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ADDITIVE MANUFACTURING (AM) OF POLYMER NANOCOMPOSITES: APPLICATIONS AND CHALLENGES

Physics

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ABSTRACT

Polymer nanocomposites have attracted increasing interest in research and development with several current and potential industrial applications due to their wide margin of superiority over conventional materials. Polymer composites provide a higher strength-to-weight ratio, easily customizable product properties, flexible manufacturing processes, high resistance to corrosion or erosion, and lower cost. The recent progress in additive manufacturing (AM) methods has paved the way for even a broader range of flexibilities in design and materials in several industrial sectors, including aerospace, biomedical, construction, electronics, telecommunication, mechanical, and defense. However, some hindrances remain in the synthesis of polymer composites and their fabrication through AM technologies. A comparative review of AM processes for polymer composites and their applications is presented in this study. This study aims to provide with an updated understanding of the underlying issues, barriers, limitations, and opportunities. It will also help to systematically reveal the research problems and future directions related to materials synthesis and AM processes.

KEYWORDS

Additive manufacturing, 3D printing, Polymer composites, Nanocomposites, Fused filament fabrication, Powder bed fusion, Vat photopolymerization

1. INTRODUCTION

There has been an increasing need, hence a research interest, to utilize polymer composites. Polymer composites provide flexibility in terms of final product properties, design, starting raw material options, and continuously expanding progressive innovations in synthesis techniques, in addition to their wide commercial availability and cost-effectiveness compared to other materials, such as metals and ceramics, for low-cost applications. They are becoming prime choices even for critical applications (e.g., aerospace, automotive, and medical device) due to enhanced and tailored properties. Polymer-based nanocomposites provide a wide margin of superiority over conventional materials due to their higher strength-to-weight ratio, easily customizable product properties, flexible manufacturing processes, and high corrosion resistance properties. Furthermore, the recent development of AM methods has provided a new production era in several industrial sectors, including biomedical, construction, electronics, telecommunication, mechanical, and defense. AM processes allow a higher degree of freedom in the design and fabrication of customized parts, rapid manufacturing, waste reduction (in some cases elimination), lower chances of human error, high precision, and accuracy at low costs.

Generally, polymers exhibit inferior mechanical, thermal, and electrical properties compared to metal and alloys for mechanical strength, thermal conductivity, and electrical conductivity, which are prime considerations for selecting materials in various applications. These properties can be improved by adding suitable additives to polymers to synthesize polymer composites of an unlimited number of kinds and properties. Combining such unlimited flexibility of polymer composites with AM fabrication provides rapid, inexpensive, efficient, and multi-functional production and products.

Despite extensive research in synthesis and AM of polymer nanocomposites, some limitations persist, requiring further investigation. For example, particles-reinforced polymer composites face problems of inhomogeneous dispersion, inconsistent feedstock material, unlike the nature of most filler material to polymers, and little awareness of the shape and size of filler material. Resolution, repeatability, and printability need to be enhanced by optimizing AM process conditions and starting material parameters together. Finally, interfacial interaction/bonding, the material's composition, and structural defects need to be analyzed in detail to ensure excellent product performance.

In this article, an up-to-date review of polymer nanocomposites, their manufacturing potentials, issues, and opportunities using AM (also widely known as 3D printing- 3DP) technologies and their applications in different sectors will be discussed.

2. Additive manufacturing (AM) processes

Several AM processes currently exist, which have been utilized for rapid prototyping applications. Depending upon the working principles, AM processes can be broadly classified into seven categories, vat polymerization (VP), powder bed fusion (PBF), material extrusion (ME), material jetting (MJ), binder jetting (BJ), directed energy deposition (DED), and sheet lamination (SL). Generally, AM technologies use computer-aided design (CAD) models. Cross-sections of the geometries are defined from these CAD models, and material is added to each layer to make a tangible and intricate part. This article focuses on three AM techniques: vat polymerization, powder bed fusion, and material extrusion, as these processes are most widely utilized for polymer nanocomposites. Plenty of materials have been developed for each AM process commercially available with AM technologies in the market.

