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Vat polymerization-based 3D printing of nanocomposites: A mini review

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Vat polymerization, the earliest and most established 3D printing technology, offers abundant advantages of high-precision fabrication and rapid printing speed, among others. This technology is often applied to fabricated objects with complex and delicate structures, which are of specific interest in numerous fields. However, it suffers from poor mechanical properties of the resultant printed parts due to layer-by-layer manufacturing patterns and the absence of functionality, restricting the broader application of printed objects. Integrating nanomaterials with vat polymerization-based 3D printing endows the creation of products with enhanced properties and unprecedented functional adjunction with flexible designs. Giving a brief description of 3D printing technology, this review illustrates the principles and characteristics of vat polymerization technology. In this mini-review, we emphasize recent advances in nanocomposite fabricated using vat polymerization, predominantly focusing on creating nanocomposites with enhanced mechanical, thermal properties, and electrical conductivity. Finally, we summarize the article with the challenges being faced and future perspectives of nanocomposites fabricated from vat polymerization.

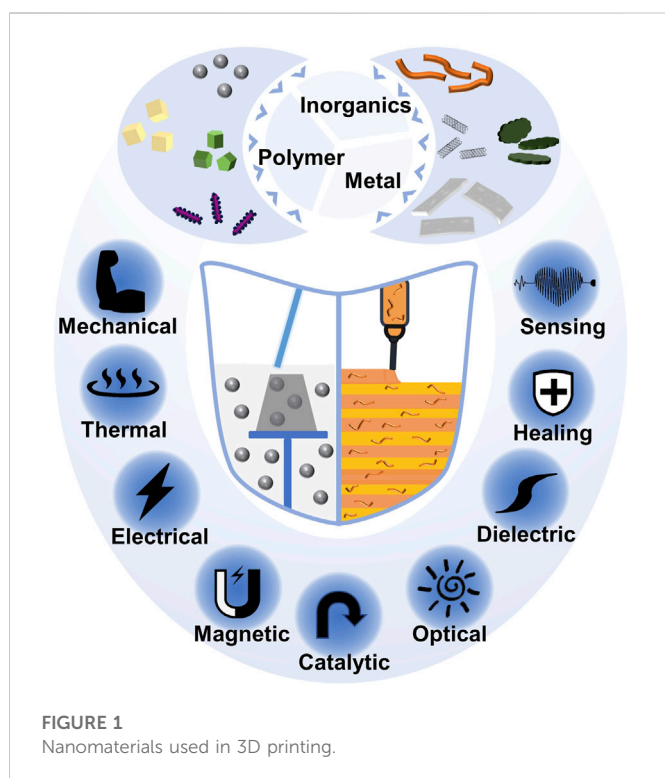
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1 Introduction

3D printing, often referred to as additive manufacturing, is a disruptive manufacturing technology in which replica physical objects with complicated internal structures are fabricated directly from interacted digital models in a layer-by-layer fashion. In recent times, 3D printing has been employed in various fields of research due to the enormous advantages of product-design freedom, simple customization, and the waste minimization of raw materials, including catalysis (Lawson et al., 2021), electronics (Espera et al., 2019), aerospace (Joshi and Sheikh, 2015), and tissue engineering (Distler and Boccaccini, 2020). According to the principle of pattern formation, 3D printing employs diverse technologies, including material extrusion, binder jetting, material jetting, directed energy deposition, sheet lamination, powder bed fusion, and vat photo polymerization (Lee et al., 2017). Each technology has its pros and cons in terms of practical applicability. Nevertheless, choosing a unique technology often considers the manufacturing material, printing speed, resolution, cost, and performance requirements of the final product (Clarissa et al., 2021).

The lack of reliable load-bearing ability and functionality in 3D-printed structural work pieces makes them insufficiently fabricated as functional end-use products. To address this concern, the possibility of tuning the performances by introducing nanomaterials into 3D



