A portable diode system for the quantification of absolute VUV/UV photon fluxes in low pressure plasmas

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Abstract: The absolute determination of VUV photon fluxes is rather complex due to the necessity of a direct vacuum connection and an elaborate intensity calibration of the required spectrometer. Therefore, an easy-to-use and portable device based on a silicon diode is developed in which spectral resolution is achieved with band and long pass filters. The diode system is calibrated against an absolutely intensity calibrated VUV spectrometer. The photon fluxes measured in ICP discharges are in good agreement with results of the spectrometer.

Keywords: Absolute photon fluxes, VUV spectroscopy, diode system, low pressure plasma.

1. Introduction

Photon fluxes in the vacuum ultraviolet spectral region (VUV, wavelength below 200 nm) and in the ultraviolet range (UV, wavelength 200 – 400 nm) can have significant influence during surface treatment processes with low pressure plasmas. Photons arise from atomic and molecular transitions of the respective atoms and molecules which determine the spectral composition of the emitted radiation. Figure 1 gives an overview of the photon energy ranges of molecular transitions in H_2 (green), N_2 (blue) and O_2 (grey) plasmas as well as of the resonant atomic transitions in Argon (red).

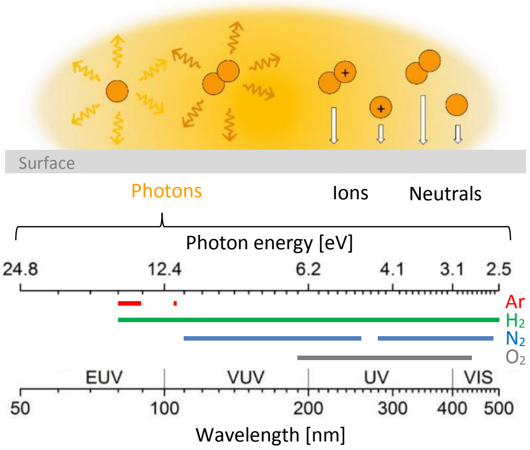

Fig. 1. Photon energies and particle species in surface treatment with low pressure plasmas. Emission ranges of molecular transitions in H_2 (green), N_2 (blue) and O_2 (grey) plasmas and of the resonant atomic lines of Argon (red) are indicated.

The effect of VUV/UV radiation on the surface material depends on the absolute flux and on the photon energy. Depending on the application, it can be beneficial or undesirable for the surface treatment. The quantitative investigation of a hydrogen discharge showed that the VUV photon flux exceeds the photon flux in the optical region and can be comparable to the ion flux [1]. That underlines the crucial role of VUV/UV photon fluxes in process discharges which must not be neglected and that an absolute determination of energy resolved photon fluxes is indispensable. Moreover, the photon fluxes strongly depend on the geometry of the specific discharge and on the position of the substrate which in turn leads to the necessity of in-situ measurements.

An absolute quantification of VUV/UV photon fluxes is rather challenging since a direct vacuum connection and large vacuum spectrometers are necessary. The absolute calibration of these spectrometers is hard to perform and must be carried out specifically at the specific plasma setup. To overcome these difficulties, an easy-to-use measurement device based on a silicon photodiode is under development. The intensity calibration of the diode system is performed against an absolutely calibrated VUV spectrometer and is then independent of the discharge setup in use. Its compact design allows measurements of photon fluxes directly at the position of interest.

Systematic measurements of absolute VUV/UV photon fluxes in ICP discharges simultaneously performed with the diode system and a VUV spectrometer are presented. For an extended benchmark of the diode system, pressure and power scans in hydrogen and nitrogen, in H_2/N_2 and in mixtures with O_2 as well as in argon are performed.

