Published by IOP Publishing for Sissa Medialab

Received: *July 2, 2019*
Accepted: *October 12, 2019*
Published: *January 8, 2020*

3rd European Conference on Plasma Diagnostics (ECPD2019)
6–10 May 2019
Lisbon, Portugal

Reversed-slit spectroscopy method for in situ measurement of H isotopes on plasma facing material

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Abstract: Optic spectroscopy methods are employed for plasma facing component (PFC) material diagnostics and H isotopes retention on various platforms. A common issue of those spectroscopy methods is the measurement sensitivity. Various improvement methods have been investigated. In the current work, a 'reversed-slit' method is explored, aiming to maximise signal input while retain a reasonably good line resolution. A thin metal wire with a diameter that is comparable to a normal slit width is placed in front of an open slit. By opening the slit the input of the emission light is maximised. The thin wire casts a line of shadow on the spectral image. The shadow line is used as the 'characteristic line' to identify and separate the spectral line of each H isotope. The result shows that an open slit of 3 mm width can bring an intensity increase of 20–30 times compared to a 100 μm wide normal slit without compromising the resolution.

Keywords: Optics; Spectrometers; Data processing methods; Detector design and construction technologies and materials

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https://doi.org/10.1088/1748-0221/15/01/C01007

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1 Introduction

Magnetic confined fusion is currently the optimal way of realising fusion reaction and producing clean energy. However the criteria for magnetic confined fusion are extremely difficult to achieve. Fusion plasma of 150 million celcius degrees is confined by magnetic coils inside the torus. The studies of the plasm-wall interaction are thus critical to many facets of fusion including the erosion of wall surface, fuel retention and safety issues. Various spectroscopy methods have been studied as analytical tools for plasma facing component (PFC) material diagnostics on plasma devices such as JET, EAST and ITER [1–3]. Laser induced breakdown spectroscopy (LIBS) is a strong candidate due to its capability of reliable, fast and in situ measurement of element content, its flexibility that allows it to be adapted and integrated into complex plasma facilities and to carry out measurements in the extreme environment (e.g., strong magnetic field, radioactive, high temperature, low pressure etc.). However, further improvement of the measurement sensitivity is much needed. Previous work [4] has shown the successful monitoring of D and H contents on W exposed in the linear plasma device PSI-2 [5]. Further development is aiming at measuring D, H, T at the same time with reasonably good measurement sensitivity. A number of improvement techniques have been studied. For example, double pulse technique is used to enhance the signal [6], time or space resolved techniques are used to reduce the background [7], advanced line fitting procedure is used to better recognise the signal [4, 8], etc. In this work, we focus on one advancement of the spectroscopic method.

The Balmer-alpha lines of H isotopes are very close to each other. H_α (656.289 nm) is roughly 0.2 nm away from D_α (656.104 nm) and D_α is only 0.06 nm away from T_α (656.045 nm). Hence the in situ monitoring of H isotopes requires an exceptionally high measurement sensitivity, especially at low concentration level. In this work, the monochromator at first order [9] and a Littrow spectrometer are used for the measurements. A thin wire is placed in the centre of the open slit to distinguish the isotope lines. The open slit let in more emission light, resulting in a more efficient

use of the grating area and greater signal. The resolution is provided by the thin wire. In this work, the monochromator is used for the test measurement with the spectral lamp and the Littrow spectrometer is used for in situ measurement. A self-developed image processing programme is developed to extract information from the 'reversed' spectrum. The work exhibits an alternative way of spectroscopy and offers a potential improvement of the measurement sensitivity.

