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Generation of soft X-rays and extreme ultraviolet (EUV) using a laser-irradiated gas puff target*

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Abstract

The paper gives a review of investigations on generation of soft X-rays and EUV using a gas puff target irradiated with high-power laser pulses performed during last few years. In the studies a newly developed double-stream gas puff target approach was used. The gas puff targets were irradiated with various lasers, including Nd:glass, Kr:F, Nd:YAG, and iodine lasers. The aim of the studies was to develop efficient and debris-free laser plasma X-ray and EUV sources for applications in microscopy, lithography, micro- and nanotechnology.

Keywords: Extreme ultraviolet (EUV); Gas puff target; Laser plasmas; X-rays

1. INTRODUCTION

A hot and dense plasma produced by interaction of nanosecond high power laser pulses with matter is an intense source of electromagnetic radiation in the wavelength range extending from tenths of a nanometer to a few tens nanometer. This wavelength range corresponds to the regions of soft X-rays and extreme ultraviolet (EUV) radiation (Attwood, 1999). Laser plasma X-ray and EUV sources are considered to be used in many applications in science and technology which are discussed in current literature (Gizzi *et al*., 2004; Faenov *et al*., 2004; Turcu *et al*., 2004; Desai *et al*., 2003; Pikuz *et al*., 2001; Marzi *et al*., 2000; Turcu & Dane, 1998; Attwood, 1999).

In laser plasma X-ray and EUV sources the laser radiation interacts with solid, liquid, or gas targets. During the laser-target interaction hot and dense plasma is created which emits strong radiation in the soft X-ray and EUV range. A great disadvantage of laser plasma X-ray and EUV sources based on solid targets is the production of target debris by laser ablation. To avoid the debris production, a new method of plasma generation by using a gas puff target instead of a solid target was developed at the Institute of Optoelectronics (Fiedorowicz *et al*., 1992, 1993). The gas puff target is formed by pulsed injection of a small amount of gas under high-pressure into a laser focus region. Moreover, the use of the gas puff target increases the absorption of laser energy in the plasma, and to improve the X-ray and EUV conversion efficiency. Strong X-ray and EUV emissions from gas puff targets irradiated with nanosecond high-power laser pulses were demonstrated in the experiments performed at various laboratories (Fiedorowicz *et al*., 1994, 1996*a*, 1998, 1999*a* or *b*; Celliers *et al*., 1996; Suzuki *et al*., 2003).

The laser plasma radiation source based on a gas puff target was successfully used in the spectral investigations of emission from multiply-charged ions. The characteristic features of the gas puff laser plasmas made possible to measure the X-ray spectra with very high spectral resolution, which was not available for other laser plasma sources. The investigations were performed in the collaborative experiments that were carried out at the Institute of Optoelectronics (Khakhalin *et al*., 1994, 1995; Skobelev *et al*., 1997, 1999; Bartnik *et al*., 1999; Doron *et al*., 1999).

It was observed that to achieve high X-ray and EUV production, the laser focus has to be placed very close to the nozzle output, causing degradation of the nozzle by the plasma. The problem with the nozzle degradation was solved with a new concept of a double-stream gas puff target approach developed at the Institute of Optoelectronics (Fiedorowicz *et al*., 2000*a*).

The double-stream gas puff target is formed by pulsed injection of high-Z gas (xenon, krypton, argon, etc.) into a hollow stream from a low-Z gas (hydrogen, helium) using an electromagnetic valve system equipped with a double-nozzle setup. The outer stream of gas confines the inner stream of gas improving the gas puff target characteristics

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(higher gas density at larger distance from the nozzle output). Using this new approach, strong enhancement of X-ray production was demonstrated (Fiedorowicz *et al.*, 2000*a*). The double-stream gas puff target makes possible to develop an efficient and debris-less laser-produced plasma X-ray sources for many applications, including microscopy, lithography, micro- and nanotechnology.

This paper describes our recent investigations on X-ray and EUV emissions from the double-stream gas puff target irradiated with nanosecond high-power laser pulses. The experiments were performed at the Institute of Optoelectronics in Warsaw, the Institute of Laser Engineering in Osaka, the Institute of Plasma Physics in Nieuwegein, and the Prague Asterix Laser System (PALS) Laboratory in Prague. In the experiments at Warsaw, the targets were irradiated with Nd:glass and Nd:YAG lasers. The experiments at Osaka were performed with the use of a Nd:YAG laser also. In the experiments at Nieuwegein, the double-stream gas puff target was irradiated using a KrF laser.

