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Research Article

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Manufacturing methods of functionally graded materials: a comprehensive review

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Highlights

- Introduction
- FGM manufacturing methods
- Classification of FGM manufacturing methods
- Comparison of FG manufacturing methods

Abstract

Various problems arise in traditional composites and the gradient of these materials needs to be controlled for more specific purposes. At this stage, functionally graded materials, which are a more specific area of advanced composites, come into play. High strength-to-weight ratio, wear resistance, thermal insulation, controlled porosity and many other features can be obtained by using functionally graded materials. A wide variety of functionally graded materials and their manufacturing methods are available. In this study, the classifications of properties of functionally graded materials and manufacturing methods in the literature are examined. The basic principles for each method are presented. In addition to the advantages and disadvantages of the methods, the difficulties in the production phase are also examined.

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1. Introduction

Composites, produced by combining two or more materials, offer versatile properties for various applications. Conventional composites generally consist of fibers that serve as reinforcement and a matrix medium in which these fibers are dispersed. Although there are lots of advantages, combining different materials can cause problems such as stresses because of the differences in thermal expansion coefficients, residual stresses occurring, and surface incompatibilities after production [1-3]. Functionally graded materials (FGM) offer solutions in various fields. FGMs are used in areas by modifying material properties, such as aerospace, electronics, military industry, biomedical areas, the textile industry and more. FGM is produced by gradually changing the material content in directions. Concrete, industrial tools, ship hulls, microchips are artificial FG materials. Bones, bamboo and teeth etc. can be given as examples of natural FGM structures. Just as the density of bone increases progressively closer to the joints, optimizing its structural integrity for higher strength. Similarly, bamboo demonstrates a gradual transition from a soft, flexible outer layer to a harder, more rigid inner core. The primary advantage of FGMs is to eliminate the stress jumps at interfaces that occur in conventional composites. FGMs have features such as high strength-to-weight ratio, the ability to be designed according to the desired feature, lightness, damping and vibration control, and resistance to high temperature differences. Despite their advantages, FGMs face challenges such as complex manufacturing processes, high costs, and limited applications.

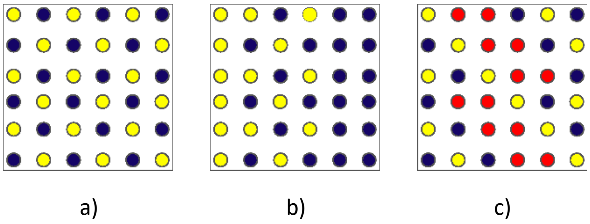

Figure 1. Variation of a) typical composite, b) single FGM, c) double FGM [7]

The mechanical properties of an object produced with an FGM, with the selection of direction or more than one direction; are defined by volume fraction and material gradient. Linear gradients promote uniform stress distribution, while nonlinear gradients can induce more localized stress fields. The differences in characteristics between typical composites and FGMs are shown in Figure 1. In contrast to typical composites, a single FGM

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can be produced by a single dispersed ingredient or phase that is not evenly distributed throughout the matrix [4-6].

Double FGM requires multiple components or phases [7]. Functionally Graded Materials (FGMs) are characterized by variations in their composition and microstructural properties. These variations, which define the material gradient, can be categorized into four main types: fraction gradient, shape gradient, size gradient, and orientation gradient, as illustrated in Figure 2, which depicts different gradient methodologies explaining material gradient [8].

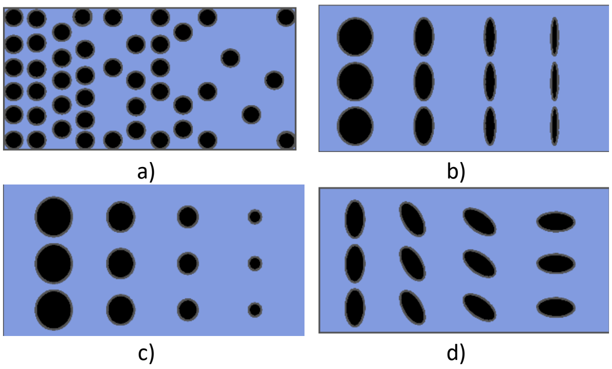

Figure 2. Different gradient types of FGM: a) fraction, b) shape, c) size, d) orientation

FGM, in terms of material composition change; It is divided into three types according to whether it changes ladder, sudden, and gradually, as shown in Figure 3 [9].

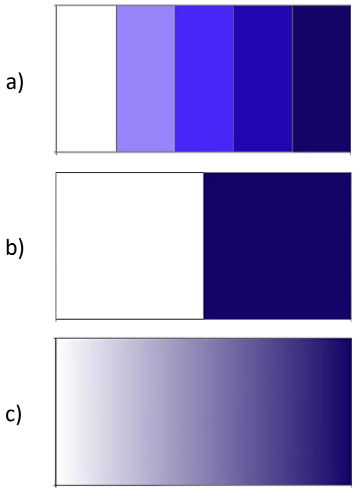

Figure 3. Variation of gradient transition types in FGM: a) ladder, b) sudden, c) gradual

Composition change of the material can be through longitudinal or the thickness of the structure as shown in Figure 4.