2. Experimental setup

2.1 Design of the VUV diode system

A sketch of the diode system is depicted in figure 2. The detector element consists of a silicon photodiode (*Opto Diode*, type *AXUV100G*) with an active area of 1 cm^2. The diode is sensitive for radiation with a wavelength between 1 nm and 1100 nm. To obtain spectral resolution, filters are selected in the mechanical filter wheel in front of the diode taking into account the investigated gas as well as the photon energy region of interest. An aperture between the diode system and the plasma chamber limits the solid angle which makes the intensity calibration independent of the specific application. As the device is attached directly to the plasma chamber, no additional pumping system is required. The signal output is connected via an amplifier to the data acquisition system.

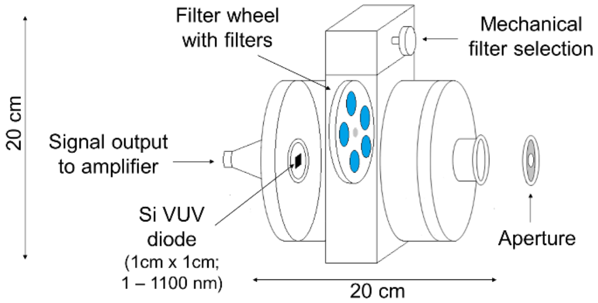

Fig. 2. Design of the VUV diode measurement system.

2.2 Calibration of the measurement setup

The absolute calibration of the diode system was performed in a planar ICP (ø15 cm, height 10 cm; 2 MHz; 0.3 – 10 Pa; RF power up to 2 kW) against an 1 m McPherson VUV spectrometer (*Model 225*, grating with 1200 lines per mm). The VUV spectrometer can be equipped either with a solar-blind photo multiplier (PMT, *EMR 51F-08-18*) or with a windowless Channel Electron Multiplier (CEM, *McPherson Model 425*, sensitive 1 – 180 nm, MgF_2 coated). The relative intensity calibration of the PMT setup is performed between 190 nm and 300 nm with a deuterium arc lamp and extended down to 116 nm by using the branching ratio method for atomic and molecular nitrogen according to [2]. The detection of helium lines in the wavelength region between 250 nm and 300 nm simultaneously with the VUV spectrometer and an absolutely calibrated optical spectrometer at the same line of sight allows an absolute calibration in the wavelength range 116 – 300 nm. An absolute calibration of the VUV spectrometer with the CEM can be obtained between 50 nm and 124 nm directly by using the radiation of a calibrated high current hollow cathode which is operated with several different gases [3]. Hence, the VUV spectrometer allows measurements of absolute photon fluxes in the wavelength range from 50 nm to 300 nm.

The calibration of the diode system against the VUV spectrometer was carried out for a set of filters in a hydrogen or in a hydrogen/oxygen discharge, respectively. The calibration as well as the later comparison of the two VUV systems are only valid for a homogenous plasma emission over the complete observation volumes. In order not to limit the vertical plasma diffusion from the coil across the plasma vessel and thus to provide a spatially uniform plasma emission, discharges with a low pressure (1 Pa) and high RF power (900 W/ 1100 W) were chosen. The calibration is performed in the way that the voltage signal measured with the diode system and a certain selected filter can be directly converted into the corresponding photon flux (photons/m^2/s) via a calibration factor (photons/m^2/s/V) which is specifically determined for each particular filter.

The filter set includes band pass (BP) and long pass filters (LP) which were chosen primarily to be applicable in H_2 plasmas. Their transmission was measured by using either a deuterium arc lamp, an Ulbricht sphere or directly a hydrogen discharge. The filters are summarized in table 1, where the centre wavelength and the FWHM for band pass or the cut-on wavelength for long pass filters, respectively, are stated. These indications are measured values except for 122-BP for which a direct measurement of a transmission curve was not possible with the available radiation sources described above. With a hydrogen discharge, however, it could be seen that the filter only transmits a small wavelength interval around the L_α line. Therefore, the peak wavelength and the FWHM given by the manufacturer are indicated in case of the 122-BP.

