COMPACT SPECTROMETER FOR SINGLE SHOT X-RAY EMISSION AND PHOTON DIAGNOSTICS

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Abstract

The design and characterization of a compact spectrometer realized for photon in-photon out experiments (in particular X-Ray Emission Spectroscopy), conceived to be used at the FERMI free-electron-laser (FEL) at ELETTRA (Italy) is here presented. The instrument can be easily installed on different end stations at variable distances from the target area both at synchrotron and FEL beamlines. Different input sections can be accommodated in order to fit the experimental requests. The design is compact in order to realize a portable instrument within an overall size of less than one square meter. The spectrometer covers the 25-800 eV spectral range, with spectral resolution better than 0.2%. The characterization on Gas Phase @ ELETTRA as instrument for XES and some experimental data of the FEL emission acquired at EIS-TIMEX @ FERMI, where the instrument has been used for photon beam diagnostics, are introduced.

INTRODUCTION

X-ray emission spectroscopy (XES) is a well-established method in surface and solid-state investigations at third generation synchrotron radiation sources [1-2]. The instrument presented here is designed for photon in-photon out experiments, in particular XES, at synchrotron and FEL beamlines. The equipment is intended to be used at the LDM (Low-Density-Matter) [3] and EIS-TIMEX (Elastic and Inelastic Scattering - TIme-resolved studies of Matter under EXtreme and metastable conditions) [4] beamlines of FERMI. Additionally, it can be used as a diagnostic tool for the real-time shot-to-shot acquisition of the FERMI spectral content (both fundamental and high-harmonics) and of the shot-to-shot fluctuations beam characteristics, especially at energies above 250 eV, where it could be complementary to the existing spectrometer used as a diagnostic of FERMI [5]. Two selectable gratings are used to cover the 25-800 eV energy range with a spectral resolution higher than 0.2% and an acceptance angle as high as 1.7×10^{-4} rad. Different input sections, with/without an entrance slit and with/without an additional relay mirror can be attached to the spectrometer to adapt it to the size of the experimental chamber.

INSTRUMENT DESIGN

The optical design of the instrument is well established both for FELs [6] and high-order laser harmonics [7,8] and has been presented elsewhere [9]. It consists of an entrance slit, a grazing-incidence spherical diffraction grating with variable groove spacing and a detector. The 25-800 eV range is covered by gratings (Hitachi cod. 001-0437, 1200 gr/mm and cod. 001-0450, 2400 gr/mm). An EUV-enhanced back-illuminated CCD camera (Princeton Instruments PIXIS-XO 400B, 1340 × 400 pixel, 20-um pixel size), is mounted on a motorized linear translation stage and is connected to the grating stage by a bellows. Since the length of his focal curve is longer than the detector size, the latter is moved by means of a motorized stage to cover the whole spectral region. Three configurations have been realized by connecting three different input stages to the grating block. Configuration A is shown in Fig. 1. It has a variable-width entrance slit. Configuration B, shown in Fig. 2, has an additional cylindrical mirror acting as a relay section between the slit and the grating. In this way, the distance between the input and the grating is increased. Configurations A and B were tailored to the needs of the experimental chambers of the Gas Phase beamline of Elettra and the LDM beamline of FERMI and were designed for measurements on gas samples. Configuration C, shown in Fig. 3, is mainly planned to be used in chambers for measurements on solid targets. It is operated without an entrance slit, since the FEL focal spot on the sample acts as point-like source of the instrument. Again, a cylindrical mirror was added to the configuration, acting as a relay section between the source and the grating to adapt the envelope of the instrument to the size of the TIMEX experimental chamber. To maintain the pressure gradient between the inner and outer parts of the shield that contains the instrument, a pumping system is connected via a dedicated pumping flange to the spectrometer.

The spectral resolving element of the instrument, defined as the energy dispersion on the 20-µm detector pixel, is shown in Fig. 4. The global response of the

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instrument, in terms of counts per input photon, is presented in Fig. 5.

