

# Versatile X-ray diagnostics of laser-produced high-temperature plasmas using an ultra-high luminosity spectrometer

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**Abstract.** Versatile diagnostics of high-temperature magnesium laser-produced plasmas is performed using an ultra-high luminosity X-ray focusing crystal spectrometer. The plasmas are produced by focusing nanosecond laser pulses (0.53  $\mu\text{m}$ , 1 J, 2 ns) on a massive magnesium target. The absolute intensities and X-ray yields are measured for the resonance lines of H- and He-like ions (spectral range:  $\Delta\lambda = 8\text{--}10 \text{ \AA}$ ), the electron temperature of plasmas is determined (range:  $T_e = 200\text{--}550 \text{ eV}$ ), and the behaviour of these parameters is established in a broad range of laser pulse energy  $E_L$  (10 mJ–1 J). This approach permits the parameters of laser-plasma radiation sources to be monitored in real time in a wide intensity range (difference in intensity: over  $10^4$ ) and in a broad range of plasma parameters.

**Keywords:** high-temperature plasma diagnostics, X-ray spectroscopy and metrology, multiply charged ions, laser-produced plasmas, X-ray spectrometers.

## 1. Introduction

Comprehensive plasma diagnostics should rely on versatile methods which make it possible to measure not only the main plasma parameters (the electron temperature and density, the ions charge state distribution, etc.) but also its radiative characteristics. This is because considerable recent attention has been focused on quantitative (absolute) spectral measurements required for different practical applications. Such metrological measurements are especially topical for inertial confinement fusion which involves the radiation-induced compression on thermonuclear targets (see, for instance, Refs [1, 2]). In this case, it is necessary to know the spectral composition of the radiation, its power, conversion efficiency, and other radiative characteristics. The same task is faced by projection EUV nanolithography – one of the most promising technologies for the mass production of integrated circuits with elements less than 20 nm [3]. The most important constituent of this technology is the use of high-intensity EUV radiation sources, on which strict requirements are imposed as regards the absolute radiation yield at working wavelengths ( $\lambda = 135$  and  $66 \text{ \AA}$ ) as well as in a broad spectral range. Absolute intensity measurements are also required in the

microscopy of biological objects in the ‘water window’, medical examinations (radiologic imaging) etc.

Developing new diagnostic and absolute calibration techniques calls for the investigation of plasma parameters and its radiative characteristics in a broad range of plasma parameters. Most minutely elaborated at present are the methods of spectroscopic diagnostics from the spectra of hydrogen- and helium-like ([H]- and [He]-like) multiply charged ions. The spectra of these ions with atomic numbers  $A_Z = 6\text{--}30$  have been adequately studied and are widely used to estimate the electron temperature  $T_e$  of plasmas in the range from 100 to 1000 eV [4–7]. For the examples of X-ray spectra of nanosecond laser-produced plasmas the reader is referred to numerous monographs and reviews (see, for instance, Refs [8–10]). To diagnose the plasmas of heavy elements, whose spectra are much more complex in structure, a new comparison method and its modifications are under development [11–14]. To determine  $T_e$  of the plasmas of heavy elements, this method proposes that the spectra under investigation should be compared with the spectra of adequately diagnosed laser-produced plasmas. The laser-produced plasmas are diagnosed from the spectra of light elements with the structure of [H]- and [He]-like ions. Improving the accuracy and reliability of the method requires data on the [H]- and [He]-like ions in a broad  $T_e$  range in order to cover the entire temperature range in which the spectra of heavy elements are studied.

Metrological investigations of high-temperature plasmas call for the use of absolutely calibrated spectrometers. To calibrate the responsivity of X-ray spectrometers in a broad spectral range, a new monochromatisation method was developed – a new method for the formation of quasi-monochromatic X-ray fluxes of laser-produced plasma line radiation with a large angular divergence [15]. These fluxes are employed for the absolute calibration of spectroscopic devices. To improve the accuracy of this calibration technique, both in intensity and wavelength, one has to know the intensity ratios for the resonance lines of [H]- and [He]-like ions for various electron temperatures.

