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Towards Compact X-Ray Microscopy with Liquid-Nitrogen-Jet Laser-Plasma Source

Per A. C. Takman*, Ulrich Vogt and Hans M. Hertz

[1]*Biomedical and X-ray Physics, Royal Institute of Technology/Albanova, SE-106 91 Stockholm, Sweden*

In this contribution we present source properties and design considerations for a compact x-ray microscope operating in the water-window spectral region at 2.48 nm. The microscope will use a liquid-nitrogen-jet laser-plasma source with sufficient brightness, uniformity, stability and reliability for microscopy operation. The source is quantitatively characterized by calibrated slit-grating spectroscopy and zone-plate imaging to determine absolute photon numbers and source size and stability. Calculations including sources parameters as well as characteristics of available x-ray optics indicate that high-quality microscope images can be obtained with exposure times in the range of few minutes.

KEYWORDS: X-ray microscopy, laser-plasma x-ray source

1. Introduction

Soft x-ray microscopy in the water-window (λ = 2.3-4.4 nm) exploits the natural contrast between carbon and oxygen for high-resolution imaging of, e.g., biological samples 1). Present compact soft x-ray microscopes operate at λ = 3.37 nm using a methanol or ethanol liquid-jet laser-plasma source 2). However, imaging of thicker (~10 μm) objects requires operation in the lower part of the water-window where the transmission is higher. In the present paper we present the path towards a compact microscope operating at λ = 2.48 nm with a liquid-nitrogen-jet laser-plasma source.

Liquid-jet laser plasmas 3),4) are attractive, compact, high brightness sources for x-ray and extreme ultraviolet (EUV) radiation. They offer reliable, spatially well defined, regenerative sources allowing high-repetition-rate operation with minimum debris emission 5). By choosing a suitable target liquid and correct plasma conditions, the emission wavelength may be spectrally tailored to suit the desired application. Liquid nitrogen is an attractive target for soft x-ray microscopy in the lower part of the water-window since hydrogen-like and helium-like nitrogen ions emit strongly at λ = 2.478 nm (Ly α) and λ = 2.879 nm (He α). Additionaly, nitrogen benefits from being relatively inert, thereby minimizing damage of and deposition on sensitive x-ray optics due to debris.

We are presently developing a compact soft x-ray microscope for operation at λ = 2.48 nm using a liquid-nitrogen-jet laser-plasma source. This microscope will be based on a zone-plate condenser 6) since present normal-incidence multilayer mirrors for this wavelength neither have the required reflectivity nor the necessary uniformity. We investigated the liquid-nitrogen-jet laser-plasma source and its applicability to x-ray microscopy 7). We found that the temporal and spatial stability as well as the reliability is sufficient for x-ray microscopy use. Calculations using measured source parameters 7) and x-ray optics characteristics 6),8) show that exposure times of few minutes will result in high-quality x-ray microscope images.

2. Source characterization

The liquid-nitrogen target delivery system employs a fused silica capillary nozzle. Up to 60 bars of nitrogen pressure is applied to the capillary, which is lead into a vacuum chamber via a liquid-nitrogen-filled cryostat, providing on-line cooling and liquefaction of the applied nitrogen gas. The resulting liquid-nitrogen jet (18 μm diameter) shows very stable operation with a remaining angular instability of < 1 mrad. Furthermore, the system routinely allows long-term operation without interrupts.

The plasma is generated by a pulsed, 100 Hz, ~3 ns, frequency-doubled Nd:YAG laser (Coherent Infinity 40-100). The beam is focused to the continuous part of the jet giving a laser full-width-at-half-maximum (FWHM) diameter of ~15 μm in the focus. Pulse energies up to ~200 mJ, corresponding to focal intensities of $\sim 4\times10^{13}$ W/cm^2, can be achieved at the liquid-nitrogen jet target.

Emission spectra and quantitative flux measurements were performed with a slit-grating spectrograph 9) in combination with an x-ray-sensitive CCD detector (Photometrics CH350 with SITe SI003AB chip). Absolute calibration of the spectrograph components and CCD efficiency was performed at the ELSA synchrotron radiation facility in Bonn. Based on the calibration process, we determine that the systematic error in the spectral flux measurements performed with the system is less than 30%. Figure 1 shows a quantitative laser-plasma emission spectrum. A total flux of $\sim 1.0\times10^{12}$ photons/(pulse×sr×line) in each of the two emission lines of nitrogen was obtained with 200 mJ/pulse laser energy. This corresponds to a conversion efficiency of ~0.5% assuming 4π sr isotropic emission. The measured flux compares well with previous work 10)-12).

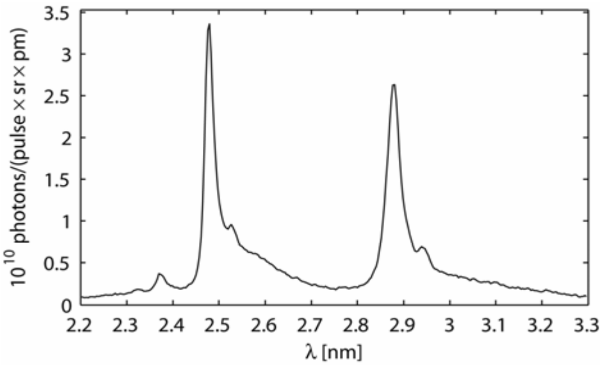

Fig. 1: Liquid-nitrogen-jet laser-plasma emission spectrum for 200 mJ laser-pulse energy.

