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## Liquid-jet laser-plasma X-ray sources for microscopy and lithography

H.M. Hertz, M. Berglund, B.A.M. Hansson, O. Hemberg and G.J. Johansson

Biomedical and X-Ray Physics, Royal Institute of Technology, 10044 Stockholm, Sweden

**Abstract.** We review the development of compact laser-plasma soft x-ray sources based on microscopic liquid-drop or liquid-jet targets. It is shown that such sources provide practically debris-free, high-flux operation at water-window and EUV wavelengths. This regenerative and solid-density target system holds promise for the generation of high-average power using high-repetition-rate lasers. Application of the source to compact x-ray microscopy, multilayer-optics characterization and EUV lithography is briefly discussed.

### 1. INTRODUCTION

The scientific and industrial interest in methods requiring high-brightness soft x-ray and EUV sources is growing rapidly. High-resolution microscopy and narrow-line-width lithography are just two of many applications. Although the synchrotron radiation source provides both high brightness and high power many applications would benefit from compact and less expensive sources. Such instruments would greatly increase the accessibility to short-wavelength technology which is currently based on synchrotron radiation, thereby increasing its impact. Laser-plasma sources have the potential to fulfill this need.

Laser plasmas are attractive table-top soft x-ray sources due to their small size, high brightness, high spatial stability and, potentially, high repetition rate and high power. This source has been developed for microscopy [1,2,3], x-ray lithography [4], and EUV projection lithography [5]. With metal targets, conversion efficiencies of several tens of per cent may be reached with laser intensities of  $\sim 10^{12}$ - $10^{14}$  W/cm<sup>2</sup> [6]. However, with such conventional non-regenerative bulk targets or tape targets the operating time is limited, especially when high-repetition-rate lasers are employed. Furthermore, conventional targets produce debris which may destroy or coat sensitive x-ray components, such as zone plates or multilayer optics, that are positioned close to the plasma. Basically, two target systems have been developed that show promise to avoid the problems: the gas/cluster-jet target [5], and the liquid-droplet [7] or liquid-jet target [8].

In the present paper we review the use of microscopic liquid droplets and liquid jets as target for table-top laser-plasma x-ray generation. This regenerative target type provides fresh target material at liquid density for full-day operation without interrupts. It allows high-repetition-rate lasers to be used, thereby having potential for high average power. Furthermore, it reduces debris production several orders of magnitude compared to conventional targets. For certain liquids debris is practically eliminated. Thus, the effective photon flux may be increased a few orders of magnitude compared to conventional targets since smaller source-component distances or effective x-ray/EUV collection optics can be employed. By choosing a suitable target liquid, the emission wavelength may be spectrally tailored to suit, e.g., x-ray microscopy or EUV lithography. In addition, it allows nearly  $4\pi$  steradian geometric access.

2. EXPERIMENTAL ARRANGEMENT

2.1. Background

The generic arrangement for laser-plasma x-ray generation using continuous liquid jets as target is shown in Fig 1. When a liquid is forced through a nozzle, a liquid jet is formed. The jet eventually breaks up into droplets. This is due to the liquid's tendency to minimize surface energy. The break-up is spontaneous and normally stochastic since it is initiated by microscopic random perturbations in the surface of the liquid jet. The hydrodynamics of such liquid jets have been described in several publications [9,17].

Two methods can be applied. In Fig. 1a the laser is focused onto the liquid jet before it breaks up into droplets (liquid-jet method) [8] while in Fig. 1b the droplets are used as target (liquid-droplet method) [7] The applicability of the two target methods is discussed in Sects. 2.2 and 2.3.

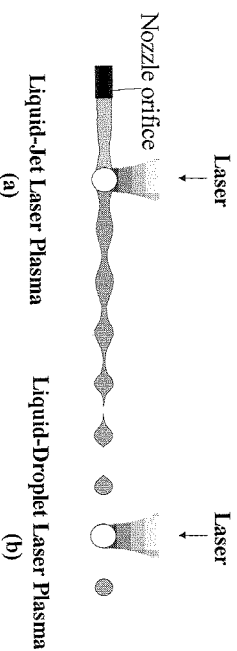


Fig. 1. Generic arrangement for a) liquid-jet target and b) droplet-target laser-plasma x-ray and EUV generation