To record the X-ray spectra of high-temperature plasmas in a wavelength range  $\lambda \lesssim 10 \text{ \AA}$ , use is primarily made of special crystal spectrometer schemes possessing a high efficiency (luminosity). The highest efficiency is inherent in spectrometers of focusing geometry, for instance, von Hamos spectrometers [16]. This scheme offers significant advantages in the recording of the radiation of quasi-point sources: an ultra-high luminosity throughout a broad spectral range, a high spectral resolution, the possibility of recording spectra with spatial resolution, linear focusing, etc. At present, the von Hamos scheme enjoys wide use in X-ray spectroscopic

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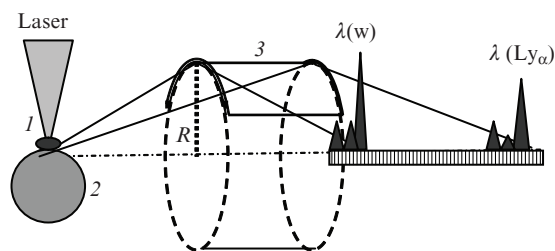
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research (see, for instance, papers [17, 18] and references therein).

The aim of this work is to carry out the versatile diagnostics of high-temperature magnesium laser-produced plasmas: to measure the absolute intensities of the resonance lines of [H]- and [He]-like ions and determine the electron temperature of the plasmas. The use of an ultra-high luminosity von Hamos spectrometer made it possible to determine the behaviour of these parameters in a broad range of laser pulse energy and plasma parameters. We discuss the significance of the resultant data for the development of a new comparison method in the diagnostics of heavy-element plasmas and of the new technique for the production of quasi-monochromatic fluxes of X-ray line radiation from laser-produced plasmas for the absolute calibration of spectrometers and their elements.

## 2. Experiment

The experiment is schematised in Fig. 1. The second harmonic radiation of a Nd:glass laser (the Phoenix facility) was used for plasma production. The laser radiation (0.53  $\mu\text{m}$ , 1 J, 2 ns) was focused on a massive magnesium target with the use of a lens with a focal length  $f = 300$  mm.



**Figure 1.** Schematic of the experiment to record the spectra of magnesium laser-produced plasmas with the use of an X-ray focusing crystal von Hamos spectrometer:

(1) laser-produced plasmas; (2) target; (3) cylindrical mica crystal with a bending radius  $R$ ; (4) detector (linear CCD array);  $\lambda(w)$  and  $\lambda(\text{Ly}_\alpha)$  are the wavelengths of the resonance lines of [He]- and [H]-like Mg ions.

The X-ray spectra of [H]- and [He]-like Mg ions were investigated using a focusing crystal von Hamos spectrometer. In this scheme [13], the crystal is bent to a cylindrical surface, the X-ray source and the detector plane being positioned on the cylinder axis (see Fig. 1). Owing to the focusing geometry, the instrument exhibits a high efficiency in a broad spectral range [17, 18]. Another significant advantage offered by this scheme is its linear focusing, whereby the spectrum is formed on the spectrometer axis. This permits the use of flat-field detectors for recording the spectra, in our case a linear CCD array with a high quantum efficiency, which also improves the spectrometer efficiency.

The instrument makes use of a cylindrical mica crystal (with double spacing  $2d = 19.84$   $\text{\AA}$ ) of radius  $R = 20$  mm. This parameter determined the compact spectrometer size (diameter, 40 mm; length, 100 mm). The function of radiation detection was fulfilled by a Toshiba TCD 1304AP absolute-calibrated linear CCD array with 3724 8- $\mu\text{m}$  wide elements of height 200  $\mu\text{m}$  and an overall active detector area of 200  $\mu\text{m} \times 29.8$  mm. The detector axis was aligned with the spectrometer

axis with an accuracy of  $\pm 20$   $\mu\text{m}$ . The X-ray spectra were recorded in the first reflection order of the mica crystal in a spectral range  $\Delta\lambda = 8.2 - 9.5$   $\text{\AA}$ . In our case the spectral resolution of the spectrometer was defined by the source size and amounted to  $\lambda/\delta\lambda \approx 600 - 1500$ . This value was found from the width of spectral lines.

