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RESEARCH

Radiation of a plasma generated by laser pulse on CO_2, CHF_3, and CF_4 gas-jet targets in the "water transparency window" 2.3–4.4 nm

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Abstract
The paper considers the emission spectra and dependences of the emission intensity of a number of carbon lines in absolute units for laser plasma generated by laser pulse on CO_2, CHF_3, and CF_4 gas jets on the gas pressure at the nozzle inlet. A pulsed Nd:YAG laser, $\lambda = 1064$ nm, $\tau = 5.2$ ns, $E_{imp} = 0.8$ J was used. Gas-jet targets were formed by a supersonic conical nozzle, the pressure at the nozzle inlet varied in the range of 5–25 bar. The emission spectra of the investigated gases were compared and the reasons for the different dependences of the radiation intensity on the gas pressure at the nozzle inlet were analyzed.

Keywords Laser plasma · Soft X-ray radiation · Water window · Gas jets

1 Introduction

Previously, a number of applications associated with soft X-rays are being actively developed, in particular, this is X-ray microscopy [1, 2]. For example, the Department of Multilayer X-ray Optics of the IPM RAS is developing an X-ray microscope for research in the "water transparency window" of 2.3–4.4 nm [3]. For the successful development of these applications, high-intensity laboratory soft X-ray sources are necessary. Laser-plasma sources of radiation (LPS) are the most convenient in terms of their properties [4].

Physics of interaction processes between laser radiation and the target substance was previously studied in a large number of works [5–12], including those of a fundamental nature [13, 14]. The main attention in these works was paid to the laser systems used. Less attention is paid to laser-plasma source targets. Various types of laser plasma sources targets were studied: solid-state, liquid-jet, and gas-jet. These sources have various advantages and disadvantages, but the most developed at the moment are gas-jet target formation systems.

As well known, the efficient emission in the "water transparency window" of 2.3–4.4 nm in laser plasma sources requires the formation of a dense high-temperature plasma. For the formation of such a plasma, the following conditions should be met: high-power laser radiation, strong absorption of radiation by the substance of the gas target, and a high density of emitting ions. Laser systems with different energy, pulse duration and operating wavelength have been studied [2, 8, 15–18]. Lasers with a nanosecond pulse duration have received the greatest practical application.

For gas-jet targets, high absorption of laser radiation and a high intensity of soft X-ray radiation can be achieved due to the high density of ions in the zone of laser spark formation. In turn, obtaining a high density can be achieved in two ways—the use of gases at high pressures or the use of polyatomic chemical compounds.

The article contains the results of a study of the emission of carbon ions. Plasma was generated by pulse laser radiation focused on CO_2, CHF_3, and CF_4 gas-jet targets. Nozzle inlet pressures were up to 25 bar. We used a pulsed gas valve and a supersonic nozzle to form a gas target. The target was excited by a laser with a nanosecond pulse duration.

The objectives of the work were investigation of the laser plasma radiation in the spectral range of the "water transparency window" 2.3–4.4 nm; measurement of absolute values of radiation intensity; study of changes in the spectra and absolute values of the radiation intensity depending on the nozzle inlet pressure of the gases; comparison of different gas-targets.

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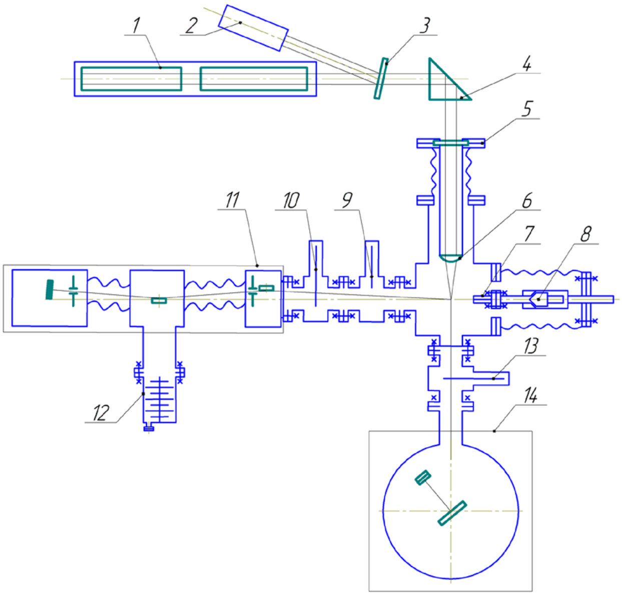

Fig. 1 Scheme of the research facility. 1—laser, 2—laser radiation power sensor, 3—dividing plate, 4—prism, 5—optical input, 6—lens, 7—nozzle, 8—quick-acting valve, 9—vacuum shutter, 10—two film free-standing filters, 11—spectrometer-monochromator RSM-500, 12—turbomolecular pump, 13—two-film free-standing filters, 14—spectrometer for measuring absolute radiation intensities

2 Experimental arrangement and operation

The scheme of the research facility is shown in Fig. 1.