2. DOUBLE-STREAM GAS PUFF TARGET APPROACH

The double-stream gas puff target is created using a special valve system equipped with a double nozzle setup. The valve system and the nozzle setup are shown schematically in Figures 1a and 1b, respectively. The valve system consists of two electromagnetic valves combined in the common body. Each valve is driven by a current pulse from the power supply synchronized with the laser power supply, to be able to set the proper time delay between the laser pulse and opening of the valve. The double nozzle setup consists of two coaxial nozzles. The inner nozzle has a form of a circular orifice of 0.4 mm in diameter, through which high-Z gas is injected into the hollow stream of low-Z gas. The hollow stream is produced by the outer nozzle in a form of a ring with inner diameter of 0.7 mm and outer diameter of 1.5 mm surrounding the inner nozzle. The outer gas stream confines the gas flowing through the inner nozzle and prevents spherical expansion of high-Z gas, which is a case for the ordinary gas puff target created in result of pulsed injection of gas into vacuum. The double-stream gas puff target approach makes possible to form an elongated gas stream with high gas density at a relatively long distance from the nozzle output.

The gas puff targets were characterized using the pulsed X-ray radiography (X-ray backlighting). The laser plasma X-ray source based on a gas puff target irradiated with a 1 ns/5 J laser pulse from a Nd:glass laser was used as a back lighter. The wavelength of the X-ray back lighting beam (near 0.7 nm) was determined by the use of krypton gas as the target and the X-ray selecting filter made from a 20-μm-thick Si filter combined with a 20-μm-thick Be foil. X-ray shadow grams of the back lighted gas puff targets were registered using a CCD camera. Typical X-ray shadow grams of the gas puff targets are shown in Figure 2. Figure 2a presents the shadow gram of the a double-stream argon/helium gas puff target, and Figure 2b shows the shadow gram of the ordinary gas puff target from argon created without injection of helium. An X-ray shadow gram of the double-stream xenon/helium gas puff target is presented in Figure 2c. Backing pressure in the valves was 10 bar.

From the shadow grams gas density spatial profiles can be obtained. The maximum gas density of xenon for the xenon/helium target at the distance of 1 mm from the nozzle output was measured at about of 5 mg/cm^3. It corresponds to the particle density of about 2.3×10^{19}. More detailed information on characterization measurements using the X-ray back lighting method are given elsewhere (Fiedorowicz *et al.*, 1999*a* or *b*, 2000*a*, 2003, Rakowski *et al.*, 2004).

3. X-RAY AND EUV EMISSIONS STUDIES WITH A DOUBLE-STREAM GAS PUFF TARGET

3.1. Nd:glass laser experiments

The experiments with a Nd:glass laser were performed at the Institute of Optoelectronics, Warsaw. The double-stream

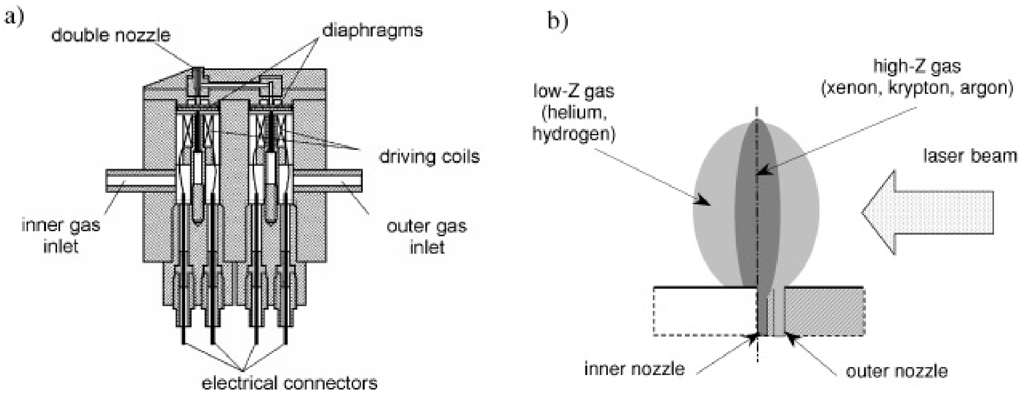

Fig. 1. Schematic of the valve system to form the double-stream gas puff targets (**a**) and the double-nozzle setup (**b**).

