Temperature measurements of liquid flat jets in vacuum

I. INTRODUCTION

X-ray absorption spectroscopy (XAS) is a powerful method for the investigation of fundamental electronic properties of matter. However, the use of XAS in the soft x-rays is experimentally challenging in bulk materials, as transmission limits the sample thickness to some hundreds of nanometers.1 This is especially an experimental challenge in vacuum, i.e., its temperature, remains mostly unknown. Alternative approaches utilizing fluorescence based XAS measurements can, due to geometrical effects and differences in absorption lengths, lead to artifacts in the measured absorbance spectra.1-3

For MeV electron diffraction experiments in liquids, ultra-thin homo-

geneous samples are required to minimize noise contributions from inelastic electron-scattering events. The small penetration depths of highly energetic electrons are <1 μm, and thin samples with around 800 nm thickness are necessary to avoid multiple-scattering events.4-6

Several methods have been developed to generate a sub-micron flat sheet in a vacuum environment, namely, two colliding cylindrical jets, 3D printed nozzles, and gas-dynamic jets.8-10 Earlier works on colliding jets have been performed under ambient conditions showing a thickness down to a μm.1-14 Recently reported thicknesses reach ~100 nm for both colliding jets and gas-dynamic jets.8-10,15 These sub-micrometer-thickness flatjets have enabled several novel achievements in science, such as investigating liquid samples using high-order harmonic spectroscopy,16,17 fs-transient XAS,18 angle-resolved photoelectron spectroscopy,19 and fs-electron scattering in liquids.20 Despite these advantages and applications, an essential property of flatjets in vacuum, i.e., its temperature, remains mostly unknown.

Liquid samples in vacuum experience fast evaporation that results in a temperature decrease potentially reaching down to the supercooled regime.21,22 For a majority of molecular systems, the temperature can have a vital impact on the properties and the evolution of the system. Ultrafast dynamics and structure of biological solutes dissolved in water and chemical reactions can be highly temperature sensitive.23,24

Although temperature characterization of supercooled water droplets and cylindrical jets have been conducted in previous studies22,23,27,28 using Raman spectroscopy, systematic investigation of liquid flatjets has not been performed yet.

In this work, we present measurements of the temperature profiles of liquid water (H2O) and ethanol (C2H5OH) in flatjets in
vacuum using Raman spectroscopy. The flatjets are generated using the two most common methods, gas-dynamic flatjets, and collision-based flatjets. A systematic investigation of the temperature dependence on the material vapor pressure, nozzle orifice size, flow rate, and initial temperature of the liquid sample is presented.

II. EXPERIMENTAL METHODS

We investigated two types of flatjets: (1) a collision-based (impingement) flatjet formed by the collision of two cylindrical liquid jets, and (2) a jet produced by a microfluidic nozzle, in which an initially cylindrical liquid jet is strongly compressed sideways by helium gas. These two configurations are the two most widely used setups for liquid-phase XAS measurements. The determination of the temperature profiles within these liquid jets is therefore of key importance for spectroscopic investigations in the x-ray domain.

In both setups, we used a high-pressure liquid chromatography (HPLC) pump to deliver the samples to the nozzles. The vacuum in the experimental chamber typically ranged between $5 \times 10^{-3}$ to $1 \times 10^{-2}$ millibar during the measurements. In order to keep acceptable vacuum conditions, the sample was captured in a cold trap at liquid-nitrogen temperature. The investigated samples were distilled water and absolute pure ethanol with at least 99.8% purity from Merck.

For the collision-based flatjet, two cylindrical liquid jets from two quartz nozzles of equal inner diameters (either 18 $\mu m$ or 60 $\mu m$) collided at an angle of 48$^\circ$ and generated a flatjet with a thickness that could be as thin as 500 nm, as assessed by white-light interferometry.

The thickness depends on several parameters, such as the orifice size of the nozzle, flow rate, type of liquid, and the measurement position. For the presented measurements, the flow rate was 2 ml/min for 18 $\mu m$ nozzles and 5 ml/min for 60 $\mu m$ nozzles, with a maximum jet velocity of 65.5 and 14.7 m/s, respectively.