The operation of the installation is carried out as follows. The gas enters the quick-acting valve 8 and further into the conical supersonic nozzle 7 and then is pumped out by cryocondensation and cryoadsorption pumps. Radiation of the laser 1 falls on the dividing plate 3, from where a small part of the radiation is reflected to the first radiation power detector 2. The main part of the radiation, passing through the prism 4 and the optical input 5, falls on the lens 6. At the focus of the short-focus lens, the laser radiation causes a breakdown and the formation of plasma in a gas jet. Polychromatic soft X-ray radiation of laser spark, passing through the electropneumatic vacuum shutter 9 and two free-standing X-ray filters 10 and enters the RSM-500 spectrometer–monochromator. Next, monochromatic soft X-ray radiation is detected by a pulsed detector. The RSM-500 is pumped out by a turbomolecular pump 12. During the work, we used spherical mirrors and gratings. The radius of curvature of the mirror is 4 m and of the grating is 3 m. The number of grooves is 600 grooves/mm. The spectral resolution of the device, measured at the L-absorption edges of silicon and aluminum, and the K-edge of beryllium free-standing filters, as well as at the zero-order half-width, was 0.04 nm. For the gratings and mirrors used, the operating wavelength range was 0.1–12 nm.

Free-standing film filters based on Al (thickness 150 nm) and Ti/Be with layer thicknesses of 3 nm/2 nm, number of periods is 30, were installed at the entrance to the RSM-500 spectrometer. These filters transmit radiation in the spectral range of the "water transparency window" and at the same time effectively absorb the long-wave noise component of the signal. Also, free-standing filters protect the detector from particles of various nature formed during the operation of the soft X-ray radiation source, which can effectively reduce background noise. The research facility is described in more detail in [19].

To study the absolute radiation intensities, a calibrated in absolute units spectrometer based on a multilayer X-ray mirror 14 was used [20, 21]. The spectrometer is a φ–2φ goniometer, in which multilayer X-ray mirror is used as a dispersing element. This device works as follows: soft X-ray radiation of a laser spark passes through the input free-standing film filter and falls on the X-ray mirror. In accordance with the Wulff–Bragg condition, radiation with a certain wavelength is reflected from the mirror. The reflected radiation passes through the second free-standing film filter and is recorded by the detector. Spectrum scanning is carried out by rotating (by an angle φ) the X-ray mirror relative to the incident beam, while the detector is rotated relative to the incident beam by a double angle (2φ). The rotation of the mirror and the detector is carried out using a stepper motor, the condition φ–2φ is provided by a gear.

In the mirror spectrometer 14, two Ti/Be free-standing film filters 13 with layers thickness of 3 nm/2 nm, the number of periods is 30, were installed. These filters effectively pass soft X-ray radiation with a wavelength of 3.1–10 nm. Multilayer X-ray mirror based on Cr/Sc was used. With such a combination of multilayer mirror and film filters, we can explore the spectral range of 3.1–7 nm. The spectral resolution of the device is determined mainly by the FWHM (full width at half maximum) of the X-ray mirror reflection curve [22].

For our research facility, filters and mirrors, in accordance with [22], the energy concentrated in the emission line of the E_{line} and the number of photons of the N_{line} can be determined as follows:

$$E_{\text{line}} = \frac{4\pi \cdot \alpha V}{\gamma \delta T^2 R}, \tag{1}$$

$$N_{\text{line}} = E_{\text{line}} \cdot \frac{\lambda_{\text{line}}}{h_{\text{c}}}, \tag{2}$$

where V is the signal recorded by the detector, in volts. For the researched spectral range, the sensitivity of the amplifier was $\alpha = 10^{-11}$ C/V; the solid angle of the detector entrance window as seen from the point EUV radiation source $\gamma = 5.45 \times 10^{-5}$ sr; sensitivity of the photodiode $\delta = 0.25$ C/J. T is the transmission coefficient of the Ti/Be film filter on the researched wavelength, R is the X-ray mirror reflection coefficient at the researched wavelength.

