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To cite this article: J Rubio-Zuazo et al 2008 J. Phys.: Conf. Ser. 100 072032

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A novel electrostatic electron analyzer for Hard X-Ray Photoelectron Spectroscopy (up to 15 keV)

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Abstract. At the Spanish CRG beamline (SpLine) located at the European Synchrotron Radiation Facility (ESRF) we have developed a complex experimental set-up dedicated to the combination of X-Ray Diffraction (XRD) and Hard X-ray Photoelectron Spectroscopy (HAXPES). For that we have developed a novel high energy electron analyzer that fulfills the requirements imposed by both techniques. The analyzer is a cylinder sector analyzer (CSA300HV) designed to operate at energies between few eV and up to 15 keV. Since one year, it is operated routinely up to 15 keV electron kinetic energy. In this work we present a detailed description of the developed electron analyzer together with the experimental determination of its intrinsic properties in terms of transmission, energy resolution and kinetic energy range achievable.

1. Introduction

HAXPES is a powerful emerging technique for bulk compositional, chemical and electronic properties determination. It benefits from the exceptionally large escape depth of high kinetic energy photoelectrons enabling the study of bulk and buried interfaces up to several tens of nanometers depth. Lindau et al. [1] made in 1974 the first HAXPES experiment measuring the Au 4f subshell at a photon energy of 8 keV. However, no other group followed this pioneering work certainly because of the strong drop of the photoemission cross-section at higher photon energies and to the lack of commercial analyzers available to handle high electron energies. For low-Z elements a drop of up to 4 orders of magnitude is observed when the photon energy is increased from 2 keV to 15 keV [2]. Very high photon fluxes requested by HAXPES became available with the third generation synchrotron sources. Due to this many laboratories are starting now research programs based on HAXPES [3].

At the CRG Spanish synchrotron beamline SpLine installed at the ESRF, Grenoble, France, we have developed a novel cylindrical sector electron analyzer (CSA300HV) which fulfills the technical requirements imposed by the combination of XRD and HAXPES. For XRD a large sample to detector distance is requested in order to keep a maximum access to the reciprocal space. The analyzer should be of both small dimensions and high luminosity, adapted to the spot size of the synchrotron radiation, as the photoemission cross-sections are extremely low at high excitation energies. The energy resolution should be comparable to standard XPS. The analyzer should also operate at low energies...
(few eV) to be able to correlate surface and bulk properties by the combination of the low energy surface sensitive data with the high energy bulk sensitive data. The CSA300HV analyzer has been installed since the beginning of 2006, and is running routinely in the full energy range from few eV up to 15 keV [4]. In this manuscript, we present the analyzer’s main characteristics and its intrinsic properties in terms of transmission, resolution and energy range.

2. Electron analyzer description
A schematic layout and a picture of the developed analyzer CSA300HV are shown in Figure 1. The analyzer is a sector of a cylindrical mirror analyzer with a five-element retarding zoom lens system. The lens is operated at fixed spatial magnification (M= 1 .. 60) and varying retardation ratios R=Ekin/Ep=0.01 .. 1500. The internal and external radii are 32mm and 130mm, and the entrance to exit slit distance is 300mm. The entrance central angle is 45° and the total deflection angle of 90° [5]. The sample-to-lens distance is 50mm and the lens and analyzer angular acceptances are ±15° and ±5.4°, respectively. Due to the large dispersion values [6] the CSA300HV has a reduce dimension and weight of ~30 kg only. The fully computer-controlled high voltage power supply with a stability <30meV can be operate continuously from few eV to 15 keV without changing the electronics setup.

Figure 1. Schematic optical layout (left) and a picture (right) of the developed CSA300HV analyzer. The entrance slit w₁ and exit slit w₂ are located at the lens image and detector position, respectively and their dimension are given in mm in the text.

