

Nano and Micron Raw Materials for Manufacturing 2D and 3D Advanced Materials: Physical Form, Dispersion, Loading, Direct and Indirect Routes, and Qualification

From a Named Powder to a Controlled Feedstock and an Accepted Functional Material

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Statement of Significance

Nano- and micron-scale raw materials can expand the chemical, structural and functional design space of additive manufacturing, coating, casting and moulding. They can also make a process less predictable. A product name such as graphene, barium titanate, silver, silicon carbide or silica does not identify the particle state entering the factory, the state dispersed in a binder, the state deposited by the machine or the state remaining after cure, drying, debinding, sintering and service. Primary particles can arrive as aggregates, soft agglomerates, granules, fibres, platelets, porous clusters, surface-treated powders or liquid dispersions. The same nominal composition can therefore produce different viscosity, sedimentation, optical attenuation, powder flow, abrasion, reaction, exposure potential, interface quality and final function. Smaller is not automatically better: increasing specific surface can increase interfacial opportunity while also increasing adsorbed moisture, dispersant demand, viscosity, reactivity and occupational-control burden. This review establishes a state-resolved route from representative sampling and raw-material characterization through wetting, deagglomeration, stabilization, loading, shaping, transformation and final qualification. It separates retained-composite routes from sacrificial-binder and indirect mould routes, distinguishes measurement outputs that are often conflated, and applies no-go gates before a material is allowed into a printer or production cell. The public 3Dresyns portfolio is used only to map the breadth of candidate material families. It is not treated as independent evidence that any listed material is printable, safe, stable or functional in a particular product.

Abstract

The industrial use of nano- and micron-scale powders, fibres, platelets and dispersions requires more than matching a material name to an additive-manufacturing category. Raw-material identity includes composition, crystalline or molecular state, primary dimensions, morphology, aspect ratio, specific surface area, porosity, surface chemistry, coating, moisture, aggregation, agglomeration, contamination, ageing and package history. Each attribute can change when a sample is split, dispersed for measurement, compounded into a feedstock, pumped, jetted, extruded, recoated, irradiated, cured, dried, debound, sintered or finished. This qualitative engineering review defines a complete chain from supplier lot to accepted 2D trace, coating, retained 3D composite, green body, sintered material or indirectly moulded component. It explains why number-, area-, volume- and intensity-weighted size distributions answer different questions; why dynamic light scattering, laser diffraction, microscopy and gas adsorption are not interchangeable; why zeta potential is a conditional indicator rather than a universal stability certificate; and why a deagglomeration process must be controlled by delivered energy, temperature, contamination and damage as well as mixing time. Process-specific sections examine vat photopolymerisation, direct ink writing, material jetting, material extrusion, powder-bed routes, films and patterned 2D structures. A route-selection framework distinguishes retained fillers from materials intended to become the continuous final phase after binder removal, and it compares direct shaping with printed-mould and precursor routes. Primary studies in ceramic stereolithography, silica-glass nanocomposites, colloidal and nanoparticle inks, fibre-filled extrusion, magnetically oriented platelets, rotational printing, conductive polymer composites and laser-assisted metal writing are used to expose mechanisms without transferring their recipes. The review concludes that the qualifying unit is not the named powder or even the formulated feedstock, but the complete material–interface–process–transformation–product state. A nano/micron route should proceed only when representative identity, dispersion state, processability, exposure control, final composition, microstructure, function, ageing and change control can all be demonstrated for the intended geometry and factory.

Keywords: *nanomaterial; micronized powder; nanoparticle; nanofibre; platelet; dispersion; agglomeration; specific surface area; rheology; photopolymer composite; direct ink writing; indirect additive manufacturing; debinding; sintering; occupational exposure; qualification*

1. Purpose, scope and terminology

This paper makes nano- and micron-scale raw materials selectable as engineering inputs rather than attractive names. It has six objectives. First, it defines the material states that must be distinguished from supplier package to accepted component. Second, it

connects physical form and surface state with measurable feedstock behaviour. Third, it establishes how sampling and measurement choices can create false confidence. Fourth, it compares retained-composite, sacrificial-binder, precursor and indirect-mould routes. Fifth, it identifies process and material no-go gates before scale-up. Sixth, it defines a qualification record that protects product function, occupational safety and scientific claims.

Corpus-level additive-manufacturing technology selection is inherited from ER-139; this review owns only the raw-material and feedstock evidence needed to apply that framework to nano- and micron-scale constituents.

ISO 80004-1 supplies core nanotechnology vocabulary [4], ISO/TS 80004-6 addresses nano-object characterization terminology [5], and ISO/TS 80004-13 provides terms for graphene and other two-dimensional materials [6]. Those vocabularies prevent three common collapses. A **nano-object** is not the same as every solid containing nanoscale structure. A **primary particle** is not necessarily the mobile entity present in a powder or liquid. An **aggregate**, with strongly bonded or fused constituents, is not equivalent to an **agglomerate**, whose constituents are more weakly bound. In this paper, *particle* is used generically only when the controlling level has not yet been established. Specifications use the more precise term whenever the distinction changes processing or risk.

The micron domain is included because many industrial feedstocks deliberately combine nanoscale and micron-scale constituents. Micron particles can improve packing, reduce total surface area, carry a surface coating, act as the structural phase or limit nozzle and layer geometry. Nanoparticles can occupy interstices, alter interfaces, change optics or provide a functional network. A bimodal or multimodal blend is not qualified by quoting the smallest constituent. Its packing, segregation, surface-area distribution and process history must be measured as a system.

The phrase **advanced material** is used instead of treating *exotic* as a scientific class. The scope includes component-scale metals and alloys; oxides and glasses; carbides, nitrides, borides and related refractory phases; carbon materials; conductive polymers; cellulose and other engineered fibres; magnetic, dielectric, ferroelectric, optical, luminescent and radiation-functional phases; and combinations designed for a declared function. The 3Dresyns web page uses *exotic materials* as a navigation and portfolio term and lists a broad candidate space [36]. That declaration defines topics that require engineering scrutiny, not a common maturity level.

The paper covers 2D and 3D manufacturing. **2D** includes a controlled coating, trace, film, patterned layer or planar functional architecture whose thickness and through-plane state remain material attributes. **3D** includes retained composites, graded or oriented structures, porous bodies, green bodies and final consolidated components. It does not include concrete, cement, geopolymer, regolith or macroscopic high-volume construction deposition. It also does not claim that a nanoscale constituent gives the printed feature nanoscale resolution.

ISO/ASTM 52900 provides the base additive-manufacturing process categories [1]. ISO/ASTM 52920 establishes production-site and process-qualification principles [2], while ISO/ASTM 52927 organizes main characteristics and corresponding test methods [3]. These standards support a common evidence architecture, but none makes a material printable merely because it can be assigned to a category. The material must survive the actual feed, machine and post-process chain.

Five delivered states are distinguished. A **raw material** is the received powder, fibre, platelet, granule or liquid dispersion. A **conditioned constituent** has undergone a controlled operation such as drying, sieving, surface treatment or pre-dispersion. A **formulated feedstock** contains binder, vehicle, dispersant, photoinitiator, reactive precursor or other constituents needed for delivery and shaping. A **shaped intermediate** can be a wet deposit, cured composite, green body, dried body, debound body, mould or sacrificial tool. An **accepted material** is the state whose composition, geometry, microstructure, surface, function and service behaviour meet the written requirement.

The review is deliberately recipe-free. It does not prescribe a particle size, nano fraction, solids loading, dispersant amount, sonication time, milling speed, zeta-potential threshold, viscosity, exposure, nozzle, layer, drying cycle, debinding profile, sintering temperature or universal acceptance value. Such values belong to a declared material, surface, binder, machine, geometry, environment and test method. Literature values are evidence that variables matter, not permission to copy them.