The high spectrometer efficiency permitted each spectrum of multiply charged Mg ions to be recorded in one laser shot even for the lowest energy of laser pulses,  $E_L \approx 10$  mJ. For a higher energy  $E_L$ , it was necessary to significantly attenuate the radiation intensity to prevent the saturation of CCD detector signals. This was accomplished with absorption filters, which were placed at the spectrometer input. To shield the CCD detector from visible radiation, use was made of a 1.2- $\mu\text{m}$  thick polypropylene film coated with an aluminium layer 0.2  $\mu\text{m}$  in thickness. A set of different foils (aluminium 9.1- $\mu\text{m}$  thick and lvsan 20- $\mu\text{m}$  thick foils) was employed to attenuate the radiation delivered to the spectrometer. The filter transmittances were determined using the data of Ref. [19], with account taken of the real geometry of radiation transmission through the filters (inclusion of the Bragg angle in the determination of the effective filter thickness). For the highest energy  $E_L \approx 1$  J, the net intensity attenuation factor was equal to  $\sim 1000$  for the resonance line  $w$  of [He]-like ions and to  $\sim 250$  for the resonance  $\text{Ly}_\alpha$  line of [H]-like Mg ions.

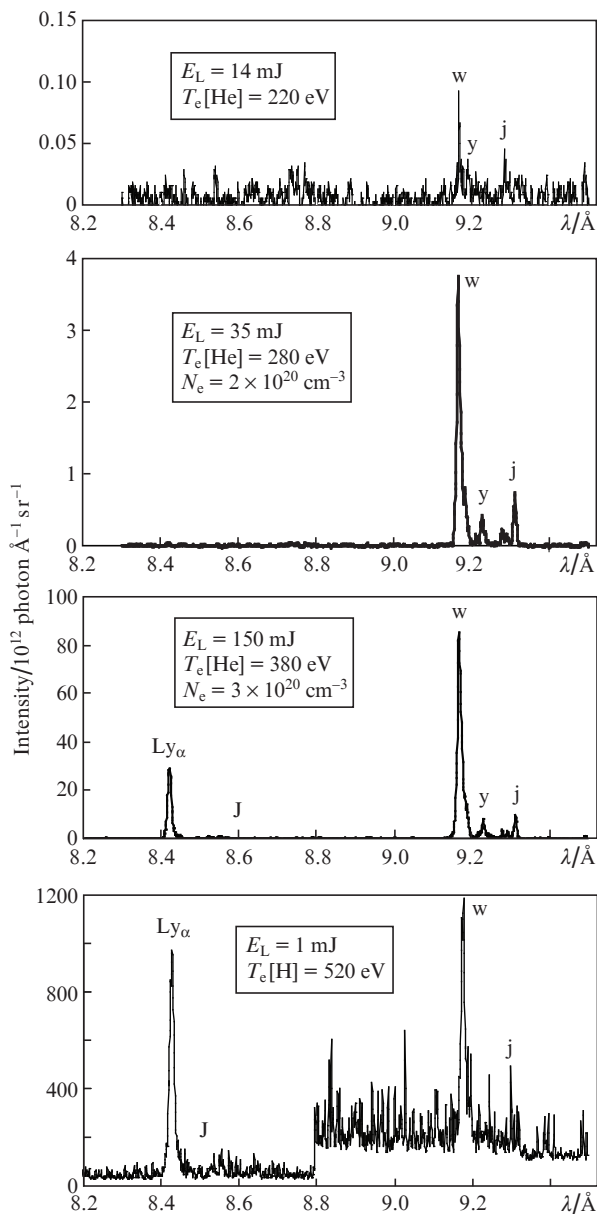
The spectrometer as a whole was absolute-calibrated in sensitivity. To this end, use was made of the calibration technique involving the formation of quasi-monochromatic X-ray fluxes of laser-produced plasma line radiation with a large angular divergence. This technique was employed to calibrate the elements of the spectrometer: the mica crystal reflectivity [17] and the CCD detector responsivity [18]. The integral mica crystal reflectivity  $\rho$  was equal to  $1.7 \times 10^{-6}$  rad and the detector quantum efficiency  $\eta$  was equal to 0.8. These data as well as the transmittance data for the absorption filters were used to determine the absolute intensity scale for the recorded spectra.

