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Electron Diffraction from Liquids Jets in TEM

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Electron diffraction from liquid jets has been observed in a transmission electron microscope (TEM). As was shown by Gañán-Calvo [1], liquid jets may be produced by gas-dynamic forces exerted on the liquid by a coaxial flowing gas. Using this technique, we have reduced the diameter of a liquid jet by two orders of magnitude, producing a jet sufficiently small to be partially transparent to a high energy electron beam from a nozzle sufficiently large to prevent clogging.

The injector used to produce the jet consists of two concentric tubes, the inner supplying the liquid and the outer the gas. A sharpened fused silica tube of inner diameter typically 20–50 µm is used to supply the liquid. This tube is centered in a borosilicate capillary, the end of which has been shaped in a flame to produce a tapered exit. As pressurized gas flows through the outer capillary, the liquid is accelerated and the jet diameter is reduced. A more detailed description of these nozzles (see Figure 1) [2] can be found elsewhere [3].

The liquid tube shown in Figure 1 is recessed from the gas exit orifice but may also be made flush with or even protruding slightly from the gas aperture to reduce the possibility that liquid might contact the walls of the gas aperture. The liquid jet is then produced entirely in the gas expansion into vacuum. While this tends to produce larger jets, the jet diameter can be reduced to a few hundred nanometers by using an asymmetric liquid feed tube, e.g. one with a jagged edge or one that has been sharpened only on one side. Asymmetric or jagged edge tubes produce small jets by reducing the liquid cross-section while the liquid is still in contact with the tube. This allows the liquid to reach the critical velocity for jetting at lower flow rates.

A TEM image of a 350 nm diameter isopropyl alcohol jet is shown in Figure 2. This image was taken at 200keV on an FEI Tecnai with a specially designed environmental chamber. Diffraction patterns for water and alcohol were observed, each showing two rings. Figure 3 shows the inner water diffraction ring surrounded by a broader second ring and a single isopropyl ring, both recorded at the same camera length. Jet diameters were approximately 500 nm and 200 nm for water and alcohol, respectively.

The ability to penetrate a liquid jet with an electron beam opens many possibilities for further work. For example, two-liquid jets may be produced to study mixing or reaction at high temporal resolution by probing the jet at various distances along its direction of flow. It is our ultimate goal to study hydrated samples within the jet. While the jet speed, 100 m/s, is far too high to permit direct imaging of particles, the brief exposure time per particle might allow diffraction patterns to be obtained from radiation sensitive samples without damage.

[1] A.M. Ganan-Calvo., *Physical Review Letters*, 1998. 80(2): p. 285-288.
[2] D. P. DePonte et al., *Micron*, 2008. doi:10.1016/j.micron.2008.12.009

[3] D. P. DePonte et al., *J. Phys. D: Applied Physics*, 2008(19): p. 195505.
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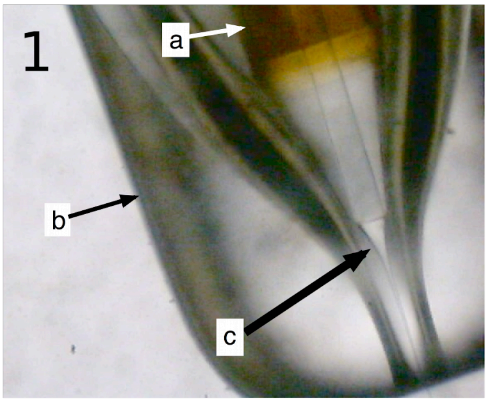

FIG. 1. Liquid injection nozzle: An inner 360-μm OD, 40-μm ID tube (a) and outer glass capillary (b) forming the gas aperture produce (c) a cone of liquid emitted from the inner liquid feed tube and accelerated by co-flowing gas.

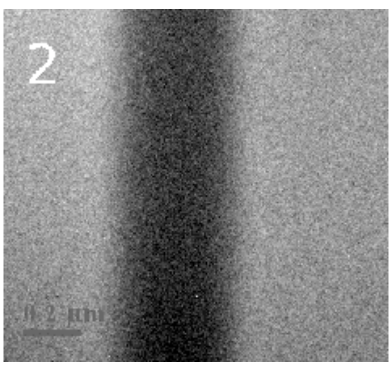

FIG. 2. ETEM image of a 350-nm-diameter isopropyl jet. Electron energy 200keV.

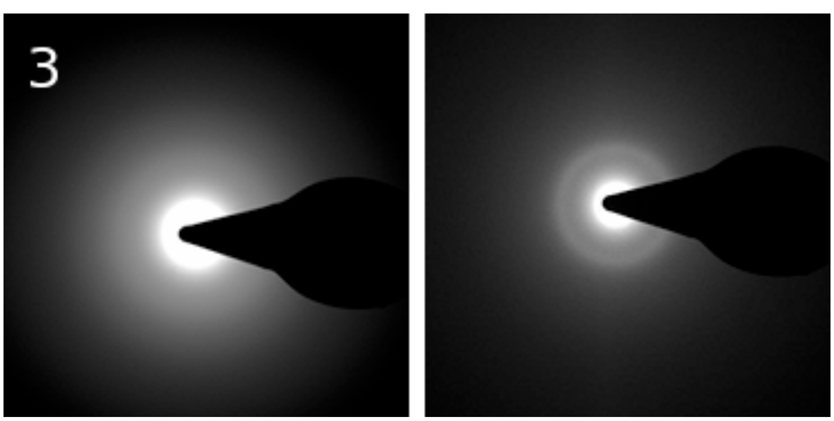

FIG. 3. ETEM diffraction rings for water (left) and isopropyl (right) corresponding to an effective molecular spacing of roughly 0.3 nm and 0.5 nm, respectively.

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