2. Material identity and the state-resolved chain

A reliable chain starts with a final requirement, not with a powder catalogue. The requirement names the delivered material state and intended function: retained polymer composite, fired ceramic, glass, metal, porous scaffold, conductive trace, magnetic element, dielectric component, optical element or another explicitly defined product. It states critical dimensions, surface, orientation, composition, residual organics, pore state, function, environment, ageing, cleanliness and permissible post-processing. If the required state is unknown, route selection is premature.

The received lot is then identified. Composition alone is insufficient. The lot record includes phase, primary dimensions, particle-size distribution at a declared weighting basis, morphology, aspect ratio, internal porosity, specific surface, surface treatment, coating, moisture, adsorbates, agglomeration, package atmosphere, manufacture date, storage, transport and relevant hazards. For a

supplier dispersion, the vehicle, concentration basis, additives, pH or non-aqueous chemistry, viscosity, sediment state and redispersibility also belong to identity.

Sampling converts a lot into a test portion. That step can dominate the validity of every later number. Fine particles segregate by size, density, shape and electrostatic history. Fibres bridge and orient. A surface crust or settled layer can differ from the centre of a container. ISO 14488 addresses sampling and sample splitting for particulate properties [8]. Its role here is not to supply one universal sampling plan, but to require a plan capable of representing the lot and the attribute being measured.

Conditioning can include quarantine, controlled acclimation, drying, sieving, low-shear homogenization, surface treatment or preparation of a masterbatch. Every conditioning step changes state. Drying can alter surface hydroxylation or harden agglomerates; sieving can remove granules without removing their source; vigorous homogenization can break fibres or coatings; opening a package can introduce water or oxygen. The conditioned state therefore receives a new batch identity and does not inherit every supplier measurement unchanged.

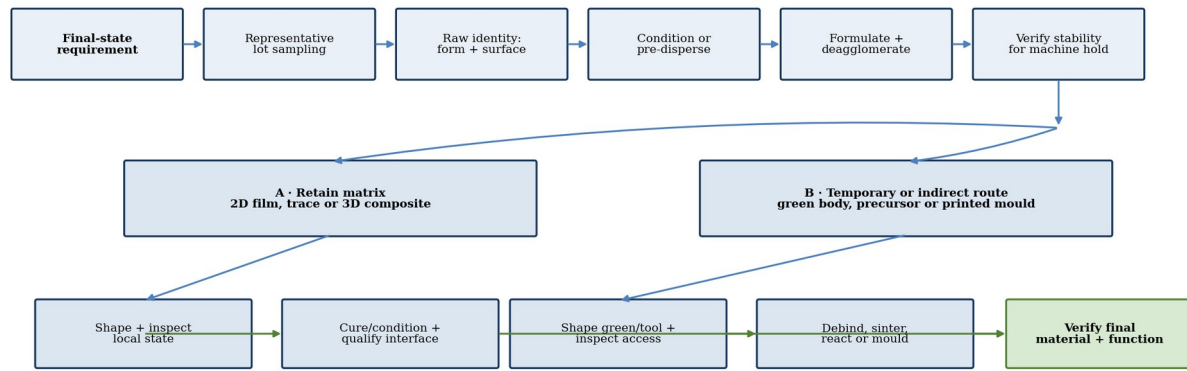
Formulation begins with wetting and incorporation. The material is exposed to binder or vehicle, dispersant, coupling agent, initiator, inhibitor, catalyst, plasticizer and any other additive. Deagglomeration distributes energy into the system. Stabilization must persist through storage and the time-temperature-shear history of processing. Air removal, filtration and transfer follow. A visually uniform feedstock can still contain a broad aggregate population, composition gradients or a metastable state that changes during the build.

Shaping introduces another material history. A vat feedstock can settle, absorb light, scatter exposure and experience repeated recoating and thermal cycles. A jetting feedstock meets filters, pumps, menisci and high extensional rates. An extrusion ink or melt experiences wall shear, pressure, residence and nozzle wear. A powder experiences handling, aerosolization, spreading and thermal or humidity history. A coating or trace experiences wet-film levelling, evaporation, coffee-ring flow, substrate wetting and local cure. These histories determine the structure actually delivered to each voxel, road, droplet or layer.

Post-processing can retain the dispersed phase inside a polymer, transform the matrix, remove the binder, sinter the particles, infiltrate pores, react precursors or transfer the material through a mould. The material name must follow the state. A cured resin containing alumina is a polymer–alumina composite until an authorised thermal route removes the organics and develops a ceramic. A silica nanocomposite can become fused silica only after the declared debinding and sintering chain, as demonstrated for a specific stereolithography system by Kotz and co-workers [25]. The demonstration establishes feasibility, not transferability to another silica, resin, geometry or furnace.

Table 1. State-resolved control chain for nano/micron materials

State	Identity that must be preserved	Dominant questions	Evidence before transition	Invalid shortcut
Final requirement	Actual delivered composition, structure, function, geometry and service state	What must the component do, and in what state?	Requirement, comparator, acceptance method and population	Selecting a powder before defining the product
Supplier lot	Chemistry, phase, dimensions, morphology, surface, aggregation, moisture, coating and package history	What physical entity was received?	Certificate plus representative verification and hazard record	Treating nominal chemistry as complete identity
Conditioned constituent	Drying, sieving, treatment, homogenization and exposure history	Did preparation improve or damage the material?	Batch record and before/after critical attributes	Assuming conditioning is neutral
Formulated feedstock	Constituent fractions by mass and volume, vehicle, additives, dispersion state, air, viscosity and stability	Can the system be delivered repeatedly?	Mixing-energy record, dispersion/rheology/stability tests and hold time	Reporting only nominal filler loading
Shaped intermediate	Local composition, orientation, cure/dry state, interfaces, defects and geometry	What state did the process actually create?	Position- and time-resolved coupons, mass, microscopy and geometry	Inferring uniformity from appearance
Transformed material	Residual binder, phase, pores, shrinkage, infiltrant, heat history and surface	What changed during cure, drying, debinding, sintering or reaction?	State-specific chemistry, mass, dimensions and defect evidence	Calling a green or filled polymer object the final inorganic material
Accepted product	Final composition, structure, function, ageing and traceability	Does the intended product meet the written requirement?	Method-labelled tests, uncertainty, population and authorised disposition	Inferring function from filler name or supplier claim



A candidate survives only when identity, delivery, transformation, exposure control and final function close in the same genealogy.

Figure 1. Material-state chain with two route decisions.

The two route decisions prevent a common category error. In a retained composite, the interface and matrix remain part of the product and must be qualified in service. In a temporary-binder route, organic removal and consolidation determine the final phase, dimensions and defects. In an indirect mould route, the printed object controls tooling geometry and removal rather than becoming the product. The best route is the one whose complete chain closes the requirement, not the one that puts the most powder into the printer.

3. Primary dimensions, morphology, aggregation and specific surface

Particle size is not one scalar. A sample can be described by a number distribution, surface-area distribution, volume distribution, mass distribution, intensity-weighted hydrodynamic result, equivalent spherical diameter or direct microscopy measurements of selected axes. Each representation emphasizes different parts of a polydisperse population. A small number of large agglomerates can dominate a volume-weighted result, while a large number of fine particles can dominate surface area. Converting between bases requires assumptions about shape, density and optical properties that may not hold.

ISO 26824 supplies particle-characterization vocabulary [7]. ISO 13320 covers laser-diffraction methods [9], and ISO 22412:2025 covers dynamic light scattering [10]. Laser diffraction estimates an equivalent-size distribution from an optical model; it does not directly count primary particles or identify aggregate bonding. Dynamic light scattering estimates hydrodynamic motion and is highly sensitive to the measurement medium, concentration and larger scattering entities. Neither method replaces microscopy when shape, fibre length, platelet thickness, coating, satellites or fused aggregates control the process.