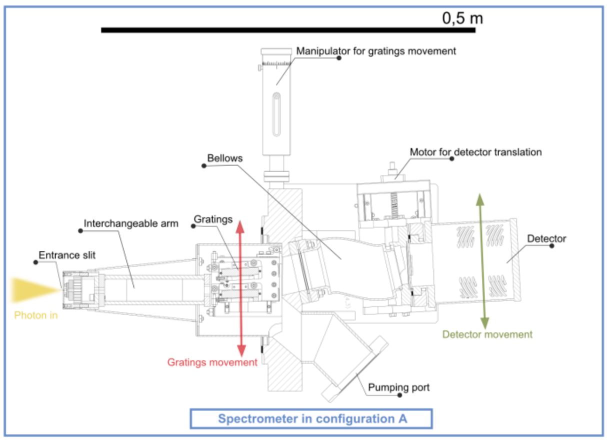

Figure 1: Spectrometer in configuration A.

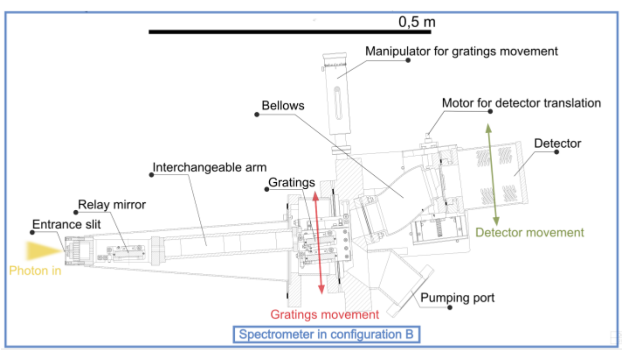

Figure 2: Spectrometer in configuration B.

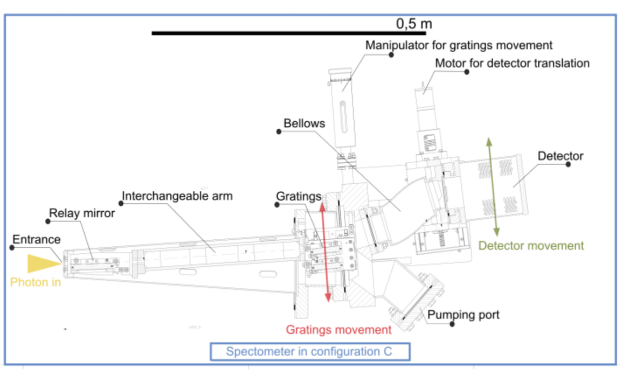

Figure 3: Spectrometer in configuration C.

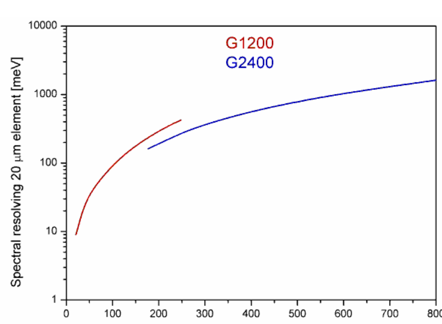

Figure 4: Spectral resolving element of the instrument.

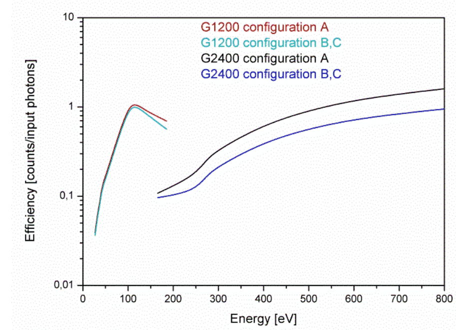

Figure 5: Global response of the spectrometer. It is defined as the product of the efficiency that has been measured for each component, namely the gratings, the detector and the mirrors.

The instrumental parameters of the instrument are finally summarized in Table1.

