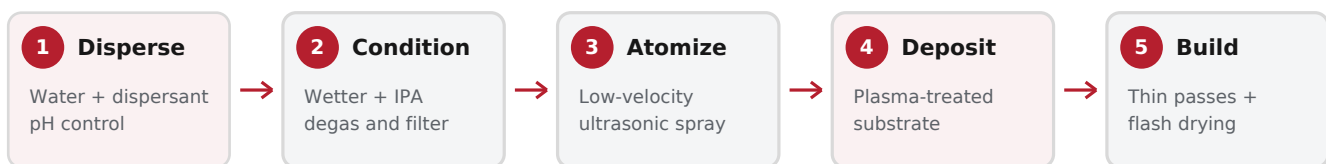


Ultrasonic Spray Deposition of Micron Scale Alumina Films

Wet process development for controlled ceramic coatings on polyimide and glass

Abstract

This white paper documents a January-February 2026 development program performed by MicroSpray Brands for Quantum Design. The work examined aqueous alumina suspensions delivered by an ultrasonic nozzle to form micron-scale ceramic films on Kapton polyimide and glass. The principal challenge was not droplet generation; it was maintaining dispersion stability, dynamic wetting, and controlled drying after droplets reached the substrate. Microscopy identified islanding, exposed substrate channels, and particle-rich rims consistent with dewetting and evaporation-driven particle transport. The resulting process strategy combined polyelectrolyte dispersion, pH control, a low-dose dynamic wetting agent, optional alcohol cosolvent, immediate plasma activation, and many thin passes separated by flash drying.



Prepared by MicroSpray Brands

Technical case study based on project records through February 2026
Website edition | September 2026

Project attribution identifies the development customer and does not imply independent validation or endorsement of the conclusions.

PROJECT OVERVIEW

Executive Summary

Ultrasonic atomization provided a controllable, low-velocity route for applying very small liquid volumes, but film quality depended on the complete liquid-substrate-particle system. The work showed that a stable spray plume can still produce a discontinuous coating when the suspension reaches a low-energy polyimide surface, retracts before immobilization, or dries with a pinned contact line. The practical solution was to treat formulation, substrate activation, deposition rate, and drying as one coupled process.

Principal findings

- Wetting was the first limiting mechanism. The early Darvan-containing, near-neutral aqueous slurry beaded on Kapton and failed to coalesce into a continuous wet film.
- Dispersion chemistry and wetting chemistry served different functions. DARVAN C-N stabilized alumina; TEGO Wet 270 and a small IPA fraction were used to accelerate spreading on the substrate.
- Drying redistributed solids. The observed web-like pattern and bright particle-rich rims were consistent with contact-line pinning and evaporation-driven outward flow described in the droplet-drying literature.^[7,8]
- Surface activation had to be used promptly. Ar/O₂ or O₂ plasma was preferred over abrasive roughening because it directly increased surface polarity and wettability while avoiding damage to thin polyimide.^[9,10]
- Thickness was best accumulated gradually. Multiple light passes with controlled flash drying reduced local flooding, edge buildup, and differential shrinkage.

Evidence classification

Category	Meaning in this paper	Examples
Observed	Recorded directly during the project or from project microscopy.	Beading, non-coalescence, exposed Kapton channels, thickened rims, web-like deposits.
Screened	Deliberately varied during formulation development.	pH, TEGO Wet 270, IPA, plasma treatment, PVA binder.
Proposed	A controlled next-step protocol derived from the observations and literature.	The 0.4 um alumina and glass scale-up process aimed at a nominal 10 um film.

The reported values define an engineering starting window. They are not a substitute for application-specific qualification because alumina surface area, particle-size distribution, substrate lot, plasma dose, ambient humidity, and the solids content of commercial additives can shift the optimum.

PROCESS SCIENCE

Why Ultrasonic Spraying Was Suitable

An ultrasonic nozzle converts a liquid film on a vibrating atomizing surface into droplets through capillary-wave breakup. Classical and later correlations show that droplet size depends principally on excitation frequency and liquid properties, while flow rate and viscosity can modify the resulting distribution.^[1,2] Unlike a pressure nozzle, atomization does not require forcing the slurry through a small hydraulic orifice. This is useful for low-flow nanoparticle deposition and reduces overspray momentum, although stable metering, suspension quality, and continuous wetting of the atomizing surface remain practical limits.