For the gas-compressed flatjet, a microfluidic gas-dynamic chip nozzle from Micronit Microtechnologies BV with an orifice of 50 $\mu m$ was used. The focusing gas was helium and its pressure was adjusted using a regulator between 5 and 10 bars. The liquid sample flow rate was 1 ml/min.

A profile characterization of both flatjets is described in Refs. 8 and 10. While the collision-based flatjet reached a minimum of $\sim 3 \mu m$ with two 50 $\mu m$ nozzles, the gas-compressed flatjet exhibited a thickness down to $\sim 20 \mu m$. Later results with smaller nozzles orifices for collision-based flatjets reached $< 1 \mu m$ thickness.

Figure 1 illustrates our experimental scheme. As Raman excitation source, we used a Nd:YVO$_4$ cw laser (Coherent Verdi-V5) with a wavelength of 532 nm, a maximum power of 2 W and a spot size of $\sim 88.9 \mu m$ on the sample. Heating of the flatjet by this excitation laser was negligible as measurements performed at different pump powers showed negligible variations in flatjet temperature.

The Raman signal was collected at an angle of 90$^\circ$ with respect to the excitation beam by an objective lens within the liquid-jet chamber. The collimated signal was then sent out of the chamber via a window and focused by another lens onto the entrance slit of a flat-field spectograph (Princeton Instruments Acton SP23000). The resulting spectrum was recorded by an EMCCD camera (Andor iXon3).

For the calibration of the temperature measurement using Raman scattering, we used the flatjet under ambient conditions and simultaneously measured its temperature with a thermocouple. The sample, liquid water, was either cooled with a cold bath or heated with a warm bath and a heating device coupled to the nozzle holder, in order to get an absolute and accurate calibration curve.

The Raman spectrum of liquid water around 3400 cm$^{-1}$ mainly consists in a superposition of five contributions (at 3050, 3200, 3400, 3500, and 3650 cm$^{-1}$), which correspond to the fundamental O–H stretching bands, other vibrational modes, and Fermi resonances. The intensity of each component changes relatively to the total intensity in response to temperature changes. For instance, with decreasing temperature, the integrated intensities of the 3200 and 3400 cm$^{-1}$ bands increase and that of the 3500 cm$^{-1}$ band decreases.

To obtain the calibration curve, we normalized the recorded Raman spectrum at the midpoint 3300 cm$^{-1}$ and integrated the total band intensities above and below 3300 cm$^{-1}$. By plotting the ratio between these two integrated bands as a function of temperature, a calibration curve can be extracted by the expression:

$$\frac{1}{T} = C_1 \ln \left( \frac{I_{<3300}}{I_{>3300}} \right) + C_2,$$

where $T$ is the temperature, $I_{<3300}$ is the integrated intensity of the band below $\Delta \nu = 3300$ cm$^{-1}$, and $I_{>3300}$ above it. $C_1$ and $C_2$ are the empirical constants determined by calibration at known temperatures.
For the temperature measurements of ethanol, the anti-Stokes and Stokes signals could be recorded simultaneously so that the temperature was derived from the ratio of the two bands. A particularly well-suited mode is the CCO symmetric stretch at 888.8 cm\(^{-1}\). The temperature was thus extracted from the anti-Stokes/Stokes ratio,

\[
\frac{I_{\text{AS}}}{I_{\text{S}}} = \left(\frac{V_l + V_{\Delta}}{V_l - V_{\Delta}}\right)^3 \exp \left(\frac{-hV_{\Delta}}{kT}\right),
\]

where \(T\) is the temperature, \(k\) the Boltzmann constant, \(h\) the Planck constant, \(V_l\) the frequency of the laser, and \(V_{\Delta}\) the frequency of the CCO symmetric stretch mode (center frequency).

III. RESULTS AND DISCUSSION

Figure 2 shows the temperature profile along the collision-based flatjets. The origin is set at the collision position. For liquid water flowing from the 18 \(\mu\)m nozzles (blue curve), the maximum cooling of 25°C is obtained just before the end of the flat sheet, leading to a final temperature of \(\sim 2^\circ C\). Due to the small nozzle diameter, only one flat sheet of about 1 mm length is formed in this case.