To excite the gas stream Nd:YAG laser was used. This laser has the following parameters: wavelength 1064 nm, variable pulse energy up to 0.8 J, pulse duration 5.2 ns, frequency 10 Hz. Laser radiation is focused on a gas target by a lens with a focal length of 45 mm. The calculated diameter of the focal spot is 66 µm. The transmitted laser radiation was detected by a laser radiation power sensor, which made it possible to estimate the absorption of laser radiation in the laser spark.

For the system of forming a pulsed gas stream, a pulse valve was used. High gas pressure (up to 25 bar) was created at the entrance to this valve. At the valve output, a conical nozzle was fixed. We used a conical supersonic nozzle, with a critical section diameter of 500 µm, 5 mm long, and a solution angle of 11°. In the experiments, the duration of the gas pulse was 0.5 ms.

Gas jets formed during the outflow of gas from conical nozzles to a vacuum, in the general case, have a complex spatial structure, determined by the parameters of the gas at the input in the nozzle and the geometric parameters of the nozzle. The tasks of describing the atomic-cluster jets that are formed with the outflow of the condensing gas from supersonic nozzle into vacuum are especially complicated. The gas-dynamic calculation of the structure of such an atomic-cluster target is very complicated and was not carried out in this work.

During the experiment, it was possible to measure the stagnation pressure along the axis of the gas jet. A capillary connected to a pressure meter, an ASM-300 type pressure meter, was introduced into the selected point of the gas flow.

3 Experimental results

3.1 CO_2 research

Initially, a gas target based on carbon dioxide was investigated. The emission spectra measured with the help of RSM-500 are shown in Fig. 2. It can be seen that with an increase in nozzle inlet pressure, the intensity of lines formed by ions with different charge increases unequally. The greatest intensity of the lines corresponds to the maximum nozzle inlet pressure of 25 bar.

Line interpretation and relative intensities of observed emission lines are given in Table 1 [23]. It is clear that with an increase in gas pressure, the intensity of the C VI lines increases faster. With an increase in gas pressure, there is a monotonous increase in the intensity of the radiation of lines, without the tendency to reach saturation. Thus, we can conclude about the prospect of increasing nozzle inlet pressure when used as a working gas CO_2.

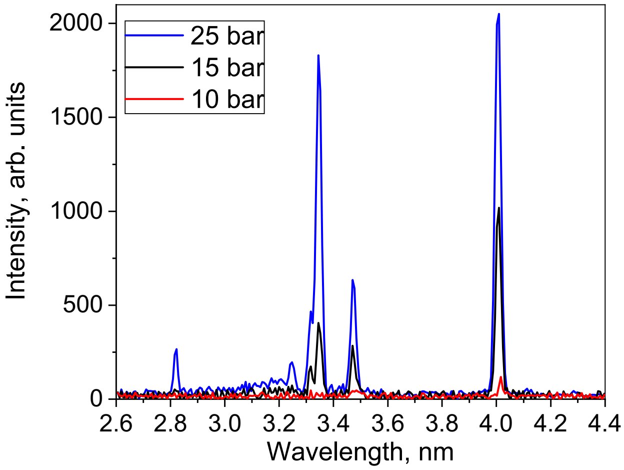

Fig. 2 The emission spectra CO_2 under pulse laser excitation at various nozzle inlet pressures

For a gas nozzle inlet pressure of 25 bar, studies of the absolute intensities of soft X-ray radiation were carried out. The spectrum measured with a mirror spectrometer is shown in Fig. 3. The resolution of the mirror spectrometer is worse than that of the RSM-500. It leads to a significant broadening of the lines on a registered spectrum. At the same time, the lines are located far enough from each other, which makes it possible to measure the absolute intensity of radiation of these lines.

The absolute intensities of the emission lines per laser pulse are given in Table 2.

Table 2 shows that the radiation intensities are very high and comparable with those obtained in [2, 8, 18]. Thus, when using targets based on CO_2, a sufficiently intense source of soft X-ray radiation can be realized. With an increase in the CO_2 nozzle inlet pressure to more than 25 bar, the intensity of the soft X-ray radiation should increase.

3.2 CHF_3 research

Next, we studied a gas-jet target based on CHF_3. The emission spectra measured with the RSM-500 are shown in Fig. 4. It can be seen that, with increasing pressure, the intensity of the lines formed by ions with different charges also increases unevenly. The highest intensity of the lines corresponds to the maximum nozzle inlet pressure of 25 bar.