The measurement preparation is part of the result. ISO 14887 addresses procedures for dispersing powders in liquids [12]. If a test portion is aggressively dispersed to reveal a primary-size distribution, that result cannot be silently used as the delivered agglomerate state entering a dry recoater. Conversely, a deliberately weak preparation may report storage agglomerates rather than the achievable formulated state. A useful report therefore declares the sampling location, medium, concentration, dispersant, energy input, time, temperature, instrument model, optical assumptions and result basis.

Morphology controls more than visual appearance. Spheres can pack and flow differently from angular particles. Platelets and fibres have multiple characteristic dimensions and can interlock, bridge, filter, align and create anisotropy. Porous particles carry vehicle and change apparent density. Hollow particles can collapse. Satellites and roughness change contact area. Granules may have excellent handling but disintegrate into a different population during compounding. The morphology specification must follow the process stage that it is meant to control.

Specific surface area connects small dimensions and porosity to formulation demand. ISO 9277 defines gas-adsorption BET determination [11]. A higher BET value can mean smaller external particles, internal porosity, roughness or a combination. It can increase adsorption of water, dispersant, initiator, inhibitor or reactive species. It can also make a mass fraction misleading: two powders at the same weight loading can impose very different interfacial area and viscosity.

Primary dimensions, aggregate dimensions and agglomerate dimensions should be separated when possible. A dispersion process may break weak agglomerates while leaving fused aggregates intact. If the controlling nozzle or layer is smaller than a tail of hard aggregates, the route can fail even when the reported median appears acceptable. Tail metrics, maximum observed structures and challenge filtration can matter more than the central percentile.

Surface treatment creates a new material identity. Silanes, polymers, surfactants, oxidation, reduction, acid treatment, coupling agents and adsorbed species can improve compatibility or create new failure modes. A coating can change the measured size only

slightly while fundamentally changing wetting, charge, cure, decomposition residue or interface strength. Treatment coverage, unbound treatment agent, ageing and desorption must therefore be controlled.

The Stefan et al. study of barium-titanate powders in stereolithography showed, within its specific materials and resin system, that powder properties affected suspension and printing behaviour [26]. Sun and Zhang demonstrated that ceramic material properties influence micro-stereolithography optics and resolution [24]. These studies support a general engineering conclusion: powder identity and process response are coupled. They do not supply a universal barium-titanate grade, loading or exposure.

4. Representative characterization: match the method to the failure

A characterization plan begins with the decision that a result must support. One instrument should not be asked to prove chemistry, size, aggregation, surface, flow and safety at once. Orthogonal methods are valuable because disagreement can reveal a state change rather than an analytical nuisance.

Composition and phase may require elemental analysis, spectroscopy, diffraction, thermal analysis or material-specific methods. Microscopy can reveal shape, coatings and large agglomerates but samples a limited field. Laser diffraction can cover a broad size range but depends on optical and shape assumptions. Dynamic light scattering is useful for suitable suspended nano-objects but can be dominated by a small population of larger scatterers. Gas adsorption measures accessible surface, not a geometric diameter. Sedimentation, centrifugation or analytical separation can reveal density and size effects but also change the dispersion.

Sampling uncertainty is propagated rather than hidden. Duplicate instrument readings from one test portion do not establish lot uniformity. Independent increments from relevant container locations, preparation replicates and day-to-day checks answer different questions. A control chart must separate instrument repeatability, preparation repeatability, container heterogeneity and lot-to-lot change.

ISO/TS 22107 addresses dispersibility of solid particles into a liquid [13]. Dispersibility is not the same as long-term stability and not the same as printability. It describes how a material enters a dispersed state under declared conditions. ISO/TR 13097 addresses characterization of dispersion stability [14]. Stability must be evaluated over the actual hold, circulation, build and interruption time, including relevant temperature and shear. A feedstock that is stable on a shelf but segregates under pump recirculation is not stable for jetting.

Zeta potential can help interpret electrostatic stabilization in an appropriate medium. ISO 13099-1 covers electroacoustic and electrokinetic phenomena for zeta-potential determination [15]. The value depends on medium, pH, ionic strength, conductivity, concentration, surface chemistry and model. Sterically stabilized non-aqueous systems, concentrated reactive resins and mixed solvents may not follow a simple magnitude threshold. A zeta-potential result is therefore evidence about one prepared state, not a universal certificate of stability.

Rheology must reproduce the deformation history relevant to the process. A single viscosity at one shear rate cannot qualify a yield-stress ink, a recoating suspension, a jetting fluid or a polymer melt. Relevant outputs can include flow curve, yield response, thixotropic loop interpreted cautiously, recovery after high shear, oscillatory response, extensional tendency, temperature dependence, ageing and wall slip. Geometry and surface of the measuring system matter when particles approach the gap or migrate.

Table 2. Measurement selection by engineering question

Engineering question	Useful evidence set	What the result does not prove	Failure if misused
What entity was received?	Representative microscopy, chemistry/phase, declared size basis, morphology, BET surface, moisture and coating evidence	Uniformity across the lot or behaviour after compounding	Wrong lot released under a matching chemical name
Are hard aggregates compatible with the delivery path?	Tail-sensitive microscopy or size method, challenge filtration, nozzle/layer comparison and pressure response	Absence of softer agglomerates or future growth	Clogging, scratches, recoater damage or weak layers
Can the material be dispersed?	Controlled preparation-energy series, size/microscopy before and after, temperature and contamination record	Stability during storage or processing	Temporary apparent improvement followed by rebound
Is the dispersion stable for the process?	Time-, temperature- and shear-resolved concentration, rheology, optical or sediment profile and redispersibility	Uniformity at every machine location	Composition drift through a long build
Is the surface compatible with the matrix?	Wetting, adsorption, spectroscopy, cure/reaction and interface tests	Final part strength or function	Dispersant starvation, cure inhibition or weak interface
Can the feedstock be printed?	Process-specific flow, filtration, recoating, exposure, droplet, extrusion or powder-spread challenge	Final material acceptance	Successful deposition with unacceptable cure, pores or anisotropy
Is free nano-object release controlled?	Task-based aerosol or surface assessment, containment verification and material-specific hazard record	Toxicological safety of every exposure scenario	Invisible exposure during weighing, cleaning or recovery
Does the final component function?	Final-state composition, structure, orientation, interfaces, method-labelled function and ageing	Transfer to another formulation, geometry or service	Filler identity mistaken for product performance

The decision rule is simple: characterize the failure-bearing entity in the state where the failure occurs. A dry-powder agglomerate relevant to recoating is not replaced by a highly diluted DLS result. A cured interface is not qualified by liquid zeta potential. A final conductive path is not demonstrated by powder conductivity. Measurement relevance is a state-matching problem.

5. Wetting, surface chemistry and additive demand

Wetting is the first formulation gate. The binder or vehicle must displace air and adsorbed species from the relevant surface under the actual mixing history. Poor wetting can appear as floating powder, persistent clusters, entrained air, unstable viscosity or unreacted regions. Apparent wetting at low loading can fail when the available dispersant or resin is insufficient for the total surface area at production loading.

Surface energy is not a permanent material constant detached from the environment. Moisture, oxidation, adsorbed organics, temperature, prior solvent and surface treatment change interaction with the vehicle. High-area oxides can adsorb polar species and alter acid-base balance. Carbon surfaces can vary with graphitization, oxidation and functionalization. Metal nanoparticles can carry oxide or ligand shells that determine chemistry more strongly than the core during formulation.