Vat Photopolymerization (VP):

The vat photopolymerization process is an AM technique in which photo-sensitive materials are exposed to radiation/light in a controlled manner to obtain polymerized material layers. Subsequent layers combine to form a 3D object; however, this process's application is limited to the materials that polymerize on exposure to light. Photopolymers and resins can be processed using this technology. Depending upon the variation in curing source, VP processes can further be classified into; Stereolithography (SLA), digital light processing (DLP), two-photon polymerization (2PP), and volumetric 3D printing.

SLA photopolymer resins mainly consist of monomers, oligomers/ binders, photoinitiators, and some additives. Monomers and oligomers are the main constituents of photopolymer resin, which solidifies due to crosslinking. Photoinitiators convert into radicals and react with oligomers and monomers, providing crosslinking to produce polymer chains on exposure to curing light. Additives may include nanoparticles or pigmentation for improved resin properties or desired coloring.

Digital light processing (DLP) also relies on UV light-induced polymerization of the resin. The significant difference between these two technologies resides in that DLP induces an instantaneous entire layer polymerization using digital micro-mirrors devices (DMD). In contrast, in SLA, a single laser point (e.g., layer pixel) is exposed at a time. As a result, DLP offers a higher printing speed than SLA and other technology competitors.

The applied light source has also evolved from standard lamps to light-emitting diode covering, thus, a more comprehensive range of wavelengths at a lower cost. The hardware setup for DLP offers higher lateral printing resolution. The z-resolution is tightly correlated with light penetration and scattering, which can be improved by adding light absorbers and polymerization facilitators. DLP achieves a resolution of about 1 μm (while the printing speed is about 30mm3 s−1) regardless of the layer's lateral area and complexity.

Material extrusion (ME):

In extrusion-based processes, the semi-solid liquid is extruded on a build surface through a nozzle, solidifying in an extruded shape, with further layers extruded sequentially to obtain a solid part. The most commonly used technique under this category is fused filament fabrication (FFF), also known as fused deposition modeling (FDM). In the FFF process, the material is supplied in the form of filaments. It is heated in a heating chamber to attain the required viscosity and extruded through a nozzle over a platform. FFF sales in 2016 shared 96% of the global AM market; therefore, it is the most widely used AM technology. Thermoplastics, metal particles reinforced polymers, and hydrogels have been fabricated using extrusion processes. Generally, the feedstock material in FFF is provided in the form of a filament spool. A mechanism of roller and gear is used to feed filament material into a heating chamber, which heats and deposits the material onto the build platform. FFF 3D printers may have dual extruders to allow multi-material fabrication or 3D fabricates support structures for overhanging structures.

Powder bed fusion (PBF):

In powder bed fusion processes, the powdered material is carried in a bed, and a layer of material is solidified using a heat source. Once the fusion of one layer is performed, a new layer of powdered material is added for the subsequent layer. Different heat sources are utilized to perform the melting or sintering process of powder. Selective laser melting (SLM) and selective laser sintering (SLS) processes utilize the laser source, an electron beam is used in electron beam melting (EBM), and a thermal print head performs the function of material sintering in the selective heat sintering (SHS) process. PBF processes provide higher resolutions compared to extrusion-based techniques. Polymer powders, metal alloys, and ceramics can be processed using these processes.

SLS is the most widely used PBF process, in which 3D objects are fabricated using powder material in a layer-by-layer manner. Thermal energy (focused laser radiations) selectively fuses the material depending upon the design of the fabricated object. SLM process differs from SLS in terms of solidification of material and resulting bonding mechanisms. SLM process relies on the melting of the powdered material to the liquid phase; however, in SLS, partial liquefaction is practiced. Different laser sources are implemented depending upon the absorptivity of the powder material. CO_2, Nd: YAG, short-pulse Cu-vapor, and fiber laser are the most widely reported thermal energy sources for SLS.

PBF processes do not require building support material, as the unsintered powder material acts as support material to complex or overhanging structures. Besides, this material can be recycled for use in the fabrication of parts without significant changes in properties. The major drawback of these processes is post-processing treatments for better surface finish and heat-treatments for improved mechanical properties.