The relative intensities of observed emission lines are given in Table 3 [23]. It is clear that with an increase in gas pressure, the intensity of the C VI lines increases faster. With an increase in nozzle inlet pressure, there is a monotonous increase in the intensity of the radiation of lines, with the

Table 1 Relative intensity of the emission lines of CO_2 at various nozzle inlet pressure

Wavelength (nm)	Ion	Transition	Intensity (arb. units)					
			25 bar	20 bar	17.5 bar	15 bar	12.5 bar	10 bar
4.026	C V	$1s^2$–$1s2p$	2050	1590	1400	1020	360	120
3.49	C V	$1s^2$–$2s3p$	630	480	450	280	100	50
3.37	C VI	$1s$–$2p$	1830	1220	970	400	130	–
3.343	C V	$1s^2$–$1s4p$	460	300	300	170	70	–
3.27	C V	$1s^2$–$1s5p$	200	140	130	–	–	–
2.84	C VI	$1s$–$3p$	260	140	120	–	–	–

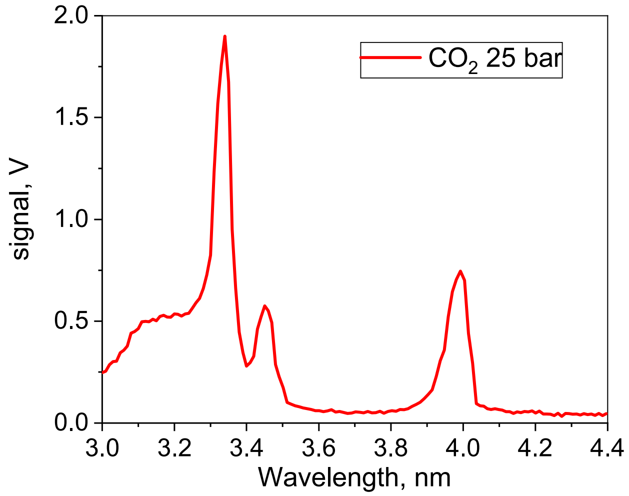

Fig. 3 Spectrum measured with a mirror spectrometer, obtained for CO_2 at a nozzle inlet pressure of 25 bar

Table 2 Absolute intensities of CO_2 emission lines at a nozzle inlet pressure of 25 bar

Wavelength (nm)	Ion	Transition	E_{line} (J)	N_{line} (ph/pulse)
4.026	C V	$1s^2$–$1s2p$	4.9×10^{-4}	9.9×10^{12}
3.49	C V	$1s^2$–$2s3p$	1.54×10^{-4}	2.7×10^{12}
3.37	C VI	$1s$–$2p$	4×10^{-4}	6.9×10^{12}

tendency to reach saturation. Thus, we can conclude that there is low prospect of increasing the gas nozzle inlet pressure when CHF_3 is used as the working gas.

For a nozzle inlet pressure of 25 bar, studies of the absolute intensities of soft X-ray radiation were carried out. The spectrum measured with a mirror spectrometer is shown in Fig. 5.

The absolute intensities of the emission lines per laser pulse are given in Table 4.

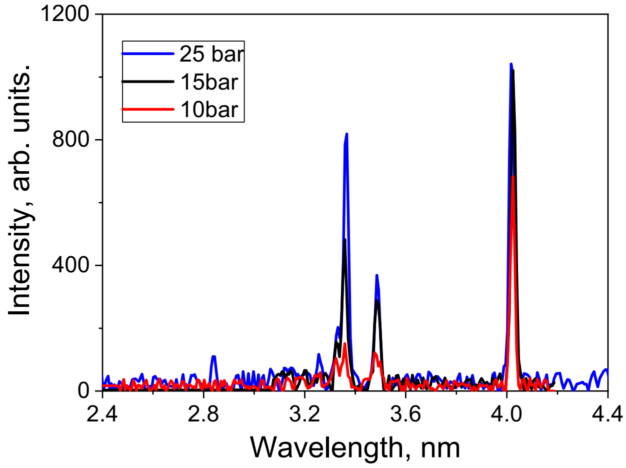

Fig. 4 Emission spectra of CHF_3 under pulsed laser excitation at various nozzle inlet pressures

You can see from Table 4 that the radiation intensities are quite high, but approximately two times less than the values obtained for CO_2. Increasing the nozzle inlet pressure when CHF_3 is used will not lead to a significant increase in the intensity of soft X-ray radiation.

3.3 CF_4 research

Next, we studied a gas-jet target based on CF_4. The emission spectra measured with the RSM-500 are shown in Fig. 6. It can be seen that, with increasing pressure, the intensity of the lines formed by ions with different charges also increases unevenly. The highest intensity of the lines corresponds to the maximum nozzle inlet pressures of 25 bar.