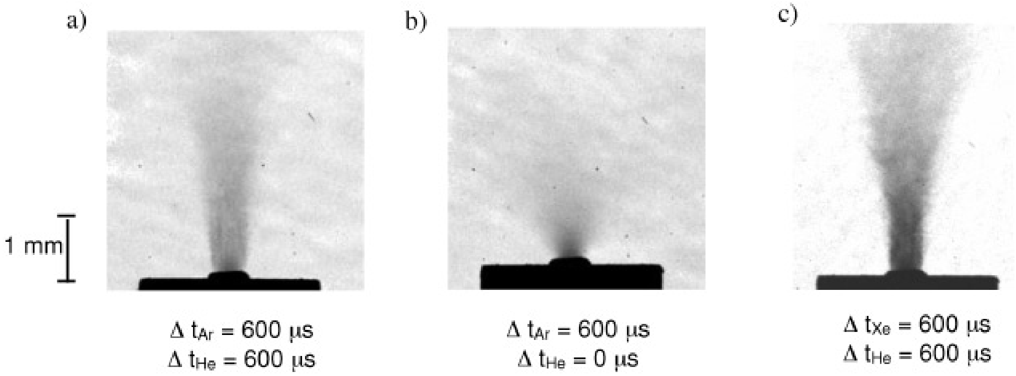

Fig. 2. Typical X-ray shadow grams of the gas puff targets. (**a**) The double-stream argon/helium target. (**b**) The ordinary argon gas puff target. (**c**) The double-stream xenon/helium target.

gas puff targets were irradiated with laser pulses from a high-power nanosecond Nd:glass laser. The time duration of the laser pulses was about 1 ns and energy to 10 J. The laser beam was focused onto the target perpendicularly to the flow of gas. The distance between the laser focus and the nozzle output was varied from 0.5 mm to 1 mm. Soft X-ray emission at the wavelength near 1 nm (photon energy about 1-keV) was measured using various diagnostic instruments, including a pinhole camera, a crystal spectrograph, and a semiconductor detector. Strong enhancement of soft X-ray emission from the xenon/helium target was observed as compared to the emission from the ordinary xenon target. This effect is caused by higher gas density for the double-stream gas puff target, the distances between the laser focus and the nozzle output is larger than 0.5 mm which makes possible to increase absorption of laser energy in the plasma. The detailed results of these studies are presented and discussed in other papers (Fiedorowicz *et al.*, 1999*a* or *b*, 2000*a*).

In the next experiments, the main instrument to measure emission in the soft X-ray and EUV range was a transmission grating spectrometer equipped with free-standing grating of 1000 lines/mm placed inside of the 50 μm pinhole. Spectral images of the plasma source were registered with a CCD camera with a back-illuminated CCD array. The spectrograph measured spectra in the wavelength from 1 nm to 15 nm. Additionally, the absolutely calibrated silicon photodiode with a filter made from a 1 μm thick Y layer onto a 0.2 μm thick SiN_4 membrane was used to measure energy of X-ray pulses. The detector was placed at the distance of 26 cm from the source. The signals from the detector were registered using the 500 MHz digital oscilloscope.

Spectral images for the xenon/hydrogen, krypton/hydrogen, argon/helium, and nitrogen/helium targets were measured for various parameters (laser energy, laser focus position, gas pressure). From the images spectral distributions in the soft X-ray and EUV range were obtained. The spectral distributions for the xenon/hydrogen and the krypton/hydrogen targets are presented in Figure 3. The spectra was measured for various positions of the target in respect to the laser focus determined by the distance Δx between the nozzle centre and the laser focus along the laser beam.