3. Additive manufacturing of polymer nanocomposites

The commercially available polymers for AM cannot perform adequately in high-performance applications. Therefore, it is vital to synthesis functional polymer materials compatible with AM processes discussed above. Generally, polymers do not exhibit significant mechanical, thermal, and electrical properties as they do not demonstrate substantial strength, heat transfer capabilities, and conductivity. Therefore, they are altered by introducing filler particles or reinforcement to achieve desired properties for functional applications.

Polymer nanocomposites are obtained by adding nanofillers dispersed within the polymer matrix. These nanoparticles typically lie in the nanometer range and act as reinforcement to achieve functional properties. These materials have attracted the research focus due to their extraordinary enhancement in mechanical, thermal, electrical, or chemical properties even at very low reinforcement content**.** Polymer nanocomposites provide an added benefit of homogeneity than fiber-reinforced composites, as they are less prone to anisotropic effects induced due to uni-directional reinforcement. Various nanoparticle-based composites have been developed for AM processes and mainly focused on improved mechanical and electrical properties. For example, nanoclay for improved mechanical, carbon nanotubes, and graphene for improved electrical conductivity are reported in the literature.

Polylactic acid (PLA) – based polymer nanocomposites:

Polylactic acid (PLA) is a thermoplastic polyester, generally obtained from biodegradable resources, and exhibits excellent mechanical and thermal properties, good processability, and low impact on the environment. PLA is globally accepted as biodegradable due to its origin from plants and has proven safe for biomedical applications and food industries. Therefore, being explored in tissue engineering, biomedical implants and food packaging sectors for their performance and processing through AM processes.

Polycaprolactone (PCL) – based polymer nanocomposites:

PCL is also an approved biodegradable material by the US Food and Drug Administration (FDA); therefore, its applications in biomedical engineering have been explored. Some processes are also reported in the literature to 3D print this material owing to its suitability in bone tissue engineering. PCL matrix containing the bioactive reinforcing phase of HA and strontium substituted HA (SrHA) were printed directly, eliminating the need to produce filaments, thus avoiding a multi-step manufacturing process. The resulting composites with varying reinforcing content were characterized for their physical, mechanical, and biological properties. Different architectures were designed to mimic the bone tissue structure, and printing parameters were optimized for different material configurations

High-density polyethylene (HDPE) – based polymer nano composites:

HDPE is the most widely used polymer in the consumer industry. It exhibits a high strength-to-density ratio as compared to LDPE. However, HDPE's two most essential concerns include; its reusability and lower mechanical strength. HDPE-based composite filaments incorporated with cardboard dust particles were prepared for the FFF process to overcome the problem of recycling. Different cardboard dust contents (20, 50, and 75%) were added to HDPE, and resulting nanocomposite filaments were analyzed for their microstructural and mechanical properties. The filament was extruded successfully without considerable deviation in diameter and density for lower cardboard content.

Acrylonitrile butadiene styrene (ABS) – based polymer nano composites:

ABS is an exciting material for the fabrication of polymer composites; due to its solubility in acetone, which assists the synthesis of composites, it can hold higher filler content and is easily extrudable. It has the advantages of good flexural and impact strength, resistance against the chemical environment, and good physical stability. However, to improve its thermal and electrical performance, the addition of metallic particles to ABS evidences its utilization in electrical and thermal conductivity applications

Polyvinylidene fluoride (PVDF) – based polymer nanocomposites:

Typically, the extrusion process for PVDF is challenging due to the high coefficient of thermal expansion. The inclusion of materials with a low thermal coefficient could make it feasible for AM by tailoring the composite material properties. Zirconium tungstate facilitated the provision of improved printability of the composites by lowering the overall thermal expansion. Although improved dimensional accuracy was achieved using reinforcing components, it was attained at the expense of reduced mechanical strength and elongation compared to pure PVDF.

Thermoplastic polyurethane (TPU) – based polymer nanoco mposites:

TPU is a biocompatible material with excellent mechanical properties and is widely used as a coating material to biomedical implants; however, it can be further enhanced with nanoparticle reinforcement for functional applications.

Nylon–based polymer nanocomposites:

Polyamide (also referred to as Nylon) is utilized due to its significant mechanical properties. Nylon reinforcement with nanoparticles can provide heat and chemical resistance. Nylon 12, also known as polyamide 12 (PA12) exhibits a semi-crystalline structure and is the most widely used material in PBF processes due to its higher melting point and glass transition temperature. PA12 can provide high impact strength but prone to be affected by moisture.