3. Experimental results
The analyzer capabilities have been tested in terms of resolution and transmission using the synchrotron radiation offered by the SpLine beamline [7]. Figure 2a shows a sequence of Cu 2p_{3/2} peaks obtained at ~10 keV photon energy (~9 keV electron kinetic energy) for different pass energies E_p. The photon flux was 10^{11} Photons/s and the acquisition time used 1 sec per point. The FWHM ∆E_exp and the peak intensity I dependence with E_p are shown on Figures 2b and 2c for different slit sets. The dependence of ∆E_exp with E_p has been fitted by the function ∆E_{exp}=(∆E_{Xray}^2+∆E_{X,LW}^2)^{0.5}, where ∆E_A is the analyzer resolution and (∆E_{X,LW}^2=(ΔE_{X-ray}^2+(ΔE_{Cu-LW}^2))^2 is the quadratic addition of the X-ray bandwidth and the natural Cu 2p_{3/2} line width, assuming that the three contribution are Gaussians. Figure 2b shows the obtained fit of ∆E_{exp} (solid line) and the corresponding extrapolation (dashed line) for the intrinsic analyzer resolution ∆E_A, ∆E_A/E_p=0.039 and ∆E_A/E_p=0.015 values are obtained for w₁/2=9mm and w₁/2=3mm slits, corresponding to an analyzer resolution of ∆E_A=390meV and ∆E_A=150meV at E_p=10eV, respectively. ∆E_{exp} is limited by the X-ray bandwidth value of ∆E_{X-ray}=1.63eV, not by ∆E_A. This will be improved with the future installation of a post-monochromator. The behaviour of I with E_p is dependent on the lens transmission T. T is proportional to (r_o sin α_o)^2, where r_o and α_o are the space and angular dimension at the sample, respectively. r_o is defined by the lateral lens magnification M and the entrance slit w₁ (r_o = w₁/M). α_o is related to the retardation R = E_{kin}/E_p, the analyzer acceptance angle α_A and M through sin α_o = M sin α_A R^{-0.5} (Liouville’s law), thus T is given by T=(w₁ sin α_o R^{-0.5})^2=(w₁ sin α_o)^2 (E_p/E_{kin}). Hence, for a given kinetic energy the transmission is proportional to E_p and w₁^{-2}, as α_A is constant. This prediction is in perfect agreement with the obtained data as it is show in Figure 2c. The symbols are the experimental data and the fitted intensities (solid lines) follow the expected behavior as function of E_p.
The analyzer’s capability to cover a wide energy range (few eV to 15 keV) keeping its outstanding performances is demonstrated in figure 3. Figure 3a shows a valence band spectrum (VB) from a polycrystalline Ag sample obtained at 21.21 eV photon energy at room temperature. Slits of \(w_1=w_2=3\) mm and \(E_p=1\) eV were used. The inset shows the expanded Fermi cut-off, the continuous line illustrates the results from the best fit of a Fermi function. A pseudo kT value of 39 meV is obtained, that correspond to a \(\Delta E_{\text{exp}}=130\) meV (3.32 times pseudo kT-value). The difference to the expected RT value (\(\Delta E_{\text{exp}}=84.4\) meV) is due to broadening induced by the analyzer. Assuming a Gaussian analyzer function an analyzer intrinsic resolution of \(\Delta E_A=100\) meV is obtained. The analyzer resolution is reduced to \(\Delta E_A=40\) meV (not shown here) making a similar analysis at the same conditions but with 1 mm slits (\(w_1=w_2=1\) mm). Hence, values of \(\Delta E_A/E_p=0.1\) and \(\Delta E_A/E_p=0.04\) are obtained at low energy and \(E_p=1\) eV for \(w_1/2=9\) mm and \(w_1/2=1\) mm. These two values are larger than the expected values as deduced from the high energy data (s. above). This discrepancy can be explained by the power supply ripple getting discernible at very low pass energies. However, it must be stressed that the same power supply is used in both cases and the analyzer resolution limit is better than 40 meV. Figure 3c shows the gold 3s, 3p and 3d core level spectra obtained with the CSA300HV analyzer for a photon energy of \(\hbar \nu=9\) keV and \(\hbar \nu=17\) keV, corresponding to an kinetic energy range of \(E_{\text{kin}}=5-15\) keV. These spectra in combination with the spectrum shown in Figure 3a demonstrate the wide electron kinetic energy range covered by the analyzer. Hence, the developed energy analyzer maintains its characteristic absolute energy resolution for a wide range of kinetic energies, i.e., from few eV up to 15 keV.
Figure 3. (a) VB spectra obtained on a polycrystalline Ag sample. Inset shows the measured Fermi cut-off with a HeI source. The inset shows the expanded Fermi cut-off, the solid line corresponds to the best fit of a Fermi function to the measured data. (b) Representative HAXPES 3s, 3p and 3d core level spectra obtained with the CSA300HV analyzer measured on a polycrystalline Au sample for a photon energy of $h\nu = 9$ keV ($E_{\text{kin}} = 5.4 \ldots 6.9$ keV) and $h\nu = 17$ keV ($E_{\text{kin}} = 13.4$ keV $\ldots 14.9$ keV). The bottom spectrum is multiplied by a factor 3. Note the absolute and relative cross section differences in both spectra.

4. Conclusions and outlook
We have developed a novel outstanding electron analyzer that fulfills requirements imposed by the XRD and HAXPES techniques. It is the only commercially available analyzer capable to handle kinetic energies up to 15 keV down to few eV. Spectra over the full range are obtained with the same setup of the analyzer and power supply. The presented results demonstrate that the analyzer’s energy resolution and transmission perform as expected over the full energy range. The implementation of a 2D-detector for parallel data acquisition is currently in preparation to increase the measurement speed and overcome the restrictions imposed by the low cross sections at high kinetic energies.

Acknowledgements
We would like to thanks the SpLine and Focus GmbH staff for their valuable help in the development of the electron analyzer. Financial support for this research was provided through Spanish ministry of Education and Science (MEC) Grants nos. FAP-2001-2166 and MAT1999-0241-C01.

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