

Polyimide and Polyimide-Precursor Photopolymers for Vat Photopolymerisation: Chemical Identity, Organogel Formation, Imidisation, Shrinkage, Void Formation and High-Temperature Qualification

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Statement of Significance

A vat-printed polyimide precursor is not yet a polyimide part. The printed state can be a solvent-rich photocrosslinked organogel, a polyamic acid or ester derivative, an ionic polyamic-acid salt, or a commercial photosensitive precursor whose final imide identity appears only after controlled drying and thermal cyclodehydration. Confusing these states transfers high-temperature properties to a temporary scaffold and hides the mass loss, shrinkage, stress, void formation and chemistry changes that determine final geometry. This review establishes a state-resolved identity gate and a qualification chain from liquid precursor through print fixation, solvent removal, imidisation and final service verification.

Keywords: vat photopolymerisation; polyimide; polyamic acid; polyamic ester; polysalt; organogel; imidisation; shrinkage; void formation; thermal conversion; high-temperature qualification.

Abstract

All-aromatic polyimides combine thermal stability and mechanical performance with poor melt or solution processability in their final imidised state. Light-based additive manufacturing therefore commonly prints a soluble and photosensitive precursor, fixes geometry by a temporary photocrosslinked scaffold and then uses solvent removal plus thermal imidisation to form the target polyimide. Primary studies demonstrate mask-projection stereolithography of photoacrylated polyamic esters, photocurable polyamic-acid salts, concentrated supramolecular polysalts, two-photon writing of a commercial negative photosensitive precursor and structure–morphology differences between polyimide variants. The process can retain complex geometry, but reported linear shrinkage includes approximately 52% in a dilute organogel strategy and $19 \pm 1\%$ for a two-photon precursor under its cure schedule. These route-specific values cannot be pooled or transferred to concentrated polysalt systems without primary confirmation. Solvent transport, crosslink removal, cyclodehydration by-products, thermal gradients and constraint determine warping, cracking and internal voids. A 2026 morphology study observed prevalent internal voids at and above 350 °C under its conditions, with chemistry-dependent severity. ER-105 separates precursor, gel, partially imidised and final states; defines mass, dimension and chemistry closure; and requires final-state thermal, mechanical and component verification. It does not grant Kapton equivalence, universal service temperature, electrical qualification, low outgassing, biocompatibility or fire performance.

1. Scope and boundaries

ER-105 owns polyimide and polyimide-precursor chemical identity in vat and closely related light-based additive manufacturing. It covers photoactive polyamic esters, polyamic acids and salts, concentrated supramolecular polysalts, commercial photosensitive polyimide precursors, organogel formation, drying, thermal imidisation, shrinkage, volatile release, void formation, morphology and final-state qualification.

ER-20 retains generic thermal-property and service-temperature qualification; ER-41 owns general chemical resistance; ER-59 owns generic sequential dual-cure logic; ER-65 owns cationic ring-opening photopolymerisation; ER-72 owns general environmental ageing; ER-73 owns generic interlayer cohesion; ER-92 owns residual-reactant hazard; and ER-100 owns corpus-level evidence architecture [C1–C8]. ER-105 narrows these domains to precursor-to-polyimide conversion and the claim-transfer risks created by that conversion.

The review does not treat every imide-containing photopolymer as an all-aromatic polyimide, call a photocrosslinked organogel a finished thermoplastic, infer final properties from precursor supplier data, certify electrical insulation, establish flammability or space outgassing, or assign an intended-use approval. Polyetherimide, polyamide-imide, bismaleimide and carbonised polyimide require their exact final chemistry to be named rather than absorbed into a generic polyimide label.

2. State-resolved identity gate

Polyamic acid is a ring-opened precursor whose cyclodehydration forms imide rings. A polyamic ester modifies that precursor and can carry photocrosslinkable groups. A polyamic-acid salt introduces ionic counterions, including polymerisable ammonium species, to provide photoreactivity or processability. A polysalt is not merely a blend name: counterion identity, stoichiometry, solids concentration, water or solvent state and temporal stability belong to the formulation specification. Photosensitive polyimide is an operational label that still requires disclosure of whether the printed object is precursor-rich, partially imidised or fully converted.