Photopolymers & epoxies – based polymer nanocomposites:

Photopolymers are light-sensitive materials, normally polymer monomers, which solidify on exposure to radiation. The range of wavelength, which causes these materials to cure depends upon their nature. Therefore, a variety of light sources could apply to the vat polymerization technique. In addition to the development of processes, biodegradable materials have attracted researchers owing to their sustainability. One of these green materials is cellulose, which has proven to be a significant reinforcement to polymer-based materials. The vat photopolymerization process was utilized to cure photo-sensitive PU epoxy.

Shape memory polymer composites (SMPCs):
There has been an increasing interest in shape memory polymers (SMPs) to synthesize composites for functional applications. These materials can regain their original shape after deformation caused by any external stimuli. SMPs can be reinforced with nanoparticles to obtain shape memory nanocomposites with improved recovery mechanisms, mechanical properties, and controlled actuation. SMP composites have a massive potential for their applications in aerospace, biomedical, electronics, and 4D printing.

4. Applications

Biomedical sciences:
AM processes are being rapidly employed in the biomedical field for tissue engineering and patient-specific implants. Different biomedical science materials include bio-inks, biomaterial inks, synthetic hydrogels, thermoplastic resins, ceramics, and metals. The materials for such applications must exhibit biocompatibility and significant mechanical properties. Medical applications require implants, in most cases, with biocompatible materials, complex shapes, and a high degree of customization. In tissue engineering, precise control over porosity and fabrication of interconnected networks is quite challenging. FFF processes have been used for better dimensional and porosity control.

3D printed PLA/HA trabecular bone models displayed comparable mechanical strength compared to commonly used polymeric foam bone models.

Ongoing research also aims to adopt the bio-inspired design to the medical field to synthesize soft materials (polymers, rubber, and leather) that can be utilized for the fabrication of delicate organs and tissues. In this regard, AM would be a potential candidate for integration to realize advanced materials. Besides, research has also focused on the fabrication of smart materials for 4DP, which can change their shape over time, which has attracted biologists' and material scientists' attention for their potential use in biomedical applications.

AM has found widespread use in biomaterials, regenerative medicine, tissue engineering, drug delivery, and laboratory device prototyping. In the medical field, AM can also be used for aims such as teaching, functional flow modeling, procedural planning, and device engineering. Although 3DP medical applications are still in early-stage, past and current studies show promising results and imply a bright future for the field. One of the main advantages of employing AM in the medical field is its ability to produce patient-specific devices and therapeutic approaches.

Construction:
The development of AM processes in the construction industry has recently urged researchers to develop novel materials, designs, and procedures due to limitations posed by the utilization of traditional construction materials in current AM processes. Polymers and their composites are being considered as an alternative to conventional materials and reinforcements. 3D Printed ABS structures were utilized as reinforcements to conventionally casted fine-graded cement mortar.

Electrical/Electronics:
The outlook for producing advanced electronic components and systems using traditional manufacturing processes is grim, and such production might even be impossible. Therefore, AM processes are being utilized for cost-saving and environmentally friendly printed electronics. AM techniques facilitate wide electronics applications, including the printing of resistors, capacitors, transistors, and circuits on a flexible substrate, as well as the production of flexible wearable gadgets and a wide variety of mechanical, optical, electromagnetic, medical, and chemical sensors. AM processes can fabricate micro-level monolithic devices; however, the applicability of these processes is limited, owing to materials' biocompatibility. Polymer composites responsive to the magnetic field are now widely employed in the electrical and electronics sectors. Titanium dioxide and calcium copper titanate polymer composites fabricated for the vat photopolymerization process revealed higher resolution, improved dielectric permittivity, and no dispersion at high frequencies. Materials with such properties could be utilized to manufacture electromagnetic devices operating at higher frequencies.

3D printing processes capture the interest in manufacturing telecommunication components with complex geometrical structures that are difficult, if not impossible, to realize with traditional manufacturing techniques.

