

Cationic Ring-Opening Photopolymerisation in VPP: Photoacid Generation, Dark Cure, Humidity and Epoxy/Oxetane Network Formation

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3Dresyns Engineering Review ER-65 · Version 1.1 · 25 August 2026 · No proprietary formulation composition disclosed

Statement of Significance

Cationic VPP can continue evolving after illumination because photoacid-generated active centres may persist. The same dark reaction that improves conversion can enlarge features and shift dimensions, while water, basic impurities and temperature alter propagation. This review separates photoacid generation from ring-opening kinetics and defines a qualification framework for humidity, post-exposure conversion, dimensional drift and hybrid radical/cationic systems.

Keywords: vat photopolymerisation; cationic photopolymerisation; photoacid generator; epoxy; oxetane; dark cure; humidity; iodonium salt; sulfonium salt; hot lithography

Abstract

Vat photopolymerisation is dominated by free-radical acrylate and methacrylate chemistry. Cationic photopolymerisation provides a distinct route in which light generates a strong acid or an acid-forming species that initiates ring opening of epoxides and oxetanes or chain growth of vinyl ethers. The active ionic centres can persist after illumination, enabling continued dark reaction and conversion in regions no longer receiving light. This persistence is simultaneously an opportunity and a confinement problem. Continued reaction can improve through-depth conversion and reduce dependence on oxygen at the propagation stage, but it can also enlarge features after exposure, shift dimensions with waiting time and weaken the direct relation between projected optical boundary and final polymer boundary. Water, alcohols, amines, basic fillers, pigments, counterion chemistry and temperature can alter acid availability, propagation, transfer and termination. Recent cationic VPP research spans visible-light one-component iodonium salts, carbon-dot and metalloporphyrin sensitisation, oxetane-accelerated epoxy resins, hot lithography, cationic ceramic slurries, dual-wavelength acrylate-epoxy systems and cationic photoinhibition lithography. These studies demonstrate printing at 365, 405 and longer wavelengths, but also show that spectral absorption alone is insufficient: acid-generation quantum yield, monomer class, formulation basicity, temperature, humidity and post-exposure time jointly determine the realised material state. This review develops a VPP-specific qualification framework for cationic ring-opening systems. It separates photoacid generation from monomer propagation, distinguishes dark cure from thermal secondary cure, and requires post-exposure conversion, humidity response, dimensional evolution and chemical-specific conversion to be reported. Hybrid radical/cationic systems are included only where they clarify cationic behaviour; their cure-sequence architecture remains the scope of ER-59.

1. Introduction

In conventional radical VPP, polymerisation is initiated through radical generation and commonly becomes limited by oxygen at exposed interfaces. Cationic systems use a different active species. Onium salts or

sensitised photoacid generators produce strong Brønsted or Lewis-acidic species capable of initiating cationic chain growth or ring opening [1].

The distinction changes the process logic. Oxygen does not quench cationic propagation in the same manner as carbon-centred radicals, but water, alcohols, amines and basic impurities can transfer, neutralise or otherwise redirect the reaction. The material can also continue reacting after the illumination ends.

light -> photoacid generation -> monomer activation -> cationic propagation -> dark reaction / transfer / termination

The engineering question is therefore not whether an epoxy resin can be photocured. It is whether acid generation, ionic propagation and post-exposure evolution remain sufficiently fast, spatially confined and chemically stable for the intended printer and final application.

2. Reaction classes that must not be conflated

Class	Dominant mechanism	Qualification consequence
Cationic ring-opening polymerisation	Photoacid activates epoxide or oxetane; successive ring-opening reactions propagate the network.	Measure monomer-specific conversion, dark reaction, humidity and temperature.
Cationic vinyl-ether polymerisation	Cationic chain growth consumes vinyl ether double bonds.	Do not assume epoxy-like kinetics or network structure.
Radical-induced cationic initiation	Light-generated radicals decompose an onium salt and produce acid.	Separate radical-generation efficiency from cationic monomer conversion.
Sensitised photoacid generation	A photosensitiser transfers energy or electron density to an onium salt.	Spectral overlap is necessary but not sufficient; measure acid generation and cure.
Hybrid radical/cationic network	Radical acrylate and cationic epoxy reactions coexist or are wavelength-selected.	Report each functional-group conversion and the actual orthogonality.
Thermal secondary cure	Heating accelerates remaining cationic or separately latent chemistry.	Do not label every post-exposure conversion increase as a new cure mechanism.