The collection angle in the direction of the spectral dispersion is limited by the angular acceptance of the gratings, which is 10 mrad for G1200 and 5 mrad for G2400. In the direction perpendicular to the dispersion, the collection angle is limited by the size of the detector, being in the range of 9-17 mrad depending on the instrument configuration.

Table 1: Instrumental Parameters

Grating G1200	Spherical VLS	
Central groove density	1200	mm^{-1}
Photon energy range	25-250	eV
Incidence angle	87	deg
Grating G2400	Spherical VLS	
Central groove density	2400	mm^{-1}
Photon energy range	180-800	eV
Incidence angle	88.7	deg
Grating entrance arm	237	mm
Grating-to-detector distance	235	mm
CCD detector		
Format	1340 × 400	
Pixel size	20 × 20	µm
Detector area	26.8 × 8	mm
Configuration A: entrance slit + grating		
Slit-to-grating distance	237	mm
Configuration B: entrance slit + relay mirror + grating		
Slit-to-grating distance	457	mm
Configuration C: relay mirror + grating		
Source-to-grating distance	637	mm

INSTRUMENT AS X-RAY EMISSION SPECTROMETER

The spectrometer as instrument for X-ray emission experiments was fully characterized at the low-energy branch of the Gas Phase beamline of Elettra synchrotron by acquiring fluorescence spectra from solid and gas targets. The instrument was assembled in configuration A and mounted perpendicular to the direction of the synchrotron beam with the entrance slit 10 mm away from the focal point and opening of 200 µm. The photon

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flux on the sample was in the 10^9-10^{11} ph/s range, with the lower flux measured above 150 eV, where the plane mirror of the branch line has its cut-off in reflectivity.

Fluorescence from Solid Target

Measurements on solid targets were performed on silicon and on boron or boron nitride powder. The samples were positioned on the focus of the branch line at an incidence angle variable in the range 60-80 deg, in order to have the elastic contribution out of the spectrometer. A Si fluorescence spectrum taken at the Si L-edge is shown in Fig. 6. The spectrum is in overall agreement with literatures on crystalline Si films [10]. B and BN fluorescence spectra have been taken at the B K edge. In Fig. 7 the main emission peak due to a B(2p)-B($1s^{-1}$) transition is presented for B, in agreement with the data in the literature [11].

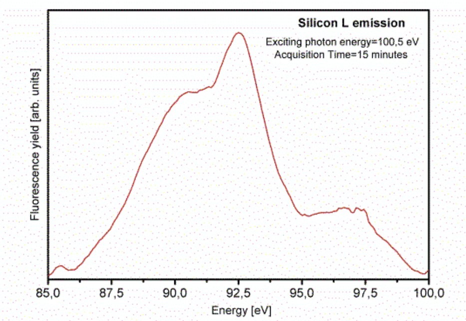

Figure 6: Si L emission fluorescence spectrum for crystalline Si.

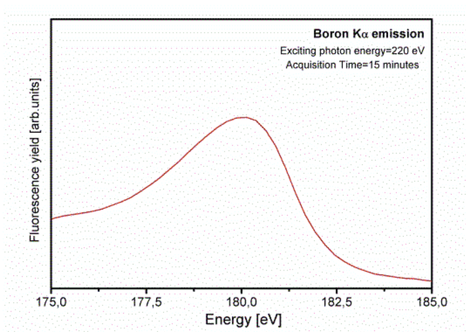

Figure 7: B Kα emission fluorescence spectra.

Fluorescence from Gas-Phase Targets

To measure fluorescence from gas targets, a suitable gas cell was mounted close to the entrance slit of the spectrometer, having entrance and exit holes for the synchrotron beam and a lateral slit (120-µm wide and 5-mm long) parallel to the entrance slit of the spectrometer, to maximize the throughput of the fluorescence that is collected by the instrument. The pressure in the experimental chamber was measured to be few-10^{-3} mbar with a pressure inside the gas cell in the several-10^{-1} mbar range.