Mechanical Engineering:
Typically, polymers exhibit inferior mechanical properties, thus limiting their direct utilization for structural or thermal applications. Therefore, different composites have been fabricated and tested for their mechanical and thermal performance. In this regard, HDPE/cardboard dust composites could be a sustainable and economical solution to low-cost engineering applications, where high strength is not required. These materials could be utilized in architectural/civil models, interior designing, and indoor paneling.

Aerospace/defense:
The aerospace industry has adopted AM processes to manufacture lightweight and high-performance polymer composite spare parts, as weight reduction in the aerospace industry leads to lower fuel consumption and cost savings. There is a high degree of freedom for producing custom parts. AM techniques are being used to manufacture jet engines and commercial plane parts, owing to the flexibility of the processes, the ability to design and realize complex structures with internal linings, customization of components, waste material reduction, multi-material utilization, and *in-situ* fabrication.

FFF can provide quicker and cheaper manufacturing of complex components of unmanned ariel vehicles (UAVs). Most of the parts for this lightweight UAV were fabricated through the FFF technique.

Magnetite reinforced polymer-based composite materials can absorb different wavelengths. These materials can be utilized to manufacture submarines and drones to avoid radars. Hollow glass spheres reinforced polymer composites demonstrated very low dielectric constants for many frequencies. Such materials could also provide appealing applications in the military and defense sectors.

5. Challenges

A most critical requirement in feedstock material synthesis is the homogenous dispersion of reinforcement particles within the matrix, which plays a significant role in the composite's resulting properties. Also, the accumulation of filler particles could affect the process, uncured photopolymer in the VP process, and the nozzle's clogging in the ME process. The uniform distribution is vital to achieving the required results and avoiding local stress concentrations, which result in product failure. Nanoparticles play an influential role in improving mechanical properties; however, it comes at the cost of reduced ductility or failure strain in nanocomposites due to poor interfacial adhesion and agglomeration of nanoparticles. The agglomerated nanoparticles act as stress concentrating defects, resulting in a deviation from expected material properties. Homogenous dispersion of nanoparticles is vital for optimum performance of the nanocomposites. A high-shear mixing process, like a twin-screw extruder, should be utilized to ensure adequate dispersion.

The physical phenomena involved in AM process result in nanoparticles' alignment in the printing direction. However, depending upon the functional application of nanocomposites, it could be beneficial to achieve improved electrical or thermal conductivity in the direction of oriented nanoparticles.

Printing orientation strongly affects the mechanical properties of 3D printed nanocomposites. Anisotropic effects induced due to printing direction are widely reported**.** The mechanical properties of vertically printed parts can reduce up to five times than the parts printed in XYZ direction. However, tailored mechanical properties with lower mechanical anisotropy can be achieved using a polymer blend.

The nature of filler material also affects its homogeneity within the composite matrix; polymers are generally hydrophobic. It is difficult, if not impossible, to mix a polymer solution with hydrophilic filler. Graphene, for example, can be dispersed using an aqueous suspension of Nano-fibrillated cellulose and so can be used in cellulose composites. Graphene has been proven to be an excellent filler material and promising for mechanical, thermal, and electrical applications. Therefore, it requires innovative approaches and processes for their proper utilization. The grafting of polymers has been demonstrated to be an effective method to improve dispersion. However, there remain some challenges in the effective distribution of particles within the polymer matrices.

Process conditions, including printing temperature, ambient temperature, infill pattern, infill density, and printing speed, significantly impact the printed part's properties. Processes carried out under high temperatures would lead to fewer voids and porosity. The printability of material is an essential factor for consistent printing. Simple polymers can be printed conveniently; however, AM of composites is challenging for several reasons, such as gradients in temperature and flow characteristics of composite materials.

6. CONCLUSION:

Research in material science and AM processes has urged scientists to quest for novel materials for functional applications, resulting in the synthesis of advanced nanocomposites with higher design flexibilities. It is worth mentioning that the AM processes should not be considered the full replacement to conventional manufacturing processes; instead, they can complement these processes for a synergic output. However, to utilize the advantages offered by AM processes, the designers must follow the design for AM (DfAM) approach. In this way, the interlinked parameters of process-material-design can serve several industries, including aerospace, automobiles, biomedical, and electrical, in a better way.

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