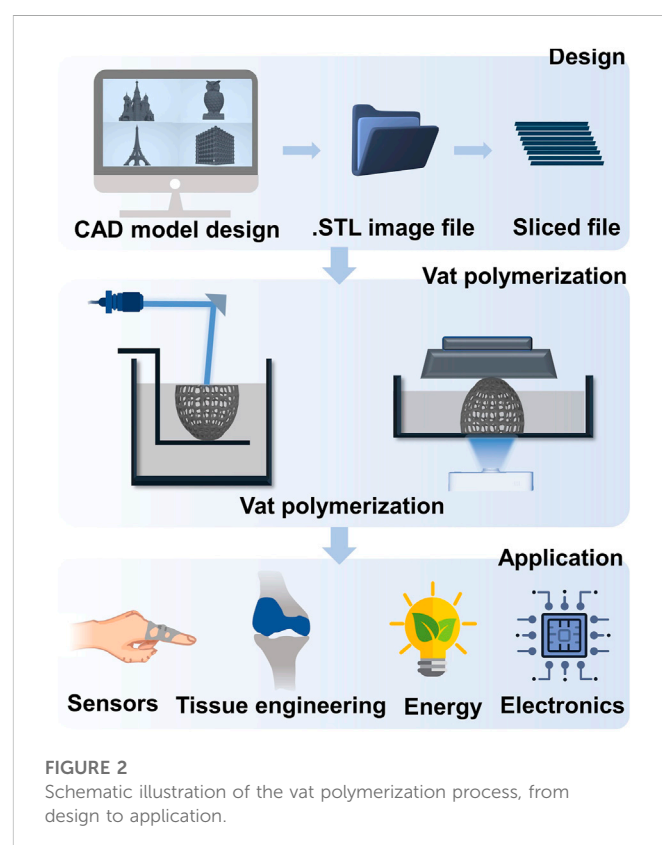
printed constructs furnishes an attractive path for achieving high performance and multi-functional integration (Kankala et al., 2018; Valino et al., 2019). Due to the advancement of nanotechnology, incorporating nanofillers (in the range of 1–100 nm) produces different properties than their micro- and macro-sized counterparts (Roduner, 2006). Nanomaterials have a wide range of application prospects owing to their electrical, magnetic, thermal, optical, sensitive properties, and surface stability (Baig et al., 2021). These unique interactions give rise to the optimal control of polymer performance. Several efforts have been dedicated to developing nanocomposites based on 3D printing by incorporating ceramic, metallic, and/or organic fillers to improve thermal, electrical, mechanical, and other properties to overcome the limitations of traditional single-component materials (Campbell and Ivanova, 2013; Hassan et al., 2019; Wang et al., 2020) (Figure 1).

2 Vat polymerization

Vat polymerization, the most mature and widely used 3D printing technology, offers the advantages of high resolution and printing efficiency. It can be subdivided into stereolithography appearance (SLA), digital light processing (DLP), liquid crystal display, continuous liquid interface production, two-photon 3D printing, and computed axial lithography (Sampson et al., 2021). Vat polymerization is based on photopolymerization, where a photosensitive liquid resin can be utilized as the raw material. As illustrated in Figure 2, three processes are commonly used: The design of a computer-aided model, the development of vat polymerization, and dispensing for practical applications. During the printing process, laser emitters or a projector projects a pattern of light onto a layer of liquid photosensitive resin that hardens in the desired shape. The

platform engaged as the printed part moves in the layer-thickness, and the resin is replenished. This procedure is repeated until the successful construction of the final 3D object. The light source and imaging systems differ for different vat polymerization technologies to some extent, while the control and stepping systems remain similar. The photosensitive resin can be cured under light irradiation at a corresponding wavelength. While in places without irradiation, it can be maintained as a liquid. Hence, parts can be easily separated from the liquid resin. It is noteworthy to acknowledge the high precision of light irradiation and the rapid photocuring polymerization of vat polymerization for the quick printing of complex objects.

Photosensitive resin for vat polymerization is typically composed of initiator, oligomer, monomer (active diluent), and additives (Skliutas et al., 2018). The initiators in photosensitive resin are shifted to highly active intermediates under irradiation at specific wavelengths. These trigger cross-linking reactions between oligomers and monomers in the photosensitive resin, transforming it from the original liquid resin into a solid object. The typical raw materials and photocuring mechanisms are chosen depending on the laser wavelength and the printer projector. Acrylate resins, which undergo a free-radical polymerization mechanism, are the most popular photosensitive resin for vat polymerization technologies due to their high photopolymerization curing speed. However, acrylate resins exhibit large volume shrinkage after photopolymerization, resulting in a reduction in printing precision and the deformation of printed objects. To overcome the problem of volume shrinkage, epoxy resin has been incorporated into cationic photopolymerization (Crivello, 1999). However, the photopolymerization induction period for the cationic photosensitive resin remains longer and has a relatively slower



curing rate. Hence, a hybrid photosensitive resin containing acrylate and epoxy resin is an optimal choice when considering volume shrinkage, curing speed, and cost when the light source matches. Although numerous studies have been devoted to obtaining multiple printed parts by adjusting and designing segments constructed by photosensitive resin polymer backbones (Peng et al., 2020; Zhou et al., 2020; Yiming et al., 2021), currently, vat polymerization products generally suffer from several disadvantages, such as fragility, ease of deformation, poor thermal resistance, and low biocompatibility. However, there is an urgent need for higher-performance products to meet high-end application demands.