In the xenon spectra (see Fig. 3a), three wavelength bands are dominating in the spectrum. The X-ray emission near 1 nm, which is considered to be useful for X-ray proximity

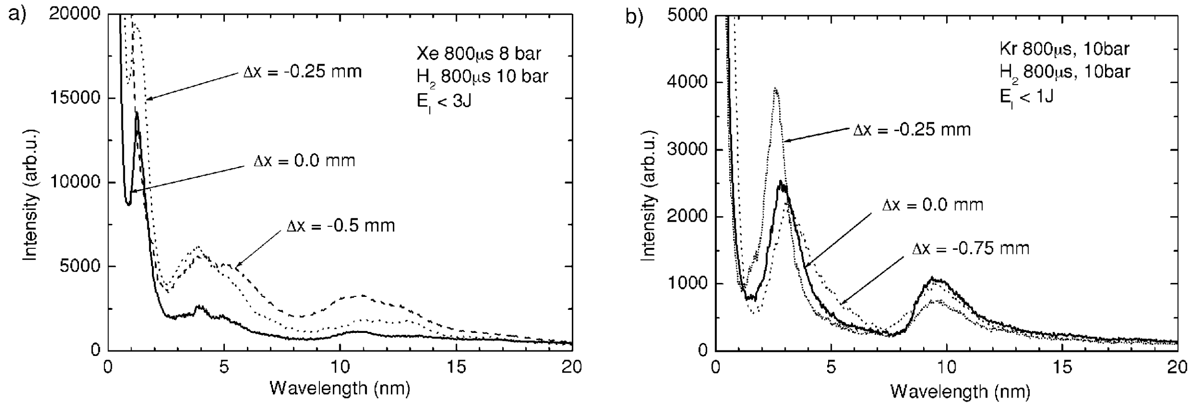

Fig. 3. The spectral distributions for the xenon/hydrogen (**a**) and the krypton/hydrogen (**b**) gas puff targets obtained for various distances between the laser focus and the nozzle output Δx.

printing lithography, is much stronger as compared with the soft X-ray emission near 4 nm and EUV radiation near 11 nm. Emission in the latter band is considered to be used in the projection lithography (EUV lithography). Intensity of emission in these bands strongly depends on the position of the laser focus. It means that X-ray and EUV production can be optimized by using proper intensity of laser radiation in the interaction region. It was found that intensity of emission near 13 nm, which is important for the projection EUV lithography based on Mo/Si optics, could be increased in respect to the dominating spectral feature near 11 nm.

The spectrum for the krypton targets (see Fig. 3b) exhibits two dominating spectral bands. The intense soft X-ray production in the "water window" can be useful for X-ray microscopy of biological samples. Relatively strong EUV production is not applicable for EUV lithography because the wavelength of the emission is too short.

The emitted energy in X-ray and EUV pulses was measured using the calibrated photodiode. The efficiency of conversion of laser energy into X-ray energy, determined as ratio between the energy of X-ray radiation emitted into 4π angle and the energy of laser pulses, was about 6% for the laser energy of about 3 J. The time duration of X-ray pulses was usually 2–3 times longer than the pulse duration of the laser pulse. The details of the experiments and the results of measurements for different gases (argon, nitrogen) are presented elsewhere (Fiedorowicz *et al.*, 2001*a*, 2001*b*).

3.2. KrF laser experiments

The experiments with a KrF laser were performed at the Institute for Plasma Physics in Nieuwegein, The Netherlands. In the experiments the double-stream xenon/helium and xenon/hydrogen targets were irradiated with a KrF laser generating laser pulses of 27 ns duration and energy to 0.9 J. EUV emissions in the wavelength band near 13 nm was measured using the calibrated silicon photodiode combined with a multilayer molybdenum/silicon mirror. The EUV detector signals for various targets and different distances between the laser focus and the nozzle output are shown in Figure 4.

It can be seen that emission near 13 nm from the double-stream gas puff target was an order of magnitude higher than in a case of the ordinary gas puff target. Moreover, emission observed for the xenon/hydrogen target is higher as compared to the xenon/helium target. It is caused by lower absorption of EUV radiation in hydrogen than in helium. The experiment and the obtained results were described in details by de Bruijn *et al.* (2000)

3.3. Nd:YAG laser experiments

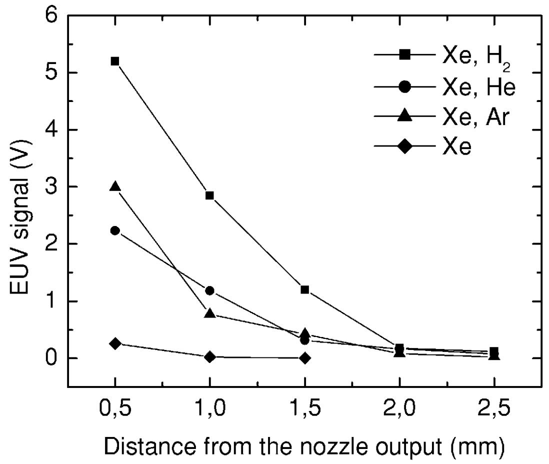

Fig. 4. EUV detector signals for various targets and different distances between the laser focus and the nozzle output.

