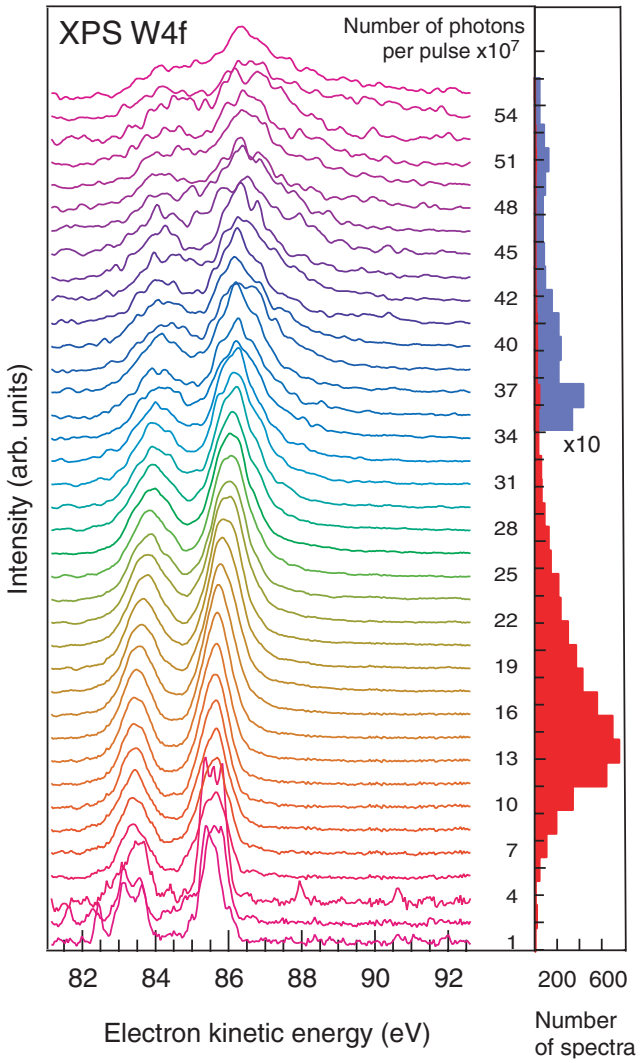

Figure 1. W 4f photoemission as a function of incident x-ray intensity. Focal size $395 \pm 23 \times 274 \pm 14\,\mu\text{m}^2$.

on in ESCA. Our objective is to find suitable x-ray fluences per pulse for electron spectroscopy and derive a generally valid model based on the photon energy dependent photoionization cross-section and the target density. We have thus measured energy distribution curves (EDC) for W 4f emission as a function of x-ray photons per pulse, presented in figure 1. For smaller values of the number of photons, we observe the characteristic W $4f_{7/2}$ and $4f_{5/2}$ spin-orbit pair with 2.2 eV splitting and a 4 : 3 intensity ratio. The statistics of the spectra vary due to the differences in the number of pulses accumulated with the respective number of photons per pulse, as displayed in the right-hand panel of figure 1, with the latter simply reflecting the probability distribution of pulse energies of FLASH resulting from the self amplified spontaneous emission (SASE) process. We observe little modification of the EDC up to 13×10^7 photons per pulse. However, above 30×10^7 photons per pulse, notable peak broadening and shifting occurs.

We can now quantify our experimental findings regarding peak shift and peak broadening of the W 4f lines as a function of the number of electrons created. In figure 2, the peak shift

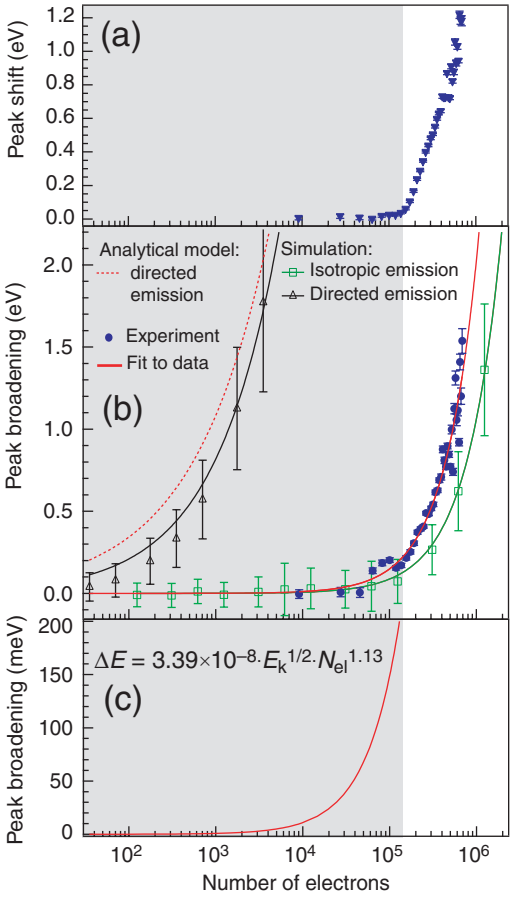

Figure 2. Evolution of peak shift (a) and peak broadening (b) depending on the number of electrons created determined with least squares fitting to the experimental and simulated photoemission spectra using equation (1). The error bars correspond to one standard deviation from the fit. The results are compared to an analytical model of directed emission. (c) Zoom to the experimental data fit for small peak broadening.