2 Experimental

The LIBS system consists of a nanosecond laser, the optical system, the emission collecting components and the spectrometer based detecting system. The system is described in our previous work [4]. Two spectrometers are used in the experiments. One is a Czerny Turner spectrometer (Acton SpectroPro-500) equipped with three gratings (600/1200/2400 grooves/mm). 2400 grooves/mm grating is used for better resolution. The other one is a Littrow spectrometer, operating at second order for better resolution. The settings and parameters are optimised for the in situ measurement of H isotopes on W samples [4]. The Czerny Turner spectrometer is used for the test with D/H lamp and the Littrow spectrometer is used for W and D line measurement. For this new spectroscopy method, the slit of the spectrometer is wide open (3 mm), a copper wire (∅50 µm) or a molybdenum wire (∅100 µm) is used. The thin wire is attached in front of the slit and fixed as close as possible to the entrance window of the spectrometer. A long fibre (35 metres, ∅1.5 mm) is equipped to transmit the emission light from the chamber to the spectrometer. Two optical lenses (Plano Convex, ∅25 mm) are inserted between the fibre and the entrance slit of the spectrometer, to homogenise and magnify the output of the fibre by a factor of 2, so that the emission spot (∅1.5 mm × 2) matches the width of the open slit, resulting in a more efficient use of the emission light and the grating area. The experimental structure for LIBS on main chamber setup are shown in figure 1.

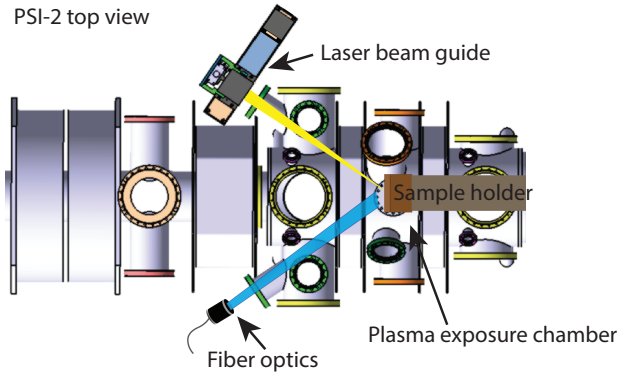

Figure 1. Experimental set up of LIBS system on PSI-2.

3 Results and discussion

3.1 The 'reversed' spectrum with a spectral lamp

This setup is first tested with a D/H lamp of H : D ≈ 45% : 55%. To obtain stronger signal from a stable light source, the exposure time is extended to 2–5 ms. Figure 2 shows the process

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of obtaining the open slit image and how to interpret the data. Figure 2(a) shows the spectral image of the open slit without the thin wire. The emission light from H_α and D_α pass through the open slit, dispersed by the grating (2400 g/mm) and projected on to the camera. As shown in the spectrum, the spectral 'line' as usually seen on the camera image becomes the spectral 'square', i.e., from 10 pixels (≈ 0.08 nm, line width) to 300 pixels (≈ 2.4 nm, square width) because of the extremely widened entrance slit (from 50 µm to 3 mm). The separation between the two emission lines/squares is defined by the grating property, thus is the same as before (about 30 pixels apart on one frame). Since the line/square width is much larger than the separation, the images appear overlapping. Next the spectral image is taken with the thin wire (50 µm) in the centre of the open slit, as shown in figure 2(b). The thin wire is projected on to the spectral 'squares' from H_α and D_α, leaves a sharp and clean mark on each of the emission image. The position and the darkness (reversed intensity) are then used to identify the two isotope lines and determine the intensity ratio. Figure 2(c) shows the spectra obtained from the images in (a) and (b). Subtract the spectrum with wire from the spectrum without wire, the result is the 'reversed' spectrum. It has the shape that is very similar to a spectrum obtained from a narrow slit.

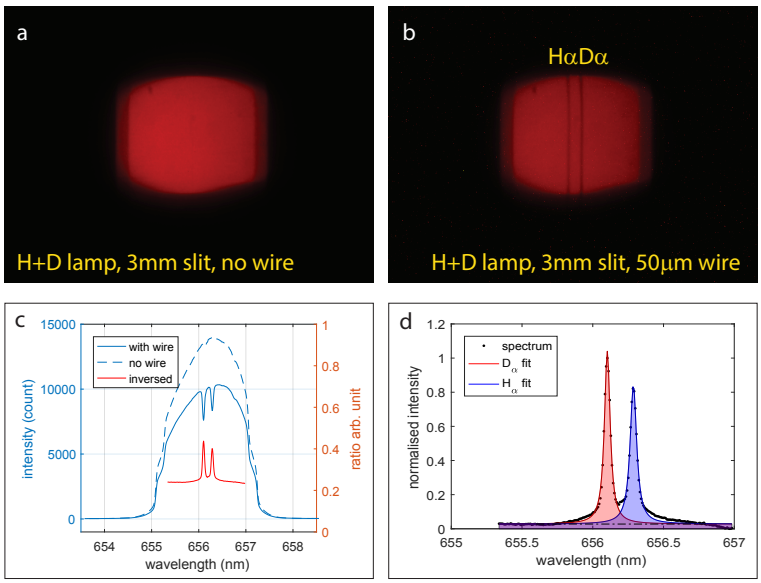

Figure 2. Reverse-slit D and H spectral image and reversed spectrum.