Two material systems were addressed

Development stage	Powder and substrate	Primary objective
Initial wetting study	30-60 nm Al ₂ O ₃ in water on Kapton polyimide.	Convert discrete droplets into a continuous, uniform wet film without islanding or exposed channels.
Follow-on thickness build	Nominal 0.4 um Al ₂ O ₃ on 1 in x 1 in glass.	Develop a repeatable multipass route toward an approximately 10 um dry coating.

Coupled process architecture



Figure 1. The film is governed by the entire sequence from dispersion through drying. Atomization alone cannot correct dewetting or particle migration after impact.

Thickness by mass balance

For a uniform dry film, the first-order relationship is:

$$t = (m_{\text{feed}} \times C_s \times \text{eta}) / (A \times \rho_{\text{film}})$$

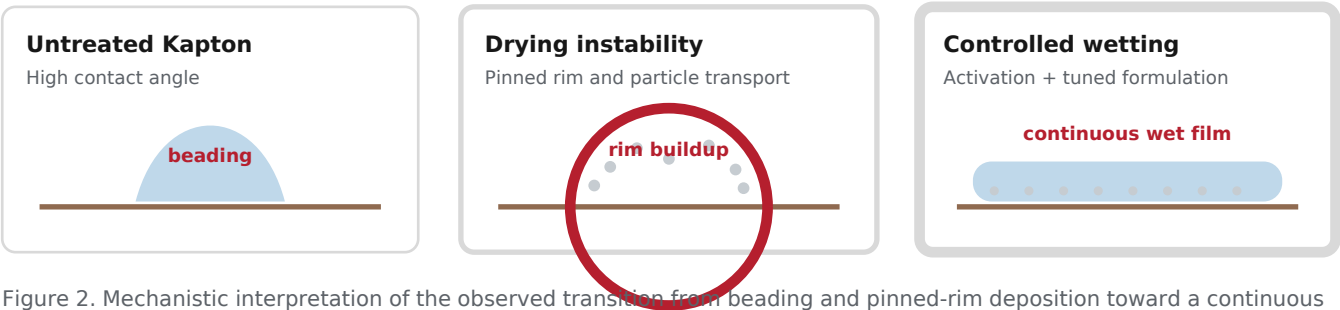
where t is dry thickness, m_{feed} is liquid mass delivered, C_s is the mass fraction of nonvolatile solids, eta is transfer efficiency, A is coated area, and rho_{film} is the dry film bulk density. Using the theoretical density of alumina, 3.95 g/cm³, a dense 10 um film requires 3.95 mg/cm², or about 25.5 mg on a 1 in x 1 in coupon. A porous green film will have a lower effective density, so profilometry and mass gain must be measured together.

Published ultrasonic spray studies likewise show that uniformity is a multivariable optimization problem involving delivery rate, scan motion, spacing, drying, and material properties.^[3,4]

OBSERVED FAILURE MODES

What the Kapton Coatings Revealed

The initial waterborne alumina slurry contained DARVAN C but was near pH 7. At 10x magnification, the dried coating showed particle-rich islands separated by exposed brown Kapton channels. Bright, thickened rims surrounded portions of the deposit. Later trials at 3 wt% alumina, 1 wt% DARVAN C-N relative to alumina, 0.05 wt% TEGO Wet 270, and pH 10 still showed web-like, coffee-ring morphology.



Mechanistic diagnosis

Observation	Most likely mechanism	Process implication
Rounded islands and open channels	Insufficient dynamic wetting followed by wet-film rupture or retraction.	Raise substrate surface energy and lower dynamic liquid surface tension.
Bright or thickened rims	Pinned contact line with outward capillary flow during evaporation. ^[7]	Use thinner wet deposits, control flash drying, and avoid strong local evaporation gradients.
Web-like deposits	Coalescence occurred only locally; solids accumulated along retreating or pinned liquid boundaries.	Balance spreading against overspreading; reduce wet-pass loading and tune surfactant dose.
Sensitivity to pH and age	Alumina surface charge and polyelectrolyte adsorption changed the agglomeration state. ^[5,6]	Control addition order, pH, mixing history, and time from preparation to spraying.