For some energy ranges of interest corresponding band pass filters are available and can be applied. In the other cases the photon fluxes cannot be directly determined. For this purpose, combinations of several measurements with long pass filters and/or without any filter are necessary. Table 2 shows the wavelength ranges and the corresponding spectral region including the atomic or molecular transitions of several gases which are accessible with the diode system and which are calibrated against the spectrometer. The wavelength interval of the particular filter or filter combination is defined by an absolute transmission higher than 5 %. Without any filter selected in the filter wheel the accessible wavelength range is determined by the silicon diode. The manufacturer indicates that the diode is sensitive from 1 nm to 1100 nm.

Table 1. Set of band and long pass filters with measured peak and cut-on wavelength.

Filter name	Type of filter	Peak wavelength	FWHM	Filter name	Type of filter	Cut-on wavelength
122-BP	Band pass	122 nm	14 nm	MgF_2	Long pass	113 nm
151.5-BP	Band pass	154 nm	30 nm	Fused	Long pass	153 nm
228.5-BP	Band pass	230 nm	38 nm	BK7	Long pass	281 nm
313-BP	Band pass	313 nm	10 nm	400-LP	Long pass	397 nm
				500-LP	Long pass	500 nm

Table 2. Applied combination of filters and assigned spectral ranges.

Combination of filters	Wavelength interval	Energy interval	Gas	Assigned spectral region, atomic or molecular transition
No filter – fused silica	< 153 nm	> 8.1 eV	H_2, N_2, …	VUV
400-LP – fused silica	153 – 397 nm	3.1 – 8.1 eV	H_2, N_2, …	UV
No filter – MgF_2	< 113 nm	> 12.1 eV	H_2	Werner band (C-X), Lyman series ($L_{\beta,\gamma,\ldots}$)
122-BP	122 ± 7 nm	10.2 eV	H_2	L_α (n=2 – n=1)
151.5-BP	146 – 186 nm	6.6 – 8.4 eV	H_2	Lyman band (B-X)
			N_2	atomic lines
228.5-BP	196 – 267 nm	4.8 – 6.3 eV	H_2	Continuum (a-b, H_2)
313-BP	308 – 321 nm	3.7 – 4.0 eV	H_2/O_2	OH (A-X)
BK7 – MgF_2	113 – 281 nm	4.4 - 12.1 eV	N_2	Lyman-Birge-Hopfield (a-X; N_2)
500-LP – BK7	281 – 500 nm	2.5 – 4.4 eV	N_2	2[nd] positive system (C-B; N_2)

Due to the same reason mentioned above, the FWHM given by the manufacturer was used to state an accessible wavelength range for the 122-BP filter. Nevertheless, no energy interval but the energy of the photons arising from the centre wavelength of the L_α line is given in Table 2. Furthermore, it has to be kept in mind that no filters are available with a cut-on wavelength of 200 nm which represents the transition from the VUV to the UV spectral region. That, in turn, means that wavelength ranges assigned as VUV and UV, respectively, in Table 3 are slightly different to the primary definition in figure 1.

As an example, figure 3 shows an absolutely calibrated spectrum of a H_2/O_2 plasma (black curve, intensity on left axis) and the transmission curves of some filters (coloured lines, right axis). The spectrum was measured with the spectrometer equipped with the PMT. The corresponding wavelength ranges accessible with the filters applied in the diode system are indicated.