Although the 'reversed' slit changes the way emission light is collected, it does not alter the atomic emission process in the laser produced plasma. The broadening effect of the emission 'lines' still consists of Gaussian (Doppler) and Lorentz (pressure). Figure 2(d) shows that a Voigt profile is used to fit the 'reversed' spectrum. The positions of the two lines match the central wavelengthes of H_α and D_α lines (656.3 nm and 656.1 nm). The intensity ratio calculated from the reversed spectrum matches the light source composition (H : D ≈ 45% : 55%). The increase of the total intensity is proportional to the increase of the slit width. When slit width is increased from 50 µm to 3 mm, the signal intensity is nearly 60 times more. The linearity of such increase needs to be further confirmed. The result is a proof-of-principle for this method.

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3.2 Reversed-slit spectroscopy on W line

After a successful proof-of-principle with the spectral lamp, the reverse-slit spectroscopy method is used for in situ measurement of D on W samples after plasma exposure. W I 400.8 nm line is chosen to evaluation the method.

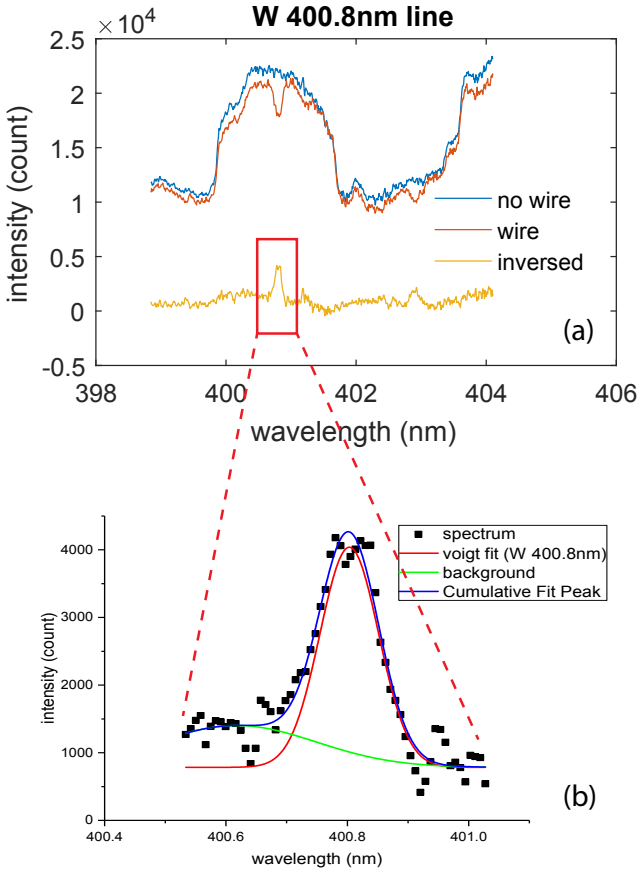

Figure 3. W spectrum with reversed slit and voigt profile.

Figure 3 shows the W spectrum with reversed-slit method and a voigt fit of the reversed spectrum. The spectra were taken in pairs, one with wire (100 µm) and the other without wire and the reversed spectrum is then generated by subtracting one from the other. The reversed spectrum is shown as the yellow line in figure 3(a). The fitting is shown in figure 3(b). The noise level is significantly reduced by averaging a large number of spectra.

3.3 Reversed-slit spectroscopy for D measurement

As observed in the previous section 3.2, the wire thickness is a key factor to the success of the new method. A quick test of the two available wires (Tungsten — 50 µm and Molybdenum — 100 µm)on D_α line with in situ setup is carried out before the recording. With Tungsten — 50 µm, the reversed spectrum is almost invisible and thus Molybdenum — 100 µm is used for the in situ measurement of D. A pair of spectra were taken for each measurement, one with wire and one without. The reversed spectrum is generated by subtraction of those two spectra.