State	Minimum evidence	Claims withheld
Liquid precursor solution	Backbone precursor, photoactive group/counterion, solvent, solids, water and age	Polyimide properties and final dimensions
Photocrosslinked organogel	Gel fraction, solvent content, conversion and self-support criterion	Final thermoplastic identity or service temperature
Dried precursor scaffold	Residual solvent/water, mass and dimensions after controlled drying	Complete imidisation and void-free morphology
Partially imidised object	Imide conversion versus temperature/time and remaining volatile evolution	Final Tg, CTE, strength or chemical resistance
Final polyimide object	Conversion, composition, mass/dimension closure, morphology and retained properties	Reference-film equivalence without matched testing
Pyrolysed derivative	Carbonisation schedule, carbon identity, mass yield and microstructure	Polyimide identity after destructive conversion

Table 1. Identity gate across precursor, organogel, imidisation and final-material states.

3. Demonstrated light-based routes

3.1 Photoacrylated polyamic ester organogels

Hegde et al. printed a soluble PMDA–ODA polyamic diacrylate ester in NMP by mask-projection stereolithography [1]. Photocrosslinking fixed an organogel, followed by controlled solvent removal and thermal imidisation. The study reported approximately 52% isotropic linear shrinkage, with most shrinkage associated with solvent removal, while preserving geometric integrity under its optimized schedule. Complete imidisation was supported at 350 °C and final thermal and mechanical results approached conventionally prepared PMDA–ODA in that study. None of these values transfers to another precursor loading, geometry or thermal path.

3.2 Polyamic-acid salts

Herzberger et al. converted commercially available PMDA–ODA polyamic acid into ultraviolet-curable salt solutions by adding 2-(dimethylamino)ethyl methacrylate [2]. The formulations showed gel times below 5 s and produced self-supporting organogels that were thermally converted to all-aromatic PMDA–ODA. The counterion and temporary photocrosslinked scaffold belong to the precursor route; they are not assumed to remain as the final load-bearing chemistry.

3.3 Concentrated supramolecular polysalts

Arrington et al. investigated concentrated photoactive supramolecular polysalt routes for PMDA–ODA and BPADA–mPD polyetherimide systems [3]. The work demonstrates that precursor concentration and supramolecular design can change processability and shrinkage. Numerical concentration, viscosity and shrinkage values are withheld here because they were not independently confirmed at the primary record.

3.4 Two-photon writing

Pagliano et al. used a commercial negative photosensitive polyimide precursor for two-photon polymerisation and subsequent oxygen-free curing [4]. They reported $19 \pm 1\%$ linear shrinkage for selected cantilevers, sub-micrometre precursor features and a micro-hotplate operated above 300 °C. Features attached directly to a non-shrinking substrate were more prone to distortion than structures built on a co-shrinking precursor base. Device operation supports that micro-hotplate and process, not a universal continuous-use rating for the material class.

3.5 Chemistry-dependent morphology

Jackson et al. compared additively manufactured PMDA–ODA and PMDA–DDS precursor routes through post-processing [5]. Their specimens were homogeneous below 350 °C, while processing at and above 350 °C produced prevalent internal voids, more severe in PMDA–DDS under the reported conditions. Scattering and atomistic analysis associated the difference with chemistry-dependent packing and interaction strength. The finding prevents a single thermal schedule from being transferred across polyimide structures.

3.6 Pyrolysis is a new material route

Further heating of printed PMDA–ODA to 1000 °C has been used to form disordered carbon objects [6]. The retained geometry and carbon response are important adjacent evidence, but the product is no longer polyimide. ER-105 treats carbonisation as a destructive material-conversion boundary and does not use carbon properties to qualify the polyimide state.

