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Materials Science in Additive Manufacturing

REVIEW ARTICLE

A review of advances in additive manufacturing and the integration of high-performance polymers, alloys, and their composites

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Abstract

In recent years, additive manufacturing (AM) has emerged as the most revolutionary technology in manufacturing, playing an indispensable role in many important areas due to its outstanding precision, ability to fabricate complex structures, and short production cycles. At the same time, the development of this technology has been accompanied by a constant search for materials suitable for it. These materials play important roles in the industry, have excellent properties but are difficult to process using traditional manufacturing methods, or are newly developed materials specifically for AM. While these explorations are being undertaken, attention to standards in the field will ensure that research is accelerated and on the right track. This paper presents each of the seven technique categories of AM. The focus of this paper is on the emerging materials for AM, such as polyetheretherketone, polyimide, high entropy alloys, and composites. Finally, international standards in the field of AM with perspectives on research in this area are also presented.

Keywords: Additive manufacturing; 3D printing; Polyetheretherketone; Polyimide; High entropy alloys; Standards

1. Introduction

With the progress and development of science and technology, people are increasingly investing in and paying attention to cutting-edge key areas such as aerospace, biomedical, and so on. The development is accompanied by the increasing complexity of the structure of the parts and the increasing requirements for strength, corrosion resistance, biocompatibility, etc., of the parts. Therefore, upgrading and improving manufacturing technology and actively expanding the adaptability with high-performance materials are the focus of prevailing research.

Additive manufacturing (AM) has caught the attention of researchers and the industry in recent years due to its rapid prototyping capabilities and design flexibility[1-3] compared to traditional manufacturing. These interests have led to the development of many types of AM methods, with significant results for a wide range of materials. As a key component of Industry 4.0, AM has been endowed with the ability to prepare products for the future with higher customization, shorter cycle times and

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costs, and more outstanding capabilities than traditional manufacturing processes.

According to the ISO/ASTM52900-21 standard[4], AM techniques are classified into seven categories, as shown in Figure 1, namely, binder jetting (BJT), directed energy deposition (DED), material extrusion (MEX), material jetting (MJT), powder bed fusion (PBF), sheet lamination (SHL), and vat photopolymerization (VPP). The technical characteristics, application scenarios and applicable material types vary among these techniques. Given that, these techniques have developed in different aspects in recent years, culminating in the current status. The current summary and overview of these seven technologies is relatively rare, which is unparalleled to the rapid development of the industry.

On the basis of continuous technological advances, the adaptability of high-performance raw materials is another key issue, and even, empowering and trying to further explore the potential of materials through AM technology will be the focus of attention of the application end of AM field in the future. This review focuses on two core classes of materials, namely polymers and metals, and highlights a representative high-performance material from each of them. The review also focus on those materials that have a fit with AM technology in terms of application areas and have already demonstrated significant research value in conventional processing to explore their latest developments in the AM field as well as further possibilities for the future. Engineering plastics such as polyimide and polyetheretherketone (PEEK), as well as alloys such as high entropy alloys (HEAs), are among the star materials that fit this profile. We believe that tracking the development of these high-performance materials in the AM field will help us further understand the growth trajectory of AM technology.

Therefore, the final section of the review includes the relevant, up-to-date standards in the AM field. This multidimensional summary is intended to provide another perspective on the development of the technology.

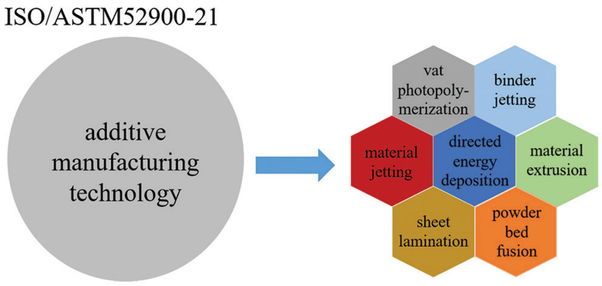

Figure 1. Seven different categories of AM techniques[4].

2. Additive manufacturing techniques

2.1. Binder jetting

Binder jetting additive manufacturing (BJT) is an inexpensive AM technique that uses liquid adhesives to bond and cures specific parts of a powdered material layer by layer into the green state, followed by different types of post-processing to obtain a finished product, depending on the nature of the material[5,6].

