

Ultrasonic Spray Deposition of MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) Thin Films: Process Parameters and Published Results

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Scope note: the studies cited here were carried out by independent research groups on their own equipment. They are cited as evidence for what spray coating and ultrasonic atomization can do with MXene dispersions, not as demonstrations of MicroSpray hardware. The process window in Section 4 is a starting point drawn from that literature and from MicroSpray nozzle data, not a reported result.

Note on MicroSpray's own MXene work. MicroSpray has carried out complimentary (no-charge) ultrasonic spray testing of MXene dispersions for Drexel University. Those trials were exploratory feasibility work; their results are not reported here, and nothing in Sections 3 to 5 should be read as describing them.

Summary

MXenes, two-dimensional transition-metal carbides and nitrides of which $\text{Ti}_3\text{C}_2\text{T}_x$ is the workhorse, are supplied and processed as aqueous colloidal dispersions of flakes a few nanometres thick and one to ten micrometres across. Turning that dispersion into a continuous, uniform film is the step that decides whether the material's metallic conductivity, optical transparency and electromagnetic shielding survive into a device. This paper reviews the published evidence that spray coating, and ultrasonic spray in particular, is a viable and scalable route for MXene films; explains why the physics of ultrasonic atomization suits the feedstock; proposes a starting process window drawn from the literature and from MicroSpray nozzle data; and lists the open problems that still limit sprayed MXene films.

1. The feedstock problem

A typical $\text{Ti}_3\text{C}_2\text{T}_x$ ink is water, sometimes water/ethanol, at 0.1–10 mg/mL solids. It is Newtonian at low concentration, near-zero in viscosity, and the dispersed flakes are large, thin, and easily fragmented by shear. Three properties of this ink drive the choice of deposition method:

1. **Flake integrity.** Inter-flake contact resistance dominates conductivity in thin films. Large flakes (above 5 μm) are worth protecting; high-shear atomizers and small orifices break them.
2. **Cost.** Research-grade MXene dispersions cost hundreds of dollars per gram. Transfer efficiency matters even at lab scale.
3. **Oxidation.** $\text{Ti}_3\text{C}_2\text{T}_x$ oxidizes to TiO_2 in water over days to weeks, faster when warm and aerated. Short residence time in the nozzle and controlled drying are both desirable.

Spin coating handles flake integrity but wastes more than 90% of the ink and cannot address textured or large substrates. Vacuum filtration makes excellent free-standing films but not conformal coatings.

Blade coating gives the densest films (Ref. 10) but needs a flat substrate and a higher-solids ink. Spray coating is the method that scales, and the question is which atomizer.

2. Why ultrasonic atomization fits

An ultrasonic nozzle atomizes at an open, vibrating surface. Liquid arrives by gravity or low-pressure feed, spreads into a film on the atomizing face, and is broken into droplets by capillary waves whose wavelength is set by the drive frequency. Consequences for MXene inks:

- **No orifice, no clogging.** There is no small pressurized channel for flakes or aggregates to bridge.
- **Low droplet velocity.** Droplets leave the face at roughly 0.1–0.3 m/s and are carried by a low-pressure gas shroud. They land, wet, and dry rather than splash. For a plate-like particle this means flakes tend to lie flat on the substrate, which is the orientation that gives in-plane conductivity.
- **Narrow droplet size distribution.** Median droplet diameter follows the frequency: for water, roughly 31 μm at 60 kHz and 12–15 μm at 120 kHz. A tight distribution gives repeatable film build per pass.
- **Low shear.** Capillary-wave breakup imposes far less shear on the liquid than pressure or two-fluid atomization, so flake size is preserved.
- **Thickness control by pass count and flow rate.** Film thickness scales linearly with the number of passes at a fixed feed rate, which the electrode and electrolyte literature (Refs. 11, 13) uses as the primary control variable.
- **High transfer efficiency.** A focused or shaped ultrasonic spray places most of the material on the substrate, which matters when the ink costs more than the nozzle.

3. What the literature shows

Continuity and transparency. The foundational spray-coated MXene film work from Drexel (Ref. 1) showed that delaminated $\text{Ti}_3\text{C}_2\text{T}_x$ sprayed from aqueous or ethanol dispersions forms homogeneous films without pinholes over large areas on glass and on flexible polyester, with sheet resistance stable to a 5 mm bend radius.

Conformal coverage of textured surfaces. The clearest demonstration of ultrasonic spray on MXene is the KAUST work on silicon heterojunction solar cells (Ref. 4). An automated ultrasonic spray coater deposited ~55 nm $\text{Ti}_3\text{C}_2\text{T}_x$ films at ~8000 S/cm over random-pyramid textured wafers, conformally, at 243 cm^2 industry-size cell area, with above 20% power conversion efficiency and 99% retention after 600 days in air. This is the reference to cite when a customer asks whether ultrasonic spray can coat something that is not flat.