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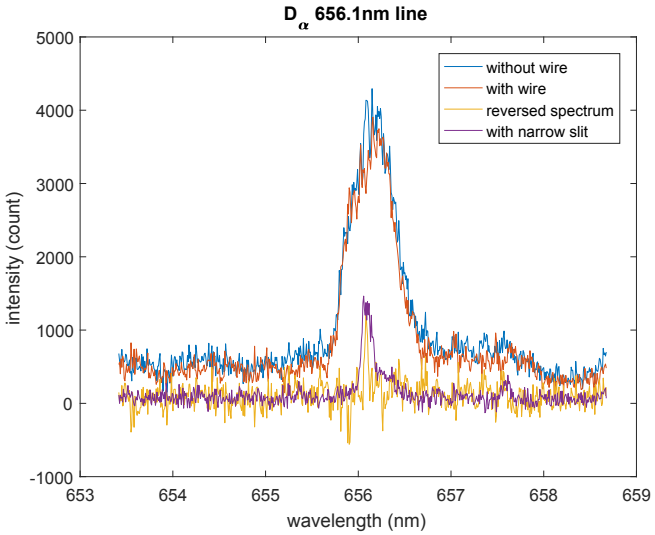

Figure 4. D/H spectrum with reversed slit.

Figure 4 shows the pair of the spectra taken from in situ measurement and the generated reverse spectrum. A spectrum with a normal slit (500 µm) is included for comparison. The centre of the reversed line is an exact match of the normal line, suggesting that the new method is able to precisely recognise the emission lines. The intensity peak-to-base values of the normal line (500 µm slit) and the reversed line (100 µm wire) are roughly the same, while the normal line is about 3 times wider than the reversed line. The line intensity of the normal line is the integration over the normal line area. The line intensity in the new method is the integration over the spectral 'square' in the spectrum image with wire, not the reversed line in the reversed spectrum. The reserved spectrum obtained by subtracting the spectrum pair is used to resolve close isotope lines and calculate the intensity ratio in case more than one isotope lines appear at the same time. Hence the estimated resolution of the new method on D_α line is 3 times better and the intensity (integration over the spectrum with wire, not the reversed spectrum) is more than 10 times higher. In the current measurement, H content is below the detection limit and so the focus is on the accurate identification of D_α line and precise estimation of its intensity. A more detailed comparison is provided in section 3.5.

3.4 'Background' on the reversed spectra

As can be noticed on the reversed spectrum from the lamp (section 3.1), there is a raised 'background' under the two isotope lines. Unlike the background in a normal optical emission spectrum which is mainly due to the continuum emission, the 'background' in the reversed spectrum comes from the subtraction of the spectrum pair (with and without wire). The wire not only casts a shadow in the centre of each emission spectrum, but also blocks part of the input light source, resulting in a reduced overall intensity in the corresponding spectrum. The thicker the wire is, the more the input light is blocked, the higher the 'background' is. However, the wire (50 or 100 µm) is considerably thinner (50 times in average) than the width of an open slit (2–3 mm), only a small amount of light is blocked by the wire. The high 'background' is caused by an integration over time. The longer the exposure time is, the larger the difference between the spectrum pair is.

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In the case of the *in situ* measurement of W and D lines, the 'background' on the reversed spectra is much lower and not evenly spread across the spectrum range when compared to that in the lamp case. The lower level of 'background' is mainly due to the exposure time. To measure the emission signal from a laser induced plasma (LIP) which lasts typically less than 1 μs in vacuum, t_{expo} = 500 ns is used for the *in situ* measurement. When using a continuous light source, t_{expo} = 2 ms is used in the lamp case. The intensity difference is as large as 10^4 fold and that results in the apparent 'high background' in the lamp case and not so obvious 'background' in the in situ case. The uneven shape of the 'background' on the in situ spectra, especially the W 400.8 nm spectrum, is mainly due to the continuum emission from the LIP emission source.