The relative intensities of observed emission lines are given in Table 5 [23]. It is clear that with an increase in gas pressure, the intensity of the C VI lines increases faster. With an increase in gas pressure, there is a monotonous increase in the intensity of the radiation of lines, with the tendency to reach saturation. Thus, we can conclude that there is low prospect of increasing the nozzle inlet pressure when CF_4 is used as the working gas.

Table 3 Relative intensity of the emission lines of CHF_3 at various gas nozzle inlet pressure

Wavelength (nm)	Ion	Transition	Intensity (arb. units)			
			25 bar	20 bar	15 bar	10 bar
4.026	C V	$1s^2$–$1s2p$	1040	1090	1020	680
3.49	C V	$1s^2$–$2s3p$	370	330	290	120
3.37	C VI	$1s$–$2p$	820	720	480	150
3.343	C V	$1s^2$–$1s4p$	200	180	160	100
3.27	C V	$1s^2$–$1s5p$	120	–	–	–
2.84	C VI	$1s$–$3p$	110	–	–	–

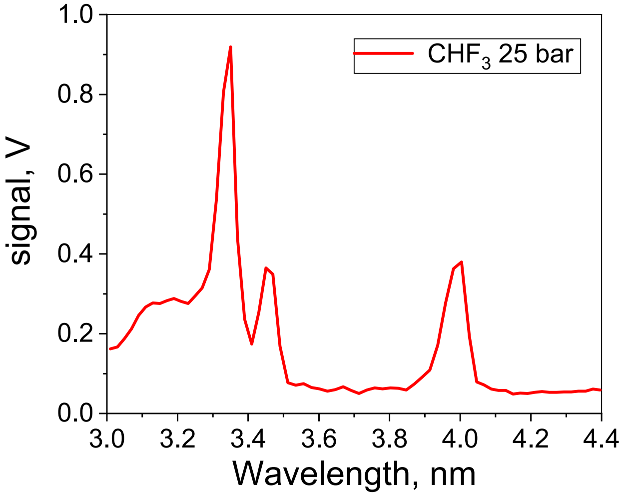

Fig. 5 Spectrum measured with a mirror spectrometer, obtained for CHF_3 at a nozzle inlet pressure of 25 bar

Table 4 Absolute intensities of CHF_3 emission lines at a nozzle inlet pressure of 25 bar

Wavelength (nm)	Ion	Transition	E_{line} (J)	N_{line} (ph/pulse)
4.026	C V	$1s^2$–$1s2p$	2.5×10^{-4}	5×10^{12}
3.49	C V	$1s^2$–$2s3p$	9.7×10^{-5}	1.7×10^{12}
3.37	C VI	$1s$–$2p$	2×10^{-4}	3.3×10^{12}

For a nozzle inlet pressure of 25 bar, studies of the absolute intensities of soft X-ray radiation were carried out. The spectrum measured with a mirror spectrometer is shown in Fig. 7.

The absolute intensities of the emission lines per laser pulse are given in Table 6.

You can see from Table 6 that the intensities of the emission lines are quite high, approximately two times less than the values obtained for CO_2 and approximately correspond to those for CHF_3. Increasing nozzle inlet pressure when using CF_4 will not lead to a significant increase in the intensity of soft X-ray radiation.

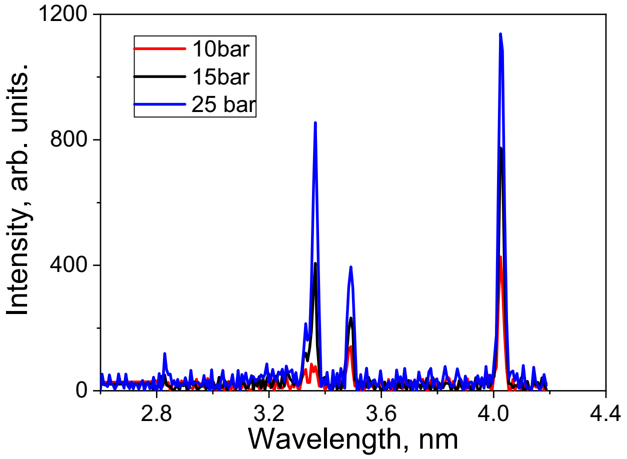

Fig. 6 Emission spectra of CF_4 under pulsed laser excitation at various nozzle inlet pressures

3.4 Emission of soft X-ray radiation as a function of pressure

Additional measurements were made of the relative intensity of the 4.026 nm line of the C V $1s^2$–$1s2p$ ion and the 3.37 nm line of the C VI $1s$–$2p$ ion depending on the gas pressure for various target gases. The 4.026 nm line is the brightest and very convenient for testing the radiation source, the 3.37 nm line is less intense, but is of considerable interest for soft X-ray microscopy studies. The line intensities were studied on a RSM-500 spectrometer-monochromator.