The BJT technology is an AM process conducted entirely at room temperature. As there is no deformation due to thermal effects, it can be used to build large parts and there is no need to design additional support structures when using BJT technology. In some cases, it is even possible to fabricate colored products. This technology is now used to print finished parts with complex internal structures and geometries in a variety of materials including metals[5,6], ceramics[7], and polymers[8].

The major disadvantage of BJT as compared to several other AM technology is that it is uncompetitive in many commercial cases due to shrinkage problems when machining metals and the difficulty of achieving fully dense parts directly[9]. Furthermore, the fabricated products are very fragile in their green state before post-processing, limiting the use of the AM process to a certain extent. Figure 2 clearly reflects several typical product defects when using BJT techniques[10].

Improvements and refinements to address this feature are ongoing. One of the main treatments is the use of additives to provide additional treatment to the green parts before sintering. This process modifies the internal structure of the green parts, increasing the number of particle alignments and the size of inter-particle contacts to make it denser and more stable[11]. This approach has been demonstrated with several different types of additives, including metal salts[12], metal-organic inks[13], and sol-gel[14].

Building on this foundation, related research to gain insight into the effects of reactive binders on the creep and densification mechanisms of binder-jet 3D printed parts has also been conducted recently. Grant *et al.* investigated the intrinsic mechanism of aqueous titanium bis-ammonium lactato dihydroxide (TALH) as a binder to alter the creep of the samples and achieved significant deformation improvement[11].

2.2. Directed energy deposition

DED technology melts metal powders or wires during the manufacturing process and deposits them layer by layer onto specified areas and cooled them to form the

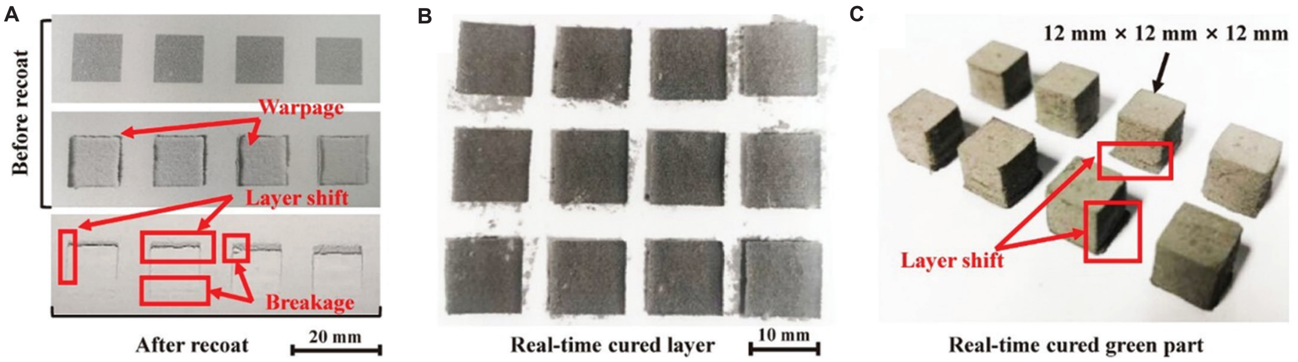

Figure 2. Typical vital defects in real-time curing binder jetting additive manufacturing. (A) Real-time cured layers during printing (inside powder bed). (B and C) Real-time cured layers and green parts after an ultraviolet irradiation (outside powder bed)[10].

structure. The molten material is released through the nozzle and the direction of deposition is changed in the process by adjusting the relative movement of the platform or nozzle[15,16]. Figure 3 presents the working principle of a general laser powder DED (LP-DED) system[17].

DED technology is well suited for the preparation of high-performance metallic materials and is also suitable for the processing of some ceramic materials[18]. Compared to other AM technology, DED technology offers a number of advantages, including faster build rates, the capability to produce large parts, the ability to fabricate under non-horizontal conditions, the ability to print in weightless environments under certain conditions, and the ability to deposit multiple materials simultaneously on some models[18,19]. This makes DED technology a promising technology for a wide range of applications.

The DED technology can also be used for the repair and addition of materials onto existing components. With DED technology, it is possible to achieve higher precision, lower residual stresses and a more flexible repair process than with conventional welding repair methods[20]. Research into DED technology is becoming increasingly advanced due to its outstanding advantages, with recent studies showing, for example, that the final performance of products can vary considerably under different scanning strategies[21]. Nonetheless, there are disadvantages of using DED technology. In most cases, the components formed by DED technology have a relatively low resolution and often require post-processing to complete the final product. The anisotropy of the product itself is significant and requires dynamic adjustment of parameters to reduce the impact of this on the performance of the fabricated product[20,22].