3. Photoacid generation is a separate materials variable

A cationic photoinitiator must absorb or be sensitised at the printing wavelength and generate an acid species with adequate quantum yield. Absorption maximum alone does not determine initiation efficiency. Photolysis, electron transfer, counterion stability, side-product basicity and acid strength all affect the productive pathway.

Visible-light cationic 3D printing has been demonstrated using zinc tetraphenylporphyrin with an iodonium salt at 405 nm [5], coumarin-scaffold iodonium salts [6], carbon-dot photosensitisers [7] and more recent one-component stilbene iodonium salts [11]. The 2024 stilbene systems generated superacid with substantially improved quantum yield and initiated cycloaliphatic epoxides, oxetanes and selected glycidyl ethers under 365, 405 and, for selected structures, 430 nm illumination [11].

These results do not establish universal visible-light activity. A photoacid generator that absorbs at 405 nm can still produce inadequate acid, create basic or absorbing by-products, bleach, colour the print or interact adversely with fillers and pigments.

4. Monomer class controls the propagation window

Cycloaliphatic epoxides, glycidyl ethers, oxetanes and vinyl ethers should not be treated as interchangeable cationic monomers. Their ring strain, nucleophilicity, viscosity, hydrogen-bonding environment and tendency toward transfer produce different induction periods and conversion ceilings.

Hydroxyl-bearing species can accelerate some epoxy systems through an activated-monomer pathway, while other protic species can increase chain transfer or reduce effective network growth. Oxetanes can act as lower-viscosity reactive diluents and can be accelerated by selected epoxide structures. Crivello reported that selected alkyl-substituted epoxides can “kick-start” oxetane polymerisation by substantially reducing or eliminating the induction period. The resulting kick-started oxetane systems polymerised at rates at least two-fold higher than the analogous biscycloaliphatic epoxide ERL under the investigated conditions [4].

The recent stilbene-iodonium study further illustrates monomer dependence: glycidyl ether printing required elevated temperature near 60 °C in the reported system, whereas selected cycloaliphatic epoxy and oxetane formulations reacted at lower temperature [11]. A wavelength-compatible photoacid generator therefore does not remove the need to qualify each monomer family.

5. Dark cure and post-exposure evolution

Cationic active centres can persist after light removal. Decker and Moussa reported extensive dark polymerisation for approximately 30 min after a short UV exposure in a model diepoxy system, with the post-exposure reaction representing up to 80% of the total process under the study conditions [2]. Those values are system-specific, but the mechanism is central to VPP interpretation.

Dark cure can improve conversion in optically attenuated regions. It can also produce delayed feature growth, interlayer evolution and dimensional drift after the projected image has ended. The relevant material state is therefore time-dependent:

$$\text{state} = f(\text{light history, acid population, temperature, humidity, elapsed dark time})$$

The term dark cure should be reserved for verified post-exposure chemical conversion. A delayed increase in hardness can also reflect cooling, solvent loss, physical ageing or a separate thermal reaction and must not be assigned to cationic propagation without chemical evidence.

6. Humidity, nucleophiles and basic impurities

Cationic propagation is not oxygen-inhibited in the conventional radical sense, but it is not environmentally insensitive. Water and alcohols can participate in chain transfer, change network structure or reduce effective propagation. Amines, bases and basic filler surfaces can neutralise or adsorb the photogenerated acid.