and the additional peak broadening with respect to the measurements at the lowest intensities obtained from a least squares fit are shown, using Gaussians at 2.2 eV splitting and a 4 : 3 intensity ratio representing the W 4f spin-orbit pair. We convert the incident x-ray photon numbers into a system-independent number of electrons N_{el} using the W 4f photoionization cross-section of 2.5 Mbarn per atom at 118.5 ± 0.2 eV [9] and assuming a 3 Å electron escape depth [47].

As seen in figure 2, space charge induced peak shifting (panel a) and peak broadening (panel b) both occur with an increasing electron density. As peak broadening sets in at lower electron numbers than significant peak position shifts, we focus on the peak broadening as the limiting factor for fs-ESCA with FEL pulses. In figure 2(b), we present the results from numerical modelling of electron propagation. With the ASTRA code [48] the phase space of an electron cloud (radius 335 μm) created on a solid surface and propagated to a detector at 10 cm distance from the surface has been computed, assuming 50 fs duration of the incident

Table 1. Fitting coefficients of equation (1) for the simulations and the analytical model in comparison with the experimental data. The errors correspond to one standard deviation from the fit.

	a	b
Experiment	$3.39 \times 10^{-8} \pm 3.2 \times 10^{-8}$	1.13 ± 0.07
ASTRA simulation		
Isotropic emission	$3.14 \times 10^{-8} \pm 2.9 \times 10^{-9}$	1.09 ± 0.01
Directed emission	$1.45 \times 10^{-3} \pm 1.9 \times 10^{-4}$	0.59 ± 0.01
Analytical model		
Directed emission	$3.63 \times 10^{-3} \pm 1.4 \times 10^{-10}$	0.50 ± 0.001

pulse. Photoelectrons of 90 eV kinetic energy and a Lorentzian energy distribution of 1 eV FWHM have been considered for initial momenta directed perpendicular to the surface (directed emission) and for isotropic initial momentum distribution into a half sphere. In addition, we consider an analytical model of space charge effects [44, 45], describing the propagation of an instantaneously created electron disk at a nominal electron kinetic energy E_k, relating line broadening ΔE to the electron number in the pulse N_{el} and the initial radius of the electron disk r_0 through

$$\Delta E = a\sqrt{E_k} N_{el}^b \tag{1}$$

with $a = \left[\frac{2e}{\epsilon_0 \pi r_0}\left(\frac{2}{3\pi} + \frac{1}{6}\right)\right]^{1/2}$ and $b = 0.5$, the electron charge e and the permittivity of free space ϵ_0. From these model considerations, we directly see in figure 2(b) how the initial electron phase space affects the space charge induced peak broadening.

The experimentally observed peak broadening is close to the isotropic simulation, reflecting the fact that the W 4f photoemitted electrons have a wide angular distribution. The onset and the dependence on the number of electrons are significantly different for directed emission, which is also the assumption of the analytical model. We can now summarize the experimentally found peak broadening and the ASTRA simulations by fitting the space charge broadening to the power law from equation (1) through parameters a and b. The respective values are given in table 1. We note that the experimental results and the simulation in the case of isotropic emission show an almost linear dependence on the electron number similar to what has been observed by Zhou *et al* [46].

After determining the working range for electron spectroscopy on a solid surface with pulses from FLASH, we can now embark on femtosecond time resolved electron spectroscopy, using delayed optical pulses for excitation. With this set-up we have studied the appearance of sidebands in the W 4f photoemission spectrum. It is well known from gas phase spectroscopy that the overlap of an optical pump and an XUV probe pulse in time leads to the appearance of sidebands in the photoemission spectrum originating from the dressing field experienced by the photoemitted electrons [49]–[52]. The magnitude of these sidebands as a function of time delay between the laser and XUV pulses represents a cross-correlation measurement, which has been used at FLASH to determine the statistical jitter (250 fs RMS [50]–[52]) between the synchronized optical and XUV pulses. On a solid surface, a first observation of sidebands has

