Capillary nozzles for liquid-jet laser-plasma x-ray sources

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(Received 8 April 2003; accepted 29 April 2003)

We describe a method to fabricate tapered glass nozzles suitable for liquid-jet-target generation in laser-plasma soft x-ray and extreme ultraviolet sources. In the method, a tapered nozzle is formed as an integral part of a flexible capillary glass tubing. The method makes use of inert materials, extending the possible choice of target liquids compared to current nozzles. It also provides flexibility as regards nozzle diameter and pressure, thereby allowing optimization of the target size and extending the range of applicability for the liquid-jet-target laser plasmas. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1589156]

Liquid-jet laser plasmas are becoming increasingly important as compact x-ray sources since they provide a high-density regenerative target in combination with negligible-debris operation.[1,2] To date, commercial ink-jet-tapered glass capillary nozzles are often used to produce the liquid-jet target, resulting in limited flexibility in the choice of jet dimensions and liquids. In addition, metal orifices are used, making the nozzle assembly at the orifice more complicated.[3,4] In the present Note, we describe a glass nozzle fabrication method that increases the flexibility in the choice of jet parameter and provides uncomplicated formation of stable liquid-jet targets.

Laser-plasma x-ray sources based on microscopic liquid jets feature high flux and brightness, negligible debris, and allow long-term operation without interrupts. Spectrally tailored emission for a specific application can be produced by selecting a target liquid with proper elemental contents. This includes, e.g., hard x-ray generation[5] and narrow-bandwidth water-window emission[1,3] appropriate for zone plate optics in x-ray microscopy.[6] Other applications are reflectometry[7] and extreme ultraviolet lithography.[8,9] Depending on the hydrodynamic properties of the liquid, the source may be operated either in the liquid-jet mode[2] or the droplet mode,[1] i.e., the target for the laser plasma is either the liquid jet itself or droplets formed by the jet. For applications, the liquid-jet mode is often preferable due to its higher stability and simpler triggering.

Unfortunately, the commercial ink-jet glass nozzles[10] limit the range of usable target liquids. Often organic and other materials in the nozzle assembly have shown to dissolve in the liquid, creating jet instabilities and clogging. This has been observed for different hydrocarbons, liquid N_2O, ammonium hydroxide, but also from long-term use of ethanol. Furthermore, these nozzles have a fixed diameter (approximately 10 μm), making it difficult to optimize the target geometry for maximum emission given the laser pulse parameters.[11] In addition, liquid-jet operation[2] is difficult for many common liquids (ethanol, water, etc.) since their high surface tension in combination with the nozzle geometry and jet speed results in a short distance to the drop-formation point (typically few mm). In the liquid-jet mode this is the maximum distance from the nozzle to the plasma, which, thus, may damage the nozzle orifice. To overcome the above limitations, we have developed a new nozzle fabrication method. It is based on small-diameter fused silica capillary tubing with an integrated nozzle and other inert materials. It provides increased flexibility as regards liquids, orifice geometry, and extends the applicability of liquid-jet mode to many common liquids by allowing an increase in the drop-formation distance. In addition, the method facilitates simple feedthrough into the vacuum chamber and allows operation at higher pressures. Next, we describe this fabrication method and its properties.

The starting point of the nozzle fabrication is an approximately 50 cm long synthetic fused silica capillary tubing with a polymide coating. The inner and outer diameters are typically 100 μm and 375 μm, respectively. This type of capillary tubing is used in capillary electrophoresis and has shown to be sufficiently clean for our fabrication process. The capillary tubing is connected to a metal inline filter (0.5 μm) with help of standard high performance liquid chromatography components. These components are made of polyetheretherketon (PEEK), a material compatible with the most common solvents except for some strong acids, like concentrated nitric and sulfuric acid. The components also extend the range of operating pressure compared to the previously customary 50 bars. The small diameter of the fused silica tubing allows for operation up to several 1000 bars while the other components are specified up to 350 bars.