As you can see from Fig. 8 that with an increase in the pressure of CHF_3 and CF_4 at the entrance to the nozzle, the radiation intensities at a wavelength of 4.026 nm are close in magnitude and increase with reaching saturation. When using a target based on CO_2, the intensity of radiation at a wavelength of 4.026 nm varies in a complex way. Up to 14 bar, the intensity is relatively low; after 14 bar, a sharp increase is observed. The increase in intensity continues up to 25 bar without saturating. Thus, the behavior of the intensity of radiation of the CO_2 gas-jet target is fundamentally different from that of the CHF_3 and CF_4 targets.

Table 5 Relative intensity of the emission lines of CF_4 at various gas nozzle inlet pressure

Wavelength (nm)	Ion	Transition	Intensity (arb. units)			
			25 bar	20 bar	15 bar	10 bar
4.026	C V	$1s^2$–$1s2p$	1140	970	770	430
3.49	C V	$1s^2$–$2s3p$	400	340	230	140
3.37	C VI	$1s$–$2p$	850	660	400	80
3.343	C V	$1s^2$–$1s4p$	210	180	120	70
3.27	C V	$1s^2$–$1s5p$	–	–	–	–
2.84	C VI	$1s$–$3p$	120	–	–	–

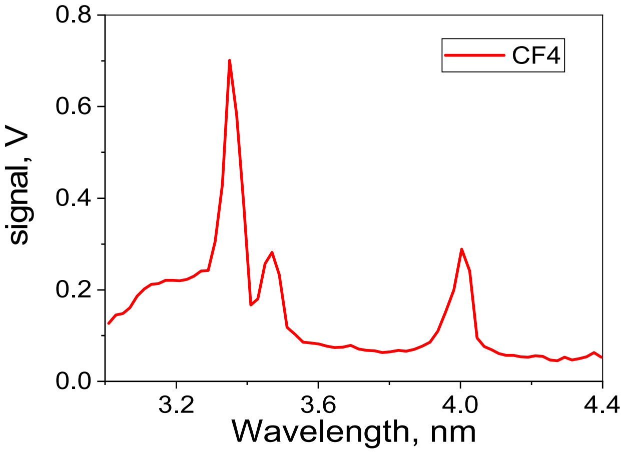

Fig. 7 Spectrum measured with a mirror spectrometer, obtained for CF_4 at a nozzle inlet pressure of 25 bar

Table 6 Absolute intensities of CF_4 emission lines at a nozzle inlet pressure of 25 bar

Wavelength (nm)	Ion	Transition	E_{line} (J)	N_{line} (ph/pulse)
4.026	C V	$1s^2$–$1s2p$	1.9×10^{-4}	3.85×10^{12}
3.49	C V	$1s^2$–$2s3p$	7.5×10^{-5}	1.3×10^{12}
3.37	C VI	$1s$–$2p$	1.5×10^{-4}	2.5×10^{12}

Figure 9 shows that the behavior of the radiation intensity at a wavelength of 3.37 nm qualitatively corresponds to that for a wavelength of 4.026 nm. As the nozzle inlet pressure increases, the emission intensities of the CHF_3 and CF_4 targets are close and tend to saturate. When using a CO_2 target, the intensity of the 3.37 nm line is low up to a pressure of 14 bar and then sharply increases.

Thus, the form of the dependence of the intensity of soft X-ray radiation on the gas pressure for the CO_2 target differs significantly from the form of the dependence for the CHF_3 and CF_4 targets. The reason for these differences is not entirely clear.

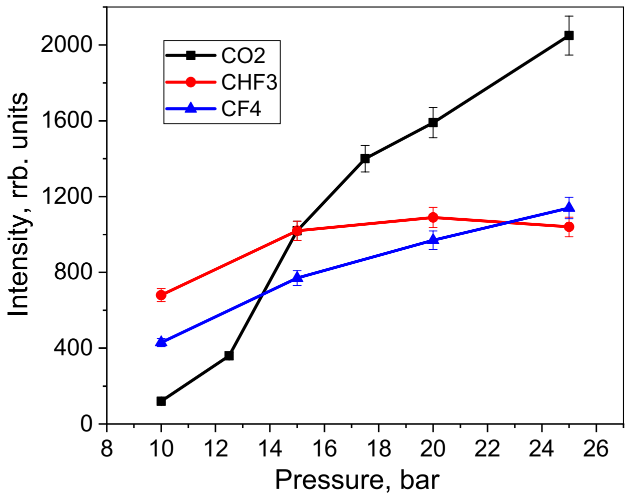

Fig. 8 Relative intensities of radiation at a wavelength of 4.026 nm for various gas targets depending on nozzle inlet pressure