A dispersant is a functional component with its own dose basis, adsorption, compatibility and fate. Dose per mass can be misleading when surface areas differ. Under-dosing leaves attractive contacts; over-dosing can leave free species that plasticize, inhibit cure, foam, migrate, contaminate a furnace or weaken the final interface. The optimum is conditional on surface state, medium, solids concentration, competing additives and processing temperature.

Coupling agents and reactive surface treatments can improve matrix interaction but may also bridge particles, change shelf life or alter thermal decomposition. Their effect should be tested in the formulated system and in the final state. A surface treatment that produces low liquid viscosity but decomposes to harmful residue in a dielectric ceramic has not improved the complete route.

Raghavan, Walls and Khan experimentally demonstrated that hydrogen-bonding interactions changed the rheology of silica dispersions in organic liquids [27]. The transferable lesson is that vehicle chemistry and solvation can control particle interactions. The reported liquids and surfaces are not a universal dispersant recipe. The state-specific relation between surface, medium and rheology must be established anew.

Reactive and precursor routes provide an important comparator. Walker and Lewis demonstrated a particle-free reactive silver ink for mild-temperature conductive patterning in a specific system [35]. That result does not displace particle inks universally. It shows why route selection must compare powder dispersion with dissolved, molecular, sol-gel or reactive alternatives when those alternatives can create the required final phase with less clogging or sedimentation. The precursor route introduces its own conversion, shrinkage, residue and chemistry controls.

Air is a constituent until removed. Nanopowders and high-area micron powders can entrain large volumes of air during incorporation. Vacuum alone may expand bubbles faster than the matrix can release them. De-airing must be validated for batch depth, viscosity, temperature and subsequent transfer. Foam that disappears at the surface can remain as microvoids or re-enter during pumping.

6. Deagglomeration, dispersion stability and history

Dispersion comprises at least three operations: wet the surface, separate unacceptable agglomerates and prevent unacceptable re-association. Homogeneous colour or a smooth drawdown does not prove any of them. The target state is process-specific. Complete separation to primary particles may be unnecessary, impossible or damaging; the requirement is a bounded population that can be delivered, shaped and qualified.

Energy input should be controlled as delivered energy and local history, not only mixer speed or elapsed time. Rotor-stator mixing, planetary mixing, bead milling, three-roll milling, ultrasonication and extrusion distribute energy differently. Scale changes vessel geometry, circulation, cooling and residence distribution. A laboratory time cannot be multiplied directly into a production batch.

Deagglomeration competes with damage. Fibres shorten, platelets fragment, coatings abrade, crystal defects can increase, polymer chains can degrade and reactive surfaces can oxidize or hydrolyse. Milling media and vessel surfaces can add contamination. Ultrasonication can heat the batch and create cavitation chemistry. A valid development study therefore follows both dispersion improvement and material damage across an energy series.

Smay, Cesarano and Lewis demonstrated rheologically designed colloidal inks for direct assembly of periodic structures [28]. Li and Lewis demonstrated high-volume-fraction nanoparticle inks in a specific directed-assembly system and connected solids state with drying behaviour [29]. These studies show that dispersion, viscoelastic design and post-deposition dimensional change are coupled. Their compositions and loading values are not universal targets.

Stability has several mechanisms. Gravitational settling or creaming depends on density contrast, effective entity size, medium viscosity and interactions. Brownian motion becomes more relevant as entities become small, but it does not guarantee stability against aggregation. A weak particle network can suppress settling while creating unacceptable yield stress or ageing. Density

matching can reduce gravity-driven separation while changing cure, volatility or safety. A stable average concentration can hide vertical changes in size distribution.

Process circulation can create stability that differs from the storage bottle. Pumps can break agglomerates, promote flocculation, filter large entities or heat the material. Dead zones accumulate settled solids. Return jets entrain air. A feedstock must be sampled at machine-relevant locations and times, not only from the formulation vessel.

Redispersibility is not permission for uncontrolled settling. If redispersion is part of the process, the instruction defines equipment, energy, duration, temperature, maximum cycles and post-redispersion checks. Hard cake, irreversible viscosity drift or loss of functional response is rejection. Manual shaking is not a scalable control unless validated as a defined operation.

Shelf life, pot life, open-vat life and machine hold are different claims. Shelf life concerns the sealed package. Pot life begins after activation or mixing. Open-vat life includes air, light and machine contamination. Build hold includes continuous process history. Each needs its own acceptance test and genealogy.

7. Loading and process-specific windows

Loading must be reported on an unambiguous basis. Weight fraction and volume fraction are not interchangeable, especially when constituent densities differ strongly. A formulation record includes every solid, liquid and volatile constituent and declares whether the basis is as-received, dry, inorganic-after-conversion or final product. A high weight fraction of a dense metal can occupy a modest volume and may not form a continuous network. A low weight fraction of a low-density, high-aspect-ratio filler can dominate rheology.

Increasing solids can reduce drying or sintering shrinkage and increase the amount of functional phase, but it also reduces free matrix, increases crowding, changes heat and mass transfer and can make interfaces or voids dominant. Increasing nanoparticles at fixed solids volume can raise surface-area demand sharply. A maximum mixable loading is not necessarily a printable, debindable or functional loading.

Vat photopolymerisation requires a coupled optical–rheological–chemical window. Particles and agglomerates can scatter or absorb the exposure wavelength; surface species can adsorb initiator or inhibit polymerization; sedimentation can change local loading; high viscosity can impair recoating and bubble release. Cure depth, lateral growth and conversion are separate outputs. Griffith and Halloran established ceramic stereolithography using a particular suspension and thermal chain [23], while Sun and Zhang showed how material properties affect ceramic micro-stereolithography [24]. These primary demonstrations support mechanism control, not generic exposure settings.

Direct ink writing requires flow through the nozzle, stable filament formation, shape retention, inter-road bonding and controlled drying or cure. Yield stress can support geometry, but excessive elasticity or thixotropic recovery can create pressure variation and poor fusion. Particle size and hard-aggregate tails must be compatible with nozzle, while fibres and platelets can align under shear. Compton and Lewis demonstrated extrusion of fibre-filled cellular composites and showed that printing can organize reinforcement [30]. The resulting orientation is a product attribute, not merely a print aid.

Material jetting and inkjet impose tighter constraints on particles, agglomerates, viscosity, surface tension, filtration, volatility and nozzle wetting. A material can pass a nominal pore-size filter yet foul through adsorption, soft agglomeration or drying at the meniscus. A filter can also change formulation by selectively retaining a functional fraction. Droplet formation must be tested through ageing and recirculation, not inferred from low-shear viscosity.

Material extrusion of filled thermoplastics or reactive pastes adds compounding, pellet or filament uniformity, melt viscosity, thermal stability, die swell, pressure, fibre breakage and abrasion. Gnanasekaran and co-workers printed carbon-nanotube- and graphene-filled polybutylene terephthalate in a specific fused-deposition system and reported both functional behaviour and abrasive-filler challenges [33]. The comparison illustrates that two carbon forms at nominally similar purpose can behave differently. It does not establish a universal carbon-filler ranking.

Powder-bed routes require dry handling, spreadability, packing, thermal or optical response, powder recovery and exposure control. Nanopowders commonly have high cohesion and dustiness; granulation or coating may be required to create a processable secondary particle. That secondary particle becomes the feedstock identity. Breaking it during reuse or recoating can change both flow and exposure potential. ISO/TS 12025 provides a method framework for quantifying nano-object release from powders by aerosol generation [16]; a dustiness result must still be related to the actual handling task.