3 Nanocomposites from vat polymerization

3.1 Mechanical enhancement

The demand for lightweight, high-strength materials is increasing in various engineering sectors. Polymeric composites, including nanofillers, offer outstanding improvements at relatively lower loadings than micro-sized fillers, helping achieve high-level performance improvements across various advanced applications (Quaresimin et al., 2012). Recently, the mechanical properties of engineered structural components, including structural integrity and durability, produced by vat polymerization have been enhanced by incorporating second-phase nanofillers.

Nanoparticles are widely used to enhance the mechanical properties of vat polymerization polymers because of their low cost and the significant enhancements they deliver, of which, SiO₂ nanoparticles are the most widely used. In an instance, (Weng et al., 2016) and colleagues demonstrated that the absorption and scattering of UV light by SiO₂ nanoparticles were weaker than that of other 1D and 2D materials containing silicon elements (i.e., attapulgite and montmorillonite), which improved the tensile strength and elastic modulus of SiO₂-containing nanocomposites. In another instance, Chiappone and coworkers (Chiappone et al., 2016) introduced the precursor tetraethoxysilane to a photosensitive resin and post-treated samples with acidic vapor to generate SiO₂ nanoparticles *in situ* by the sol-gel reaction of the precursor in the polymer network after DLP printing. The generation of a SiO₂ inorganic phase contributed to significant enhancement of the tensile and compressive strengths, modulus, and surface hardness of the fabricated samples due to the preferential growth of induced nanoparticles on the surface of the samples. Moreover, different surface treatments create diverse nanoparticle interfacial adhesion and dispersion stability, leading to altered mechanical properties of various nanocomposites (Song et al., 2018). Some other 1D nanomaterials, including nano-TiO₂ (Duan et al., 2011), Cu nanopowder (Vidakis et al., 2022), and polyhedral oligomeric silsesquioxane (Li et al., 2019), have been used as reinforcing phases for photosensitive resins. In addition, the specific orientation of reinforcing fillers is believed to supply the creation of composites with design properties (Martin et al., 2015).

Carbon nanotubes (CNTs) are structurally stable, long, thin carbon columns with high aspect ratios. These CNTs often exhibit excellent polymer reinforcement due to their ability to cause the formation of higher-order interphase polymer layers and promote mechanical strength through interfacial stress transfer between nanotubes and polymers (Wong et al., 2003; Spitalsky et al., 2010).

Creating CNT composites with photosensitive resins greatly improves the performance of the matrix. Sandoval et al., (2007) demonstrated that the ultimate tensile and fracture stress of SLA epoxy-based resins increased by 17% and 37%, respectively, with the introduction of .05% (w/v) multiwall CNTs (MWCNTs). Introducing MWCNTs would create strong interfacial bonding between the SLA epoxy matrix and buckled MWCNTs. However, the resultant mechanical properties of nanocomposites depended on the arrangement of CNTs in resins (Chavez et al., 2019). In addition, other 1D nanofillers, such as boehmite nanowires (Han et al., 2018), Al₂O₃ nanowires (Han et al., 2017), and cellulose nanocrystals (Kumar et al., 2012), have been incorporated into photosensitive resins for vat polymerization to enhance mechanical properties.