Investigations on X-ray and EUV emission from a double-stream gas puff target irradiated with high-power laser pulses from Nd:YAG lasers were performed at the Institute of Laser Engineering, Osaka in Japan, the Laser Laboratorium, Goettingen in Germany, and the Institute of Optoelectronics, Warsaw.

In the experiments performed at Osaka, the xenon/helium targets were irradiated with a commercial Nd:YAG laser (Continuum YG682) producing 10 ns laser pulses with energy to 0.7 J at 1.06 μm wavelength. The laser beam was focused onto the gas puff target perpendicularly to the flow of gas, at the distance of 1 mm from the nozzle output. X-ray and EUV emissions in the 8–18 nm wavelength range were measured using two grazing incidence flat-field grating spectrometers. One of the spectrometers, equipped with a toroidal mirror, allowed measuring the spectra with spatial resolution. The spectra were registered with CCD cameras. Spectral and spatial distributions of EUV emission from the xenon/helium gas puff target were obtained. Figure 5 presents typical spectra for the double-stream xenon/helium target and for the ordinary xenon target.

The xenon spectrum shows a characteristic dominant spectral feature centered near 11 nm, originating from $4d$–$4f$

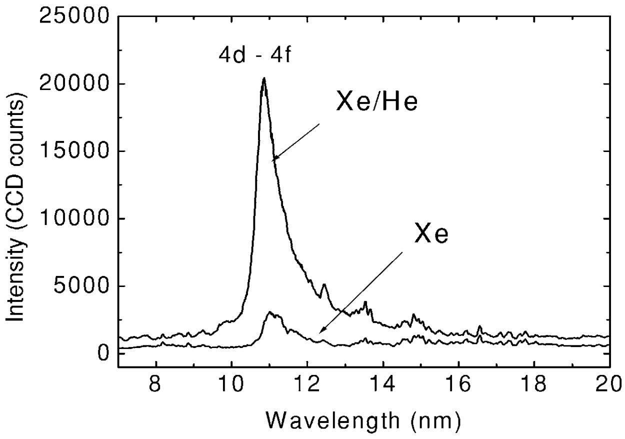

Fig. 5. Typical spectra for the double-stream xenon/helium target and for the ordinary xenon target.

transition array in XeVII-XeXII ions. Strong enhancement of EUV emission from the double-stream target comparing to the ordinary target can be seen. The enhancement is caused by higher gas density in the interaction region, sharper density gradients, and smaller thickness of the cold gas surrounding the hot plasma region. Smaller size of the source in the case of the double-stream target was also observed.

To compare EUV production from a double-stream gas puff target with EUV production from a solid target, the spectra from solid copper, iron, and tin targets irradiated with a Nd:YAG laser in the same conditions were measured with the same grating spectrometer. The spectra (see Fig. 6) clearly show that emission from the xenon target near 11 nm is much stronger than EUV emission from solid aluminum, copper, and iron targets, and comparable to emission near 13 nm from a solid tin target, that was found to be the best radiator in this wavelength range (Spitzer *et al*., 1993).

More detailed descriptions of the experiments performed at Osaka and the results of EUV emission measurements from different gases are presented in other papers (Fiedorowicz *et al*., 2000*b*, 2001*a* or *b*; Bartnik *et al*., 2000).

The same gas puff valve system was used in the experiments performed at Goettingen where a Nd:YAG laser producing 6 ns laser pulses to 0.5 J of energy. EUV emission from various gases, including xenon, krypton, and oxygen were studied carefully using the reflection grating spectrograph with the varied space grating and a CCD camera, the EUV pinhole camera and the calibrated silicon photodiodes. The measured characteristics of the source were described by Kranzusch & Mann (2001). The source was used in the EUV metrology (Kranzusch *et al*., 2001).