Chen and co-workers identified environmental humidity as a major interfering variable in cycloaliphatic-epoxide cationic curing and reported higher conversion for more hydrophobic formulations under elevated humidity [3]. The correct engineering conclusion is not that all water is detrimental, but that water activity and formulation hydrophobicity must be controlled and reported.

Humidity qualification should include the complete resin-conditioning and exposure history. A dry-cabinet resin tested in a low-humidity laboratory cannot be assumed equivalent to material exposed in an open vat for several hours.

7. Temperature and hot lithography

Temperature changes monomer mobility, acid-mediated propagation, diffusion and vitrification. It can also determine whether a nominally active glycidyl ether formulation reaches useful conversion. Hot lithography deliberately prints at elevated temperature to widen the reaction and flow window.

Taschner and co-workers evaluated sulfonium borate initiators in BADGE and demonstrated hot lithography at 90 °C. Their selected initiator showed high conversion, thermal and storage stability, and improved layer quality and coloration relative to reference systems in that study [8]. The formulation also used a sensitizer and an acid scavenger to represent printing conditions and restrict overpolymerisation.

Elevated-temperature results cannot be transferred to room-temperature VPP. Heating changes viscosity, diffusion, acid mobility, working-curve behaviour, separation mechanics and the post-exposure reaction window.

8. Spatial confinement, acid migration and overpolymerisation

Cationic VPP can continue polymerising after and beyond the optical event that generated the acid. Spatial resolution therefore depends on more than photon absorption. Acid generation, transport, monomer mobility, reaction time and neutralisation determine the final boundary.

Recent one-component cationic prints showed slight signs of overpolymerisation even when the photoinitiator itself was sufficiently wavelength-matched [11]. Hot-lithography formulations have used acid scavengers to limit superacid migration [8]. These examples support treating confinement as a reaction-transport problem rather than only an optical-absorber problem.

Islam, Sangermano and Klar demonstrated STED-inspired cationic photoinhibition of sensitised epoxy polymerisation in a two-photon direct-laser-writing configuration. Under maximum depletion, residual polymerisation was reduced to approximately 30–40% of the uninhibited value while a horizontal residuum (“pedestal”) remained [10]. This result — an adjacent cationic-confinement demonstration in 2PP, not a layerwise VPP experiment — illustrates the persistence of competing cationic pathways even under active photoinhibition.

9. Hybrid radical/cationic systems

Hybrid acrylate-epoxy formulations can combine rapid radical green-state formation with cationic ring opening. They can also use different wavelengths to select different network states.

Cazin and co-workers reported 405 nm radical acrylate curing and 365 nm activation of an additional cationic epoxy reaction. Increasing epoxy and cationic-photoinitiator content changed morphology and compromised reaction orthogonality in the reported system [9]. Mason and co-workers later used visible-light radical polymerisation to form a soluble thermoplastic and UV-activated cationic epoxy crosslinking to form an insoluble thermoset within one vat [13].

These studies are included here only to clarify cationic initiation, acid generation and monomer response. The sequencing and network-state architecture of dual cure remains the scope of ER-59.

10. Cationic ceramic slurries and indirect processing

Particle-filled ceramic systems add acid adsorption, scattering, high viscosity and green-body requirements to the cationic reaction. Park and co-workers combined oxetane with a cycloaliphatic epoxide in a photo-

cationic ceramic slurry, reporting improved photocuring speed, lower viscosity and higher solid content. Post-light heat-induced polymerisation of remaining groups increased green-body crosslinking in the reported workflow [12].

This evidence is formulation-specific. A cationic binder does not automatically solve slurry stability, debinding or sintering. ER-37 remains responsible for debinding-defect qualification, while ER-65 addresses the chemical state entering that process.