Graphene, a single layer of carbon atoms, consists of a backbone of sp² hybrid bonds filled in a honeycomb lattice. 2p orbitals forming the π state bands delocalize over the carbon layer and result in the highly stiff characteristic graphene (Papageorgiou et al., 2017), which is often applied in photosensitive resins (Li et al., 2018). The combination of enhanced strength and flexibility in SLA manufacturing is realized by introducing graphene oxide (GO), which is related to increasing the crystallinity of nanocomposites (Lin et al., 2015). Nanocomposites prepared by the bottom-up vat polymerization exhibit significant anisotropic mechanical properties because graphene nanosheets tend to be arranged more parallel in resins during 3D printing (Markandan and Lai, 2020). The annealing process after the completion of printing also affects the mechanical properties of nanocomposites (Manapat et al., 2017). In addition, Ti₃C₂ MXene, a novel 2D ultrathin nanomaterial, is an excellent candidate for enhancing the tensile strength of photosensitive resins to obtain reliable elastomers (Li et al., 2022a). The dispersion effect of nanofillers in photosensitive resins contributes directly to the strengthening development of 3D printed objects. A uniformly dispersed nanofiller can effectively absorb and dissipate energy, improving the mechanical properties of nanocomposites in terms of fracturing. Agglomerated nanofillers induce the formation of micro flaws, deteriorating the mechanical properties. Therefore, there is an optimum loading level for incorporated nanofillers to increase the mechanical strength of nanocomposites from vat polymerization.

3.2 Thermal enhancement

A growing task in the design and application of advanced devices is heat management. In this context, an expanding demand for high thermal conductivity materials that can dissipate waste heat from the operation processes of devices exists. The preparation of composites by introducing fillers, such as graphene, metal, and inorganic nanomaterials, is a promising strategy for improving the thermal conductivity of photosensitive resins (Lee et al., 2021; Pezzana et al., 2021). Carbon fibers, due to their remarkable thermal conductivity and high aspect ratio, are also widely used to enhance the thermal conductivity of polymers, which is beneficial for forming nanocomposite percolating networks (Hong et al., 2010). In a case, acidified vapor-grown carbon nanofiber (VGCF)-supplemented dual-cure photosensitive resins were prepared by the SLA technology (Li et al., 2022b). The addition of 2% w/w VGCFs increased the thermal conductivity of a composite by 79% compared to control through the transfer of thermal energy by VGCF phonons.

High filler loadings are typically necessary to achieve appropriately enhanced thermal conductivity in polymeric matrices. It has been reported that the 7.4 wt% loading of a polymer with graphene caused nozzle clogging in a fused filament fabrication printer (Wei et al., 2015). For vat polymerization, using liquid resin would avoid the problem of the repeated clogging of nozzles or spraying devices due to the introduced nanofillers. This would create a quantum leap in the thermal conductivity of printed products through the high loading of nanofillers. However, vat polymerization remains a significant challenge due to the processing requirements of photoactivity and viscosity. Yang et al., (2022) prepared a nanocomposite with a high loading (60 wt%) of micro silica, resulting in the improved thermal conductivity of $.44 \text{ W m}^{-1} \text{ K}^{-1}$, with 196.91% compared with the pure matrix. Nanocomposites assembled with high thermal conductivity due to closely arranged phonon transportation pathways in resin matrices could be applied to fabrication injection and vacuum casting molding to extend the service life. In addition, the fabrication of ultralow thermal conductivity thermoelectric materials can be realized by mixing thermoelectric materials, including $\text{Bi}_{0.5}\text{Sb}_{1.5}\text{Te}_3$ (He et al., 2015) and Ag_2Se (Park et al., 2021) materials, with photosensitive resins.

Incorporating nanoparticles also offers the possibility of improving the thermal stability of vat-polymerized printed fabrications. However, it should be noted that the thermal stability of the composite is influenced by the organic modifier, filler content, and structural features of nanocomposites (Leszczyńska et al., 2007). The introduction of nanoparticles can also effectively enhance the thermal resistance of nanocomposites (Chiu and Wu, 2008). Zhang et al., (2015) and colleagues dispersed SiO_2 nanoparticles, which modified the silane coupling agent, into a photosensitive resin by ultrasonic and a planetary ball mill treatment, followed by 3D printing. The glass-transition temperature of the nanocomposites increased from 67.28°C for the pure resin to 80.18°C for the nanocomposites with the addition of .7 wt% SiO_2 . Similarly, the addition of a 1D nanomaterial, microcrystalline cellulose, had a formative influence on the thermal properties of the photosensitive resin, which increased the degradation temperature of the nanocomposite, and the β -relaxation of the photosensitive resin tended to move toward higher temperatures (Han et al., 2017).

3.3 Conductivity enhancement

3D printing has been used as an alternative approach for manufacturing conductive composites due to the advantages of reduced cost and eco-friendly features, which are of unique interest in various functional applications, such as electrodes (Ahn et al., 2009; Rymansaib et al., 2016), wearable devices (Hao et al., 2021), and sensors (Leigh et al., 2017). This method balances a low-cost, fast, and versatile production process with the excellent performance of the resultant products.