For larger nozzle diameters (60 \(\mu\)m), the thickness of the flat sheet increases and the flow is more stable. This allows the formation of additional flat sheets, as shown in Fig. 2(a) (red and green curves and photograph). The first sheet is about 2 mm long and has a minimum temperature of \(\sim 8^\circ C\). As it collapses into a single jet, the
temperature increases because of water masses from the rim of the flat sheet mix with the central region. On the rim, the flatjet is thicker, less prone to evaporative cooling and warmer than the center. The second flat sheet is slightly shorter (about 1.5 mm), with a minimum temperature of \(-2^\circ\text{C}\). The overall cooling rate is thus lower for the 60 \(\mu\text{m}\) nozzles (10 K/mm or 1.75 \(\times\) 10\(^5\) K/s) than for the smaller 18 \(\mu\text{m}\) nozzles (28 K/mm or 1.7 \(\times\) 10\(^6\) K/s). This is consistent with the results from Ekimova et al., who estimated that evaporation yielded a mass loss of 5\% under conditions comparable to ours, so that the thickness of the second flat sheet is still thicker than the single flat sheet from the 18 \(\mu\text{m}\) nozzles. It has to be noticed, however, that the flow rates had to be adapted for obtaining stable jets for the 60 \(\mu\text{m}\) (5 ml/min) and for the 18 \(\mu\text{m}\) (2 ml/min) nozzles.

Interestingly, supercooled water (\(-2^\circ\text{C}\)) can thus be produced, when using collision-based flatjets with small nozzles. Supercooled water can, however, also be produced using 60 \(\mu\text{m}\) nozzles, if the water is previously cooled to around \(12^\circ\text{C}\) instead of room temperature, as shown by the green plot in Fig. 2(a). Moreover, the length of the cold region zone is extended over several mm.

As compared to water, ethanol experienced a much stronger cooling rate [Fig. 2(b)], due to its faster evaporation. Even with the 60 \(\mu\text{m}\) nozzles, an average cooling rate of \(~30\) K/mm \((4.0 \times 10^5\) K/s) is observed, which is \(3\) times faster than water under the same conditions. One sheet with about 2.4 mm longitudinal extension was formed and the lowest temperature found in this sheet reached \(-20^\circ\text{C}\).

For the gas-dynamic flatjet, as shown in Fig. 3, the measurements start at approximately 0.1 mm just below the nozzle. Note that the focusing gas starts compressing the liquid sample in the nozzle tip itself. By holding the flow rate constant at 1 ml/min and increasing the gas pressure, one observes an increase in the flatjet size up to 10 mm at which point the flatjet starts disintegrating. The overall flat sheet is also much shorter than for the collision-jet source, with a maximum length of about 1 mm. As shown in Table I, for gas pressures of \(6–9\) bar, the cooling rates reach as much as \(54.7, 51.9, 52.6,\) and \(58.1\) K/mm \((4.4 \times 10^5–4.9 \times 10^5\) K/s), respectively, and an initial exit temperature of \(21.5, 18.4, 14.7,\) and \(10.3^\circ\text{C}\), respectively. While the average cooling rate appears to have little-to-no dependence on the focusing gas pressure, the initial exit temperature is strongly dependent on it. This suggests that while the use of a focusing gas limits the effect of evaporative cooling, heat is still lost through conductive cooling between the gas and the liquid.

### IV. MODEL CALCULATIONS

To explain the thermal evolution of the colliding flatjet, we developed a basic model to fit our measurements. No modeling was performed on the gas-dynamic flatjet experiment, due to its complex geometry and some unknown geometrical parameters in this commercial device. To begin, we use the Hertz–Knudsen equation to describe the mass flux \(J_{\text{HK}}\) from a liquid surface, which is generally given in the form\(^\text{33,34}\)

\[
J_{\text{HK}} = \frac{1}{A} \frac{dN}{dt} = \frac{z_e P_g}{\sqrt{2 \pi m k_B T_{1L}}} \left( \frac{P_g}{T_{1L}} - \frac{P_V}{T_{1V}} \right),
\]

where \(A\) is the area, \(N\) is the number of molecules, \(k_B\) is the Boltzmann constant, \(T_{1L}/T_{1V}\) is the interfacial liquid/vapor temperature, \(m\) is the mass of a molecule, \(P_g\) is the saturation vapor pressure, \(P_V\) is the gas pressure, \(z_e\) is the evaporation coefficient, \(\sigma_e\) is the condensation coefficient, and \(z_s\) is the sticking coefficient of the gas onto a surface.