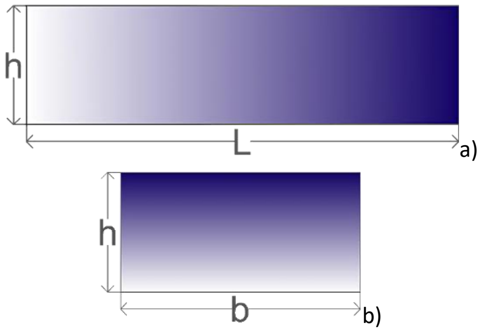

Figure 4. Variation of material properties along a) longitudinal direction, b) thickness direction [10]

Additionally, composition, microstructure, and porosity are the types FGMs based on grading [6,10]. For FGMs with porous structure, porosity can be divided into two different types as shown in Figure 5.

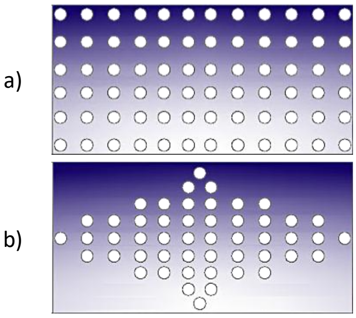

Figure 5. Porous FGM with a) even, b) uneven distribution

In some cases, superior properties need to be achieved, such as thermal stress. Multi-directional (MD) FGMs are designed by changing material properties in more than one direction as can be seen in Figure 6. MD FGMs vary from simple 1D variations observed in layered structures like the thickness of a plate, where material properties gradually transition, to 2D variations like those seen in the surface of a plate with varying thermal conductivity, and complex 3D gradients found in advanced components such as turbine blades with intricate internal cooling channels, where material composition graded to withstand complex stress and temperature gradients [12].

However, the successful realization of FGMs hinges on the development of advanced manufacturing techniques that can accurately control the gradient profile and microstructure.

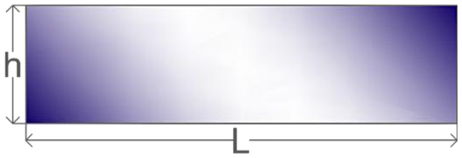

Figure 6. A 2D FGM in which material properties are varied in the longitudinal and thickness direction

This review covers state-of-the-art manufacturing processes for FGMs, including traditional techniques such as vapor deposition methods, and powder metallurgy, as well as emerging additive manufacturing methods such as fused deposition modeling. Furthermore, it compares characteristics of products such as porosity, improved strength, high cost, production difficulty and more. By understanding the strengths and limitations of these techniques, the design and manufacturing of FGMs can be optimized to meet specific performance requirements [12,13-15].

2. FGM Manufacturing Methods

FGM manufacturing methods are comprehensively examined in this section. The strengths and weaknesses of each approach are analyzed, and the characteristics of FGM products produced by different methods (such as porosity, strength, cost, and production difficulty) are compared to guide the selection of the most suitable manufacturing process for specific applications [6].

2.1. Vapor deposition methods

Vapor deposition methods are used for coating purposes to increase material properties, such as corrosion and wear resistance. While offering precise control over composition, these methods can be slow and energy intensive. These methods sometimes produce hazardous by-products and mostly not suitable for bulk production [16-19]. A comparison of these methods is shown in Table 1.

2.1.1. Chemical vapor deposition [CVD]

In CVD, a gas or vapor undergoes a chemical reaction on the surface, forming a solid coating [19]. CVD is widely used in the electronics industry for thin film deposition. A schematic of CVD setup is shown in Figure 7.

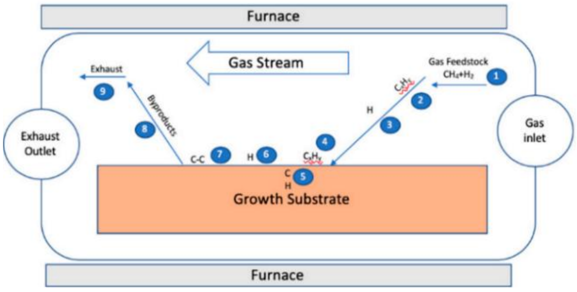

Figure 7. A schematic representation of thermal CVP growth of graphene, taken from [20]

2.1.2. Physical vapor deposition [PVD]

PVD involves deposition onto a surface through physical reactions. Pure coatings can be obtained using PVD. Automative and aerospace industries products made by this method [17]. A schematic of PVD setup is shown in Figure 8.