2D coatings and traces add substrate wetting, levelling, evaporation, edge pinning, coffee-ring redistribution, film stress, adhesion and through-thickness gradients. A uniform bulk dispersion can segregate during drying. Film thickness can be comparable to agglomerate or fibre dimensions, making rare large entities decisive. Skylar-Scott, Gunasekaran and Lewis demonstrated laser-assisted direct writing of metal architectures in one controlled ink and process system [34]; their result shows the value of coupling deposition and conversion, not a transferable metal-ink recipe. Function must be mapped across and through the film after the complete cure or conversion.

The practical process window is the intersection of seven requirements: formulation stability, delivery reliability, geometric fidelity, local composition, acceptable interfaces, downstream transformability and final function. Optimizing only viscosity or loading moves rather than closes the problem.

8. Retained-composite routes

In a retained-composite route, the printed matrix and nano/micron constituent remain in the product. The matrix carries load, separates or connects particles, controls environment and often determines ageing. The product must be named as a composite, not as the filler alone.

Retention can be advantageous when the target function emerges below full inorganic density, when low-temperature processing matters, when the matrix supplies toughness or flexibility, or when spatial control of composition and orientation is valuable. It can avoid debinding and sintering shrinkage. It also preserves every matrix limitation: glass transition, creep, moisture uptake, chemical resistance, flammability, UV ageing, residual monomer, post-cure gradient and interface degradation.

The interface is a volume of material, not a mathematical boundary. At high specific surface, an interphase can occupy a significant fraction of the composite. Adsorbed dispersant, coupling agent and constrained polymer alter local mobility and transport. A bulk matrix data sheet cannot be assigned unchanged to a highly filled composite.

Anisotropic constituents add orientation as a manufacturing variable. Kokkinis, Schaffner and Studart used magnetic fields to orient magnetized platelets during direct ink writing in a specific multimaterial system [31]. Raney and co-workers used rotational printing to control reinforcement orientation and damage response [32]. These studies establish that orientation can be programmed and can alter mechanics. They also show why a powder certificate cannot qualify the final part: local orientation, mixing, deposition and cure are part of the material architecture.

Retained conductive, thermal, dielectric, magnetic and optical systems are developed in ER-157–ER-159. ER-156 controls the upstream evidence. A claimed functional filler must have a measured dispersion and orientation state, matrix compatibility, spatial distribution and final-state function. Percolation, field alignment, poling, contact resistance, spectral response and service ageing are not inferred from the raw-material name.

Retained nanofibres and platelets can improve one direction while degrading another. They can increase viscosity, create weld-line sensitivity, reduce elongation, abrade hardware or act as stress concentrators when agglomerated. Benefits must be reported against a matched unfilled matrix and at the same print, cure, conditioning and test state.

9. Temporary-binder, precursor and indirect-mould routes

When the final requirement is an inorganic metal, ceramic or glass rather than a polymer composite, a temporary-binder route may be appropriate. The nano/micron material is dispersed in a photocurable, thermoplastic, aqueous or other binder, shaped as a green body, debound and consolidated. The high surface area that assists sintering can also increase oxidation, adsorbed species, binder demand and reaction. The route is qualified through mass transport and final phase, not through green appearance.

Kotz and co-workers' fused-silica work [25] is a useful state-resolved example: a photocurable silica nanocomposite was shaped, thermally converted and characterized as glass. The result belongs to that silica surface, nanocomposite, geometry and heat treatment. Substituting another oxide, colourant, resin or furnace can change scattering, cure, decomposition, devitrification, bubbles and shrinkage.

For ceramics and metals, nanoscale constituents can fill interstices or promote densification, but they can also segregate during binder flow, grow during firing, react with setters or atmosphere and change grain-boundary chemistry. A mixture that is uniform in the liquid can become non-uniform after drying, binder migration or differential sintering. Phase and spatial composition are therefore checked after every transformation that can redistribute constituents.

A precursor route begins with dissolved, molecular, sol-gel or reactive species rather than a particle dispersion. It can reduce sedimentation and nozzle blockage, but conversion creates volatile loss, shrinkage, porosity, cracking and chemistry-control requirements. Walker and Lewis's reactive silver ink [35] demonstrates why precursor and particle routes should be compared on complete delivered function. It does not establish that precursor routes are always safer, denser or more scalable.

Indirect mould routes use a printed durable or sacrificial mould, pattern or core to shape a separately optimized feedstock. They can decouple printer optics and rheology from the final high-solids material. They add mould manufacture, filling, vents, pressure, thermal history, demoulding or sacrifice and interface control. A route becomes attractive when final material quality and production scale benefit more from established moulding or casting than from loading the printer feedstock directly.

The 3Dresyns page declares direct printing, sintered routes, casting and injection through printed moulds as candidate paths for its broad nano/micron portfolio [36]. Those are manufacturer-declared opportunity classes. Selection still requires independent evidence for the actual material, binder, tool, conversion and product.

Route selection should include a no-powder comparator where plausible. Molecular precursors, in-situ precipitation, electroless deposition, infiltration, atomic-layer coating, solution casting or post-print functionalization can create a desired phase after

geometry is made. These routes introduce their own limitations, but they may avoid forcing an unstable or opaque powder into a printer.

10. Material-family gates and boundaries

Chemical families cannot share one process window merely because their particle dimensions overlap. Each family adds no-go conditions before general dispersion and print tests begin.

Metals and alloys require control of oxidation, passivation, moisture reaction, ignition or explosion potential, composition, surface ligands and contamination. Nanoscale metal can have reaction and melting behaviour unlike a micron powder. A conductive powder does not guarantee a conductive composite because oxide, ligand, polymer separation and contact resistance can dominate.

Oxides and glasses vary in refractive index, surface hydroxylation, acid-base behaviour, phase stability and sintering. Transparent final materials require control of pores, second phases and index inhomogeneity, not merely small starting particles. Dielectric or ferroelectric powders require final phase, density, orientation or poling evidence rather than catalogue permittivity.

Carbides, nitrides, borides and other non-oxides require oxygen, water, carbon, nitrogen, atmosphere and reaction control. A dispersant or resin selected for low viscosity can leave residue or change stoichiometry. Furnace and setter compatibility become part of the material system.

Carbon nanotubes, graphene-related materials, graphite and carbon fibres are distinct forms. ISO/TS 80004-13 helps control graphene and 2D-material terminology [6]. Layer number, lateral size, thickness, defect state, functionalization, residual catalyst and agglomeration change both processing and function. The label *graphene* does not establish a specific nanoform or performance.

Fibres and nanofibres require length and diameter distributions, curvature, bundles, surface sizing and breakage history. Aspect ratio can reduce a percolation threshold or enhance reinforcement while increasing viscosity, bridging and filtration. The length distribution after mixing and deposition, not only as received, controls the final architecture.

Quantum dots, luminescent particles and rare-earth phases require composition, shell, ligand, spectral state, photostability and relevant hazardous-element control. Emission from a dilute liquid is not final-part optical qualification. Absorption, scattering, reabsorption, oxygen, cure radicals and matrix ageing can change response.

Bioactive or biologically intended materials add sterility, endotoxin, extractables, degradation, dose and biological-evaluation requirements. Nanoscale identity can change biological interaction. ISO/TR 13014 provides physico-chemical characterization guidance for toxicological assessment of engineered nanoscale materials [17]; it does not by itself authorize a medical claim.