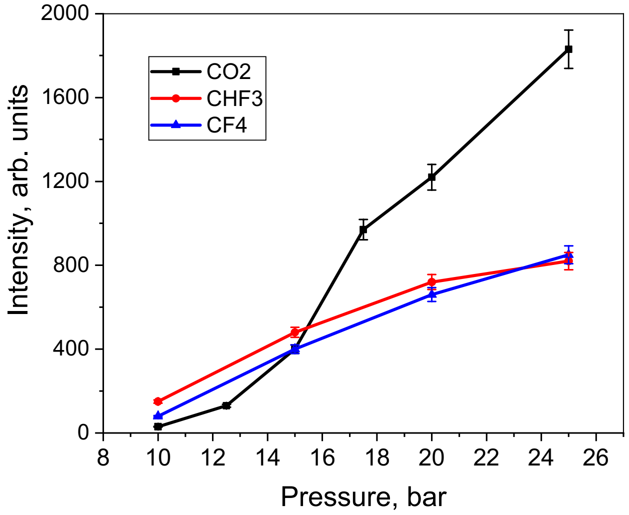

Fig. 9 Relative intensities of radiation at a wavelength of 33.7 A for various gas-targets depending on nozzle inlet pressure

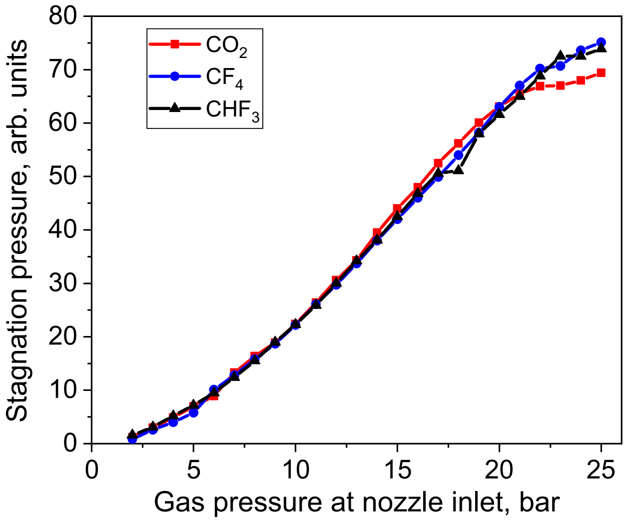

Fig. 10 Stagnation pressures of CO_2, CHF_3 and CF_4 jets near the laser spark formation zone depending on the nozzle inlet pressure

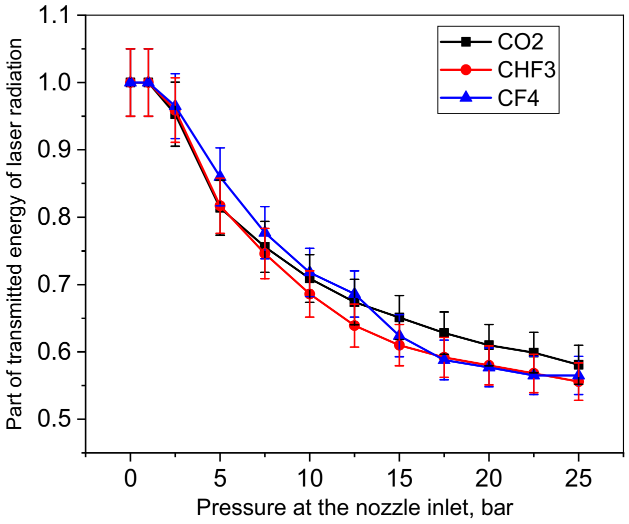

Fig. 11 Part of transmitted energy of laser radiation depending on the pressure at the nozzle inlet

4 Discussion

We tried to understand why the dependence of soft X-ray radiation on gas pressure for the CO_2 target differs significantly from the dependence for the CHF_3 and CF_4 targets. The emission of soft X-ray radiation in the first approximation is determined by the concentration of ions with the corresponding degree of ionization. Therefore, we carried out additional measurements of the concentrations of carbon atoms near the zone of laser spark formation. Figure 10 shows obtained dependences of the stagnation pressure in the laser spark formation zone on the gas pressure at the nozzle inlet.