In response to the above problems, researchers have also attempted to solve them in recent years by improving and perfecting the hardware of DED devices. Novel devices such as rolling-assisted laser directed energy deposition (L-DED) have emerged. With the aid of *in situ* rolling, the product has a unique initial microstructure, which results in relatively low uniform elongation and relatively high ultimate tensile stress[23].

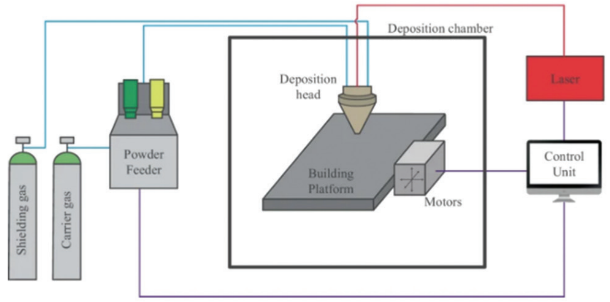

Figure 3. Demonstration of the working principle of a general laser powder directed energy deposition system[17].

2.3. Material extrusion

MEX technology is a very established AM technique. It builds 3D parts by heating a linear composite or thermoplastic material to soften it, followed by depositing and curing it layer by layer[24]. In particular, while printing biomaterials by means of MEX, the material is extruded from the nozzle and then forms the structure[25]. The process is shown in Figure 4.

MEX technology is well suited for use in domestic or non-professional applications, and it also has applications in the medical field due to its high degree of customizable flexibility[26]. At present, this technology has proven to be very compatible with more than 20 thermoplastics, such as acrylonitrile butadiene styrene (ABS), polyamide 66 (PA66), and aliphatic polyamides (PA, also known as nylon). New research has shown that MEX technology is also suitable for polymers such as PEEK or ceramic materials, which will be described in more detail in subsequent sections of this paper[24].

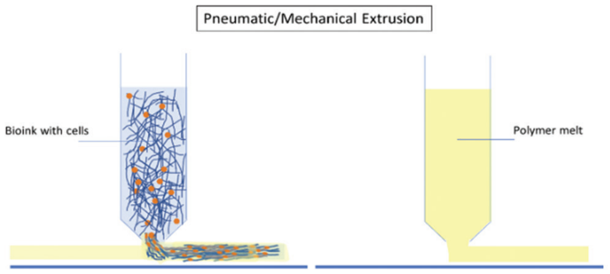

Figure 4. Illustration of material extrusion with or without cells (permission obtained from authors)[25].

One of the advantages of MEX technology is that it is inherently less restrictive in terms of raw materials. Most of the feedstocks are very inexpensive, and a considerable proportion of the model material is open source, reducing the cost of learning for the user themselves, and making it highly adaptable and relevant for the home market. At the same time, 3D printers using MEX technology are generally less expensive to maintain and run and are therefore used in some commercial scenarios to produce prototypes.

On the other hand, there are limitations to the strength of the finished products printed by MEX technology, especially for the perpendicular Z-axis direction (along the build direction), where there is a large deficit in strength. At the same time, if the user tries to increase the accuracy of a 3D printer using MEX technology, the corresponding fabrication time increases, which limits the value of this technology to a certain extent.

Most importantly, the thermoplastic material in a MEX process can be heated to a maximum of 500°C, thereby emitting harmful gases and ultrafine particles that can lead to a reduction in indoor air quality in domestic scenarios. This poses a potential health problem for the occupants of the house, considering that some components take more than a few hours to fabricate[27].

It is worth noting that MEX technology may be developed for medical applications because it is compatible with most materials. This certainly broadens the potential market for this easy-to-use technology. Recent studies have successfully used medical-grade polyamide (PA)12 polymer as matrix material to complete the print; it was also found that by adding silver nitrate, polyethylene glycol or polyvinylpyrrolidone, the printed samples could further exhibit higher tensile strength or sterilization properties[28].

2.4. Material jetting

MJT technology is an AM technology that is somewhat similar to conventional 2D printers[29]. 3D printers using this technology build complex 3D structures by spraying a photosensitive liquid material layer by layer over a specified area with a nozzle and curing it with ultraviolet light.