Metal fillers with good conductivity have been used for conductive materials in vat polymerization. Fantino and coworkers (Fantino et al., 2016) introduced silver nitrate to a photosensitive resin. The silver nitrate was reduced to silver nanoparticles *via* a UV post-curing process after DLP printing to prepare conductive nanocomposites. The direct loading of the filler affected the electrical conductivity of the composites: Increased amounts of silver nitrate increased the silver nanoparticle content in the matrix, thereby promoting the formation of charge movement paths between the nanoparticles, which led to a

decline in the electrical resistance of the nanocomposites. However, due to its prohibitive cost and ease of oxidation, the application of metal has been limited.

In comparison, carbon fillers exhibit considerable stability and high conductivity, which makes them easier to obtain and more widely used (Gonzalez et al., 2017; Zhang et al., 2017). Therefore, CNTs are widely used due to their excellent electrical properties arising from the freedom of the movement of unpaired electrons in orbitals perpendicular to the π - π plane within the plane. Mu et al., (2017) and colleagues combined commercial photosensitive resin with MWCNTs to obtain conductive composite structures using a DLP printer. To optimize printability and conductivity, .3 wt% of MWCNTs was added, increasing the nanocomposite conductivity to $.027 \text{ S m}^{-1}$. Guo and coworkers (Guo et al., 2020) prepared a DLP-printed conductive nanocomposite with 2 wt% carboxyl CNTs that exhibited an electrical conductivity of $.13 \text{ S m}^{-1}$. These shreds of evidence indicated that the composites with high conductivity, high gauge strain sensors, and superior cycling stability could provide stable signal information for human motion monitoring.

The use of graphene is also an effective strategy for increasing the electrical conductivity of photosensitive resins. Since GO has better compatibility with photosensitive resins, GO can be used directly as a filler. It is reduced to graphene during post-processing after printing, and different post-processing processes affect the electrical conductivity of the samples (Chiappone et al., 2017). Nanocomposites post-cured by UV light exhibit a non-linear electrical correspondence due to uneven internal curing, while homogeneous thermally post-cured models show a linear electrical response. In addition, the combination of 1D and 2D nanofillers provides effective conductive pathways in nanocomposites and has been used to produce conductive structures (Han and Cho, 2018).

4 Challenges and outlook

Although numerous designs of vat polymerization-based 3D printed nanocomposites have been prepared, there is still a gap in meeting the demands of industrial applications, and many impediments, from molding craft to product performance, remain unsolved. First, the viscosity of photosensitive resin tends to increase with increased nanofiller content. Meanwhile, the polymer self-diffusion coefficient is reduced (Mu et al., 2009), challenging the leveling properties of photosensitive resin and thus affecting the surface quality of printed products (Gurr et al., 2008). Second, the random scattering of UV light in a photosensitive resin observed in vat polymerization has been attributed to either the aggregation of nanoparticles or the incorporation of overlarge nanofillers (Cho et al., 2005), resulting in reduced UV penetration depth and lateral resolution. Nevertheless, it should be noted that slower speeds and/or higher laser power are required to remedy the degeneration of curing depth and group conversions (Cheah et al., 1999). Third, it is difficult to disperse nanofillers uniformly with high surface energy in photosensitive resins, and weak interfacial interaction usually leads to poor performance of the fabricated parts. At the same time, the density of nanofillers and photosensitive resins usually differs, resulting in a tendency to settle in the resin without forming a stable dispersion. Therefore, the chemical compatibility of nanofillers with photosensitive resins must be considered to improve the stability of the resins, which contain nanofillers, and enhance the performance of printed parts.

Overcoming the current bottleneck in the vat polymerization-based 3D printing of nanocomposites requires a further reduction in the production costs of nanofillers and the improved compatibility of nanofillers and resin matrices. These aspects will undoubtedly result in the excellent performance and competitiveness of fabricated products towards the further development of the main directions. Various new vat-polymerized nanocomposites with outstanding performance, including magnetic, catalytic, and dielectric properties and so on, should be developed to meet new frontiers and evolving technology requirements. The evaluation criteria of vat-polymerized nanomaterials are necessary to establish and improve, favoring the development of new composites and selecting suitable materials for various applications.

Author contributions

YL investigated and processed the data, pointed out the framework of the paper, made a summary and analysis, supported the project and completed the writing of the article. WW and FW participated in proofreading and part of the writing. At the same time, RKK reviewed the paper.

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