Table 3. Material-family opportunity and mandatory no-go gates

Family	Legitimate opportunity	Early process gate	Final-state evidence	No-go example
Metals and alloys	Conductive traces, structural or catalytic phases, sintered or infiltrated parts	Oxidation, ligands, reactivity, dustiness, vehicle compatibility and atmosphere	Composition, oxide/residue, contacts, pores, microstructure and function	Uncontrolled reactive powder or insulating interfaces defeat the requirement
Oxides and glasses	Structural ceramics, dielectric, optical, wear, thermal and bioactive systems	Surface chemistry, refractive-index contrast, moisture, dispersion and cure/dry response	Phase, pores, grain or glass state, transparency/dielectric/mechanical test	A printable suspension cannot reach required phase or optical quality
Carbides, nitrides and borides	Wear, thermal, electrical and extreme-service materials	Oxygen/water sensitivity, binder carbon, atmosphere and reaction	Stoichiometry, secondary phases, pores and service property	Organic route creates unacceptable carbon or oxide
CNT, graphene-related and graphite	Electrical, thermal, barrier, sensing and reinforcement networks	Nanoform identity, bundles, functionalization, filtration, viscosity and exposure	Orientation, network, interface, contact resistance and ageing	Raw-filler conductivity is used as part conductivity
Fibres, nanofibres and platelets	Reinforcement, anisotropy, barrier and directed function	Aspect-ratio distribution, breakage, alignment, nozzle/film scale and surface sizing	Local orientation, interfaces, voids and direction-labelled properties	Mixing destroys length or deposition creates unqualified anisotropy
Magnetic, dielectric and ferroelectric phases	Field-responsive, sensor, actuator and energy components	Agglomeration, field response in feedstock, cure interaction and alignment window	Final orientation, hysteresis, permittivity, loss, poling and device test	Material name substitutes for device-state evidence
Optical, luminescent and radiation-functional phases	Spectral conversion, shielding, imaging and photonic function	Absorption/scattering at process wavelength, ligands, dose and matrix chemistry	Spectral/dose response, spatial uniformity, ageing and safety	Cure succeeds only by destroying the intended optical response
Biopolymer, cellulose and bioactive phases	Soft, responsive, scaffold, reinforcement and biomedical concepts	Water activity, contamination, degradation, rheology and biological boundary	Final chemistry, mechanics, degradation and application-specific biological evidence	A general bio label is treated as biocompatibility

This table is a screening framework, not a maturity ranking. A material can pass one route and fail another. The same powder can be a retained filler, a sintering constituent, a coating precursor or an unacceptable hazard depending on state and factory controls.

11. From filler presence to final function

The presence of a constituent is the beginning of a causal chain, not proof of a property. Function depends on amount, volume, dispersion, orientation, contacts, interface, matrix, porosity, cure, thermal history, geometry, electrodes or optical path, environment and ageing. A compositional assay can confirm that a filler exists while the device still fails.

For structural reinforcement, load transfer requires an interface and geometry capable of carrying stress. Agglomerates can create local stiffness and global weakness. A mean modulus can rise while strength, fatigue resistance or elongation falls. Tests must match orientation, conditioning, surface and defect population.

For electrical and thermal transport, network continuity and contact state matter. Polymer films, oxide shells, ligands and voids add resistance. Percolation observed in a moulded coupon may shift after extrusion-induced alignment or layer interfaces. The Gnanasekaran study [33] demonstrates material-form-specific differences between CNT- and graphene-based printed composites; it does not provide a transferable threshold.

For dielectric, magnetic and optical function, loading alone can be actively misleading. Dielectric loss, magnetic hysteresis, optical attenuation and spectral response can worsen while the nominal active fraction increases. Field alignment and poling can be necessary. Cure wavelength can overlap filler absorption. Final thickness and internal path length change the measured response.

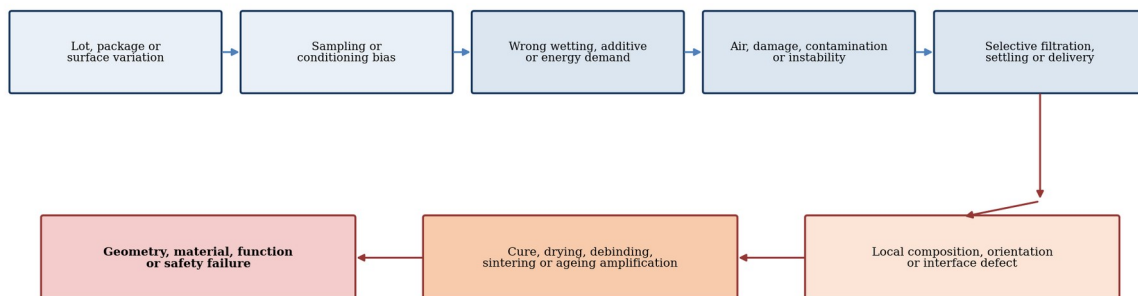
Kokkinis et al. [31] and Raney et al. [32] show that process-controlled orientation can become a designed material variable. Compton and Lewis [30] show that flow can orient fibres in printed composites. Orientation therefore belongs in the build file and inspection plan when it affects function. A change in nozzle, toolpath, speed, field or viscosity can be a material change even when formulation remains fixed.

The final comparison uses a matched reference. A claim of improvement declares the unfilled or alternative material, print orientation, cure or heat treatment, conditioning, geometry, test method, population and uncertainty. A supplier's powder property is not the comparator for a printed part.

12. Failure causality and diagnostic order

Failures should be traced in process order. Beginning with the final broken part and adjusting exposure or loading without genealogy can hide the source.

Raw-material variation can change wetting and additive demand. Incomplete wetting leaves persistent clusters and air. Excess or inadequate deagglomeration changes entity size, fibre length, coating and contamination. Instability creates concentration gradients. Delivery filters, nozzles, recoaters or vats then sample that changing population selectively. Shaping creates local cure, orientation and interfaces. Drying or thermal conversion amplifies the non-uniformity into pores, cracks, phase gradients, shrinkage or weak paths. The final test observes the accumulated chain.



Diagnosis moves backwards from the failed state to the earliest observable and controllable cause.

Figure 2. Causal chain from raw-material variation to product failure.

The diagnostic order follows the arrows. Confirm lot and sample identity. Reconstruct conditioning and formulation energy. Compare top, middle, bottom and machine-location concentration where relevant. Inspect filters and retained material. Compare shaped states before and after cure or drying. Record mass and dimensions through transformation. Only then assign a final failure to a process parameter.

Common interpretation failures include:

- quoting a primary particle size when hard aggregates control the nozzle or layer;
- treating a dilute DLS result as the concentrated production dispersion;
- using a single zeta-potential magnitude as a stability pass/fail value;
- reporting weight loading without volume, density and surface-area context;

- inferring homogeneous distribution from colour or bulk concentration;
- increasing dispersant without measuring unbound additive and cure or residue;
- calling a filled polymer by the name of the filler;
- calling a green body a ceramic, metal or glass;
- using raw-powder function as evidence of composite or device function;
- transferring a laboratory mixing time to a different vessel or batch size;
- treating no visible sediment as proof of unchanged particle population;
- assuming nanoscale feedstock creates nanoscale print resolution;
- claiming a direct route is simpler while ignoring washing, debinding, sintering, finishing or exposure controls;
- claiming an indirect route is superior while ignoring mould filling, sacrifice, tooling life and interface risk.

Each failure is preventable only when the relevant state is measured. More characterization is not automatically better; the correct characterization closes a defined causal uncertainty.

13. Factory scale, occupational control, economics and change management

Free nano-objects deserve explicit task-based exposure control. NIOSH notes that worker risks from engineered nanomaterials remain incompletely understood and recommends caution where exposure to free, unbound nanoparticles can occur [18]. The control objective is not to label every nanocomposite as equally hazardous. It is to identify the material form and task that can release or contact a hazardous entity.

Weighing, package opening, transfer, dry mixing, sieving, spill response, filter change, powder recovery, sanding and cleaning can have different release mechanisms. A liquid dispersion can reduce airborne powder during charging but introduce splash, skin or aerosol risks and can release particles after drying. A bound pellet can reduce routine dust yet generate fines during attrition. A cured composite can release a different particle–matrix fragment during machining.