The high spectral resolution of the spectrometer permitted observations of the entire structure of the X-ray spectra: the resonance lines of the [H]- and [He]-like lines of Mg ions and their surrounding satellites. The plasmas were diagnosed from the relative intensities of these spectral lines. The electron temperature  $T_e$  [He] was measured from the intensity ratio between the dielectronic satellites  $j, k$  and the resonance line  $w$  of the [He]-like ions (henceforward the notation is borrowed from Refs [4–7]). The temperature  $T_e$  [H] was measured from the intensity ratio between the dielectronic satellite  $J$  and the  $\text{Ly}_\alpha$  resonance line of the [H]-like ions. In both cases, use was made of the data of theoretical calculations [7] to determine  $T_e$ . The electron density  $N_e$  was determined from the intensity ratio between the intercombination line  $y$  and the resonance line  $w$  of the [He]-like ion [20].

## 3. Results and their discussion

The main results of the investigation are outlined in Figs 2–4. Figure 2 shows the spectra of the [H]- and [He]-like Mg ions in the vicinity of their resonance lines  $\text{Ly}_\alpha$  and  $w$  for different laser pulse energies  $E_L$ . The absolute intensity scale in units of photon  $\text{\AA}^{-1} \text{sr}^{-1}$  was reconstructed with allowance for the calibration of the spectrometer and its elements as well as for absorption filter transmittances. The [He]-like ion spectra became observable for energies  $E_L \geq 10$  mJ. Also reliably

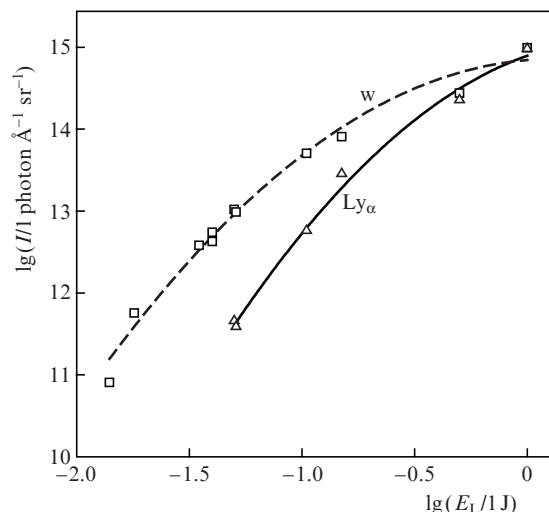
recorded in the spectra for  $E_L \geq 50$  mJ was the resonance  $\text{Ly}_\alpha$  line of [H]-like ions. For  $E_L \approx 1$  J the  $\text{Ly}_\alpha$  and w line intensities became almost equal. Figure 3 depicts the maximum absolute intensities of the w and  $\text{Ly}_\alpha$  lines in relation to the energy  $E_L$ . The uncertainty of intensity measurements amounted to 10%–15%. These dependences permit determining the intensity ratio for the lines under consideration for any  $E_L$  value in the energy range under study.



**Figure 2.** Examples of the spectra of [H]- and [He]-like Mg ions in the vicinity of their resonance lines for various laser pulse energies  $E_L$ .

For energies  $E_L > 0.5$  J, a quasi-continuous spectrum was observed in a spectral range  $\Delta\lambda = 8.8$ – $9.5$  Å, which corresponded to the radiation of hollow ions arising from transitions to the empty K shell:  $2s^22p^n - 1s2s^22p^{n-1}$ . This emission from the laser-produced plasmas of aluminium and magnesium was first recorded in Refs [21, 22].

The high luminosity of the spectrometer and the application of a high-efficiency CCD detector allowed the spectra



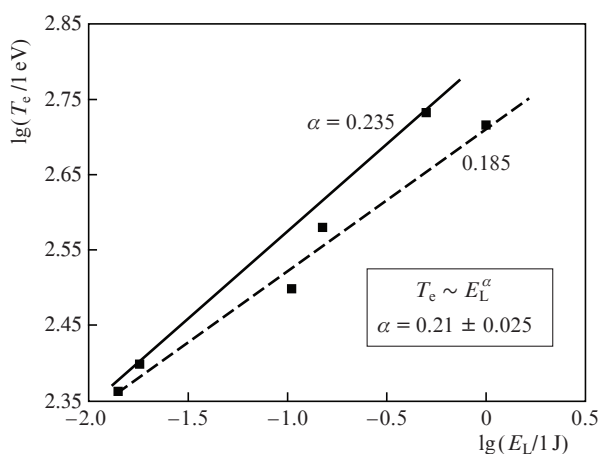
**Figure 3.** Absolute intensities  $I$  of the resonance lines w and  $\text{Ly}_\alpha$  of the [He]- and [H]-like Mg ions as functions of laser pulse energy  $E_L$  in logarithmic coordinates.