3. Measurement of photon fluxes

3.1 Comparison with VUV spectrometer

An integration over the wavelength region of interest in the absolutely calibrated spectrum measured with the VUV spectrometer results in the emissivity (photons/s/m^3). For the corresponding averaged photon flux onto a surface (photons/s/m^2) the geometry of the present discharge setup has to be taken into account. Therefore, the emissivity is multiplied by the volume to surface ratio assuming homogenous and isotropic emission. For the purpose of an extensive benchmark of the diode system against the VUV spectrometer, the photon fluxes in several molecular ICP discharges were investigated with a variation of pressure and power. Figure 4 exemplarily shows the comparison of the photon fluxes of several spectral ranges obtained with the diode system (full symbols) and the VUV spectrometer (open symbols) for a power scan in H_2 at 1 Pa (fig. 4a) and for a pressure scan in H_2/O_2 at a generator power of 700 W (fig. 4b). An error of ± 20 % is assumed for the VUV spectrometer due to its calibration procedure. As the diode system is calibrated against the spectrometer, this error also counts for the photon fluxes measured with the diode system. Furthermore, the reproducibility of the diode system has to be taken into account. It was determined in a hydrogen discharge for the 122-BP filter to be below ± 5 % and is assumed to be equal for all other filters. In figure 4 the corresponding error bars of ± 5 % lie within the displayed diode symbols and are therefore not shown. For

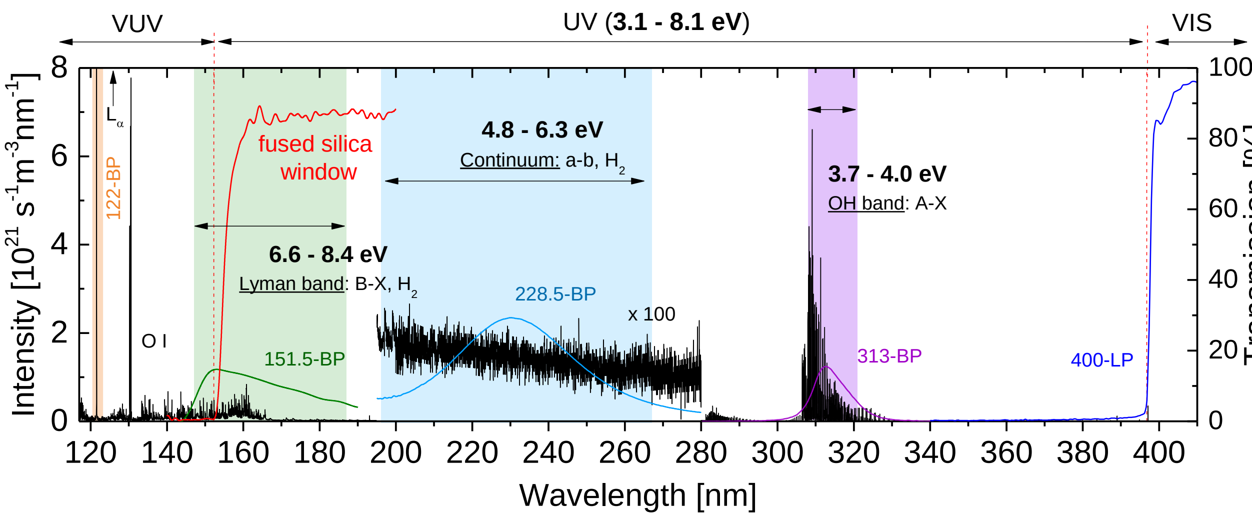

Fig. 3. Example of an absolutely calibrated H_2/O_2 spectrum (black line, intensity on left axis) with transmission curves of some filters (coloured lines, right axis) and their corresponding wavelength ranges. The Continuum is displayed with an amplification factor of 100.

the sake of clarity, the errors resulting from the calibration of the diode system are neglected in figure 4 as well.