The taper is produced in a laser-based micropipette-pulling machine (Sutter Instrum. P-2000). Before pulling, approximately 2 cm of the polymide coating is removed by heating. The area without the polymide coating is mounted in the laser focus and the capillary is pulled to a taper. The taper typically has an outer diameter of 5 μm after the pull. The geometry of the taper can be varied by adjusting the pulling parameters. According to Ref. 12, the taper angle is not critical for a stable liquid jet as long as it varies between 15° and 90°. We haven chosen a taper angle of ~20°, since a slow taper allows better control of the nozzle diameter during the following polishing process. After the pulling process is

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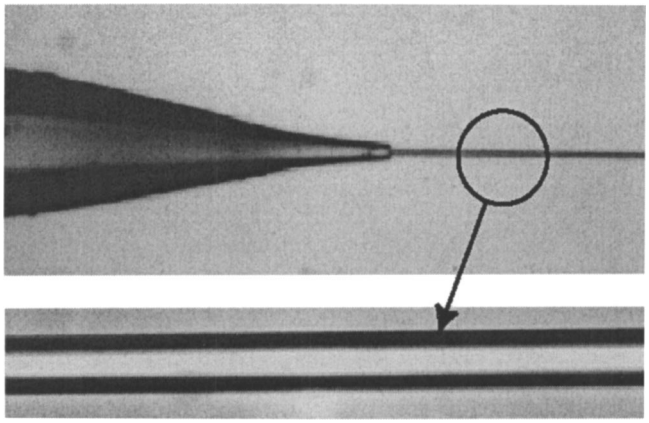

FIG. 1. Photograph of tapered nozzle with a 13 μm diameter ethanol jet.

completed, the nozzle is polished at right angles to the capillary with diamond lapping film in flowing water down to the required inner diameter. Jet diameters from 7 μm up to 50 μm have successfully been fabricated with an accuracy of ± 2 μm.

Figure 1 shows a photograph of a 13 μm diameter liquid ethanol jet produced by the novel nozzles. It is noted that the jet is free of swirls, which is important for the stability. Determination of the stability is, however, best performed by measuring the x-ray flux from a laser-plasma experiment with the jet as the target. The liquid jet is then mounted in a 10^{-3} mbar vacuum chamber. In the present experiment the laser plasma is generated when 65 mJ, $\lambda = 532$ nm, and 3 ns pulses from a 100 Hz Nd: YAG laser (Coherent Infinity) are focused onto the target jet with a 50 mm focal length lens. The emitted x rays are detected with a filtered (200 nm Ti on 200 nm SiN and air) x-ray diode resulting in detection of carbon-ion water-window x-ray flux. More details about the basic experimental arrangement can be found in Refs. 1 and 2. Figure 2 shows the emitted x-ray flux for an ethanol jet with a diameter of 8 μm when operating 2 mm from the nozzle tip. The ethanol pressure is typically 100 bars and the jet speed is approximately 80 m/s. Each data point represents a moving average over 1 s (100 pulses). The standard deviation in the measured x-ray flux is 2%. Similar results are obtained for larger-diameter jets, although the noise in the x-ray flux is slightly larger.

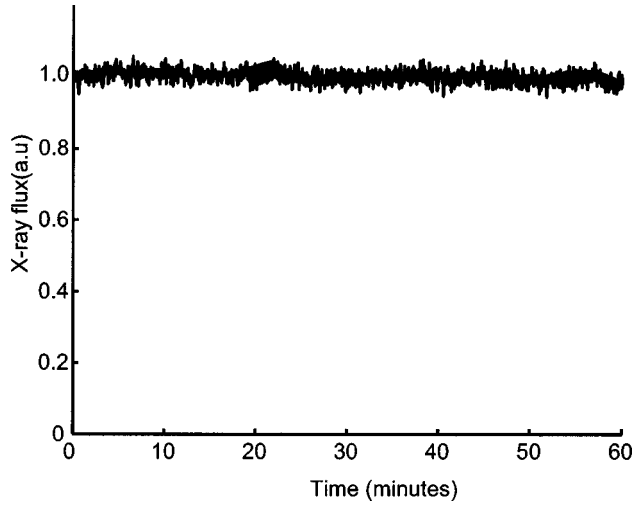

FIG. 2. Stability of water-window x-ray emission from laser-plasma operation of a 8 μm diameter ethanol-jet target formed by a tapered capillary nozzle.