You can see that the stagnation pressure near the zone of laser spark formation depends almost linearly on the gas pressure at the nozzle inlet. Small deviations from linearity are observed for low pressures, less than 5 bar, and for high pressures, more than 20 bar. Deviations from linearity at high pressures can be associated with the inertia of the moving parts of the valve. At low pressures, deviations from linearity can be associated with a change in the gas flow regime, a transition from the classical flow regime to the flow with condensation. When the gas pressure at the nozzle inlet is more than 2–3 bar, the flow regime is characterized by developed condensation with the formation of a large fraction of cluster condensate in the jet. Due to the long duration of the laser pulse, on the order of – 5 ns, the effect of the condensate in the target jet is reduced to a change in the local gas density in the zone of laser spark formation. Thus, we neglect all specific cluster effects in the interaction of laser radiation with clusters.

As you can see from Fig. 10, for all studied gas-targets, the outflow processes are approximately the same, and the concentrations of particles in the laser spark zone are also approximately equal. Thus, the different types of dependences of the intensity of soft X-ray radiation for CO_2 on the one hand, and CHF_3/CF_4 on the other hand, are associated not with the processes of gas outflow, but with the processes that take place during the formation of a laser spark.

An important characteristic of the process of interaction between a gas jet and laser radiation is the amount of absorption of laser radiation energy in a spark. Therefore, we carried out additional measurements of the part of laser radiation energy transmitted through the laser spark zone. The results of measurements are shown in Fig. 11.

You can see that the absorption of laser radiation is also approximately the same for various gases, without a sharp change in the form of the dependence. The absorption of laser energy by the CO_2 target is somewhat less by the CHF_3 and CF_4 targets.

From the above data, we can assume that at pressures at the inlet to the nozzle for CHF_3 and CF_4 more than 15 bar (see Figs. 8, 9), the energy losses for the formation of a laser detonation wave [3, 4], gas ionization and radiation become comparable with the gain absorbed energy of laser radiation. With a further increase in pressure, the number of C V and C VI ions practically does not increase, respectively, the emission intensities of the 4.026 nm and 3.37 nm lines are practically constant.

A different picture is observed for the CO_2 target. With comparable absorption of laser radiation and an increase in the energy of absorbed laser radiation with an increase in gas nozzle inlet pressure, the energy loss for the CO_2 target is not so large. So CO_2 has a smaller number of atoms in a molecule, which reduces the energy costs for gas ionization. The radiative energy loss under our conditions for the oxygen atom is also less than for the fluorine atom. Other differences are also possible in the processes during the formation of a laser spark, which lead to such large differences in the number of C V and C VI ions for targets based on CO_2, on the one hand, and for CHF_3 and CF_4, on the other hand.

Based on the foregoing, we can conclude that it is promising to continue the study of other molecular gas-targets for the source of soft X-ray radiation in the “water transparency window”. Light gas-targets with an increased carbon content in the molecule, such as CO, CN or C_2H_2, are very promising.

5 Conclusion

The novelty of the conducted research is conducting comparative studies of the emission spectra of various carbon-containing gas targets in the spectral range of the water transparency window. The absolute intensities of radiation of carbon ions C VI and C V were also measured at wavelengths of 3.37 and 4.026 nm. In addition, we investigated the dependences of the absolute radiation intensity on these lines on the gas pressure at the nozzle inlet; absorption of laser radiation energy in the spark formation zone; measuring the stagnation pressure of the gas jet in the zone of laser spark formation.

It has been established that the intensities of the carbon lines at 4.026 nm and 3.37 nm for CO_2 at pressures above 15 bar are significantly higher than those for CHF_3 and CF_4. Additional measurements of the concentration of gas-target particles and the absorption of laser radiation in a laser spark were made. Based on the studies performed, we concluded that the observed differences in the intensities of soft X-ray radiation for these target gases are not related to gas dynamic processes. These differences are due to the molecular composition of gas-targets. We believe that, it is possible to increase the intensity of the soft X-ray radiation of laser plasma using light gas-targets with an increased carbon content in the molecule. In this case, the power of the laser excitation system can be left at the same level.

The spectra obtained and the measured absolute intensities of carbon ions radiation are of practical importance and will be used in the development of a pulsed laser-plasma radiation source in the “water transparency window” of an X-ray microscope.

Dependences of the intensity of emission lines on gas nozzle inlet pressure can be used to study the formation of a laser spark on various molecular gas-targets.

Author contributions AN and AP conducted experimental studies and wrote the main text of the work. NS and NC critically examined/monitored each step of the work and further reviewed the manuscript.

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Data availability The datasets generated and analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests The authors declare no competing interests.

Conflict of interest The authors declare no conflict of interest.

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