X-ray and VUV reflectometry and metrology of plasma radiation sources

A P Shevelko
P.N. Lebedev Physical Institute of the Russian Academy of Sciences, 53 Leninskii Ave., Moscow, 119991, Russia

E-mail: apshev51@gmail.com

Abstract. Comparison of various methods for quantitative (absolute) measurements of intensities in soft X-ray and VUV spectral ranges is considered. These methods include calibration of separate spectrometer elements, the use of absolutely calibrated spectrometers and formation of quasi-monochromatic with a large angle of divergence X-ray beams. Their use together with spectroscopy methods allows a providing complete plasma diagnostics: determination of plasma parameters and measuring of plasma radiation characteristics.

1. Introduction

Recently quantitative (absolute) measurements of intensities in soft X-ray and vacuum ultraviolet (VUV) spectral ranges acquire special significance. Particularly important are these measurements for studies of radiation heating of targets in inertial confinement fusion and for developing of new radiation sources for various practical applications (EUV nanolithography, microscopy of biological objects in the “water window” spectral range, etc.). Carrying out absolute measurements is one of the topical problems of soft X-ray and VUV spectroscopy. In these ranges, all substances possess large and varying sharply absorption coefficients as functions of the wavelength, which greatly affects the reflectivity of crystals, diffraction gratings and mirrors, the sensitivity of detectors, and other characteristics of devices.

Traditionally synchrotron radiation is considered as “calibration source #1.” Synchrotron radiation facilities allow calibration of spectrometer elements with a high accuracy and in a wide spectral range. These are very big advantages. Nevertheless there are also some essential restrictions and disadvantages too. In particular parallel synchrotron beam is not quite suitable for calibration of X-ray focusing crystal spectrometers intended for detection of divergent angular beams from laboratory sources. Usually synchrotron radiation sources are located far from users, and their utilization is expensive. Another approach is the use of various laboratory soft X-ray and VUV reflectometers. Calibration of separated spectrometer elements (dispersive elements and detectors) allows determination of absolute spectrometer efficiency under known device geometry. Another advantages are compactness and possibility to have the equipment in nearest vicinity from users. This approach requires the further development of new calibration and metrology methods.

2. VUV reflectometry

Laboratory VUV reflectometry and metrology are intensively developed from the end of previous century what was caused by extraordinary progress in soft x-ray and extreme ultraviolet (EUV)
technology [1]. This progress was driven by advances in high-brightness soft x-ray and EUV sources, in new optical elements, especially multilayer mirrors, and in new types of detectors. Due to this technology a variety of novel practical applications has recently become feasible. One of the most prominent applications is EUV lithography that approaches the resolution goal of less than 20 nm for use in integrated circuit chip production for the next generation of semiconductor devices [2]. A great necessity of precise calibration of these EUV sources, optical elements and detectors was originated. One of the first laboratory reflectometers were used laser-produced plasmas [3, 4] as a source of VUV radiation. Later a big progress was achieved in development of EUV reflectometers intended for measurements of multilayer mirrors and diffraction grating efficiencies, transmissions of thin filters and other parameters of optical elements.

Various sources of VUV radiation are used in the reflectometers: laser-produced plasmas [2–4], Z-pinch plasmas [5], X-ray tubes [6, 7]. VUV reflectometer based on capillary discharge plasma source and grazing incidence monochromator is described in Ref. [8, 9] (see figure 1). This reflectometer was used for calibration of diffraction grating reflectivities, photographic film and CCD detector sensitivities.

![Scheme of reflectometer based on capillary discharge plasma source and double beam monochromator (on the left) and scheme of monochromator exit (on the right).](image)

**Figure 1.** Scheme of reflectometer based on capillary discharge plasma source and double beam monochromator (on the left) and scheme of monochromator exit (on the right).