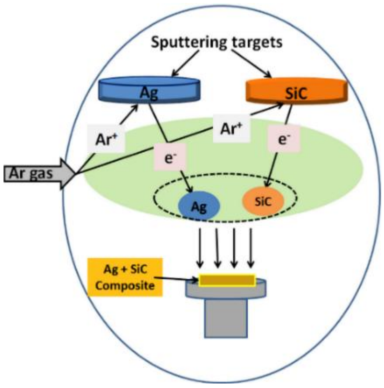

Figure 8. A schematic representation of manufacturing Ag based composite prepared by PVD, taken from [21]

2.1.3. Jet vapor deposition (JVP)

In JVD, a carrier inert gas is used to deposit the material. The vaporized material is directed to the material surface with a high-speed jet flow. It can be applied in a low vacuum environment and that allows use of multiple jet tips, making this method relatively faster [16].

2.1.4. Directed vapor deposition (DVD)

DVD enables the efficient use of electron beams on various materials, including highly reactive metals. The gas is delivered by focusing on a specific area. Provides more controllable and better targeted coverage [18].

2.2. Plasma spray (PS)

PS involves plasma sputtering of metals, ceramics and polymers. Different types of materials can be deposited simultaneously with different melters, but obtaining a homogeneous distribution can be challenging. Polymers, ceramics and metals can be coated. Additionally, PS can be combined with vapor deposition and other methods [20-22].

Table 1. Comparison of vapor deposition methods [16-21]

Method	Advantages	Disadvantages
CVD	Suitable for thin films, req. low temps. proc.	Complex equipment, toxic byproduct
PVD	High quality coatings, strong adhesion, low cost	Low deposition. rate, unfit for comp. geometry
JVD	Fast coating, suitable in low vacuum conditions	Complex equipment, energy consumption
DVD	Precision coating, high efficiency	Complex equipment, high cost

Table 2 presents an overview of the temperature ranges and typical precursors employed in various vapor deposition techniques. CVD is operated within a range of 200-1600°C, depending on the reaction temperature. PVD has a very broad temperature range, 25-1200°C. JVD is generally operated between 100-300°C for organic molecules, and it can increase for inorganics. DVD is operated between 200-600°C [16-19,23,24].

Table 2. The comparison of applied temperature range and precursors of vapor deposition methods

Method	Temperature Range (°C)	Typical Precursors
CVD	200-1600	Halides, Metals, Organics
PVD	25-1200	Metals, Ceramics, Alloys
JVD	100-300	Organic/Inorganic Molecules
DVD	200-600	Organic/Inorganic Molecules

2.3. Powder metallurgy (PM)

PM is used in the production of metals with varying melting points. The method involves combining, compressing and sintering two or more different materials, resulting in a porous structure. High cost of powder, and relatively low strength are drawbacks of this method. However, it results in fewer defects and cracks. PM enables the production of metal-ceramic composites, as well as machine parts and magnetic materials [28-31]. A schematic of PM process is given in Figure 9.

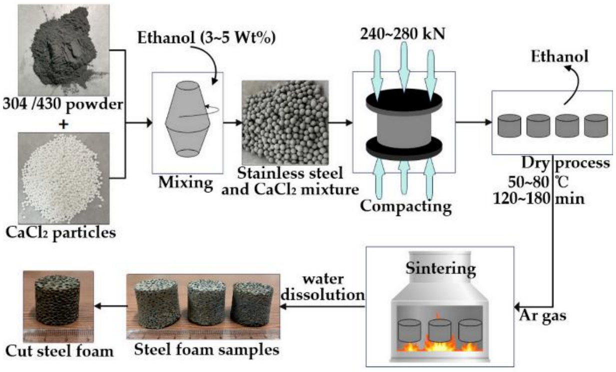

Figure 9. A schematic representation of manufacturing stainless steel foam using PM, taken from [32]

2.4. Self-propagating high-temperature synthesis (SHS)

SHS is a method based on an exothermic reaction occurring in an ignited mixture. It is applied to ceramics, metal alloys, and nanocomposites. Metallic foams are used in the production of ceramic composites and for surface hardening of metals. Although SHS requires higher temperatures and longer processing times relatively, its results are more pure products [26,33-35].

2.5. Friction stir processing (FSP):

FSP is a solid-state processing method used to modify the microstructure of metal. A tool is immersed into the workpiece and rotated to generate frictional heat, the resulting heat and pressure cause the material to deform plastically, forming a new microstructure with new properties. FSP offers detailed control over the materials microstructure, resulting in improving its mechanical properties, while at the same time providing relatively less time and low energy consumption. The method is used in the production of metal and ceramic-reinforced composites, and graded foams. However, surface defects on product and wear may occur in the tools used [36-37].

2.6. Casting methods

Casting methods offer ability to produce different shapes and varying sizes, low tooling costs, and high production rates, but have limited microstructure control, potential for defects, and often require post-processing. A general comparison of casting methods is provided in Table 3.