to be recorded for a very low energy of laser pulses  $E_L$ . In our case, the detection threshold was determined by the dark current noise of the linear CCD array, which amounted to  $\pm 10$  counts. The line intensities which exceeded this noise level corresponded to  $1.6 \times 10^1$  photon  $\text{Å}^{-1} \text{sr}^{-1}$ . With allowance for the transmittances of absorption filters, the recorded lines differed in intensity by more than four orders of magnitude. The high detection capacity of the spectrometer makes it the spectrometer of choice for recording low-intensity quasi-point radiation sources – femtosecond laser-produced plasmas and the sources with a low temperature  $T_e$ . Examples of the application of this spectrometer are found in our works [12, 23, 24]. We note that the detection capacity of the spectrometer may be improved by cooling the CCD detector due to lowering the level of dark current noise as well as by accumulation of the signal from high-repetition-rate laser-plasma radiation sources.

The resultant data make it possible to estimate the photon yield  $Y$  and the conversion efficiency  $\text{QE} = Y/E_L$  for the spectral lines under investigation. The photon yield  $Y$  is defined as the product of the highest line intensity, its spectral width, and the solid angle of emission. The linewidth varied from pixels ( $4 \times 10^{-3}$  Å) to 20 pixels ( $14 \times 10^{-3}$  Å) with increasing  $E_L$  from 14 mJ to 1 J. Assuming the source to be isotropic and radiating in a solid angle of  $2\pi$  sr, we obtain the following figures for the radiation yield in the resonance line w of [He]-like ions:  $Y = 2.3 \times 10^9$  photons for  $E_L = 14$  mJ and  $Y = 8.6 \times 10^{13}$  photons for  $E_L = 1$  J. The radiation conversion efficiency  $\text{QE}$  amounted to  $4 \times 10^{-3}\%$  and 2%, respectively. The photon yield and the conversion efficiency for the resonance line  $\text{Ly}_\alpha$  of [H]-like ions were determined in a similar way. For the highest energy  $E_L$  these quantities were  $Y = 8.2 \times 10^{13}$  photons and  $\text{QE} = 2\%$ .

The electron temperature  $T_e$  was determined from the intensity ratio between dielectronic satellites and the resonance line. The dependence of the estimated  $T_e$  on the laser pulse energy is plotted on logarithmic axes in Fig. 4. The  $T_e$  measurement uncertainty was equal to  $\sim 4\%$  for a low  $E_L$  and to  $\sim 8\%$  for the highest  $E_L$ . The uncertainty in the determination of  $T_e$  rises with increasing  $E_L$  due to a sharp decrease in the intensity of dielectronic satellites. The dependence of  $T_e$

on  $E_L$  is nicely described by the power function  $T_e \sim E_L^\alpha$  with an exponent  $\alpha = 0.21 \pm 0.025$ . This dependence differs from the canonical dependence  $T_e \sim q^\alpha \sim (E_L/S\tau)^\alpha \sim E_L^\alpha$  with exponent  $\alpha = 4/9 = 0.44$  (here,  $q$  is the intensity of laser radiation at the target,  $S$  is the focusing spot area, and  $\tau$  is the laser pulse duration). The dependence  $T_e \sim q^\alpha \sim E_L^{4/9}$  was investigated in many papers and is beyond doubt. The weaker dependence  $T_e \sim E_L^{0.21}$  is supposedly due to an increase in the area of the laser focal spot on the target with an increase in  $E_L$ . This is borne out by an increase in radiation source size, which varied from 40  $\mu\text{m}$  for the lowest  $E_L$  to 160  $\mu\text{m}$  for the highest  $E_L$ .



**Figure 4.** Dependence of the electron temperature on the laser pulse energy plotted on logarithmic coordinates.

The electron density  $N_e$  was determined from the intercombination-to-resonance line intensity ratio of the [He] ion [20]. The measured  $N_e$  value hardly changed and was equal to  $(1-3) \times 10^{20} \text{ cm}^{-3}$  throughout the  $E_L$  variation range.