In many practical applications absolute measurements of intensities in spectra, both in individual spectral lines and in given narrow and wide spectral intervals, are needed. For this purpose it is necessary to use absolutely calibrated VUV spectrometers. One way is to calibrate all spectrometer elements using synchrotron radiation or VUV reflectometers. Another way to perform absolute measurements especially in a wide spectral range is to use a transmission grating spectrometer (TGS) or an amplitude grating spectrometer (AGS). The efficiency of the gratings is usually determined theoretically, and the use of absolutely calibrated radiation detector allows, in principal, absolute measurements. Detailed description and comparison of these spectrometers and appropriate references can be found in [10].

### 3. X-ray metrology

For X-ray calibration of focusing crystal spectrometers it is necessary to use a quasi-monochromatic, with a large solid angle of divergence beams. For this purpose a special method are developed using X-ray fluxes from laser-produced plasmas [11]. This method uses a special combination of the laser target elements and K-absorption filters. The laser targets with average atomic numbers $A_Z \approx 10\div20$ are selected to ensure the excitation of hydrogen ([H]-) and/or helium-like ([He]-) ions (spectral range $\lambda=0.2\div1.0$ nm) in laser-produced plasmas. The selected K-absorption filters transmit light in a narrow spectral range which only contains the resonance lines of [H]- and [He]-like ions. Other spectral lines of these ions are located beyond the K-edge and completely absorbed by the filter. The resonance lines of ions with lower ionization degrees ([Li]- and others) occupy the long-wavelength range and are also completely absorbed by the filter.

An important condition for applicability of this method is small contribution of the intensity of continuum radiation, which can lead to decrease in the monochromatisation degree. For experimental
evaluation of this contribution, a new method is developed [11], which also employs a special combination of the K-absorption filters and two laser target elements with adjacent atomic numbers: \( Z_a \) and \( Z_a+1 \).

The quality of the beam is estimated by a parameter \( \eta \) equaled to the product of degree of monochromatisation \( \lambda/\Delta\lambda \) into angular divergence \( \Omega \). Experimental and theoretical studies [11] have shown that, in the fluxes formed, the degree of monochromatisation \( \lambda/\Delta\lambda \) may reach \( \sim100 \) at the radiation contrast ratio of 5±10 and angular divergence of \( \Omega\sim\pi \) sr. As a result, the maximum measured value of the parameter \( \eta \) constituted \( \sim300 \). Another advantage of this method is its simplicity: the tuning of the working wavelength requires only a change of the laser target and K-filter. Such a change can be made without vacuum violation in the chamber. So the use of this method ensures absolute \textit{in situ} \ calibration of spectrometers at different wavelengths.

This method is used for absolute calibration of X-ray focusing crystal spectrometers and their elements [12]. The experimental scheme for calibration of the focusing crystal von Hamos spectrometer as a whole is shown in figure 2. Absolute intensity was measured by a Siemens BPX-66 pin diode. Various K-filters and laser target elements were used to form the quasi monochromatic beams in a spectral range of \( \Delta\lambda=1.8-10 \) Å. Mica crystal (\( 2d=19.84 \) Å) with a radius of curvature \( R=20 \) mm was installed in the von Hamos spectrometer. The calibration was performed using X-ray photographic film Kodak 2492 and CCD linear array (Toshiba TCD 1304 AP) as X-ray detectors. Examples of X-ray spectra recorded in absolute intensity scales are shown in figure 3.

\[\textbf{Figure 2.} \text{Experimental scheme for calibration of the focusing crystal von Hamos spectrometer.}\]

\[\textbf{Figure 3.} \text{X-ray spectra recorded in absolute intensity scales: Ge laser-produced plasma (spectral lines of [Ne]-like ions are marked, detector – photographic film) (on the left); Ti laser-produced plasma (spectral lines of [He]-like ion and satellite lines are marked; detector – CCD linear array) (on the right).}\]
4. Conclusion
Development of new experimental and theoretical metrology methods together with spectroscopy methods allow a providing of complete plasma diagnostics: determination of plasma parameters as well as measurements of plasma radiation characteristics that is extremely important for numerous practical applications.

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