Defect-free thin layers on porous supports. A PVA/ $\text{Ti}_3\text{C}_2\text{T}_x$ pervaporation membrane (Ref. 5) was built by ultrasonic spraying onto an electrospun PTFE nanofiber mat; the authors attribute the ~1.5 μm , defect-free separation layer to the gentle deposition.

Functional layers built by spray. Spray-coated MXene overcoats weld silver-nanowire junctions and raise EMI shielding from 21 dB to 34 dB at constant nanowire density (Ref. 7); sprayed MXene on polycarbonate under a hydrophobic topcoat survives 1000+ bend cycles and 100 days outdoors (Ref. 8);

sprayed Ti₃C₂ dipole antennas at ~5 µm reach 1.1 dBi against 1.7 dBi for copper (Ref. 2); micrometre-thick sprayed Ti₃CNT_x and Ti₃C₂T_x films on glass retain adhesion through a 350 °C anneal (Ref. 3).

Why uniformity matters. Broadband ellipsometry of spray-coated Ti₃C₂T_x (Ref. 9) shows that thickness heterogeneity and roughness measurably shift the optical constants, and that the deposition technique strongly affects electrochemical behaviour. Uniform droplet delivery is not cosmetic; it changes the numbers a device sees.

4. Starting process window

The following is a starting point, assembled from the ultrasonic spray pyrolysis patent (Ref. 12), the ultrasonically sprayed electrode and electrolyte papers (Refs. 11, 13), and MicroSpray nozzle data. It is a place to begin a design of experiments, not a recipe.

Parameter	Starting value	Notes
Nozzle frequency	120 kHz	~12–15 µm median droplet; 60 kHz acceptable for thicker films or higher flow
Ink concentration	0.5–2 mg/mL Ti ₃ C ₂ T _x in DI water	Higher solids risk aggregates; lower solids means more passes
Flow rate	0.1–0.5 mL/min	Below the nozzle's flooding limit; lower is more uniform
Shroud/carrier gas	Nitrogen, 0.5–2 psi	N ₂ rather than air reduces oxidation during flight and drying
Standoff	30–60 mm	Shorter for a narrow line, longer for a wider fan
Substrate temperature	40–90 °C	Drive water off between passes; keep well below oxidation onset
Raster speed	50–150 mm/s	Set so each pass deposits a wet film that dries before the next
Passes	5–50	Thickness scales roughly linearly; measure by four-point probe every 5 passes
Post-treatment	Vacuum or N ₂ dry; optional 100–200 °C	Removes intercalated water; Ref. 3 annealed sprayed films at up to 350 °C under conditions that preserved adhesion

Two process notes from experience with oxide and 2D inks:

- **Wet vs. dry film regime.** With a warm substrate and low flow the droplets dry on contact ("dry film"), giving a granular deposit; with a cooler substrate they merge into a continuous wet layer that levels before drying ("wet film"). For conductive MXene films the wet regime generally gives lower sheet resistance; the ellipsometry data in Ref. 9 supports aiming for the smoother film.
- **Surfactant.** The spray pyrolysis patent (Ref. 12) adds a surfactant to aqueous precursors to improve wetting. For MXene, added surfactant will sit at flake–flake contacts and raise resistance; prefer substrate plasma treatment over surfactant.

5. Open problems

- **Oxidation.** Sprayed films have far more surface area during drying than filtered films. Nitrogen shroud gas, short drying, and prompt encapsulation are the current mitigations; quantitative data on oxidation versus spray parameters is thin.
- **Flake orientation and restacking.** Whether droplet impact velocity affects the fraction of flakes lying flat is not well characterized for ultrasonic spray; this is a good XRD/ellipsometry study.
- **Inter-flake resistance.** Blade-coated films from large-flake inks (Ref. 10) still set the conductivity benchmark. Sprayed films must close the gap through drying control or a mild post-densification step.
- **Line vs. area deposition.** Most reports use raster-scanned round sprays. Fan-pattern or line-shaped ultrasonic sprays for wide-web MXene coating are largely unreported.

6. Where MicroSpray fits

MicroSpray supplies standalone ultrasonic nozzles and generators at 60 and 120 kHz, with focused, fan (air knife) and vortex (swirl-gas) spray shaping, for integration into a customer's own motion system or glovebox. For MXene work the relevant configuration is a 120 kHz focused nozzle with a nitrogen shroud, fed from a syringe pump, on a heated stage. MicroSpray has already run complimentary spray trials of MXene dispersions for research groups (see the note at the head of this paper) and can do the same for a new ink. Application engineering support and a starting parameter set are available on request.

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