This technology offers high cost-efficiency and application potential. In addition to being suitable for traditional thermoset photopolymers, MJT has shown adaptability to a variety of materials such as gels and bio-based materials[30]. It has also received increasing interest from the market as well as industry in recent years because it can be used in informal settings such as office scenarios due to its similar working characteristics to traditional printers[29]. On the other hand, the ability to print simultaneously with multiple materials is a major advantage of MJT technology. The high dimensional accuracy and low surface roughness of the finished products fabricated using MJT technology make it potentially useful in areas such as cutting-edge customized medicine, aerospace, and other applications[31].

The current shortcomings of MJT technology are divided into two principal areas: material and technology. The layer-by-layer method has gaps and defects that can have a significant impact on the final performance of certain elastomeric materials, such as fatigue life[32]; on the other hand, the raw materials used in MJT technology are photosensitive, the mechanical properties of the finished product could potentially deteriorate over time[33].

Targeted research has been followed up. By studying the aging of different color resins printed by MJT over time, the researchers found that the stiffness of the material first increased and then decreased over time, and the aging process has a potential relationship with the color and appearance of the product itself[34]. At the same time, various tests were carried out; the structure of the whole experiment is shown in Figure 5.

These studies provided the basis for understanding the long-term factors affecting the changes in the appearance and performance of objects printed using MJT technology. In the future, on this basis, through a series of research on the parameters of the entire industry chain and product design, it may be possible to greatly increase the lifespan of the product and enhance the actual application value of the technology.

2.5. Powder bed fusion

Powder bed fusion (PBF) technology is an important branch of AM. Especially in the field of metal additive manufacturing, PBF occupies an absolute advantageous position. Figure 6 illustrates the market share held by different technologies in the metal additive manufacturing segment in 2019[17]. PBF technology, as

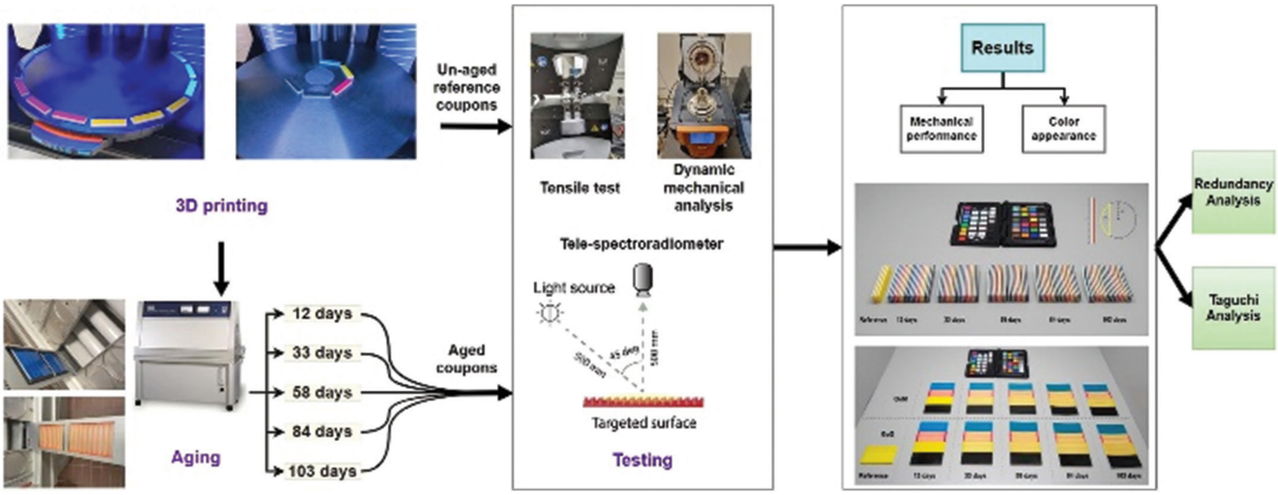

Figure 5. Schematic diagram of the experimental framework[34].