The experimental arrangement (cf. Fig. 2) is similar for both methods. An approximately 10 μm diam. liquid jet is formed inside an ~10<sup>-4</sup> mbar pressure vacuum chamber by forcing the target liquid through a small glass capillary nozzle. In liquid-droplet-target experiments the nozzle is piezo-electrically vibrated in order to achieve stable droplet formation. The laser is focused on the jet or droplets with a focal spot diameter close to the diameter of the target. Due to the small target and focus size, the laser pulse energy can be kept at reasonable levels while still achieving sufficient intensity in the focused spot to allow x-ray and EUV generation. For this reason the stability of the drop- or jet-generation method is very important. Furthermore, debris emission is significantly reduced with this microscopic target type

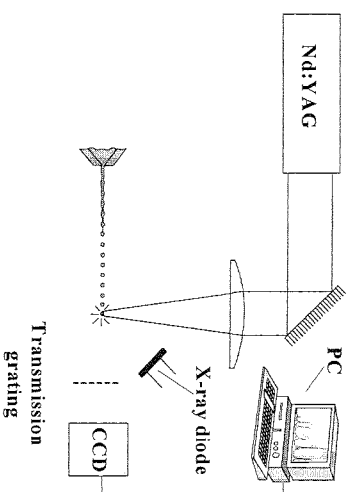


Fig. 2. Experimental arrangement for droplet-target laser plasma generation

The emitted spectrum is typically characterized by a free-standing transmission grating (10000  $\ell$ /mm) in combination with a back-illuminated CCD detector [10]. The arrangement is calibrated to allow absolute photon number determinations. High-resolution spectral measurements have been performed with an off-axis elliptical zone-plate spectrometer [11].

2.2 Droplet-target laser plasma

Liquid-droplet-target laser-plasma generation has been discussed in several publications referenced below. This target type is suitable for liquids with normal surface tension, resulting in drop formation a few mm from the nozzle. We have performed experiments on ethanol [7] water [9,12] ammonium hydroxide [13] fluoro carbon [14] and solutions [13]. Employing the liquid-jet method on such liquids may require a short nozzle-plasma distance, which, in turn, result in a risk of nozzle damage.

In a typical experiment, the beam from a pulsed, frequency-doubled, 10 Hz, 100 ps, 70 mJ/pulse SHG Nd:YAG-laser is focused to a spot diameter of ~15 μm. The ~15 μm diam. liquid droplets are generated by a ~1 MHz piezo-electrically vibrated capillary nozzle. The laser is synchronized with the piezo-electric vibration, thereby allowing each laser pulse to hit a single droplet. The resulting source diameter (FWHM) has been determined to ~15 μm [15]. The debris emission is reduced several orders of magnitude compared to conventional solid targets[16] and is completely eliminated for certain liquids [13] Improved flux may be obtained with a prepulse technique [15].

Figure 3 shows the water-window spectrum from ethanol. Note that the radiation is dominated by hydrogen and helium-like ion emission in the water window. The flux is typically  $0.3\text{-}1 \times 10^{12}$  photons/(sr×line×pulse). Given the narrow bandwidth ( $\lambda/\Delta\lambda \approx 700$ ) [11] these sources are suitable for zone-plate-based water-window x-ray microscopy (cf. Sect. 3.1). Figure 4 shows the spectrum from a water-droplet laser plasma in the EUV wavelength range. Here a frequency-doubled 750 mJ, 8 ns Nd:YAG laser was used to produce oxygen ion emission.

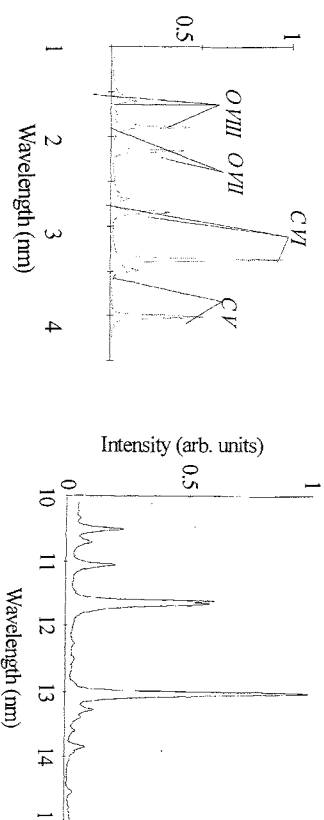


Fig. 3. Emission spectra from ethanol laser plasma.