The detailed characterization and optimization studies of EUV emission from the double-stream gas puff target irradiated with a Nd:YAG laser was performed at the Institute of Optoelectronics under the research project in the frame of the MEDEA+ program. The aim of the project was to develop a compact laser plasma radiation source for EUV metrology. In the experiments commercial Nd:YAG lasers producing 4 ns laser pulses with energies to 0.5 J or 0.7 J were used to irradiate the gas puff target created with the improved version of the valve system. The targets were characterized using the pulsed X-ray radiography (X-ray back lighting). The laser plasma X-ray source based on a gas puff target irradiated with a 1 ns/5 J laser pulse from a Nd:glass laser was used as a back lighter. X-ray shadow grams of the back lighted gas puff targets were registered using a CCD camera. Gas densities spatial profiles for various time delays between the opening of the valve and the X-ray back lighting pulse were obtained.

To study EUV emission from the source various measurement instruments were developed. EUV spectra with high resolution in the wavelength range between 5 nm and 20 nm were measured using flat-field reflection grating spectrometer. The spectrometer was equipped with a 1200 line/mm grating with varied groove spacing. The entrance slit of was mounted in the light-tight labyrinth. The spectra were registered using a back-illuminated CCD camera. Typical EUV spectrum for the xenon/helium target is presented in Figure 7.

It was found that EUV emission at 13.5 nm, which is important for EUV lithography technologies based on Mo/Si optics, can be optimized by changing the time delay Δt_{He} between the laser pulse and the opening of the helium valve. The optimum time delay Δt_{He} at about 350 μs was measured. The optimum time delay Δt_{Xe} between the laser pulse and the opening of the xenon valve was determined at 800 μs.

Temporal and EUV yield measurements were performed using the calibrated silicon photodiode AXUV-HS1. The diode was mounted in the detection head equipped with a filter made from a 1 μm thick Y layer onto a 0.2 μm thick SiN_4 membrane and a light-tight labyrinth. The signals from the detector were registered with a 500 MHz oscilloscope. The detector was mounted to the additional vacuum chamber coupled to the source chamber. In the additional chamber a Mo/Si mirror was placed to select EUV emission at 13.5 nm. Additionally, EUV yield was measured with the silicon photodiode AXUV-100/Zr covered with a Zr filter

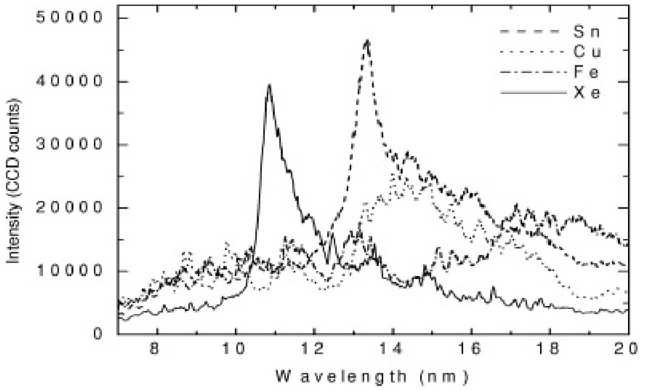

Fig. 6. EUV spectra for the xenon/helium gas puff target and solid iron, copper, and tin targets irradiated in the same conditions.

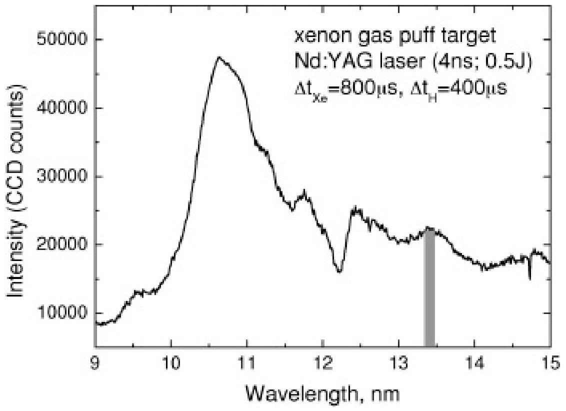

Fig. 7. EUV spectrum for the xenon/helium target.

on the detector surface. The photodiode was mounted in the detection head with a special electronic circuit allowing changing the sensitivity of the detector head. The signals from the detector head were registered with the data acquisition card. The detection system can be used for monitoring of the laser plasma EUV source operating with repetition.