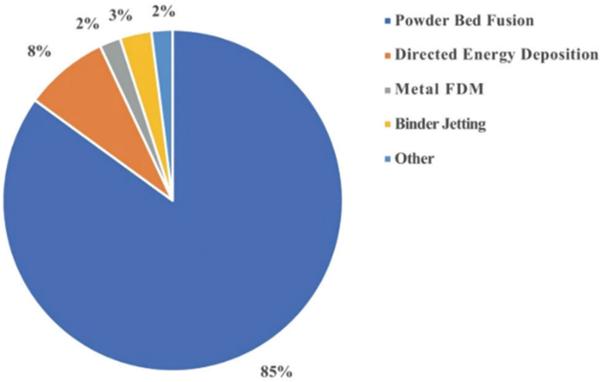

Figure 6. Schematic representation of the market share held by different technologies in the metal additives manufacturing segment in 2019[17].

the name suggests, is a technique in which various parts of the powder laid on a bed are heated by different heat sources to sinter or melt the powder and then solidified to form a 3D structure[35].

Over time, four derivatives of PBF technology have been developed to suit different raw materials and heat sources for applications; these are selective laser sintering (SLS), selective laser melting (SLM)[36], electron beam melting (EBM)[37] and multi jet fusion (MJF).

These four technologies are different in their technicalities and use different raw material form. For thermoplastics, SLS and MJF are used[37]. Of these, SLS is by far the most established PBF technology for polymers[38]. The main difference between SLS and MJF lies in the heat source, with the SLS technology using a laser for powder sintering and the MJF technology using an infrared (IR) lamp to activate the solidification process. Both types of technology are currently limited by the range of polymers available and researchers are expanding their database of raw materials to continue exploring their potential applications in industry[37,38].

Another category within the PBF technique comprises the SLM technique and the EBM technique. These two techniques are suitable for most types of metal powders[39]. The heat source for SLM technology is a laser, while the heat source for EBM technology is an electron beam[40]. Figure 7 illustrates the basic working principle and structure of SLM technology by presenting a schematic of the working process of SLM technology.

The EBM technology has a larger scanning beam diameter and a faster scanning rate, allowing for the rapid construction of larger volumes and the processing of thicker powder layers compared with the SLM technology. At the same time, the EBM technique produces products with better fineness and surface roughness compared to the SLM technique[41,42].

Both techniques are currently attracting significant interest in the academic circles, for their complex thermal processes during processing and their eventual impact on the microstructure of the finished product[43]. In fields such as aerospace, medical, and military defense, these two technologies have been more widely researched and applied due to their suitability for finished complex structures and the ability to rapidly prepare prototype products[44].

The extensive development of LPBF is matched by the ongoing development of targeted and applicable alloys to raise the application value of this technology. For instance, by adding Ti, Cr, and Fe to the Al-Cu alloy, a

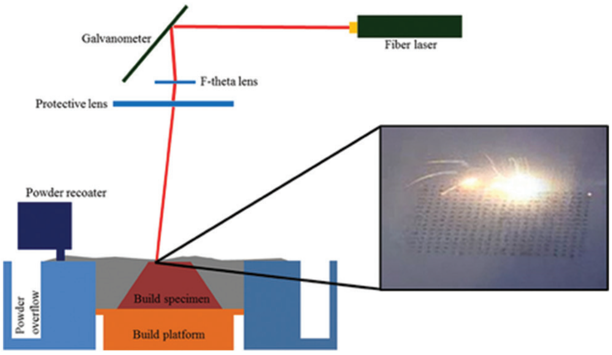

Figure 7. Schematic of selective laser melting build chamber and process[36].

new aluminum alloy with low thermal crack sensitivity suitable for PBF printing technology was fabricated and was shown to achieve the highest strength currently known for aluminum alloys printed by PBF[45]. For other aluminum alloys such as Al-Zn-Mg-Cu-Nb alloys, the effect of Zn content on the formability and aging precipitation that may result from LPBF preparation has also been studied[46]. It has been demonstrated that the content of the Zn element affects the aging kinetics, the order of elemental precipitation and the crystal size of the alloy, provided that the PBF printing technique is used. It was also explored how the processing window changes as the Zn content rises to ensure that it can be stabilized for fabrication[46].

There are numerous studies exploring in this regard, indicating that the intensive investigations of the PBF technology are a testament to its current dominance.

2.6. Sheet lamination

Sheet lamination (SHL) technology is known to be the first commercialized AM technology, but its market share is still very limited today[47,48]. The raw material requirement for SHL technology is sheet or plate. During the process, these raw materials are stacked layer by layer and tooled to cut out specific sections for gluing or welding, which are combined to form the structure.

