

Ultrasonic Atomization for Direct Liquid Injection CVD: Precursor Delivery Without the Volatility Requirement

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Scope note: the studies cited here were carried out by independent research groups on their own equipment and are cited as evidence for what ultrasonic atomization can do as a precursor-delivery method — not as demonstrations of MicroSpray hardware. Process guidance reflects MicroSpray's engineering experience with ultrasonic atomization components.

Summary

Chemical vapor deposition lives or dies by precursor delivery. Only about 10% of industrially available precursors are gases at ambient conditions; roughly 80% are solids, and most of the useful metalorganics have low vapor pressure, limited thermal stability, or both [1]. Classical bubblers demand volatility; capillaries and syringes clog when solvent flashes at the heated tip. Direct liquid injection (DLI) reframes the problem: keep the precursor as a safe, room-temperature liquid or solution, and convert it to vapor only at the last moment. The efficiency of that conversion is governed by one variable — droplet size — which makes the atomizer the heart of the delivery system. This paper reviews the physics of ultrasonic atomization for DLI-CVD, aerosol-assisted CVD (AACVD), and pulsed-pressure MOCVD (PP-MOCVD); the precursor flexibility it buys; the engineering considerations (resonance drift, flow turndown, thermal isolation) that determine reliability; and the film systems demonstrated in the literature.

1. The delivery problem in CVD

In a CVD reactor, precursors must arrive at the heated substrate as vapor. Bubbler delivery works only for precursors with convenient vapor pressure; heating a bubbler to compensate restricts use to thermally robust compounds and forces heated lines everywhere downstream [1]. Solid precursors sublime at rates that drift with grain size and remaining charge. Capillary and syringe feed into a heated evaporator adds fine flow control but risks tip clogging when the solvent evaporates ahead of the solute [1]. DLI approaches sidestep these constraints by storing the precursor solution at ambient temperature — protecting thermally fragile chemistry — and performing vaporization on demand. The remaining question is how to turn a metered liquid stream into vapor fast enough, and that is an atomization problem.

2. Why droplet size governs vaporization

Evaporation scales with free surface area. Dividing a given liquid flow into smaller droplets multiplies surface-to-volume ratio, so the same feed rate vaporizes faster and at lower wall temperature when the droplets are finer [1]. In flash-evaporation zones this is the difference between clean vapor delivery and precursor decomposition on hot surfaces: smaller droplets vaporize before they reach a wall.

Atomization quality is therefore the key parameter used to discriminate between liquid delivery devices in the DLI-CVD literature [1].

3. Ultrasonic atomization: droplet size set by frequency

When a liquid film rides a surface vibrating normal to itself, capillary waves form; above a critical amplitude the wave crests collapse and eject droplets [2, 3]. The resulting droplet population follows a narrow distribution whose median diameter is set by the capillary wavelength — a function of the liquid's surface tension and density and the operating frequency [3, 4]:

$$\text{wavelength} = (8\pi \text{surface_tension} / (\text{density} * \text{frequency}^2))^{1/3} \quad \text{median droplet diameter} = 0.34 * \text{wavelength}$$

Two consequences matter for CVD delivery:

- **Frequency is the size dial.** Higher frequency gives finer droplets [5]. For aqueous and alcohol solutions, a 60 kHz nozzle produces a median near ~30 μm ; 120 kHz roughly halves that — faster flash evaporation for the same flow.
- **The distribution is narrow.** Uniform droplet size means uniform evaporation time, which stabilizes vapor delivery and, in spray-pyrolysis-type operation near the substrate, uniform film formation.

Because an ultrasonic nozzle has no fine hydraulic orifice, it tolerates solutions that would clog pressure atomizers, and corrosion issues with organic precursors and solvents are generally absent [1].

4. Reactor architectures using ultrasonic delivery

Aerosol-assisted CVD (AACVD). The precursor solution is atomized continuously into a carrier gas and evaporates near the hot substrate. The decisive advantage: volatility stops being a selection criterion for the precursor — solubility in a suitable solvent takes its place [1]. This opens precursor classes that bubbler delivery can never use.

Pulsed-pressure MOCVD (PP-MOCVD). Discrete liquid shots are driven through an ultrasonic nozzle into a low-pressure vaporizer, producing a pressure pulse per shot; the chamber pumps back down between shots [1, 6]. Ultrasonic atomization gives the shot a reproducible fine droplet spectrum, and high conversion efficiency of alkoxide precursor to oxide film has been reported for this mode [7].

Continuous DLI with a vaporization zone. A metered liquid stream is atomized into a heated evaporator and swept to the reactor as vapor. Here the atomizer determines how small the evaporator can be and how low its wall temperature can run.

5. Demonstrated film systems

Ultrasonic-nozzle liquid injection appears across the oxide CVD literature: yttria-stabilized zirconia sealing layers on porous solid-oxide fuel cell electrodes [8]; epitaxial titanium dioxide on sapphire from titanium isopropoxide [9]; tantalum oxide [10]; and epitaxial lithium tantalate from a single-source precursor [11]. The broader DLI-CVD family — including solenoid-injector systems — delivers

composition control at the percent level and thickness control approaching 10 angstroms on wafers up to 12 inches [1], with the atomization stage setting the vaporization performance in each case.

6. Engineering considerations for reliable delivery

The review literature is candid about the failure modes of transducer-based delivery, and each has an engineering answer:

- **Resonance drift with temperature.** A piezoelectric transducer warms in operation; its resonant frequency shifts, and with a fixed-frequency drive the atomization rate drifts — historically limiting deposition runs to short cycles [1]. The remedy is a generator that tracks the transducer's resonance continuously. MicroSpray's broadband generator was designed around exactly this requirement: it follows the nozzle's resonance as thermal conditions change, holding atomization steady over long runs, and drives both 60 and 120 kHz nozzles from one unit.
- **Thermal isolation from the hot zone.** The nozzle should atomize into the heated evaporator without soaking in its heat: mount with a thermal break, keep the transducer body outside the hot zone, and let the standoff geometry — not conduction — couple droplets to the vaporizer.
- **Flow turndown.** A single atomizer's usable flow range spans roughly a factor of five [12]. Wider process windows come from adjusting solution concentration, or from selecting the nozzle frequency to match the flow regime, rather than pushing one nozzle across its limits.
- **Fluid preparation.** Filter solutions (0.2-1 μm) to protect the liquid feed path, and confirm precursor solubility margins so concentration changes made for flow reasons do not precipitate solids.

7. Integration as standalone components

Research CVD reactors are, almost by definition, custom builds. MicroSpray nozzles, the broadband generator, and liquid delivery are supplied as standalone components — BNC-driven, analog-controlled, documented for integration — so an ultrasonic delivery stage can be added to a home-built AACVD tube furnace, a pulsed-injection vaporizer, or a commercial reactor retrofit without adopting anyone's platform. Tell us the precursor solution, solvent, target flow and reactor geometry, and we will recommend a frequency and starting window.

References

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