2.6.1. Centrifugal casting

In this method, molten metal mix is poured into a rotating mold and subjected to high-pressure centrifugal force. The material is graded by utilizing the effect of rotation and the density difference of the materials. It provides continuous grading, can be applied to cylindrical shapes and is suitable for large-scale casting [38-40]. A schematic of centrifugal casting method is given in Figure 10.

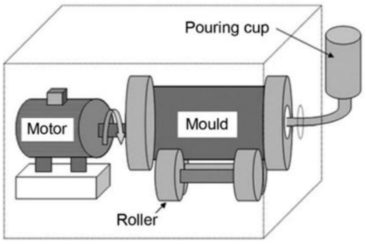

Figure 10. A schematic representation of Centrifugal Casting setup, taken from [41]

2.6.2. Tape casting

Tape casting is frequently used in the production of ceramic materials as thin layers. It also enables producing flat and thin parts over a wide area. It is particularly effective in the production of gradient materials thinner than 1 mm [42].

2.6.3. Slip casting

In this method, a pre-prepared liquid poured into a porous mold. The liquid is absorbed by the mold, leaving behind a solid layer. This process is often used for producing complex shapes and large volumes of ceramic products. Slip casting can be combined with centrifugal casting method, magnetic field or pressure. Pressure slip cast ceramics show improved mechanical properties [43].

2.6.4. Gel casting

In this method, a prepared powder is mixed with monomer solution and poured into a mold. After, the molded part is rested to be dried and proceeds to sintering. Viscosity during casting can be changed by applying heating or cooling during production. This method is environmentally friendly and can create complex shaped products [44-46].

2.6.5. Freeze casting

Freeze casting is used to obtain mostly dense ceramic that has highly controlled, complex pore structures. Metals, ceramics, polymers and composites are used in this method. The drying process of this may be time consuming. It has applications in energy storage and biomedical field [47].

2.6.6. Cast-decant-cast (CDC)

CDC based on casting layers with different material properties on top of each other. Gradient thickness can be controlled, and the thickness of the layers is limited. CDC is unsuitable for complex geometry production. Mold can be used repeatedly [48-49].

Table 3. Comparison of casting methods [34-49]

Method	Advantages	Disadvantages
Centrifugal Casting	Fit for cylindrical components, high production rates	Limited to cylindrical shapes, specialized equipment is needed
Tape Casting	Fit for thin, flat components, thin-film gradients	Limited to simple shapes, post-process is required
Slip Casting	Fit for complex shapes and large volumes, can be combined	Time-consuming, slurry control is important
Gel Casting	Eco-friendly, Fit for complex shapes, controlled viscosity	Sensitive to conditions, post-process is required
Freeze Casting	Fit for porous structures, various materials	Time-consuming, and specialized equipment is needed
CDC	Simple, inexpensive, controlled layer thickness	Limited to simple shapes and gradient profiles

2.7. Additive manufacturing methods (AM):

Complex structured products and controlled microstructure can be obtained by AM which product is modeled by CAD or CAM. An after process may be required for these methods. A comparison of AM methods, and the materials being used for each method, as well as type of process applied are given in Table 4 and Table 5, with respect. Some of the AM methods, e.g. FDM, are slow and energy consuming when considered volume produced [6,45,50].

2.7.3. Stereolithography (SLA)

SLA uses laser to selectively cure a liquid photopolymer resin layer by layer. It is able produce 3D complex models. Only one material can be printed at a time and the product may need to be supported during the process [50-52]. SLA is fitting for producing prototypes and biomedical applications. A typical SLA machine can be seen in Figure 11.

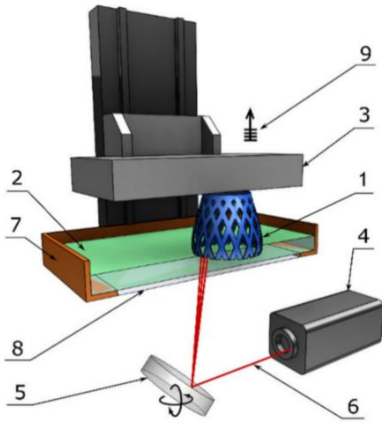

Figure 11. Components of A typical SLA machine: 1) Printed part, 2) liquid resin, 3) building platform, 4) UV laser source, 5) axis scanning mirror, 6) beam, 7) resin tank, 8) window, and 9) elevation layer-by-layer, taken from [53]

2.7.2. Solid ground curing (SGC)

SGC is curing a liquid photopolymer resin layer by layer using a projector. It's a laser-based (LB) relatively fast process but suffers from accuracy issues and material limitations [50,51].

2.7.3. Liquid thermal polymerization (LTP)

In LTP, layers of heat-sensitive polymers hardened layer by layer using laser heat. There are few research on this method in the literature [50,51].

2.7.4. Beam interference solidification (BIS)

BIS uses two laser beams to solidify liquid resin. The interference pattern of the beams creates a 3D structure within the resin. This technique is also still under development and has not yet been commercialized [50,51].