Over the years, SHL technology has evolved into various sub-categories including selective deposition lamination, ultrasonic additive manufacturing and selective lamination composite object manufacturing, depending on the type of raw materials used and the bonding process, which vary in detail but are based on the same principle[49].

Due to these adaptable variants, SHL technology can process a wide range of materials such as metals, paper, polymers, textiles, ceramics, and composites and can be used to build very large finished products[47,49]. Some of the 3D printers using SHL technology can also be deployed in office environments to fabricate paper models. The problem with SHL technology, however, is that it cannot be used to print complex structures, so the practical value of this technology is limited.

A similar technique called electron beam sheet lamination (EBSL) has recently been attempted in the manufacture of turbine disk alloys with good results[50]. EBSL technology utilizes electron beams to achieve rapid melting and solidification between multiple layers of material. This feature helps control the segregation of the alloy itself and removes impurities in the alloy to obtain products of higher purity. The alloy prepared by EBSL has a finer structure and exhibits good creep rate.

2.7. Vat photopolymerization

VPP technology is incredibly special compared to other AM technology. During the process, a light source of a specific wavelength is selectively directed at a specific part of a barrel of photosensitive liquid material to induce a curing reaction, during or after which the build platform is lowered and the surrounding liquid material re-submerges the platform and a new layer is cured[51]. Depending on the light source used and the way the platform is lowered, VPP technology can be classified as continuous direct light processing, direct light processing, stereolithography (SLA), etc.[52,53].

The principle of this technique therefore limits the range of materials that can be applied to VPP. The usual raw material is photosensitive liquid resin, but fillers such as ceramics can be added to enhance the mechanical properties of the finished product[54]. In addition, a new study has offered a solution to the problem of switching raw materials during the process, enabling the use of multiple materials in printing embedded complex structures[55].

Compared to other AM technology, VPP technology offers the advantages of high precision, fast forming, smooth surfaces and low material waste[56]. However, this technology has a few disadvantages, such as high costs, limited adaptability of raw materials and potential instability in the performance of the finished product. The field of bioprinting based on VPP technology is currently gaining traction. By varying the type of photo initiator used, VPP technology can produce finished products that are simultaneously reliable and highly biocompatible. VPP technology also has considerable potential for application in the field of tissue engineering and regenerative medicine[57].

The application of VPP technology improves the scalability and also eases the replication and manufacture of organs-on-a-chip[58,59]. Two sub-types of VPP technology – SLA and DLP – have proven to be the most favorable fabrication methods for future in-depth research in the field of organs-on-a-chip. Recent studies have demonstrated that DLP printing technology is suitable for organs-on-a-chip fabrication and development, and with the improvement and specificity optimization of the equipment hardware and preparation parameters, it is even possible to consistently reproduce them in tens of microns of printing accuracy[59].

3. High-performance polymers and polymer composites for additive manufacturing

The rapid development of AM technology will continue to revolutionize and reshape all areas of human life. The emergence of AM technology will also provide more possibilities for the application of PEEK and polyimide, which are already commonly used in aerospace, biomedical and other fields, as high-performance materials. On the other hand, the continuous experimentation with these materials has also enriched the database of materials applied in various AM technologies in different fields, further strengthening the potential of AM technologies. The following section focuses on high-performance engineering plastics – PEEK and polyimide – and their combination with AM technology.

3.1. Polyetheretherketone

PEEK, under the trademark of Victrex[60], shares many characteristics with polyether sulfone and is a semi-crystalline thermoplastic polymer with tough properties[61]. This material, which was developed in the last century, has shown enormous potential and value in several fields since its introduction.

With a high melting point of over 300°C[62], high abrasion resistance, chemical resistance, competitive dielectric strength, excellent mechanical properties[63], and potential biomechanical properties[64], PEEK is ideal for use in a wide range of industries, including military, medical, electrical and electronic. Among these, PEEK is particularly promising for medical applications as it has been demonstrated that PEEK does not exhibit cytotoxicity to biological cells, nor does it induce cancerous necrosis in cells, and therefore, it has good biocompatibility[65].

However, the progress of this field, which has been explored since 1987[66], does not stop there, and many studies exploring composite reinforcement of PEEK materials are still underway. Among the more important of these are PEEK composites containing bioactive particles such as hydroxyapatite (HA) to cater for the repair of active cells in living organisms[67] and the modification of the elastic modulus of the PEEK material by means of carbon fiber reinforcement[68]. Studies have shown that the mechanical properties of PEEK-based composites reinforced by carbon fibers have a high resemblance to those of the most pristine state of human bone than other biocompatible metals such as titanium, thus minimizing the problem of stress shielding and making them an ideal raw material for human bone repair techniques[60].