We enlarge on the conditions and limitations for the application of the methods used in our work. To carry out absolute measurements, use can be made of various calibrated crystal and diffraction spectrometers. For instance, efficiency of transmission [25] and reflective amplitude grating [26] spectrometers may be calculated beforehand in a broad wavelength range. The use of a crystal focusing von Hamos spectrometer in the present work was motivated by its high luminosity in a broad wavelength range. This made it possible to record the spectra of Mg ions with intensity difference of over four orders of magnitude. Of course, the application of this spectrometer type is not limited to the spectral range  $\Delta\lambda = 8-10 \text{ \AA}$ , which was realised in the present work. Recording the spectra in high reflection orders (Vth and above) and the available calibration data permit reaching  $\lambda \approx 1 \text{ \AA}$  [17, 18]. Using dispersive elements based on multilayer mirrors provides a possibility to advance to the long-wavelength part of the spectrum. This approach was realised in our paper [24], when the spectra were recorded in the ‘water window’ ( $\Delta\lambda = 30-40 \text{ \AA}$ ). We emphasise that the use of a von Hamos spectrometer is justified only for quasi-point radiation sources [17]. For lengthy sources, it is preferable to employ schemes in which the width of spectral lines is independent of the source size.

To determine  $T_e$  in this work, use was made of the method based on the measurement of the relative intensities of the

dielectronic satellites and resonance lines of [H]- and [He]-like ions [4–7]. Since both the dielectronic satellite lines and the resonance lines arise from the same ion states, this makes it possible to minimise the effect of nonuniformities and charge state composition of the plasmas on the results of measurements, which is an advantage of this method. However, the method is applicable to plasmas with a relatively low electron density ( $N_e \ll 10^{23} \text{ cm}^{-3}$ ), when coronal equilibrium is realised (as a rule, for nanosecond laser-produced plasmas). Excited in the plasmas in this case are the multiply charged ions with ionisation potential  $I \approx (8-10)T_e$ . This underlay the choice of a magnesium target, when [H]- and [He]-like ions are most abundant in the plasmas for relatively low  $(200-550 \text{ eV})T_e$ . With increase in the electron temperature, when  $T_e \gg I/(8-10)$ , the diagnostics of magnesium plasmas from the spectra of [H]- and [He]-like ions is hindered because of transient processes. In this case, the charge state composition of the plasmas is primarily represented by fully ionised ions and the spectra of the [H]- and [He]-like ions provide information only about transient processes rather than about the hottest plasma phase. To measure higher temperatures with this method, one has to go over to the recording of the spectra of heavier elements with higher ionisation potentials. This approach will be demonstrated in our next papers.

For femtosecond laser-produced plasmas the electron density amounts to  $\sim 10^{23} \text{ cm}^{-3}$  and the coronal distribution does not take place. Owing to the short duration of laser pulses, transient effects become highly significant, when the charge state distribution does not reach the ion multiplicities corresponding to [H]- and [He]-like ions even for light elements. Furthermore, the electron temperature in femtosecond laser-produced plasmas may be very high, up to tens of MeV. All this prohibits the application of the method of determining  $T_e$  from the spectra of [H]- and [He]-like ions. However, examples of using this method to estimate  $T_e$  of the thermal component of femtosecond plasmas may be found in the literature [27, 28].

The investigations performed in our work are of interest not only as a method for complex plasma diagnostics but also of interest for the development of new techniques for the diagnostics of heavy-element plasmas and the new methods of calibration of spectroscopic instrumentation.