2.7.5. Holographic interference solidification (HIS)

By exposing the resin to the holographic image, the entire surface is solidified in HIS. This method hasn't been commercialized [50,51].

2.7.6. Fused deposition modelling (FDM)

FDM extrudes molten materials layer by layer to create 3D objects. This process is commonly used to produce lightweight structures. Widely used in aerospace, automotive, and biomedical applications [48,49]. FDM has lesser precision compared to SLA [54-56]. A modified schematic of FDM 3D polyether ether ketone (PEEK) printer is given in Figure 12.

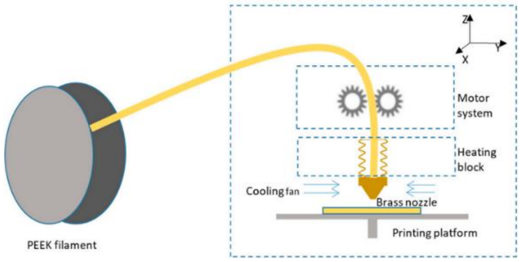

Figure 12. A schematic representation of FDM 3D PEEK printer, modified and taken from [57]

2.7.7. Material jetting [MJT]

MJT deposits tiny droplets of liquid material onto a build platform. These droplets, typically photopolymer resins, are cured with UV light to form solid structures [58]. Widely used in biomedical applications and investment casting. A schematic of MJT is given in Figure 13.

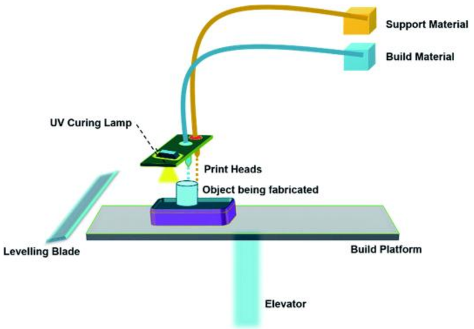

Figure 13. A schematic representation of MJT, taken from [59]

2.7.8. Binder jetting [BJ]

BJ selectively deposits a liquid binder onto a powder bed to create a 3D object. Binder jetting relies on a binding agent to adhere the particles together. It is not suitable for structural parts [58]. BJ is used to produce large cast molds and low-cost metals.

2.7.9. Selective laser melting (SLM)

In SLM, a high-powered laser is used to fuse metal powder layer by layer. This technology is increasingly being used to fabricate porous metal implants, which mimic the structure of natural bone and promote better integration with body [60]. SLM is widely used for automative and aerospace, printing metal alloys. A schematic of SLM is given in Figure 14.

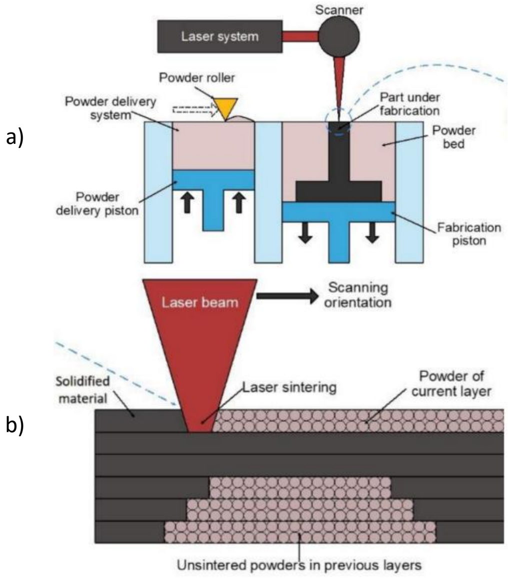

Figure 14. A schematic representation of a) SLM and b) closer look to laser-substrate region, taken and modified from [62]

2.7.10. Selective laser sintering (SLS)

SLS is a time and cost saving manufacturing process where a laser beam is used to selectively fuse powdered material under the melting point, mostly polymers, layer by layer then creating a 3D object such as detailed automative and aerospace parts. Due to sintering, the product has a porous structure, and the mechanical strength of material is lower in SLS than in SLM. [61,62].

2.7.11. Laser cladding (LC)

LC is based on melting metal powder with a high-powered laser and depositing it onto a substrate layer by layer. LC is mainly used for surface modification, repair, and coating applications of ceramics, metals and composites [63,64]**.** A schematic of LC setup is given in Figure 15.