He fluorescence spectra emitted when the exciting photon energy was fixed either to the (3,0) resonance at 64.118 eV or to the (4,-1) resonance at 64.133 eV are shown respectively in Fig. 8 and in Fig. 9. The emission lines observed in the different experimental conditions are in agreement with the literature [12]. When exciting at 64.118 eV, a peaked photon emission due to the $(2p3d)^1P$-$(1s3d)^1D$ transition is measured, whereas the emission at 40.81 eV is due to He photoionization from higher-orders synchrotron light. The emission spectrum changes appreciably when different resonances of He are populated, as in the case of the (4,-1) resonance at 64.133 eV. Fluorescence emission from Kr excited at the $M_{4,5}$ edge was also measured. Fig. 10 shows the spectrum with exciting photon energy at the Kr $3d_{5/2}\rightarrow 5p$ resonance (91.2 eV). The Kr transitions $3d_{5/2}^{-1}\ 5p_{3/2}$ (J=1) – $4p_{3/2}^{-1}\ 5p_{3/2}$ (J=0,2) are clearly visible around 80 eV.

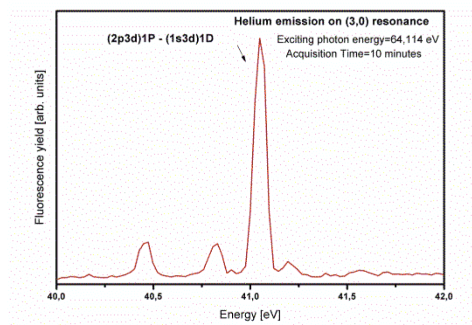

Figure 8: He fluorescence spectrum on (3,0) resonance at 64.114 eV.

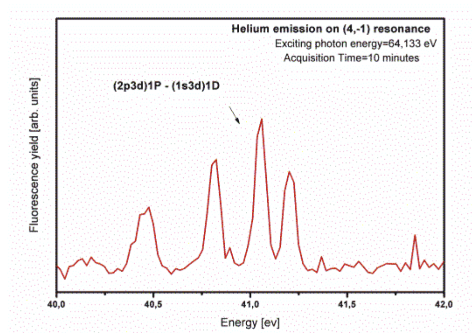

Figure 9: He fluorescence spectrum on (4,-1) resonance at 64.133 eV.

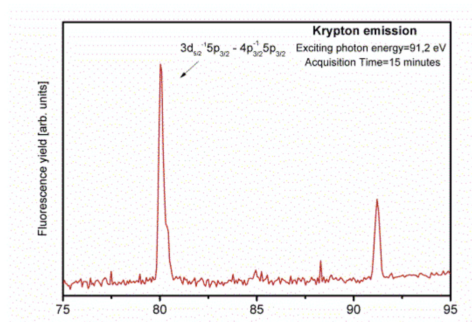

Figure 10: Krypton fluorescence spectrum with 91.2 eV exciting photon energy.

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INSTRUMENT AS A TOOL FOR FEL DIAGNOSTICS

The instrument in configuration C has been interfaced to the EIS-TIMEX chamber to perform diagnostics on the FERMI beam. Single-shot spectra have been acquired successfully. Some of the single-shot spectra acquired at the 9th harmonic (35,97 nm) are shown in Fig. 11. The intensity fluctuations reflect the shot-to-shot variations in the photon-beam parameters and are in agreement with the typical pulse-to-pulse distribution of the FEL pulse energy. The FEL emission has also been tuned over 100 eV, where the expected photon fluxes are lower. The capability to acquire single-shot spectra at this energies confirms the high sensitivity of the instrument to very low photon fluxes. The net counts per shot on the CCD camera at 103.3 eV are ≈55000 counts. Considering the CCD conversion factor (2 e^-/count) and efficiency (13 e^-/ph), the grating efficiency (0.17) and the reflectivity of the Si mirror that was used to redirect the light into the instrument ($4 \cdot 10^{-5}$ at 103.3 eV), the flux entering into the TIMEX chamber at 103,3 eV is $1{,}2 \cdot 10^9$ ph/pulse.