The X-ray plasma spectra of heavy elements (of elements with a large atomic weight  $A_Z$ ) usually have a complex structure, which is different from the structure of [H]- and [He]-like ions, and contain a wealth of spectral lines belonging to different ion charge states. The overlap of the multitude of spectral lines in the spectrum gives rise to a quasi-continuum, which significantly complicates the spectroscopic diagnostics of these plasmas. The structure and intensity distribution in the spectra are nevertheless highly sensitive to  $T_e$ . This property is used in the new comparison technique to estimate  $T_e$  of the plasmas of heavy elements [11–14]. This method proposes that the spectra under investigation should be compared with the spectra of nicely diagnosed laser-produced plasmas. Its diagnostics is performed using the spectra of light elements with the structure of [H]- and [He]-like ions. The spectra of heavy elements are investigated at the same laser intensities at the target (or at the same energies of laser pulses) as for the light elements. This makes it possible to determine the dependence of the structure of heavy-element spectra on the electron temperature and eventually estimate  $T_e$  for each spectrum. To use this method requires data on the [H]- and [He]-

like ions in a broad  $T_e$  range that covers the entire temperature range in which the spectra of heavy elements are studied. These data, like the dependence of  $T_e$  on the laser pulse energy for light elements, significantly improve the accuracy and reliability of the comparison method.

To calibrate the responsivity of X-ray spectrometers and their elements in a broad spectral range, we developed a new monochromatisation technique – a new technique for the formation of quasi-monochromatic X-ray fluxes of laser-produced plasma line radiation with a large angular divergence [15]. This technique involves the use of a special combination of elements in the laser target and K-absorption filters: excited in the laser-produced plasmas are the spectra of [H]- and/or [He]-like ions, and the K-absorption filters extract from the line spectrum only the resonance and corresponding satellite lines of these ions. The radiation of these lines is recorded behind the K-filter using an absolute-calibrated detector (a pin diode in our case). Measured therefore is the absolute intensity of quasi-monochromatic X-ray fluxes from the laser-produced plasmas, which is used for the calibration of the spectroscopic instrumentation.

An advantage of this technique is that it permits calibrating spectrometers as a whole. Specifically, an X-ray spectrograph with photographic recording was calibrated in Ref. [17] and the absolute responsivity of a spectrometer with a CCD radiation detector was measured in Ref. [18]. When the resonance lines of [H]- and [He]-like ions are simultaneously present in the plasma emission, this entails additional uncertainties of the technique, since the pin diode does not separate out the radiations of the resonance lines of these ions and records the sum of their absolute intensities. In this case, the spectral interval of the calibration becomes broader (the distance in wavelength between the resonance lines), and the uncertainty in intensity measurements in this spectral interval therefore becomes larger. The accuracy of the technique in use may be significantly improved in the presence of additional information about the intensity ratio between the resonance lines. This may be accomplished with an additional spectrometer or even with the spectrometer under calibration itself. The use of an additional channel makes it possible to calibrate the spectrometer at individual wavelengths corresponding to the resonance lines. However, a complication arises in this case: the laser-produced plasma radiation is recorded in both channels with different weights. In the first channel (the channel with the pin diode) account must be taken of the K-filter transmission and the detector responsivity. In the second channel (the spectrometer channel) one must take into account the crystal reflectance, the filter transmittances, and the detector responsivity. The detector responsivities and the filter transmittances may be assumed to be known (they are determined in a different way), while the crystal reflectance at different wavelengths is frequently unknown. This applies especially to the mica crystal  $K_2O \cdot 3Al_2O_3 \cdot SiO_2 \cdot 2H_2O$ , which exhibits a complex structure of reflectance, which varies strongly near the K absorption edges of its constituent elements. Therefore, there arises a problem with several unknown quantities: the absolute resonance line intensities, their real intensity ratio, and the ratio between the crystal reflection coefficients. They may be found by solving the system of equations in the radiation recording in both channels for different  $T_e$ . The resultant data permit performing complete analysis: to determine the absolute line intensities and even find the ratio between the reflection coefficients at the resonance lines. Iterative mathematical

techniques may also be employed to solve the system of equations (see, for instance, Ref. [29]).

In conclusion we list the main results of the work. We have demonstrated the possibility to perform complex diagnostics of high-temperature laser-produced plasmas using a compact ultra-high luminosity X-ray focusing crystal spectrometer. The absolute intensities and X-ray yield of the resonance lines of [H]- and [He]-like ions were measured by the example of magnesium laser-produced plasmas, the electron temperature of the plasmas and the dependences of these quantities were determined under the variation of laser pulse energy in a broad range. Our approach makes it possible to perform complete diagnostics: to determine not only the plasma parameters, but also its radiative characteristics. These data are required for the development of new techniques for plasma diagnostics and calibration of spectroscopic devices. The possibility of performing complex diagnostics is important in numerous practical applications.

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