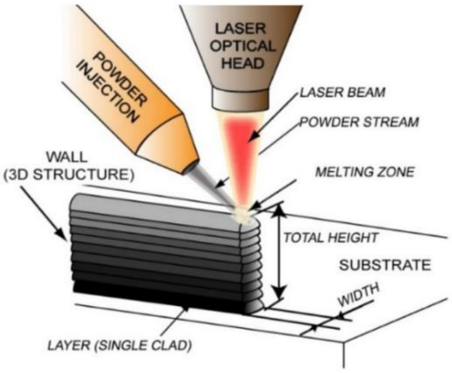

Figure 15. A schematic representation of LC setup, taken from [64]

Table 4. Comparison of AM methods [50-52,58,60,61]

Method	Advantages	Disadvantages
SLA	Detailed surfaces, biocompatible materials	High cost, limited volume
SGC	Fit for large parts	No commercial use
LTP	Fit for large parts, fast production	Less accuracy,
BIS	Fit for complex geometries, high precision	No commercial use
HIS	Fit for large surfaces, fast production	No commercial use
FDM	Desing flexibility, low cost	Slow production, poor surface quality
MJT	Fit for complex geometries, high precision, variety part shapes	High cost, slow producing, low strength
BJ	Fit for large parts, fast production., extensive materials, low cost	Low detailing, post processing might need
SLM	Fit for complex geometries, high strength, porosity	High cost, limited part size
SLS	Fit for complex geometries, extensive materials, high strength	Unfit for complex geometries, slow producing
LC	Surface coating-repair, wear resistance	High cost, porosity, inaccuracy
LENS	Fit for complex geometries, porosity	Poor surface quality, manufacturing defects
SPS	Rapid process, low sintering temps.	High cost, limited product size
LOM	Eco-friendly, size flexibility, low cost	Narrow material choice
EBM	Pure product, high strength	High cost, high energy consumption, relies on powder
Infiltration	Fit for thin layer production, high strength	Production difficulty
Electrospinning	High precision, high tensile strength	Degradation, dissolution, low strength

2.7.12. Laser engineered net shaping (LENS)

LENS uses a high-powered laser, melting metal powder and depositing it layer by layer to create a 3D object. LENS allows for the direct fabrication of complex shapes with high precision. Powder characteristics, thermal properties and laser absorption influences the process. Larger volume can be produced with LENS, compared to SLM LENS is also used for repair and doesn’t require post-processing [65]. A schematic of LENS given in Figure 16.

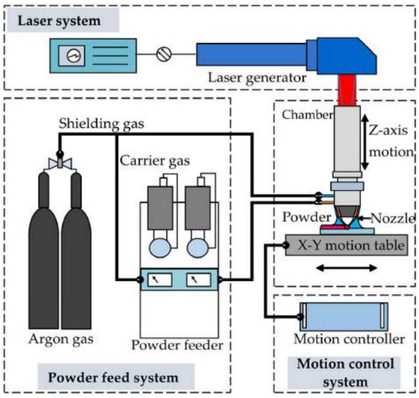

Figure 16. A schematic representation of LENS, taken from [66]

2.7.13. Spark plasma sintering (SPS)

SPS conjoins powdered materials with high density in a very short time and at low temperatures. Electricity is used to create sparks, and bonds are formed between materials. It is used to produce ceramics, metals, and composite materials [61]. High strength and high-wear resistance machine parts and materials can be produced. A schematic of SPS is given in Figure 17.

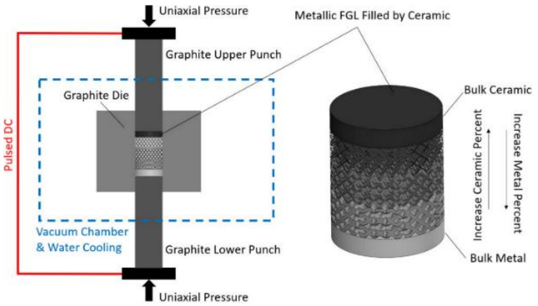

Figure 17. A schematic representation of ceramic-metal composite produced by SPS, taken from [67]

2.7.14. Laminated object manufacturing (LOM)

2D and 3D objects can be produced by LOM, layering sheets of material, and then cutting away the excess material. LOM relies on adhesives for bonding and widely used producing polymer, metal and paper laminates [68].

2.7.15. Electron beam melting (EBM)

EBM uses electron particle beam to melt powder metals, fusing the melted powder into a solid layer. It requires minimal post-processing [69]. EBM is being used in automative, aerospace and biomedical industry. A comparison between composite material produced using EBM and SLM is given in Figure 18.

Beam-based AM methods exhibit a range of processing temperatures and power levels depending on the energy source and material. SLA uses UV light to cure liquid resins at ambient temperatures and very low power. LENS and SLM are used for melting metals, operate at high temperatures (higher than 1000°C) and utilize high-power lasers in the near-infrared spectrum. SLS operates at sintering temperatures below the material's melting point. LOM, bonds sheet materials at much lower temperatures. EBM, distinct from the laser-based processes, utilizes a high-power electron beam to melt metal powders in a vacuum, operating at elevated temperatures [6,45-52,54,55,58].