Why DARVAN alone was insufficient

DARVAN C-N is an ammonium polyacrylate or polymethacrylate-type dispersant used to create electrosteric repulsion between alumina particles. Its main task is suspension stability, not rapid substrate wetting. Alumina-polyelectrolyte adsorption is strongly dependent on pH, powder surface area, and dose; an underdosed polymer can even create patch or bridging flocculation before surface saturation is reached.^[5,6] A separate dynamic wetting additive was therefore required for the short time scale between droplet impact and drying.

FORMULATION STRATEGY

A Stable and Wettable Aqueous Slurry

The working formulation was organized around four independent controls: particle dispersion, liquid surface tension, evaporation rate, and green-film cohesion. The ranges below consolidate the project screening history and are expressed on the basis noted.

Component or control	Development window	Function and caution
Alumina	3-6 wt% for low-solids screening; 3 wt% recorded baseline.	Lower solids reduce wet-pass loading. Nanopowder surface area increases dispersant demand.
DARVAN C-N	0.6-1.5 wt% on alumina; 1.0 wt% recorded baseline.	Electrosteric stabilization. Optimize against viscosity, sedimentation, and particle-size data rather than dose alone.
pH with NH₄OH	9.5-10.5; pH 10 recorded baseline.	Moves the suspension away from the near-neutral condition and changes polymer and alumina surface charge. Verify after equilibration.
TEGO Wet 270	0.02-0.08 wt% total; 0.05 wt% recorded baseline.	Dynamic spreading on Kapton. Excess can promote over-wetting, edge pullback, foam, or reduced adhesion.
IPA	2-5 vol% screening range.	Reduces surface tension and accelerates drying. Excess can destabilize the dispersion or dry droplets in flight.
PVA	0.05-0.20 wt% total as a follow-on screen.	Adds green strength and can modify internal flow. It also raises viscosity and leaves organic residue.

Recommended order of addition

1. Charge deionized water. 2. Add DARVAN C-N and mix. 3. Adjust pH with dilute ammonium hydroxide. 4. Add alumina gradually under controlled high shear. 5. Allow the suspension to equilibrate and verify pH. 6. Add TEGO Wet 270 at let-down. 7. Add IPA last. 8. Degas and filter through a medium selected to remove contaminants without excluding the intended particles.

Substrate preparation

The preferred preparation was Ar/O₂ or O₂ plasma immediately before coating. Oxygen-containing plasma introduces polar functionality and can alter nanoscale roughness on polyimide, reducing water contact angle and improving adhesion in published studies.^[9,10] Because activated polymer surfaces can partially recover their hydrophobic character with time, the delay between treatment and spraying should be fixed and minimized. Sandblasting was not selected as the primary wetting treatment: it may increase mechanical keying but can damage thin film, generate debris, and does not reliably supply the surface chemistry needed for an aqueous coating.

Practical acceptance checks before spraying

- Record pH immediately after mixing and again before spraying.
- Inspect for rapid sedimentation, visible agglomerates, foam, or viscosity drift.
- Measure a water or formulation contact angle on untreated and plasma-treated coupons at a fixed delay.
- Dry a single controlled droplet to separate wetting and drying defects from raster-motion effects.

THICKNESS BUILD

Development Protocol for a 10 um Alumina Film on Glass

The follow-on work targeted a nominal 10 um coating on a 1 in x 1 in glass coupon using approximately 0.4 um alumina. The following settings were established as a controlled development protocol. They should be treated as a starting matrix until thickness, roughness, adhesion, and cracking are measured across replicate coupons.

Thickness is built by controlled accumulation, not one heavy wet pass

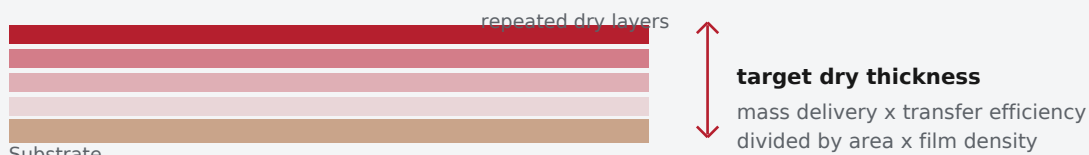


Figure 3. Many low-mass passes allow solvent removal and particle immobilization to proceed incrementally, reducing the stress and redistribution associated with a single flooded layer.