Conversion efficiency of about 1.6% of the laser energy into EUV radiation emitted at 13.5 nm was measured with different instruments. The detailed results of the studies are presented in other papers (Fiedorowicz *et al*., 2003, 2004*a*). The results of the studies were used for the design of the prototype of a laser plasma radiation source dedicated for EUV metrology applications. The source is presented in Figure 8. Preliminary experiments of the characterization of molybdenum/silicon multilayer mirrors and their degradation under irradiation with EUV radiation were performed. The source was recently used in micromachining of polymers by direct X-ray photo-etching (Bartnik *et al*., 2004).

3.4. High-power iodine laser experiments

The experiments were performed at the Joint Research Laboratory PALS of the Institute of Physics and the Institute of Plasma Physics, Czech Academy of Sciences, Prague, Czech Republic. The aim of the studies was to verify possibilities to develop high-intensity laser plasma X-ray sources for applications in X-ray microscopy and micro-processing of solids with soft X-ray pulses.

It is known that a single shot exposure soft X-ray microscope based on a Fresnel optics requires about 10^{15} photons of soft X-rays in a single line in the wavelength range between the K-absorption edges of oxygen ($\lambda = 2.34$ nm) and carbon ($\lambda = 4.38$ nm). This wavelength range is called "water window." In the previous studies, we proposed to use a nitrogen/helium double-stream gas puff target irradiated with the PALS laser to produce soft X-ray radiation in a single line at 2.5 nm from hydrogen-like nitrogen ions (Fiedorowicz *et al*., 1998). The aim of the experiments performed at the PALS facility was to verify if it is possible to produce such amount of photons using this setup.

In the experiment, gas puff targets were produced with the same valve system as presented in previous sections, which was equipped with a modified double-nozzle setup. The nozzle setup had the inner nozzle diameter of 2 mm and the outer nozzle in a form of a ring with inner/outer diameters 2.5 mm and 3 mm, respectively. The target was irradiated with high-power iodine laser PALS (Rus *et al*., 1999; Jungwirth *et al*., 2001) producing 0.5 ns laser pulses at a wavelength of 1.3 μm. Maximum energy of the laser pulses was about 700 J. Spectral measurements of the soft X-ray emission in the "water window" was performed using the transmission grating spectrograph. The grating with 10000 lines/mm was coupled to a 100 μm wide slit. The spectra were registered with a CCD camera equipped with the back-illuminated non-AR coated CCD array and 16 bit A/D converter. The spectrograph was developed at the University of Applied Sciences, Remagen. Additionally, soft X-ray production in the "water window" was measured with the absolutely calibrated silicon photodiodes. The experimental arrangement is described in details in another paper (Fiedorowicz *et al*., 2001*d*).

Soft X-ray spectra from the nitrogen/helium gas puff targets irradiated with PALS laser was measured. The results are under evaluation and will be published in a separate paper. Typical soft X-ray spectrum in the "water-window" for the nitrogen/helium target is shown in Figure 9. Rough estimation of the X-ray production in a single line at 2.5 nm gave a number of about 5×10^{15} photons for the laser energy of 540 J. This result was confirmed by the measurements using the silicon photodiodes. Soft X-ray production was estimated at about 1 J in 4π for the laser energy of 630 J. This energy corresponds approximately to 10^{16} photons. This value is comparable with the photon number measured with the grating spectrograph, and should be enough for a single shot exposure X-ray microscope.

The same experimental setup was used in another experiments performed at Prague. The aim of these experiments was to perform the first direct photo-etching of organic polymers using a laser plasma X-ray source based on a laser-irradiated gas puff target. High-intensity nanosecond pulses of soft X-ray radiation in the wavelength range from about 1 nm to 8 nm were produced by irradiation of the xenon/helium double-stream gas puff target with the PALS facility. The soft X-ray spectrum from the xenon/helium