4. Optical fixation and organogel mechanics

Print success requires enough light-induced connectivity to support solvent-rich geometry without locking in excessive gradients. Cure wavelength must be matched to precursor and initiator absorption, while nonreactive solvent, ionic species and aromatic chromophores alter attenuation and reaction rate. Gel time alone is insufficient: storage modulus, conversion, cure depth, voxel or layer dimensions, working time and wash survival must be reported on the complete formulation at controlled temperature and age.

The photocrosslinked state is usually a temporary scaffold. Its role is spatial fixation through washing, drying and thermal conversion. A stronger scaffold may improve green handling yet increase stress or leave more removable organic content; a weaker scaffold may sag or dissolve. Qualification therefore separates gel-state self-support from final-state strength and records the mass fraction that must leave before the target polyimide remains.

Control variable	Failure mechanism	Minimum report
Precursor solids and solvent	Large mass/volume loss, diffusion gradients and collapse	Mass fractions, density, water, viscosity and age
Photoactive group/counterion	Temporary network survives or decomposes nonuniformly	Identity, stoichiometry, conversion and thermal fate
Dose and optical attenuation	Under-gel, cure-through or internal conversion gradient	Wavelength, irradiance/power, exposure/scan speed and feature transfer
Gel mechanics	Sagging, wash damage or trapped stress	G'/G'' or defined self-support criterion and solvent state
Development and wash	Swelling, extraction, cracking or geometry loss	Developer, time, agitation, temperature and mass/dimensions

Table 2. Controls for printing a temporary polyimide-precursor scaffold.

5. Drying, water and solvent closure

Drying is a geometry-forming process, not a housekeeping step. Solvent concentration, vapour pressure, path length, exposed area, support and heating rate control skin formation, capillary stress and internal gradients. Hegde et al. reported that direct heating of highly solvated structures caused warping, shrinkage and breakage, whereas staged ambient removal on a perforated support preserved integrity before further heating [1]. The safe schedule remains geometry- and formulation-specific.

Water has two roles: uncontrolled moisture can alter precursor chemistry and processing, while cyclodehydration can generate water during imidisation. ISO 15512:2019 distinguishes water content from absorption and remains current, although a replacement edition is under development [S10]. ISO 62:2008 anchors water-absorption measurements for final specimens [S9]. Neither standard by itself establishes solvent closure, imide conversion or outgassing; volatile-specific mass balance and analytical verification remain necessary.

6. Imidisation and thermal-conversion window

Thermal conversion can include solvent removal, deprotection or crosslink cleavage, cyclodehydration, counterion loss, relaxation and morphology development. These processes overlap in temperature and can create gas inside a stiffening object. A furnace set point is therefore not a conversion metric. Report specimen temperature, atmosphere, ramp, holds, geometry, support and exhaust conditions, then verify imide formation and remaining precursor by an analytical method such as FTIR with declared bands and calibration logic.

DSC, TGA, DMA and TMA answer different questions. ISO 11357-2:2020 addresses DSC glass-transition measurement; ISO 11358-1:2022 addresses thermogravimetry; ISO 6721-1:2019 and ISO 6721-11:2019 address dynamic-mechanical principles and Tg; ISO 11359-1:2023 and ISO 11359-2:2021 address thermomechanical analysis and linear thermal expansion [S3–S8]. A decomposition onset, a DSC step, a DMA peak and a dimensional-change event must not be merged into one service temperature. ER-20 retains the general thermal framework [C1].

7. Shrinkage, constraint and dimensional compensation

Shrinkage contains at least solvent-loss, scaffold-removal and imidisation contributions. Linear, area and volume shrinkage are not interchangeable. Isotropy must be demonstrated in x, y and z for the declared geometry and support condition. The 52% and $19 \pm 1\%$ linear values in the cited routes begin from different precursor states and schedules

[1,4]; using them as a common ranking or a universal CAD scale factor would be invalid. Concentrated polysalt routes require their own independently confirmed shrinkage evidence.