Fig. 4. Oxygen ion EUV emission from water/methanol liquid-droplet target.

We have recently made a thorough investigation of the long-term stability of the droplet-target source using a high-speed laser-diode imaging system [17]. Rapid drop-to-drop fluctuations are below 1 micron and therefore sufficiently small for stable operation. However, a slow drift creates long-term fluctuations. This drift has been shown to arise from an evaporation-induced temperature change affecting the viscosity and, thus, the drop speed and position. The experimental results are explained quantitatively with a model based on the Hagen-Poiseuille's equation. The long-term drift is

compensated for by a new phase delay drop-to-laser synchronizing system. With this system highly stable unattended long-term operation is demonstrated. Figure 5 shows the droplet-target laser-plasma with and without the stabilization system.

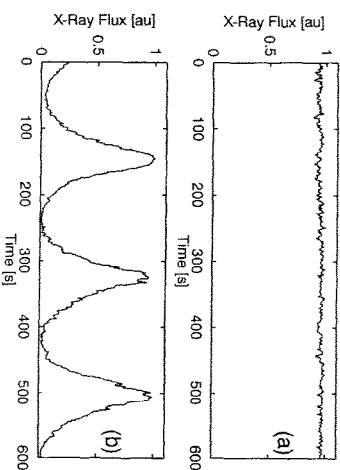


Fig. 5. Droplet-target laser-plasma x-ray flux as a function of time with (a) and without (b) the automatic control system

### 2.3. Liquid-jet target laser plasma

As mentioned above, there are several suitable target liquids that do not have sufficiently high surface tension to produce droplets close to the nozzle. Furthermore, some liquids exhibit unstable drop-formation. For these liquids, the laser-plasma may be generated in the liquid-jet instead of in the droplets. This system was first introduced on the fluorocarbon target in order to avoid a weak instability [8]. It is interesting to note that the liquid-jet configuration has the same advantages as the droplet configuration: very low debris, regenerative target for high-repetition-rate laser operation, and large geometrical access.

More important, however, is that the liquid-jet target concept allows liquefied gases at cryogenic temperatures to be used as target compounds. The hydrodynamic properties of such liquids typically do not allow laser-plasma formation in droplets. This was first demonstrated on liquid nitrogen (77 K) [18]. A nitrogen liquid jet was generated by forcing liquid nitrogen through a 5  $\mu\text{m}$  diameter metal orifice. Using the 70 ml, 100 ps frequency-doubled Nd:YAG laser, laser plasmas were produced. The spectrum is shown in Fig. 6. The flux of the helium-like nitrogen line at  $\lambda=2.88$  nm could be determined to  $\sim 5 \cdot 10^{11}$  photons/(sr $\times$ line $\times$ pulse). The source is suitable for microscopy.

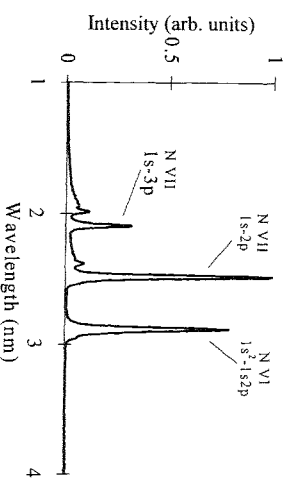


Fig. 6. Emission spectrum from laser-plasma generation in a liquid nitrogen liquid jet.

The basic scheme of the liquid nitrogen source may be applied to several different liquefied gases in order to reach new wavelength ranges, e.g., oxygen, argon, xenon, and krypton. Especially xenon has attracted recent interest since its spectral properties match the needs of EUV projection lithography. This is further discussed in Sect. 3.2.

## 3. APPLICATIONS

### 3.1 Water-window x-ray microscopy

X-ray microscopy in the water-window region ( $\lambda=2.3\text{-}4.4$  nm) is an attractive technique for high-resolution biological imaging due to the possibility to study thick unstained objects in an aqueous environment [2]. Current operational x-ray microscopes are based on synchrotron radiation sources, which limit their accessibility. The development of compact x-ray microscopy would significantly increase the applicability of the technology. We have developed the first compact sub-visible-resolution x-ray microscope [19].