Variable	Starting condition	Purpose
Alumina solids	10-20 wt%	Increase dry mass per pass while retaining atomization and raster stability.
DARVAN C-N	0.5-1.0 wt% on powder	Maintain a low-viscosity, dispersed ceramic suspension.
OPTAPIX PAF 35	1-3 wt% binder solids on powder	Improve green-film cohesion and handling before final processing.
TEGO Wet 270	0.05-0.20 wt% total	Promote glass wetting while avoiding excessive lateral flow.
pH	9-10	Support alumina dispersion with the selected polyelectrolyte.
Substrate	O ₂ plasma; 60 C	Provide reproducible surface cleanliness and accelerate controlled flash drying.
Raster	2.2 mm line spacing	Create deliberate overlap between adjacent spray tracks.
Pass structure	5 passes per block; 60-90 s flash dry	Limit wet accumulation before the next coating block.
Nominal build	Approximately 33 blocks	Initial estimate toward 10 um; calibrate by measured mass and profilometry.
Final dry	80 C for 60 min	Remove residual water and cosolvent without a high-temperature ceramic sinter.

Because the ultrasonic nozzle does not rely on pressure through a restrictive liquid orifice, the practical lower flow limit is set by the metering system and the ability to maintain a continuous liquid film at the atomizing surface. This enables low areal loading per pass and therefore finer control of total thickness.

VALIDATION AND SCALE UP

Turning the Process Window into a Production Recipe

A production-ready recipe requires measured responses, not visual appearance alone. The recommended qualification plan uses small, designed changes around the starting formulation while holding the metering hardware, nozzle condition, substrate history, and ambient environment constant.

Minimum experimental matrix

Factor	Suggested levels	Primary response
pH	9.5, 10.0, 10.5	Viscosity, sedimentation, aggregate size, film continuity.
TEGO Wet 270	0.02, 0.05, 0.08 wt%	Dynamic contact angle, coverage, edge recession, foam.
IPA	0, 2, 5 vol%	Spreading, in-flight drying, flash time, morphology.
Surface treatment	Untreated, Ar plasma, Ar/O ₂ or O ₂ plasma	Contact angle, adhesion, coating continuity, treatment aging.
Wet loading per pass	Low, center, high	Rim formation, cracking, roughness, thickness uniformity.
Flash dry	Short, center, long	Layer rewetting, interpass coalescence, stress development.

Measurements that close the loop

- Coverage and morphology: reflected-light microscopy with fixed illumination and image-analysis thresholds.
- Thickness: stylus or optical profilometry across a masked step, supported by coupon mass gain.
- Uniformity: a defined measurement grid reporting mean, range, and coefficient of variation.
- Surface quality: roughness, pinhole count, edge buildup, cracking, and particle-rich defects.
- Adhesion: a method selected for the substrate and film, with conditioning time and humidity controlled.
- Suspension state: pH, viscosity, sedimentation rate, and particle or agglomerate size before and after the spray run.

Scale-up rules

Scale by preserving dry mass delivered per unit area, track overlap, substrate temperature, and solvent-removal capacity. When coating area or nozzle count increases, confirm that exhaust flow and substrate heating can remove solvent without cooling the surface or changing the local evaporation field. Use gravimetric delivery checks and a coupon map at the start and end of each run. The result should be a statistically controlled deposition window rather than a single nominal setting. Advance a condition only when it meets four gates: continuous coverage, repeatable thickness, acceptable roughness and defect density, and adhesion after the intended thermal or environmental exposure.

CONCLUSIONS AND REFERENCES

Engineering Conclusions

The Quantum Design development program demonstrated that ultrasonic atomization is well suited to controlled alumina delivery, but a uniform micron-scale film emerges only when dispersion, wetting, deposition, and drying are engineered together. The key transition was from treating the problem as a nozzle-setting exercise to treating it as a coupled colloid and interface process.

- Maintain alumina stability with a controlled pH and a verified polyelectrolyte dose.
- Use a low-dose dynamic wetter and limited IPA only after the dispersion is established.
- Activate Kapton immediately before coating and control the treatment-to-spray delay.
- Build thickness through repeatable low-mass passes and measured flash drying.
- Calibrate the final recipe by mass balance, profilometry, microscopy, and adhesion rather than pass count alone.

This framework is transferable to other ceramic and nanomaterial coatings where small droplets must form a continuous film on a difficult substrate. The governing questions remain the same: Are the particles dispersed, does the liquid wet on the deposition time scale, and does the film immobilize before capillary flow and dewetting redistribute the solids?

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