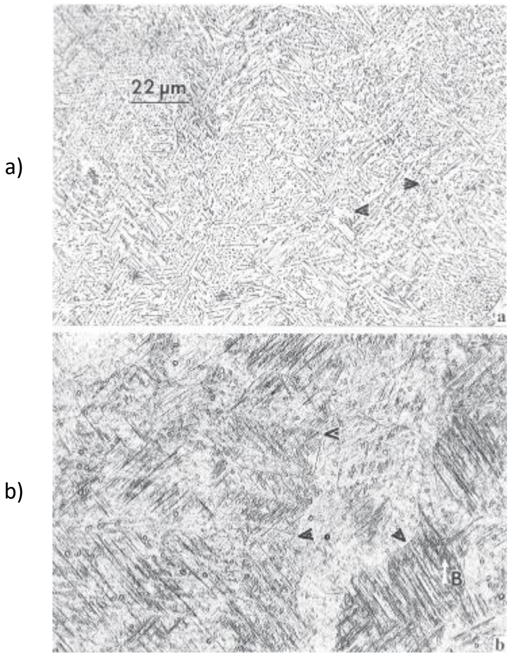

Figure 18. Comparison of microstructures for Ti-6Al-4V produced using a) EBM and b) SLM. (Arrow tips indicate columnar grain boundaries), taken from [70]

2.7.16. Infiltration

This method is used to create ceramic matrix composites by filling the pores of a ceramic preform with a molten metal. It is usually used for metal-ceramic composites. Oxidation and interface reactions may occur [71,72].

2.7.17. Electrophoretic deposition (EPD)

Charged particles suspended in a liquid are deposited onto an electrode under the influence of an electric field. EPD is used for producing automative parts and conductive ceramic and coatings, with a continuous gradient [25]. A schematic of EPD setup is given in Figure 19.

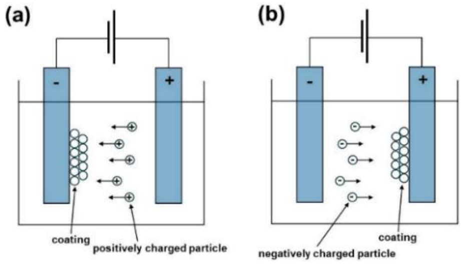

Figure 19. A schematic representation of a) cathodic and b) anodic EPD setup, taken from [73]

2.7.18. Electrospinning

Electrospinning uses a high voltage to draw a liquid substance into fibers that can be controlled at the nanometer scale. Biomedicine, nanofiber reinforced composites, electronics and optical devices are manufactured by this method. It is a relatively simple method [74-77]. A schematic of the electrospinning setup is given in Figure 20.

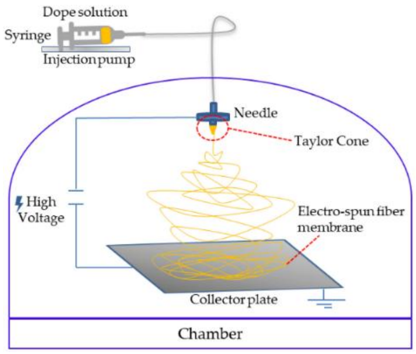

Figure 20. Schematic representation of electrospinning setup, taken from [78]

Table 5. Materials and process types of AM methods

Method	Materials	Process Type
SLA	Polymer, ceramic, composite, hydrogel	LB
SGC	Polymer	LB
LTP	Polymer	LB
BIS	Polymer	LB
HIS	Polymer	LB
FDM	Polymer, composite, ceramic, hydrogel	Thermal Extrusion
MJT	Polymer	Material jetting
BJ	Metal, polymer ceramic	Liquid binding
SLM	Polymer, metal, ceramic	LB
SLS	Polymer, ceramic, composite	LB
LC	Metal	LB
LENS	Metal, ceramic, composite	LB
SPS	Metal, ceramic	Electricity forming
LOM	Paper, polymer, metal, ceramic	Adhesive usage
EBM	Metal	Electron beam
Infiltration	Metal, ceramic	Injection molting
EPD	Polymer, ceramic, metal	Electric field
Electrospinning	Polymer, ceramic, composite	High voltage-spin forming

3. Classification of FGM Manufacturing Methods

Extensive research has been conducted on FGM methods in literature, exploring their potential for diverse engineering applications. The type of process (solid, liquid, or deposition) affects the final product's properties based on the heat, pressure, and forces involved during manufacturing. To begin, a general comparison of methods based on state of product during process is shown in Figure 21 [6,8-11,24,25,50-52,55,58,68,71]. Solid states contain powder materials whereas liquid state includes molten.