The microscope is based on the liquid-droplet laser-plasma source using ethanol as target and a 100 Hz Nd:YAG laser. This high-brightness source has a sufficiently narrow line width (typically  $\lambda/\Delta\lambda \approx 700$ ) [11] to allow high-resolution imaging with zone plates. However, since the source radiates into  $4\pi$  ster., it has to be combined with high-efficiency condenser optics in order to obtain reasonable exposure times. For this purpose we have developed a new condenser arrangement based on spherical W/B<sub>4</sub>C normal-incidence multilayer mirror [20]. This arrangement provides large collection efficiency, easy alignment, proper match of the numerical aperture to the zone-plate optics, and single-line ( $\lambda=3.37$  nm) illumination of the sample from the multi-line laser-plasma source. The imaging is performed with a 7.3% efficient ( $@ \lambda=3.37$  nm) Ni zone plate with outmost zone width of 30 nm. A back-illuminated CCD records the magnified image.

With this arrangement we have recorded good-contrast images of dry test objects and diatoms (cf. Fig. 7) showing sub-60 nm feature sizes [19]. Figure 8 shows a COS-7 cell, where the fibrous structure is interpreted as actin fibers [21]. Both images were recorded with approx. 2 minutes exposure time.

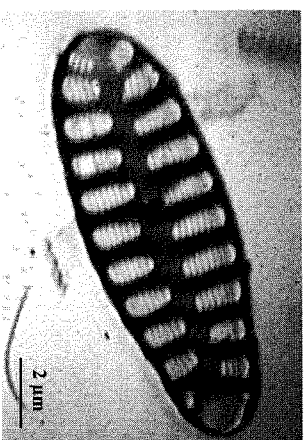


Fig. 7. Compact x-ray microscopy of a diatom

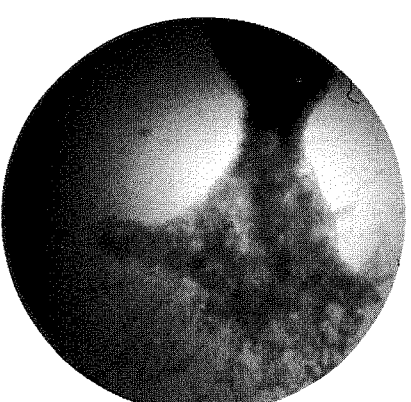


Fig. 8. Compact x-ray microscopy of COS-7 cell (image diameter 20  $\mu\text{m}$ )

Currently the multilayer condenser optic is the most important limiting factor to decrease the exposure time. In order to improve such optics we have developed a compact water-window reflectometer, which is also based on the line-emitting droplet-target laser plasma source [22]. A narrow beam of x-ray radiation is incident on the multilayer under test. The multilayer is rotatable over 90 degrees and the reflected radiation is measured by a detector rotating twice the angle of the sample. This fully automatic instrument allows accurate measurements of d-spacing and reflectivity, the two most important parameters for improved condenser optics. Calculations indicate that with realistic improvements in reflectivity and flare our compact x-ray microscope arrangement has the potential to perform imaging with exposure times only a few times longer than those of bending-magnet-based microscopes.

### 3.2 Source for EUV projection lithography

EUV projection lithography at  $\lambda=13.5$  nm is currently the strongest candidate to succeed deep-ultraviolet lithography for large-scale integrated circuit fabrication [23]. Although the synchrotron is a viable source for EUV lithography, the technology would benefit from a compact source, provided it produces sufficiently high EUV power (50-150 W inband collectable). The laser plasma has potential to scale to these power levels. Other compact sources under development include z-pinch, capillary discharge, and plasma focus [24].

Laser-plasma emission from xenon provides a spectrum suitable for EUV lithography. This was first demonstrated on gas-puff and gas-jet targets. We have developed a liquid-xenon-jet target [25]. In brief, it uses a jet of cryogenic liquid xenon as target for laser-plasma generation. The liquid xenon is formed by forcing xenon gas under 10-50 bars of pressure into a small reservoir cooled to 170-200 K. A microscopic glass capillary nozzle (diam. approx. 10  $\mu\text{m}$ ) is attached to the reservoir, producing a liquid jet of xenon in a vacuum chamber. The plasma is produced by a frequency-doubled 100 Hz Nd:YAG laser with 3 ns pulse width. The spectra are recorded by a free-standing 10000  $\ell/\text{mm}$  transmission grating and a CCD detector. Figure 9 shows a typical spectra.