11. Measurement and reporting architecture

Domain	Minimum measurement	Required interpretation
Initial formulation	Monomer classes, PAG/sensitiser/counterion, viscosity, water state and basic additives.	Defines the actual cationic system rather than “epoxy resin” generically.
Optical input	Spectrum, irradiance, exposure history and specimen path.	Separates wavelength compatibility from productive acid generation.
Acid generation	Quantum yield or validated acid proxy where available.	Identifies the initiating-species supply.
Chemical conversion	Epoxy, oxetane or vinyl-ether conversion versus time.	Prevents pooling non-equivalent functional groups.
Post-exposure reaction	Conversion versus elapsed dark time and temperature.	Quantifies dark cure rather than assuming it.
Environment	Humidity/water conditioning and matched controls.	Establishes sensitivity to protic and basic species.
Dimensions	At light-off, after dark hold, wash/dry and final conditioning.	Separates optical geometry from post-exposure growth.
Final network	DMA/DSC, gel/extractables, mechanics and application-specific function.	Links cure history to final material state.

12. Proposed engineering descriptors

The following are proposed reporting aids and are not standardised performance indices. Each requires a declared functional group, method, temperature, atmosphere and observation window.

12.1 Dark-cure fraction

$$F_dark(\Delta t) = [\alpha(t_off + \Delta t) - \alpha(t_off)] / \alpha(t_off + \Delta t)$$

This reports the fraction of measured conversion present at the stated post-exposure time that accumulated after light-off. If the denominator is at or below the analytical quantification limit, report the result as not calculable rather than assigning zero or infinity.

12.2 Humidity-retention ratio

$$R_H(RH) = \alpha_RH / \alpha_reference$$

Use matched dose, temperature, elapsed dark time and resin history. The reference humidity and water-conditioning state must be stated. The ratio does not identify the chemical reason for the change.

12.3 Post-exposure dimensional increment

$$\Delta d_post(\Delta t) = d(t_off + \Delta t) - d(t_off)$$

The measured dimension, registration method and direction must be declared. This descriptor records delayed geometric evolution but does not by itself distinguish chemical propagation from thermal or physical ageing.

12.4 Standards boundary

No published ISO, ASTM International or DIN standard directly qualifies cationic vat photopolymerisation: photoacid-generation efficiency, dark-cure conversion tracking, humidity response of cationic networks, acid migration or overpolymerisation, post-exposure dimensional evolution, and separation of radical and cationic conversion are not standardised. ISO/ASTM 52900:2021 supplies vocabulary [S1], while ISO/ASTM 52940:2025 supplies ceramic-slurry characterisation for VPP [S2]; both are mechanism-agnostic.

Protocols adopted today must therefore be documented as in-house methods. Adjacent methods may support qualitative and quantitative FTIR [S3,S4], DSC [S5], DMA [S6], tensile testing [S7,S8], water-absorption testing [S9,S10], and controlled conditioning atmospheres [S11,S12], but none validates cationic photoacid generation, dark cure, acid transport or spatial confinement.

13. Failure modes and interpretation errors

Observation	Possible error	Required control
No surface inhibition	Universal environmental robustness inferred.	Humidity, amine/base and filler-surface controls.
High PAG absorption	Efficient acid generation assumed.	Acid quantum yield/proxy and conversion kinetics.
High light-off conversion gain	Improved accuracy inferred.	Post-exposure dimensional mapping.
Room-temperature print fails	PAG described as inactive.	Monomer class and temperature-dependent tests.
Better deep conversion	Uniform network inferred.	Depth-resolved conversion and mechanics.
Feature oversize	Only optical scattering blamed.	Acid transport, dark time and scavenger controls.
Thermal post-treatment improves properties	New cure mechanism assumed.	Chemical-specific conversion before and after heat.
Epoxy label	Low shrinkage or safety inferred.	Formulation-specific shrinkage and hazard/biological assessment.

14. Scope boundaries

ER-65 is restricted to cationic initiation, monomer propagation and post-exposure evolution.