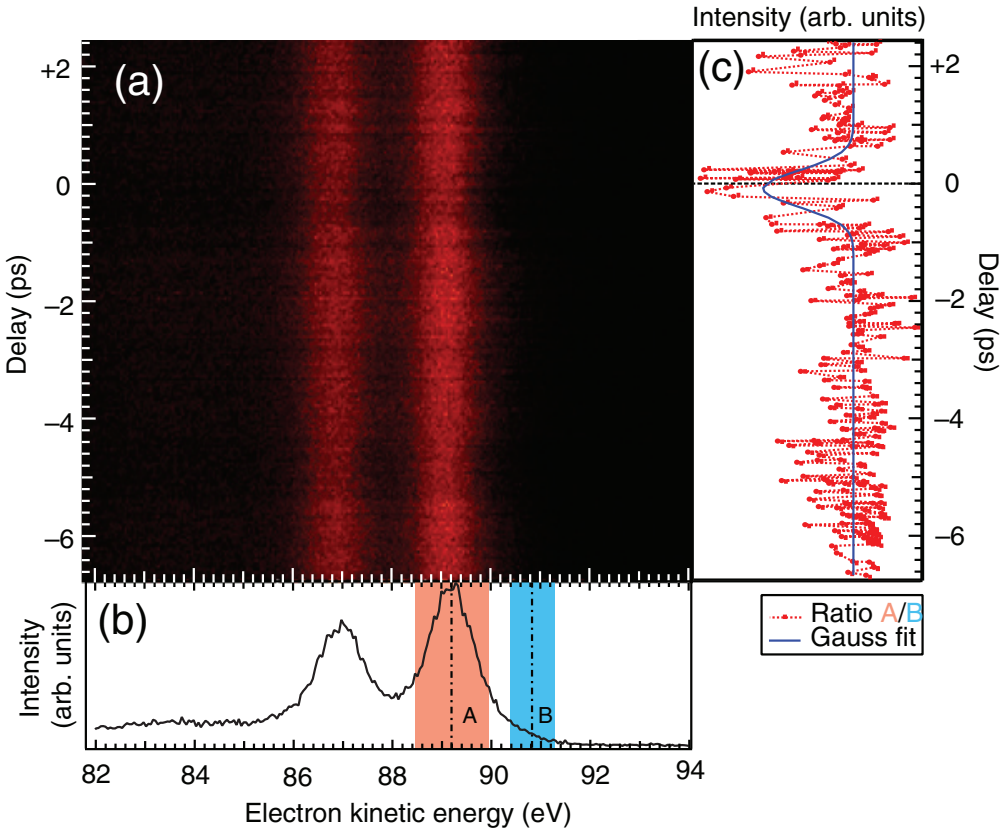

Figure 3. (a) W 4f photoemission spectra for different delays between optical and XUV pulses. (b) Single W 4f spectrum. The energy range of the sidebands is marked (B) together with the reference range (A). (c) Evolution of the ratio A/B as a function of the delay between pump and probe pulse. A dip at delay $t = 0$ indicates the appearance of sidebands. A Gaussian fit gives a width of 440 ± 90 fs with reduced $\chi^2 = 3.9$.

been reported in the ultraviolet photoemission valence spectrum of Pt(111) using 1.6 eV pulses at 2×10^{12} W cm^{-2} and 42 eV high harmonic radiation pulses [53].

In figure 3, our results on sideband generation at the W 4f edge are shown, using 1.55 eV optical radiation (4.62×10^{10} W cm^{-2}). Here, the nominal delay between the optical and the x-ray pulses has been varied. As the sidebands appear at ± 1.55 eV with respect to the photoemission peaks, we present in figure 3(c) the ratio between the unmodified photopeak (A) and the sideband intensity (B) (see figure 3(b)). At temporal overlap ($t = 0$), determined separately by x-ray induced transient optical reflectometry [54], the appearance of sidebands to the W 4f photoemission leads to a dip in the ratio A/B with a Gaussian width of $\Delta t = 440 \pm 90$ fs. The temporal width of this cross-correlation measurement is governed by the temporal jitter of the FEL with respect to the optical laser as well as temporal drifts within the FLASH macro-bunch trains of up to 800 fs caused by electronic feedback systems of the accelerator structure [54]. The temporal width of the optical pulse (120–150 fs) and the XUV pulse are negligible in our case, but their cross-correlation can be retrieved when the jitter is determined independently through electro-optical sampling [50]–[52].

4. Conclusion

In conclusion, we have conducted an investigation of space charge induced peak shift and peak broadening in W 4f core-level photoemission from a W(110) single crystal surface using femtosecond x-ray pulses from the FLASH. We have established experimentally and computationally what should be generally applicable conditions for photoelectron spectroscopy on solid surfaces using femtosecond x-ray pulses from FEL facilities. As a next step, we have studied within this working range time-resolved core-level photoelectron spectroscopy on a surface combining FLASH with a femtosecond optical laser synchronized to the femtosecond x-ray pulses. The observation of sidebands to the W 4f core level lines confirms the cross-correlation between the femtosecond x-ray and optical pulses needed for future studies of femtosecond time-resolved core-level photoelectron spectroscopy on solid surfaces.

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