In addition, the change in temperature \(dT\) due to the phase transition (evaporative cooling) of \(dN\) molecules is given by

\[
dT = \frac{H_e}{N_A C_m V} dN,
\]

where \(H_e\) is the evaporation enthalpy, \(N_A\) is the Avogadro constant, \(C_m\) is the molar specific heat, \(M\) is the molar density, and \(V\) is the volume of the liquid.

Combining Eqs. (3) and (4) yields the following expression for the change in temperature per unit length of the liquid jet in the vertical \(z\) axis (i.e., cooling rate):

| Gas pressure (bar) | Flow rate (ml min\(^{-1}\)) | Exit temperature (\(^\circ\text{C}\)) | Avg. cooling rate (\(^\circ\text{C}\) ml min\(^{-1}\)) |
|-------------------|-----------------------------|-----------------------------|-------------------------------|
| 6.00              | 1.00                        | 21.5                        | 54.7                          |
| 7.00              | 1.00                        | 18.4                        | 51.9                          |
| 8.00              | 1.00                        | 14.7                        | 52.6                          |
| 9.00              | 1.00                        | 10.3                        | 58.1                          |

FIG. 3. Measurements with a microfluidic gas-dynamic chip nozzle. (a) Gas-dynamic flatjets generated with different focusing gas pressures from 5 to 10 bar of helium, at a constant flow rate of 1 ml/min. A lower liquid flow rate is required for operation compared to collision-based flatjets, therefore much smaller flatjets are formed. (b) Thermal profiles of the gas-dynamic flatjets, at an initial position defined as 0 mm just below the nozzle tip.
where \( l_1 \) is the thickness of the jet and \( v_z \) is the velocity of the jet along the \( z \) axis.

The thickness of the jet has been estimated by Hasson and Peck\(^{1,13}\) as

\[
l_1(z) = \Lambda \frac{d^2}{dz^2} \left[ \frac{\sin^2(\phi)}{1 + \cos(\theta) \cos(\phi)} \right] = \frac{l_0}{z}, \tag{6}
\]

where \( z \) is the distance from the collision point, \( d_c \) is the orifice diameter, \( 2d_c = 48^\circ \) is the collision angle, \( \theta = 0^\circ \) is the azimuthal angle, \( \Lambda \) is an unitless proportionality constant, and \( l_0 \) is the overall proportionality constant (\( m_0 \)) at fixed \( \phi \) and \( \theta \). From white-light interferometric measurements of the flat jet thickness, a value of \( \Lambda = 124 \) is obtained as the best fit. Equation (6) predicts a linear decrease in the jet thickness with the distance from the collision point. However, it has been observed in our and other experiments\(^{4,15,16}\) that not only is the boundary rim of the flat sheet substantially thicker than its center, the size of the flat sheet also varies with the flow rate of the liquid jet, which is not captured by Eq. (6). While more complex models that take into account these effects exist,\(^{36,37}\) we decided that for our measurements along the center of the sheet, Eq. (6) was reasonably applicable once minor corrections were made.

Although the Hertz–Knudsen (HK) model is perfectible,\(^{4,38}\) we used a simplified evaporation flux model \( J_f \), based on the HK equation, where the equilibrium/saturation vapor pressure \( P_v \), is given by the Clausius–Clapeyron equation,

\[
J_f = \frac{\gamma P_v}{\sqrt{2\pi m k_B T}},
\]

\[
= \frac{\gamma P_v}{\sqrt{2\pi m k_B T}} \exp \left[ -\frac{H_e}{k_B T} \left( \frac{T_{\text{ref}} - T}{T} \right) \right],
\]

\[
= \gamma J_0 \exp \left[ -\frac{H_e}{k_B T_{\text{ref}}} \left( \frac{T_{\text{ref}} - T}{T} \right) \right], \tag{7}
\]

where \( T_{\text{ref}} = 298.15 \) K is the reference temperature, \( P_v \) is the vapor pressure at \( T_{\text{ref}} \), \( \gamma \) is an unitless proportionality constant, and \( J_0 = P_v / \sqrt{2\pi m k_B} \). From Eq. (7), we define our final simplified fitting model by