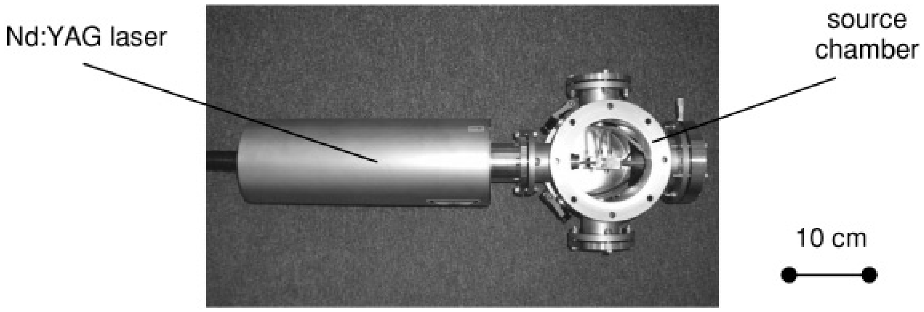

Fig. 8. Compact laser plasma EUV source based on a double-stream gas puff target irradiated with a Nd:YAG laser for metrology applications.

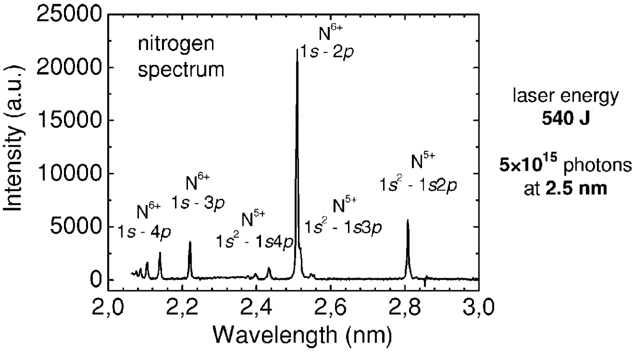

Fig. 9. Typical soft X-ray spectrum in the "water window" for the nitrogen/helium gas puff target irradiated with the PALS facility.

target measured with the transmission grating spectrograph is shown in Figure 10. The conversion efficiency of laser energy into X-rays in the 1–2 nm wavelength band (dominating in the spectrum) determined with the use of the calibrated X-ray detectors was about 30%. The source characterization and optimization measurements are described in another paper (Fiedorowicz *et al*., 2004*b*).

The produced X-ray pulses were used to irradiate samples from organic polymers and form microstructures by X-ray direct photo-etching. The results show relatively high efficiency of X-ray direct photo-etching that could be useful for micromachining of organic polymers (Juha *et al*., 2003; Fiedorowicz *et al*., 2004*c*).

4. SUMMARY

A newly developed double-stream gas puff laser target approach for efficient generation of soft X-ray and EUV radiation without debris production is described in this paper. The target is formed by pulsed injection of high-Z gas (xenon, krypton, argon, etc.) into a hollow stream from a low-Z gas (hydrogen, helium). The outer stream of gas confines the inner stream of gas improving the gas puff target characteristics (higher gas density at larger distance from the nozzle output). The electromagnetic valve system equipped with a double-nozzle setup to form new gas puff targets is presented and characterized. The new valve system is very compact and easy for operation and maintenance.

This paper investigations was on the generation of soft X-ray and EUV radiation using a laser-irradiated double-stream gas puff target, performed in various laboratories were described. Different laser systems, including Nd:glass, Nd:YAG, KrF, and iodine lasers, were used to irradiate the gas puff targets. Soft X-ray and EUV emission was measured using various diagnostic instruments, including the transmission and reflection grating spectrometers, and the absolutely calibrated silicon photodiodes. Spectral, spatial, and temporal characteristics of emissions for different gases targets were presented. The sources should be useful for applications in X-ray microscopy, EUV lithography technologies, and micro-processing of materials however, their optimization is required.

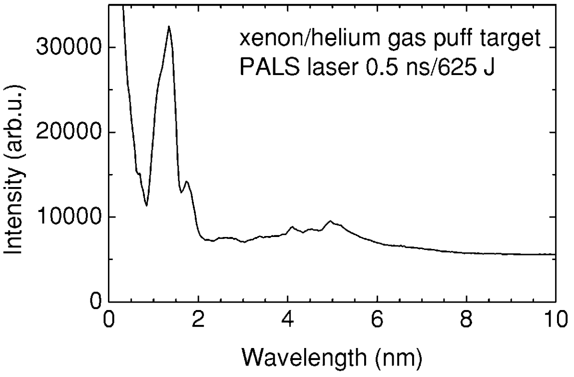

Fig. 10. Soft X-ray spectrum from the xenon/helium target measured with the transmission grating spectrograph.

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