Source size, emission distribution, brightness and stability were determined with a zone-plate imaging arrangement [13]. A calibrated 5-mm diameter condenser zone plate (CZP) [6] is combined with a central stop to form a magnified image of the source on the CCD detector. The systematic error in the brightness measurements is estimated to be less than 40%. Figure 2 shows the shape of a typical single-shot image at λ = 2.48 nm using a laser pulse energy of 200 mJ. After dark image subtraction, each plasma image was analyzed by two line plots in the horizontal and vertical directions through the pixel with maximum intensity. The full width at half maximum was used as a measure of the source size. For both nitrogen emission lines the source diameter was ~21±2 μm at 200 mJ laser pulse energy. For the determination of the spatial stability, the position of the pixel with maximum intensity was used to calculate the shot-to-shot variation of the plasma position. It was found that the spatial stability for both spectral lines was ±2 μm (1σ) horizontally, and ±2 μm (1σ) vertically.

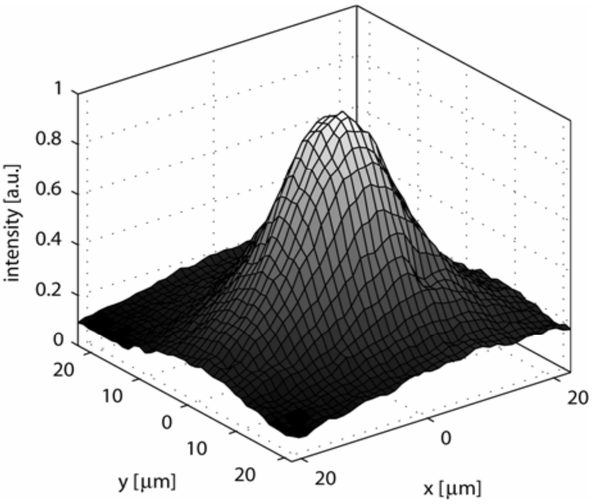

Fig. 2: Source shape at λ = 2.48 nm using a laser pulse energy of 200 mJ [7].

3. Calculations on x-ray microcope performance

The future compact soft x-ray microscope will employ a condenser zone plate (CZP) [6] to illuminate the object in critical illumination via 1:1 imaging of the source into the object plane. A hollow cone illumination of the CZP/central-field-stop arrangement in combination with the micro-zone plate imaging and CCD detector results in a field of view ~12 μm when operating the microscope at 1000× magnification. We therefore calculate the expected illumination in the object plane by the average source brightness and the uniformity within this field of view. The measured source parameters at λ = 2.48 nm using a 200 mJ laser pulse give an average brightness of 4.1×10^8 photons/(pulse×sr×μm^2×line) with a uniformity of 19%. Taking the efficiency and the collection angle of the CZP into account, this corresponds to an average intensity of 1.0×10^5 photons/(s×μm^2) in the object plane. Assuming a nickel micro zone plate with 7.6% efficiency operating at ×1000 magnification and a CCD pixel size of 24 μm, this brightness enables recording of high-quality images (signal-to-noise ratio > 14) of dry samples with an exposure time below 3 minutes. For a biological sample sustained in a 10 μm thick water layer, the exposure time will increase to 9 minutes.

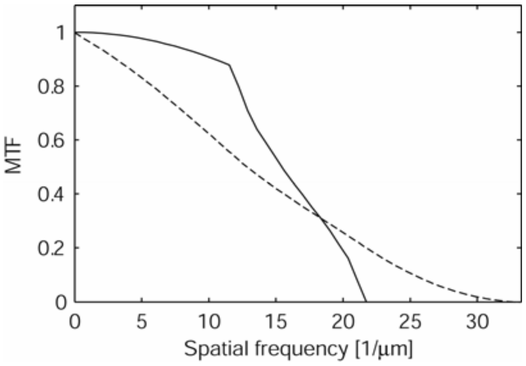

Fig. 3: Calculated apparent modulation transfer function of the x-ray microscope for a sinusoidal amplitude grating. Solid line: present condenser and objective zone plate. Dashed line: numerical aperture of condenser matched to the objective zone plate.

Additionally, the apparent modulation transfer function at ×1000 magnification for a sinusoidal amplitude grating test object is calculated [14] using the condenser and the micro zone plate parameters. The result is depicted by the solid line in Fig. 3. The expected resolution is in the range of 50 nm period, corresponding to 21 lines/μm. It is limited by the mismatch of the numerical aperture of the present condenser (60 nm outermost zone width) and the micro zone plate objective (30 nm outermost zone). For the same objective zone plate with a matched condenser the modulation transfer function is given in Fig. 3 for comparison (dashed line), resulting in a resolution given by the outermost zone width of the objective.

4. Conclusions

In summary, we have presented our work towards a compact soft x-ray microscope operating in the lower part of the water window at λ = 2.48 nm. The microscope will utilize a liquid-nitrogen-jet laser-plasma x-ray source and diffractive zone plate optics for condenser and objective. We expect exposure times in the range of few minutes for high-quality images.

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[*] e-mail: per.takman@biox.kth.se