\[
\frac{dT}{dz} = -\frac{2H_e}{N_A C_m M_l(z)} \frac{l_0}{v_z} F(z),
\]

\[
= -\frac{2\gamma}{N_A C_m M_l(z)} \frac{l_0}{v_z} \left[ \frac{H_e}{k_B T_{\text{ref}}} \exp \left[ -\frac{H_e}{k_B T_{\text{ref}}} \left( \frac{T_{\text{ref}} - T}{T} \right) \right] \right],
\]

\[
= \frac{2\gamma}{N_A C_m M_l(z)} \frac{l_0}{v_z} \left[ -\frac{H_e}{k_B T_{\text{ref}}} \exp \left[ -\frac{H_e}{k_B T_{\text{ref}}} \left( \frac{T_{\text{ref}} - T}{T} \right) \right] \right]. \tag{8a}
\]

where \( \gamma = 2\gamma_c \) is an overall proportionality constant with dimension \( m_0 \), with \( \epsilon \) a correction constant, and \( C \) is the cooling coefficient. \( F(z) \) is a correction function for the flatjet’s geometric properties. The fits to the experimental data obtained from numerical integration of Eq. 8(a) are presented in Fig. 4.

As listed in Table II, different values of \( \gamma \) and cooling coefficient \( C \) are given for the three different cases of water from 18 \( \mu \)m, water from 60 \( \mu \)m, and ethanol from 60 \( \mu \)m orifices. The ratio of the average experimental cooling-rate for different measurement sets can be approximately expressed as a ratio of cooling coefficients \( C / C \).

According to Eq. 8(c), although the cooling coefficient is not directly dependent on the nozzle-orifice diameter (given Eq. 6), it is inversely proportional to the jet velocity and therefore the flow rate, which is orifice-diameter dependent. Experimentally, flow rates of 2 and 5 \( \text{ml min}^{-1} \) were used for 18 and 60 \( \mu \)m orifices, respectively, with an experimental cooling-rate ratio of \( \sim 3 \), compared to \( c_{18 \mu m} / c_{60 \mu m} = 2.5 \). Given that the flatjets are only stable within a small flow-rate range for a given orifice size, and that their areas depend on the flow rate, it was experimentally difficult to observe a flow-rate dependence for a fixed orifice diameter. The cooling coefficient ratio for ethanol/water is \( c_{18 \mu m} / c_{60 \mu m} = 2.87 \), which is similar to the experimental cooling-rate ratio of \( \sim 2.9 \).

For the thermal evolution of the gas-dynamic flatjet, a dedicated model to the colliding jet would need to be developed. Such a model would need to take into account not just evaporative cooling, but also the strong likelihood of conductive cooling between the compression gas and sample liquid. This may be explored in a future study.

**FIG. 4.** Fitting of experimental data through numerical integration of Eq. 8(a). Blue: water with 18 \( \mu \)m nozzles. Red: water with 60 \( \mu \)m nozzles. Purple: ethanol with 60 \( \mu \)m nozzles.
From the measurements, it can be concluded that the focusing gas does not prevent the liquid sample from cooling. The use of the gas-compressed flatjet to reach the deeply supercooled regime of water is left for future studies.39

V. CONCLUSION

In conclusion, we have measured the thermal profiles of two different types of liquid flatjet systems in vacuum utilizing optical Raman spectroscopy and observed the temperature dependence on the material vapor pressure, orifice size, flow rate, and initial temperature of the liquid sample. Lower temperatures are accessible in water with the gas-compressed flatjet compared to the collision-based flatjet. Based on the results, we developed a simplified, empirical model to describe the effect of evaporative cooling on collision-based flatjets. In addition, we demonstrated that liquid water in the flatjets can enter the supercooled regime, which opens up future investigations of supercooled water using transient XAS, HHG spectroscopy, electron diffraction, attosecond spectroscopy, and many more. More generally, the measurements reported herein will facilitate the first systematic temperature-dependent studies of liquid-phase systems using these novel techniques.

SUPPLEMENTARY MATERIAL

See the supplementary material for the in-air characterization.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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