NIOSH provides occupational-exposure sampling guidance for engineered nanomaterials [19]. Material-specific NIOSH assessments for carbon nanotubes and nanofibres [20], titanium dioxide [21] and silver nanomaterials [22] demonstrate why composition, form and evidence must remain specific. They do not support one universal exposure limit or one PPE prescription for every nano/micron material.

Engineering controls are selected from the process and verified. Closed transfer, local exhaust, ventilated enclosures, contained mixing, appropriate filtration, wet cleaning, dedicated tools, controlled waste and maintenance procedures can be relevant. Their adequacy must be measured for the actual task. General room ventilation or the absence of visible dust is not evidence of control.

Housekeeping avoids dry sweeping and uncontrolled compressed air where these can redisperse material. Cleaning validation considers cross-contamination as well as exposure. A trace of magnetic, conductive, catalytic, bioactive or optically active material can invalidate a later batch even when it is not a bulk chemistry concern.

Scale-up changes more than batch volume. Surface-to-volume ratio, mixing circulation, cooling, gas removal, residence-time distribution, vessel wall, pump, hose, filter and transfer height change. High-area particles can make small moisture or additive errors disproportionately important. A production-scale batch must be qualified as a new state unless equivalence is demonstrated.

Accepted-part economics include raw-material yield, rejected lots, conditioning, masterbatch preparation, energy, cleaning, filters, tool wear, machine occupancy, post-processing, testing, waste and loss of function. Nanomaterials can be expensive per mass while contributing little mass; they can also dominate cost through handling and validation. A cost comparison that omits failed dispersion or downstream rejection is incomplete.

Change control includes supplier site, synthesis route, precursor, surface treatment, ligand, coating, primary dimensions, aggregate state, BET surface, moisture, package, dispersant, binder, mixing equipment, energy delivery, sequence, batch size, hold, filter, hose, nozzle, software, exposure wavelength, atmosphere, cure and heat treatment. A matching trade name does not waive requalification.

Genealogy links every accepted component to raw-material lot, package, sample, conditioning batch, formulation batch, equipment, operator or automated recipe, process state, machine position, post-processing load, inspection and disposition. This record is necessary for both product control and later scientific claims.

14. Qualification architecture

Qualification is performed in layers. A supplier qualification establishes capability to deliver a controlled identity. Incoming release confirms critical attributes of a lot. Formulation qualification establishes a reproducible dispersed state and hold. Machine qualification establishes delivery and shaping within a bounded material window. Transformation qualification establishes cure, drying, debinding, sintering, reaction or mould transfer. Product qualification establishes final geometry, material and function. Publication qualification establishes that the manuscript reports the correct state and evidence.

Worst cases are mechanistic. A large vat depth can challenge settling. A long recirculation time can challenge jetting. A small nozzle can challenge aggregate tail and fibres. A full-height build can challenge exposure and concentration drift. Thick–thin

transitions can challenge drying and debinding. A furnace load can challenge gas transport and temperature uniformity. A thin optical film can challenge rare agglomerates. Challenge artefacts should combine these mechanisms rather than simply reproduce an easy coupon.

Table 4. Nano/micron material qualification and release gate

Gate	Required closure	Representative evidence	Mandatory rejection condition
A. Product and route	Delivered state, function, geometry, comparator and retained/temporary/indirect route are explicit	Requirements review and state map	Filler or green-body name is used as the final product requirement
B. Supplier lot and sampling	Chemistry, phase, morphology, dimensions, surface, moisture, aggregation, package and hazards are controlled	Representative increments, certificate reconciliation and identity tests	Unrepresentative sample, unknown nanoform or uncontrolled package history
C. Conditioning and formulation	Order, energy, temperature, equipment, contamination, fractions, wetting, air and dispersion state are bounded	Batch record, before/after morphology or size, rheology and concentration	Visual mixing alone or unexplained material damage
D. Stability and delivery	Hold, circulation, filtration, settling, redispersion and machine-location composition are controlled	Time/position samples, filter record, pressure or recoating/droplet evidence	Composition drift, hard cake, selective filtration or unbounded nozzle/recoater event
E. Shaped intermediate	Local loading, orientation, cure/dry state, interfaces, defects and geometry meet limits	Position-resolved coupons, microscopy, mass and state-specific tests	Successful appearance with wrong composition, cure or internal structure
F. Transformation	Cure, washing, drying, debinding, sintering, reaction, infiltration or mould transfer is controlled	Mass/dimension by state, thermal record, phase, pores and residue	Unexplained mass, phase drift, cracking, residue or undisclosed infiltrant
G. Final function and ageing	Actual final material meets mechanical or functional requirement under service conditions	Method-labelled population, orientation, conditioning, uncertainty and ageing	Function inferred from raw powder, mean alone or unconditioned coupon
H. Factory safety and environment	Task-based exposure, containment, cleaning, waste and abnormal events are controlled	Exposure/control verification, training, maintenance and incident record	Uncontrolled free nano-object release or incompatible waste route
I. Change and publication	Genealogy, requalification triggers, independent review, author disposition and file parity are closed	Controlled change log, audit, checksums and metadata review	Open major discrepancy, DRAFT state presented as release, DOI or publication without authority

Passing an early gate never predicts a later pass. A well-characterized powder can be unformulatable. A stable feedstock can be unprintable. A printable feedstock can transform poorly. A dense material can lack the required function. A technically sound result can still be unpublishable if its sources, author control or files are inconsistent.

15. Applied route-screening example: a dielectric ceramic carrier with internal channels

Consider a component-scale dielectric ceramic carrier containing internal channels and requiring a final inorganic surface, controlled permittivity and low loss after service conditioning. Three routes enter screening: a retained barium-titanate photopolymer composite, a vat-printed barium-titanate green body followed by debinding and sintering, and an optimized ceramic moulding feedstock injected into a printed sacrificial or durable mould.

The retained composite can be shaped directly and avoids furnace shrinkage. It fails the first gate if the requirement truly demands an inorganic ceramic surface, high-temperature capability or a loss state that the polymer matrix cannot meet. It remains a candidate only if the requirement is rewritten legitimately as a polymer–ceramic composite and final dielectric behaviour is demonstrated in the printed, post-cured and conditioned geometry.

The vat green-body route matches the desired material class more directly after sintering. It survives only if the powder surface, suspension stability and process-wavelength optics permit bounded cure and recoating; the channels can be washed and inspected; binder removal is feasible through the geometry; and shrinkage, phase and pores can be controlled. Stefan et al. [26] can identify powder-property mechanisms worth testing, but their barium-titanate results cannot qualify this new feedstock.

The printed-mould route decouples the printer from the highly loaded ceramic feedstock and can use a moulding system developed for filling and thermal conversion. It survives only if the channels can be filled without unacceptable welds or trapped gas, the mould can be removed or reused as designed, and the complete tooling and conversion chain meets dimensional and dielectric requirements. At higher production volume, mould lifetime and cycle yield may favour it; at highly intertwined geometry, removal may make it impossible.

The decision is not made by assigning universal scores. The final-state requirement invalidates the retained polymer route unless a composite is acceptable. Geometry, washing and debinding can invalidate direct vat shaping. Filling and demoulding can invalidate the mould route. The surviving route is the one whose evidence gaps can be closed with the lowest complete-cell risk. If no route survives, the correct result is redesign or no-go, not a forced selection.

This example demonstrates the purpose of ER-156. Raw-material breadth creates candidates; state and geometry gates remove invalid ones; process evidence selects development work; final function makes the release decision.