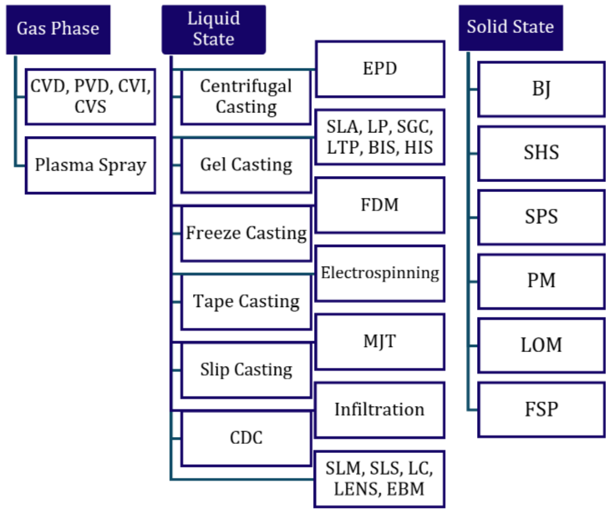

Figure 21. Classification of FGM manufacturing methods based on products state during process

As can be seen from Table 6 modified from [6], a comparison of FGM manufacturing methods based on FGM products size can be made. Bulk methods are most suited for process 3D objects, while thin methods, known as coating techniques, are inappropriate for creating structural designs [6,25,45]. Bulk and thin film methods can be combined and used to create components with optimized performance.

Additionally, based on the number of process steps, equipment requirements, and the degree of control over the microstructure and composition gradient, classification of methods are concluded, as shown in Figure 22 [109-140].

Lastly, another classification of manufacturing methods is conventional methods and additive methods. Conventional methods include casting, vapor deposition, SHS, and PM, whereas other methods are referred to as additive, as shown in Figure 23.

Table 6. Manufacturing methods based on size of FG product

Size	Methods
Thin	CVD, PVD, JVD, DVD [27]
	Plasma Spray [79,80]
	EPD [81,82]
	LC [39]
Bulk	Centrifugal Casting [83,84]
	Gel Casting [85,86]
	Freeze Casting [86]
	Tape Casting [87,88]
	Slip Casting [89-91]
	CDC [92,93]
	SLM, SLS, LENS, EBM [60,61,65]
	SLA, LP, SGC, LTP, BIS, HIS [96,97]
	MJT [6,7]
	BJ [6,7]
	FDM [98-100]
	Electrospinning [100,101]
	Infiltration [71,72]
	SHS [33,34]
	SPS [102]
	PM [28,103]
	LOM [104]
	FSP [105,106]

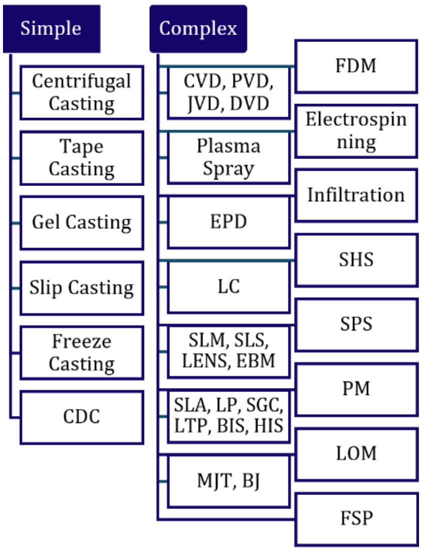

Figure 22. Relative complexity of methods

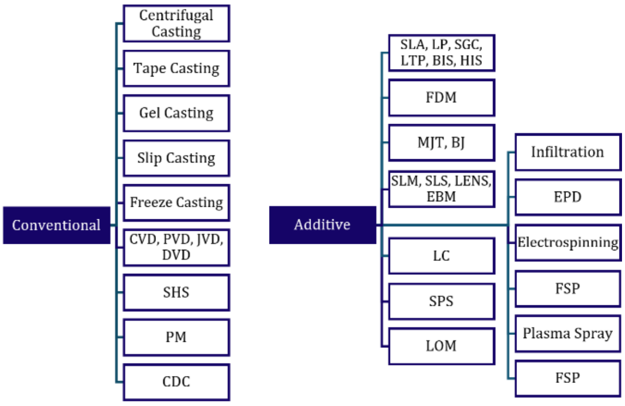

Figure 23. Conventional and additive methods, taken and modified from [6,8-11,19,21,120,125]

4. Conclusion

This review has comprehensively explored the diverse manufacturing techniques of FGMs, which is a unique approach to material design by enabling tailored properties within a component, mitigating interfacial stresses and enhancing mechanical performance compared to conventional composites. Applications of FGM span diverse fields, including aerospace, electronics, and biomedicine. This review examined a range of FGM manufacturing methods which selection of the appropriate method depends on the desired material properties, geometric complexity, production volume, and budget.

5. Discussion

The practical application of FGMs is direclty linked to advanced production methods. Each method has advantages and disadvantages. AM methods are promising FGM manufacturing methods that have excellent design flexibility and ability to create complex geometries. However, AM also has limitations, including relatively slow processing speeds and high energy consumption. Therefore, future research should focus on optimizing process parameters, developing innovative energy-efficient techniques for example, exploring hybrid manufacturing approaches, and adopting a life-cycle assessment perspective for FGM products to promote sustainable manufacturing practices. This will facilitate wider application of FGMs in varying industrial sectors.

Declaration of Interest Statement

The author declares that he has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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