Constraint changes the result. A free lattice can shrink while retaining topology; a part bonded at distributed points can accumulate tensile stress, warp or fracture; thick-to-thin transitions can dry and imidise at different rates. Qualification therefore records dimensions at print, post-development, post-drying, each thermal hold and final conditioning. Compensation is accepted only after repeatability across build positions, orientations, feature sizes and representative anchors.

Checkpoint	Mass/geometry record	Interpretation
As printed	Wet mass, bounding dimensions, feature gauges and support state	Photofixed precursor/solvent state
After development	Mass, swelling or extraction, broken/missing features	Wash survival and gel selectivity
After drying	Residual volatiles, x/y/z shrinkage, warpage and cracks	Solvent-removal contribution
During staged heating	Mass-rate events, dimensions, atmosphere and conversion marker	Overlap of removal, imidisation and relaxation
Final conditioned part	Stable mass/dimensions, density/voids and properties	Release state for qualification

Table 3. Mass and dimensional closure across the conversion chain.

8. Voids, cracking and morphology

A visually intact exterior does not prove a dense interior. Volatile release can create closed pores; thermal gradients can create skin-core conversion differences; aromatic packing and linkages can change relaxation and void stability. Cross-sections, microscopy and, where justified, X-ray methods should sample centre, surface, thick sections, nodes and build orientations. Density and porosity methods must state whether open and closed voids are distinguishable.

The Jackson comparison shows why route optimisation and polymer structure cannot be separated [5]. A schedule acceptable for PMDA–ODA did not produce the same void response in PMDA–DDS. This does not rank every ether linkage above every sulfone linkage; it establishes the need for chemistry-specific processing–morphology evidence. Claims of homogeneity require defined detection limits, sampling plan and geometry.

9. Final-state mechanical and thermal qualification

Mechanical data are valid only after final composition and conditioning are frozen. ISO 527-1:2019 and ISO 527-2:2025 provide tensile anchors [S1,S2]. Report complete curves, extensometry, orientation, porosity, surface condition, specimen dimensions and thermal history. Comparison with a reference film requires matched polymer chemistry, imide conversion, conditioning and test method; similar modulus alone does not establish equivalence.

Thermal qualification should use a functional endpoint under load and environment, not the highest temperature reached without visible collapse. DMA, TMA/CTE, thermal cycling and retained mechanical or electrical function may fail before appreciable mass loss. Moisture uptake, residual solvent and voids can shift response. ER-72 owns the generic ageing chain, while ER-105 requires every ageing specimen to be tied to its precursor and imidisation history [C5].

10. Residuals, exposure and application boundaries

NMP or other solvents, photoinitiator fragments, reactive counterions, incomplete precursor, removable acrylic scaffold products and cyclodehydration by-products can dominate exposure even when the final backbone is a polyimide. ISO 6427:2025 provides a current conventional anchor for organic-solvent-extractable matter [S11], but compound-specific identification and application-relevant extraction remain necessary. ER-92 owns generic residual-reactant hazard [C7].

High thermal stability does not prove low outgassing, clean-room compatibility, food or biomedical suitability, dielectric breakdown strength, flammability or long-term radiation resistance. The micro-hotplate demonstration above 300 °C is a device-specific validation [4]. Each intended use needs its own temperature, atmosphere, voltage or load, cycling, geometry and failure criterion.

11. Qualification architecture

Qualification block	Minimum evidence	Release question
Precursor identity	Backbone precursor, photoactive species, counterion, solvent/water, solids and age	Is the starting state reproducible and correctly named?
Photofixation	Optical window, conversion, gel mechanics, development and feature transfer	Does the temporary scaffold survive without hidden gradients?
Drying	Volatile mass balance, rate, support, dimensions and damage	Is solvent removal closed before severe heating?

Qualification block	Minimum evidence	Release question
Imidisation	Analytical conversion, temperature history, atmosphere and by-product path	Has the target imide state actually formed?
Geometry/morphology	x/y/z shrinkage, warpage, cracks, density and internal void sampling	Is compensation stable across representative geometry?
Final properties	Tensile/DMA/DSC/TGA/TMA as relevant after controlled conditioning	Does the final state meet the functional envelope?
Application closure	Residuals, cycling, interfaces and component-level duty	Do material claims survive the intended component?