- ER-81 addresses in-can stability, latency and premature cationic polymerisation in light-curable formulations — photoacid-generator counterion chemistry, impurity, pigment and filler interactions, and stabilisation strategies against premature initiation. ER-65 addresses the cationic reaction from photoacid generation onward: initiation on demand, monomer propagation, dark reaction and post-exposure evolution. Storage-state characterisation of formulated cationic vats belongs to ER-81; the reaction chemistry once light is applied belongs to ER-65.
- ER-59 addresses independently programmed dual-cure architectures. ER-65 addresses the cationic reaction within or outside such architectures.
- ER-61 addresses gelation, vitrification and mobility arrest across photopolymer classes. ER-65 addresses acid generation, ionic propagation and dark reaction.

- ER-64 addresses thiol-ene and thiol-yne radical transfer chemistry. Thiol-epoxy anionic curing is outside ER-65 unless a cationic pathway is independently demonstrated.
- ER-71 addresses in-situ monitoring and closed-loop control. ER-65 defines the cationic observables that such control may monitor.
- ER-77 addresses long-wavelength and superlinear photochemical initiation across mechanisms. ER-65 includes visible sensitisation only as it affects cationic photoacid generation.
- ER-37 addresses debinding defects. Cationic ceramic green-body chemistry is included only up to the state entering debinding.

15. 3Dresyns implementation boundary

No current standard 3Dresyns product is identified in this review as an independently validated cationic epoxy, oxetane or vinyl-ether VPP resin. Product naming, low-oxygen sensitivity, epoxy content, dual-cure positioning or thermal post-treatment must not be used to infer a cationic mechanism without controlled compositional and kinetic evidence.

A future custom cationic platform would require, at minimum:

- defined monomer class and photoacid-generator identity;
- wavelength-specific acid-generation and conversion evidence;
- humidity, basic-impurity and filler-surface compatibility controls;
- post-exposure conversion and dimensional-evolution data;
- temperature-dependent processing and storage stability;
- complete final-network, extractables and application-specific qualification.

16. Design rules

- Name the cationic monomer class rather than using “epoxy” as a universal category.
- Separate optical absorption from acid-generation efficiency.
- Report conversion after a defined dark interval, not only at light-off.
- Control humidity, amines, bases and filler-surface chemistry.
- Rebuild dimensions after post-exposure reaction and conditioning.
- Treat elevated-temperature results as a different material-process state.
- Measure radical and cationic conversions separately in hybrid systems.
- Do not infer low shrinkage, low hazard or biological suitability from cationic chemistry alone.

17. Conclusions

Cationic VPP changes the connection between illumination and final material state. Light creates acid; acid activates the monomer; ionic propagation can continue after the image has ended. The resulting advantages and risks are therefore inseparable.

A useful formulation must balance:

acid generation -> monomer-specific propagation -> dark conversion -> spatial confinement -> final network

Oxygen tolerance does not imply humidity tolerance. Visible absorption does not prove effective photoacid generation. Improved post-exposure conversion does not prove dimensional accuracy. A cationic resin must be qualified as a time-, temperature-, humidity- and chemistry-dependent material-process system.

In cationic vat photopolymerisation, the optical event starts the reaction; it does not necessarily define when or where the material stops changing.

Limitations

The evidence spans coatings, laser lithography, layerwise DLP, hot lithography, ceramic slurries and dual-wavelength systems. Quantitative cure rates, acid quantum yields, dark-cure fractions, humidity effects and print resolutions cannot be pooled across these chemistries and machines.

Several mechanistic statements rely on the established cationic-curing literature rather than VPP-only experiments. The proposed descriptors F_{dark} , R_{H} and Δd_{post} require independent review for ancestry, uncertainty and detection-limit conventions. No universal acceptance criterion is proposed.

Author contributions

Juan Seguro: conceptualisation, literature synthesis, engineering framework, writing and critical review.

Competing interests

The author is founder, CEO and CTO of 3Dresyns by Resyner Technologies S.L. No current commercial 3Dresyns product is presented as an independently validated cationic epoxy, oxetane or vinyl-ether VPP resin.

Data availability

No new experimental dataset was generated for this review.

Company information

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