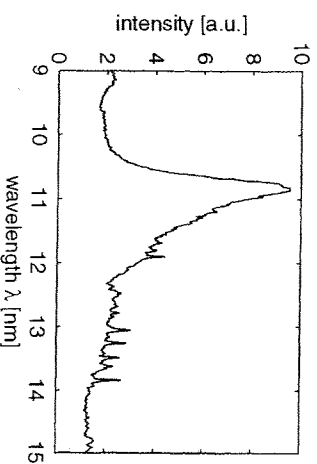


Fig. 9. Emission spectra from a liquid-jet xenon plasma.

The liquid-jet-target concept has earlier proven to be a low-emission target and its combination with xenon, an inert gas, promises good debris characteristics. In addition, the liquid-jet arrangement has the inherent advantage of allowing plasma generation at distances typically larger than 5 mm from the nozzle orifice, still providing a high-density target. Such large nozzle-plasma distance limits the debris problem due to nozzle erosion as well as thermal problems when high-power lasers are used.

The main obstacle towards the practical use of this source for EUV projection lithography system has previously been a stochastic directional instability of the xenon jet. We have recently found a remedy to this problem and we now develop this source towards high-average-power generation of EUV radiation.

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## Efficient soft X-ray generation from femtosecond-laser-produced plasma and its application to time resolved spectroscopy

N. Uesugi<sup>a</sup>, H. Nakano, T. Nishikawa and P. Lu<sup>b</sup>

*NTT Basic Research Laboratories, 3-1 Wakamiya, Morinosato Atsugi-shi, Kanagawa 243-0198, Japan*

**Abstract.** The experimental results on efficient soft x-ray generation from fs-laser-produced plasma and the application of short x-ray pulse to time-resolved spectroscopy are presented. Soft x-ray generation properties were evaluated for both flat Al targets and nanohole – alumina targets. The experimental results for flat metal targets revealed the fundamental properties of soft x-ray emission such as broadband continuum spectra and short pulse duration. By adopting nano-structured targets, a more than 30-fold enhancement of x-ray generation yield is achieved compared with that for flat targets of the same materials with a slight increase of pulse duration. The time-resolved measurement of the inner-shell absorption change of Si during the irradiation with a high-intensity fs laser pulse is achieved by using a short soft x-ray pulse as a probe pulse in pump-probe experiments for the first time. A more than 10% increase in the absorption of Si membrane at the  $L_{\text{min}}$  edge (around 100eV) was observed. The recovery time of the absorption change was measured to be about 20ps. This absorption change is assumed to be the bandgap renormalization of Si.

### 1. INTRODUCTION

The development of high-power ultrafast laser technologies opened up a new study field on high-intensity laser-matter interactions. In this research field, x-ray generation from high-density, femtosecond(fs) laser-produced plasmas are of great interest[1,2]. Although x-rays from these sources are incoherent, the high-brightness properties are very attractive to apply its to spectroscopy. Especially, the ultrashort x-ray pulses from fs-laser-produced plasmas are synchronized with the incident laser pulse, thus it is expected that the ultrafast response of optically excited materials can be observed by applying x-rays from fs-laser-produced plasmas to pump-probe type measurements. So far, time-resolved diffraction[3,4] and absorption measurements[5-7] using fs-laser-produced plasma x-rays are demonstrated. Pump-probe absorption spectroscopy using short x-ray pulses is expected to reveal the dynamics of electrons in the inner shells of optically excited atoms.

In this paper, we will review x-ray emission properties from fs-laser-produced plasmas and our recent experimental results on the enhancement of soft x-ray emissions[8-15]. Using nano-structured targets, we achieved more than a 30-fold enhancement in soft x-ray emission while keeping pulse duration less than 20 ps. Time-resolved absorption measurements in the soft x-ray region by means of pump-probe spectroscopy are demonstrated[7]. We observed a rapid

<sup>a</sup> Present address: Tohoku Institute of Technology, 35-1 Yagiyama Kasumicho Taihaku-ku, Sendai 982-8577, Japan.

<sup>b</sup> Present address: Advanced Photon Research Center, Kansai Research Establishment, Japan Atomic Research Institute, 8-1-1-2 Umemidai, Kizuchi, Souzaku-gun, Kyoto-fu 619-0215, Japan.