Table 4. Release architecture for vat-printed polyimide routes.

12. Failure-mode interpretation

- The organogel prints but collapses during wash: increase or homogenise photofixation, reduce swelling and report gel-state mechanics rather than calling the final polymer weak.
- The exterior is intact but cross-sections show pores: slow or stage volatile removal, reduce path length, review solids and verify conversion/morphology through thickness.
- Nominally isotropic compensation fails near anchors: separate free shrinkage from constraint-induced stress and redesign supports or co-shrinking bases.
- FTIR suggests imide formation but mass continues to fall: distinguish imidisation from residual solvent, scaffold decomposition and other volatile products.
- TGA onset is high but modulus falls early: use DMA/TMA and loaded functional retention; decomposition is not service temperature.
- One polyimide chemistry develops voids under a transferred schedule: re-establish the thermal window for that backbone and precursor rather than assuming process equivalence.
- Reference-film strength is not reproduced: inspect conversion, voids, molecular architecture, specimen state and test comparability before attributing the difference to printing alone.
- A pyrolysed object retains shape: rename and qualify it as a carbonaceous derivative; do not retain the polyimide material designation.

13. Minimum workflow and research priorities

- Freeze precursor backbone, photoactive group/counterion, solvent, solids, water, storage and working time.
- Map absorption, dose, conversion, gel mechanics and feature transfer on the actual formulation and platform.
- Record mass and x/y/z dimensions after printing, development, drying, every thermal hold and final conditioning.
- Verify imide conversion analytically and separate it from volatile removal and temporary-scaffold decomposition.
- Inspect internal voids and cracks at representative thicknesses, nodes, supports, orientations and build positions.
- Use chemistry-specific shrinkage models with uncertainty rather than a universal compensation factor.
- Report DSC, TGA, DMA and TMA/CTE as distinct measurements and close service temperature with a functional endpoint.
- Quantify residual solvents, extractables and volatile products for the intended exposure and thermal duty.
- Develop round-robin reference geometries that couple organogel support, free/anchored shrinkage and internal morphology.
- Publish precursor-to-final mass balances and failure maps across polymer structure, solids loading, geometry and heating schedule.

14. Conclusions

Vat and two-photon routes can fabricate complex all-aromatic polyimide structures by printing a processable photosensitive precursor and converting it after geometry fixation. The central engineering fact is that printing, drying and imidisation create a sequence of different materials. The organogel is not the final polyimide, and a final-looking shape is not proof of complete conversion or internal homogeneity.

Precursor concentration and ionic or covalent design can reduce shrinkage and improve handling, but solvent removal, by-product evolution, constraint and chemistry-dependent morphology remain controlling. Numerical shrinkage, temperature and resolution values belong to their exact precursor, geometry and schedule. They are not class constants.

ER-105 closes with a state-resolved release chain: precursor identity, photofixation, development, drying, imidisation, mass and dimensional closure, void/crack inspection, final-state properties, residuals and component duty. This chain permits disciplined comparison while withholding Kapton equivalence, universal high-temperature service and intended-use claims not demonstrated by matched evidence.

Limitations

This paper is a qualitative engineering review, not a systematic meta-analysis. The cited studies use different precursors, solvents, solids contents, printers, wavelengths, geometries, supports, atmospheres, heating schedules and analytical endpoints. Numerical values are not pooled or ranked. No proprietary formulation assay, universal imidisation schedule, service-life model, electrical or fire certification, biomedical qualification or regulatory approval is provided.

Author contributions

Juan Segurola: conceptualisation, evidence synthesis, boundary definition, engineering interpretation, manuscript preparation and final author verification.

Competing interests

The author is founder and CEO of 3Dresyns by Resyner Technologies S.L. No comparative product recommendation is made.

Data availability

No new experimental dataset was generated. Scientific statements derive from the cited primary literature and official standards. The review package includes QA and source-boundary records for independent review.

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Related Engineering Review boundaries

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