16. Discussion: why nano is neither a quality grade nor a route

The most important result of this review is that *nano* describes a dimensional condition, not a quality grade. Smaller primary particles can lower a sintering length scale, increase surface reaction or enable fine interfaces. They can simultaneously increase cohesion, viscosity, moisture sensitivity, reactivity and exposure-control demand. The optimum entity for a factory can therefore be a controlled micron granule containing nanoparticles rather than a free nanopowder.

The second result is that dispersion is a manufactured state. It has raw materials, equipment, energy, temperature, sequence, hold and acceptance criteria. It is not a one-time formulation note. A stable supplier dispersion can become unstable after dilution; a dry powder can become processable through a masterbatch; a good laboratory dispersion can fail at scale because circulation and cooling change.

The third result is that measurement methods create operational definitions. DLS, laser diffraction, microscopy and BET can all be correct while reporting different entities. Conflict should trigger a state analysis: which population is each method sensitive to, what preparation changed, and which entity causes the failure? Selecting the most favourable number is not characterization.

The fourth result is that high loading is an incomplete objective. A route needs enough intended phase for its final material or function, but loading must coexist with wetting, air removal, delivery, cure or drying, geometry, transformation and acceptance. More filler can lower one shrinkage component while increasing pores, cure gradients or weak interfaces.

The fifth result is that direct and indirect are workflow choices. Direct printing can preserve local composition control and remove a moulding step. Indirect processing can decouple printability from final feedstock density and exploit mature casting or moulding. Neither is generally superior. The correct comparator includes tooling, labour, yield, transformation, inspection and accepted function.

The sixth result is that advanced function is state- and geometry-dependent. Orientation generated by flow or field can be valuable, as shown by Compton and Lewis [30], Kokkinis et al. [31] and Raney et al. [32]. It can also produce unplanned anisotropy. Conductive behaviour in the Gnanasekaran et al. composites [33] illustrates dependence on filler form and processing. Function must therefore be measured in the final component along relevant directions and after conditioning.

The seventh result is that safety cannot be inferred from product form alone. A cured composite may bind particles during use, while its manufacture still contains open weighing and cleaning tasks. A wet dispersion can reduce dust while introducing other exposures. NIOSH's nanotechnology and material-specific guidance [18-22] supports a cautious, task-specific control strategy rather than blanket claims.

The eighth result is that the public 3Dresyns portfolio [36] is valuable as an opportunity map. Its breadth makes clear that metals, oxides, carbides, nitrides, carbon forms, magnetic phases, dielectric phases, rare-earth compounds and other materials cannot share one recipe or one performance claim. Manufacturer breadth should increase, not reduce, the discipline of family-specific qualification.

The framework has limits. It does not provide toxicological evaluation for a specific nanoform, a regulatory determination, a composition recipe or a universal measurement panel. It cannot replace application standards for medical, electrical, optical, aerospace or other regulated products. Its purpose is to expose the decisions and evidence that must exist before those specialized qualifications can be credible.

17. Conclusions and research priorities

Nano- and micron-scale raw materials should enter manufacturing through a controlled state chain, not through a material-name shortcut. The decisive object is the complete material–interface–process–transformation–product system.

The first conclusion is that composition is only one axis of identity. Primary dimensions, morphology, aspect ratio, aggregate and agglomerate state, BET surface, porosity, surface treatment, moisture, contamination and package history can change processability and function without changing the nominal chemical name.

The second conclusion is that sampling is part of measurement. A precise result from an unrepresentative increment is not lot evidence. Container location, segregation, settling and sample preparation must be controlled and propagated into uncertainty.

The third conclusion is that particle-size methods are not interchangeable. Number, volume, intensity and equivalent-diameter results answer different questions. DLS, laser diffraction, microscopy and gas adsorption should be selected against the failure-bearing entity, with preparation fully declared.

The fourth conclusion is that smaller is conditionally useful. Higher surface area can assist reaction, interfaces or densification while increasing adsorbate, dispersant, viscosity, reactivity and exposure burdens. A controlled granule or hybrid distribution may outperform a free nanopowder in the complete factory route.

The fifth conclusion is that dispersion is a process with its own genealogy. Wetting, energy delivery, temperature, contamination, material damage, stabilization, air removal, filtration and hold are controlled variables. Mixer speed and visual uniformity are insufficient.

The sixth conclusion is that stability is process-specific. Shelf, pot, open-vat, recirculation and build stability are different claims. No sediment, high zeta-potential magnitude or easy redispersion alone proves production stability.

The seventh conclusion is that loading requires mass, volume and interfacial context. Maximum incorporation is not maximum usable loading. The accepted window must preserve delivery, geometry, transformation and final function.

The eighth conclusion is that retained and temporary matrices create different products. A retained filled resin is a composite whose matrix and interface require service qualification. A temporary-binder object is a green body whose organic removal, shrinkage, phase and pores control the final inorganic material.

The ninth conclusion is that direct, precursor and indirect-mould routes must be compared at the same delivered state. A printer-success comparison is invalid when one route still requires major conversion or when another produces a different material class.

The tenth conclusion is that orientation and distribution are material attributes. Flow, field, nozzle, toolpath and cure can create local anisotropy and gradients. When these influence function, they belong in the design, build record and inspection plan.

The eleventh conclusion is that filler identity does not establish function. Mechanical reinforcement, conductivity, thermal transport, dielectric response, magnetism, optics and biological performance require final-state, geometry- and condition-matched tests. Raw-powder data and manufacturer descriptions generate hypotheses only.

The twelfth conclusion is that occupational control follows material form and task. Free-powder charging, liquid transfer, filter maintenance, sanding and cleaning require different assessments. Lack of visible dust is not evidence of controlled nano-object release.

The thirteenth conclusion is that change control must extend below the trade name. Supplier process, surface treatment, ligand, dimensions, BET area, moisture, package, mixing equipment, energy, batch scale, filter, nozzle, exposure and transformation can all require requalification.

The fourteenth conclusion is that a no-go is a valid engineering output. If representative identity, exposure control, stable delivery, transformation or final function cannot be closed, the route should be redesigned, replaced by a precursor or indirect path, or rejected.

Research should prioritize reference materials and interlaboratory methods that preserve the distinction between primary particles, aggregates, agglomerates and process-delivered entities. High-value studies should report sampling, preparation, weighting basis, optical inputs and uncertainty rather than a single nominal size.

Dispersion research should link delivered energy and contamination with both agglomerate breakup and constituent damage. Scale-up studies should compare circulation, cooling, air removal and residence distributions rather than only matching average power per volume.

Process research should map local composition and orientation through long builds. In-vat, in-line and at-nozzle measurements need validation against destructive final-state mapping. Filters and recovered residues should be treated as diagnostic samples because they reveal selective loss.

Transformation research should couple initial surface area and dispersion with drying, binder migration, debinding gases, grain growth, phase evolution, shrinkage and pores. The goal is a predictive state chain with uncertainty, not another isolated optimum coupon.

Safety research should connect standardized dustiness or release measurements with actual factory tasks and verify engineering-control performance for changing material forms. Product-life research should examine particle or fragment release during machining, wear, ageing and disposal when relevant.

Functional research should preserve the full genealogy from nanoform to device. Data sets should include final spatial distribution, orientation, interfaces, conditioning and ageing. This requirement becomes the starting evidence for ER-157–ER-159 rather than being repeated as a generic filler claim.

The practical decision is concise. Use a nano/micron material only when its physical form and surface can be sampled, formulated, delivered and transformed reproducibly; when the chosen route creates the required final state; when task-based exposure is controlled; and when final geometry and function are measured. Do not call a feedstock advanced because its ingredient list is unusual. Call it qualified only when the complete evidence chain survives independent review and the intended factory can reproduce it.

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