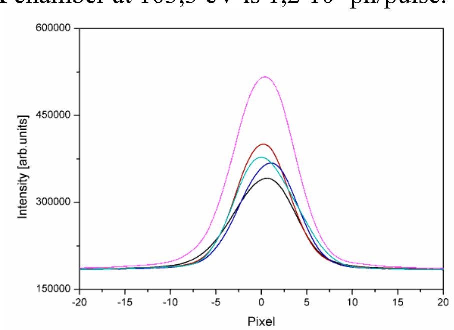

Figure 11: Single shot spectra acquired at the 9th harmonic of the seed laser set at 323.7 nm.

CONCLUSION

The portable and compact photon spectrometer to be used for X-ray emission experiments and photon diagnostics at FERMI has been presented. The spectrometer was fully characterized by measuring fluorescence spectra from solid and gas targets with synchrotron radiation at Gas Phase @ ELETTRA. The collected spectra are in full agreement with data reported in the literature, in particular the fluorescence spectra from gas targets have a signal-to-noise ratio that is higher than the data available in the literature, opening the way to the use of the spectrometer for specific gas targets where experimental data are missing. Single-shot FEL emissions have been measured at EIS-TIMEX @ FERMI. High resolution in a wide spectral region, high sensitivity and high dynamic have been tested for single-shot acquisition of the FEL beam. This opens to the possibility to use the instrument as an on-line instrument for the inspection of the spectral content of the FEL emission, complementary to the high-resolution spectrometer presently available at FERMI. Furthermore, the possibility to use the instrument at energies up to 800 eV opens perspectives to test and characterize new configurations of the FERMI FEL acceleration sections.

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REFERENCES

[1] U. Bergmann and P. Glatzel, “X-ray emission spectroscopy,” Photosynth. Res. 102, 255 (2009)

[2] J. Yano and V.K. Yachandra, “X-ray absorption spectroscopy,” Photosynth. Res. 102, 241 (2009)

[3] A. Di Cicco et al., “Probing matter under extreme conditions at Fermi@Elettra: the TIMEX beamline,” in Damage to VUV, EUV, and X-ray Optics III, SPIE Proc. 8077, 807704 (2011)

[4] V. Lyamayev et al., “A modular end-station for atomic, molecular, and cluster science at the low density matter beamline of FERMI@Elettra,” J. Phys. B 46, 164007 (2013)

[5] M. Zangrando et al., “The photon analysis, delivery, and reduction system at the FERMI@Elettra free electron laser user facility,” Rev. Sci. Inst. 80, 113110 (2009)

[6] F. Frassetto et al., “Extreme-ultraviolet compact spectrometer for the characterization of the harmonics content in the free-electron-laser radiation at FLASH”, J. Synchrotron Radiation 19, 596 (2012)

[7] L. Poletto et al., “Grazing-incidence flat-field spectrometer for high-order harmonic diagnostics”, Opt. Eng. 40, 178 (2001)

[8] L. Poletto et al.,”High-order laser harmonics detection in the EUV and soft X-ray spectral regions”, Rev. Sci. Instr. 72, 2868 (2001)

[9] L. Poletto et al., “Instrument for single-shot X-ray emission-spectroscopy experiments”, in Advances in X-ray Free-Electron Lasers II: Instrumentation, SPIE Proc. 8778, 87780W (2013)

[10] J. A. Carlisle et al., “Soft-x-ray fluorescence study of buried silicides in antiferromagnetically coupled Fe/Si multilayers,” Phys. Rev. B 53, 14 (1996)

[11] Y. Muramatsu et al., “Resonant x-ray Raman scattering in B Kα emission spectra of boron oxide (B2O3) excited by undulator radiation,” Phys. Rev. Lett. 71, 3 (1993)

[12] K.-H. Schartner et al., “Radiative cascades from doubly excited He states,” Phys. Rev. A 64, 040501 (2001)

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