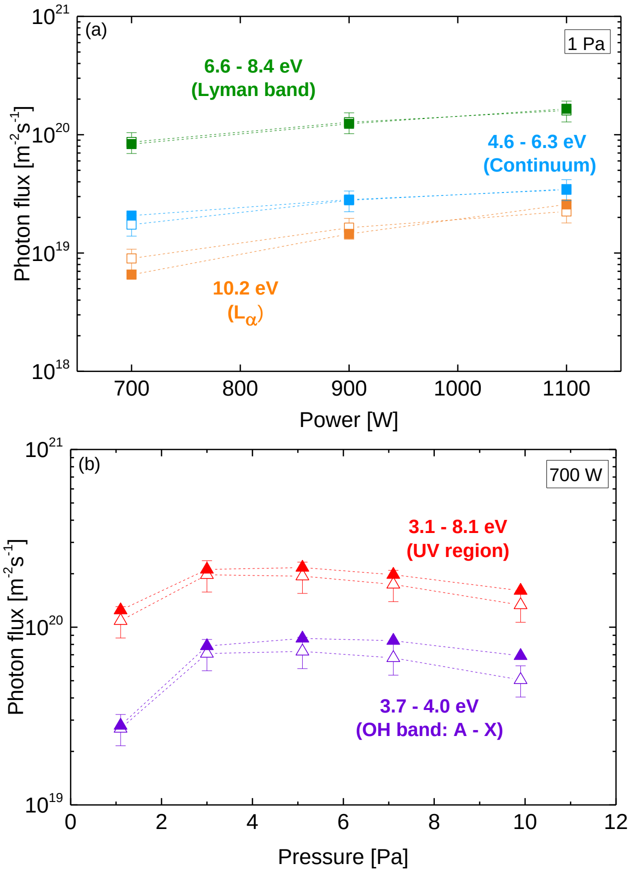

Fig. 4. Photon fluxes obtained with the diode system (full symbols, ± 5 % error bars arising from the reproducibility lie within symbols) and the VUV spectrometer (open symbols, including ± 20 % error bars). (a) Power scan in H_2 at 1 Pa. (b) Pressure scan in H_2/O_2 (85 % H_2:15 % O_2) at 700 W.

The relative power and pressure dependencies measured with the diode system are in good agreement with the VUV spectrometer. With decreasing power and increasing pressure, the trends slightly change and small absolute deviations occur. It has to be kept in mind, that the two diagnostic systems detect different sized cones of the plasma volume and therefore, an absolute comparison only holds strictly if the plasma emits homogenously. With a decrease of the power and an increase of the pressure the plasma emission turns more and more inhomogeneous due to a limited diffusion across the observation volumes. Under this consideration, the benchmark of the diode system against the VUV spectrometer is considered to be successful.

3.2 Spectral composition in a H_2/O_2 discharge

The spectral composition of the photon fluxes in the VUV/UV was investigated with the diode system in a H_2/O_2 (85 %:15 %) discharge. The measurements were performed at a pressure of 5 Pa and a RF power of 700 W and the results are presented in Table 3.

Table 3. Spectral composition of VUV/UV photon fluxes in H_2/O_2 measured with the diode system.

Assigned spectral region	Photon flux [m^{-2}s^{-1}]
UV region	$22 \cdot 10^{19}$
Lyman band	$8.9 \cdot 10^{19}$
OH band	$8.7 \cdot 10^{19}$
L_α	$2.8 \cdot 10^{19}$
Continuum	$3.0 \cdot 10^{19}$

The atomic line L_α, the Continuum and the Lyman band as molecular transitions of hydrogen were investigated as well as the OH band and the wavelength range which is assigned to the UV region. The averaged flux of UV photons onto a surface is 22 $\cdot 10^{19}$ m^{-2}s^{-1}, with 40 % arising from each the Lyman band and the OH band. The photon flux of the atomic L_α line is comparable to the photon flux emitted by the wavelength interval of the Continuum. Both ranges are about 14 % of the UV flux.

4. Conclusion and outlook

The presented intensity calibrated measurements in different plasma discharges show that the diode system allows an easy determination of absolute VUV/UV photon fluxes including information about the specific spectral region. The applied filter set was selected accordingly to energy ranges corresponding to atomic and molecular transitions in hydrogen. The applicability to other gases will be tested next. As a degradation effect by the VUV radiation can be expected, the temporal stability will be checked as well. It is also planned to demonstrate the performance and the applicability of the diode system to other plasma setups.

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