Because of the different mechanism in each case, the 'background' in the lamp spectrum is treated as a baseline offset and the voigt profile fitting is only applied to the two lines. The 'background' in the W spectrum is fitted together with the line using voigt profile and the 'background' in the D spectrum is estimated using the partial spectrum that is adjacent to the D_α line and subtracted from the spectrum. The results indicate that the 'background' elimination procedure is sufficient in each of the cases.

3.5 Measurement sensitivity comparison

The lamp case (section 3.1) uses a stable light source and a long exposure time, the result is not directly comparable to that in the *in situ* case. The *in situ* measurement of W and D using the new method is compared to the *in situ* measurement using the normal method. Table 1 listed a number of tests carried out using the new and normal methods.

The comparison of the measurement sensitivity of the W line is quite straight forward. The line intensity is proportional to the slit width (in the narrow slit case) and the increase in the line intensity corresponds to the increase in the slit width when comparing the two methods. For example, when slit width is increased from 100 μm to 3 mm (×30), the same increase intensity is applied to the use of a 50 μm slit and 3 mm slit with 50 μm wire. The small difference is due to the blocking of the light by the thin wire (section 3.4. The same increase correlation applies to the 50 μm slit. The FWHM value represents the line width of the reversed line obtained from subtraction in the new method, or the line width of the emission line produced by the narrow slit in the traditional method. FWHM provides an indication of the spectral resolution. When the FWHM is larger than the distance between two spectral lines, those two lines cannot be resolved. In general, the smaller value of the FWHM is, the better the resolution can be. The FWHM comparison shows little improvement in the resolution by using the new method. As W I 400.8 nm line is already a strong narrow emission line, the new method increases the line intensity significantly but has little impact on the resolution.

The second section of table 1 shows the measurements of isotope D_α 656.1 nm line. Data is not available for 50 μm slit or for μm wire. 50 μm slit is too narrow to allow a sufficient amount of photons into the spectrometer to form a visible spectral line. 50 μm wire is too thin to leave a visible shadow mark on the broadened spectral image. The slit and wire are both increased to 100 μm. The improvement is observed in both the intensity and the resolution. The intensity is increased by about 20 times in the new method. The resolution using 100 μm wire has a value of 0.049 and the resolution using 100 μm slit is 0.067 nm. As mentioned previously, T_α 656.045 nm is separated from D_α 656.104 nm only by 0.06 nm, which means that the traditional method wouldn't be able to resolve T_α and D_α lines but the new method is able to resolve all three H isotope lines with 20 times higher signal intensity.

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Table 1. List of experimental tests and comparison of measurement sensitivity. 'Int.' is the integrated line intensity. FWHM is the line width.

W I 400.8 nm			
Open slit with wire			
slit width = 3 mm		Int.(count)	FWHM (nm)
wire thickness	50 μm	5.01E06	0.074
	100 μm	4.85E06	0.140
Narrow slit no wire			
slit width	50 μm	1.03E05	0.066
	100 μm	1.93E05	0.148
	500 μm	7.78E05	0.675

D_α 656.1 nm			
Open slit with wire			
slit width = 3 mm		Int.(count)	FWHM (nm)
wire thickness	50 μm	—	—
	100 μm	2.76E05	**0.049**
Narrow slit no wire			
slit width	50 μm	—	—
	100 μm	1.07E04	**0.067**
	500 μm	2.60E04	0.156

4 Conclusions and future work

Measurement sensitivity is crucial for in situ measurement of fuel content on PFC materials. In the present work, a new spectroscopy method is introduced and evaluated. The new method opens the entrance slit of the spectrometer to the maximum to allow maximum light throughput. A thin wire is placed in the centre of the open slit to locate the emission from each isotope. It works as if the slit is reversed and so we call it the 'reversed-slit' spectroscopy method. The reversed-slit method is first tested with a D/H lamp and then used for in situ measurement D on W samples. The results show that this method is able to increase the light throughput without compromise the line resolution. Voigt profile is used to fit the reversed spectra. Line recognition is accurate in all three cases (D/H lamp, W and D). Isotope intensity ratio is calculated from line fitting and is in good agreement with that with normal spectroscopy method. In general, the reversed-slit method increases the light intensity substantially and is more powerful in identifying weak lines from a noisy background. Further study is planned to find out the optimum conditions for the reversed-slit method, improve its viability and explore potential applications.

Acknowledgments

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement number 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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