The aforementioned nozzle fabrication method has several advantages compared to the previously used nozzles. The use of fused silica and PEEK makes the integrated nozzle assembly chemically inert for most common chemicals, thereby extending the range of target liquids. The long capillary facilitates simple feedthrough into the vacuum chamber. Furthermore, the pressure range is improved and the fabrication of the integrated nozzle has sufficient control of nozzle size and geometry. Thus, we may operate at higher velocities and at larger diameters than before, making it possible to extend the applicability of the liquid-jet mode also to high-surface tension liquids. The distance to the drop-formation point (L) for such liquids is basically given by $L = C \cdot \nu \cdot \sqrt{\rho \cdot d^3/\sigma}$, while the Reynolds number $\mathrm{Re} = \rho \cdot \nu \cdot d/\eta$ and the Ohnesorge number $\mathrm{Oh} = \eta/\sqrt{\sigma \cdot \rho \cdot d}$ set an upper limit for laminar and nonspraying flow,[13] indicating the importance of accurate control of speed ν and diameter d. Here ρ is the density, η is the viscosity, σ is the surface tension, and C is on the order of 10. Preliminary experiments with a $v = 100$ m/s and $d = 30$ μm methanol jet ($\mathrm{Re} \approx 3.9 \times 10^3$ and $\mathrm{Oh} \approx 0.026$) show a drop-formation distance of approximately 3 cm, in good agreement with theoretical calculations. This paves the way for high-average power water-window liquid-jet operation without nozzle damage. Furthermore, the nozzles will be applied to maximize flux of a water-window source for microscopy by matching the jet diameter to the laser pulse temporal width.[11] Finally, this nozzle design can be adapted relatively easily to cryogenic liquids (liquid nitrogen, xenon, and argon) by online cooling of the fused silica capillary.

The authors gratefully acknowledge the discussions with Per Jansson and the support from the Swedish Research Council.

[1] L. Rymell and H. M. Hertz, Opt. Commun. **103**, 105 (1993); L. Rymell, M. Berglund, and H. M. Hertz, Appl. Phys. Lett. **66**, 2625 (1995).
[2] L. Malmqvist, L. Rymell, M. Berglund, and H. M. Hertz, Rev. Sci. Instrum. **67**, 4150 (1996).
[3] M. Berglund, L. Rymell, H. M. Hertz, and T. Wilhein, Rev. Sci. Instrum. **69**, 2361 (1998).
[4] M. Wieland, T. Wilhein, M. Faubel, C. Ellert, M. Schmidt, and O. Sublemontier, Appl. Phys. B: Lasers Opt. **72**, 591 (2001).
[5] R. J. Tompkins *et al.*, Rev. Sci. Instrum. **69**, 3113 (1998).
[6] M. Berglund, L. Rymell, M. Peuker, T. Wilhein, and H. M. Hertz, J. Microsc. **197**, 268 (2000).
[7] G. A. Johansson, M. Berglund, J. Birch, F. Eriksson, and H. M. Hertz, Rev. Sci. Instrum. **72**, 58 (2001).
[8] B. A. M. Hansson, L. Rymell, M. Berglund, and H. M. Hertz, Microelectron. Eng. **53**, 667 (2000).
[9] U. Vogt *et al.*, Appl. Phys. Lett. **79**, 2336 (2001).
[10] Siemens-Elema AB, Stockholm, Sweden.
[11] J. de Groot, O. Hemberg, A. Holmberg, and H. M. Hertz, J. Appl. Phys. (submitted).
[12] M. J. Mc Carthy and N. A. Molloy, Chem. Eng. (Rugby, U.K.) **7**, 1 (1974).
[13] A. H. Lefebvre, *Atomization and Sprays* (Hemisphere, New York, 1989).