The enormous potential of PEEK and its composites has also led researchers to investigate its application in the field of AM technology. By combining the two, it will be possible to expand the prospects for both. The combination of the two is first and foremost seen in the manufacture of complex medical components such as dentures, biological scaffolds, and implants, where the rapid prototyping capabilities of AM technology for complex structures are being put to great use. Several studies have recently demonstrated the successful printing of pure PEEK materials using MEX technology and good performance was observed in the printed item[69-71].

In parallel, experiments on PEEK-based composites are underway. The previously mentioned PEEK-HA composites containing HA nanoparticles[72] and PEEK-carbon fiber (CF) composites containing carbon fibers[73], as well as PEEK composites containing carbon nanotubes and graphene nanosheets[74], have all been experimented in combination with MEX technology. These experiments were generally successful; for example, biocompatible PEEK-HA composites with sufficient mechanical strength were successfully processed by AM technology[53], PEEK-graphene nanoplate (GNP) and PEEK-carbon nanotube (CNT) nanocomposites printed by AM technology also exhibited unique multiple properties, reflecting their promising applications in multiple fields such as oil extraction, automotive manufacturing, and space exploration[74], while the PEEK-CF composites processed by AM technology exhibit a controlled adjustment of the anisotropy of the thermal properties to suit potentially specific application requirements[73]. Alternative trials have also been conducted for titanium implants. Figure 8 is a schematic representation of the MEX process for replacing existing titanium products by processing PEEK as a mesh in cranioplasty[75].

Another development idea is to modify the surface of PEEK to overcome its biological inertness and further broaden its potential applications. Surface bisphosphonation of PEEK has been successfully accomplished using alendronate, and this newly obtained material has been shown to be able to direct the biological behavior of macrophages and further improve the cellular microenvironment[76]. This could significantly improve

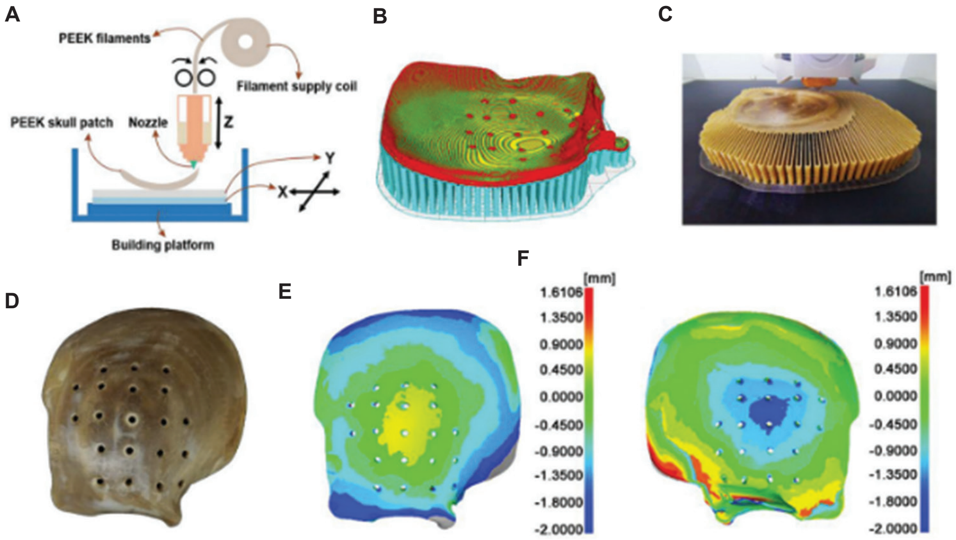

Figure 8. Schematic illustration of the process to fabricate the material extrusion (MEX)-printed polyetheretherketone (PEEK) skull implant. (A) Flow chart of the MEX printing of PEEK skull implant. (B) Printing data processing and acquiring control parameters. (C) MEX printing of the PEEK skull implant. (D) The MEX-printed PEEK prosthesis of the skull. (E and F) Comparison between the MEX-printed PEEK skull implant and the patient-specific designed PEEK skull prostheses. The front (E) and back (F) views of the printing accuracy are shown[75].

the treatment of inflammation in patients as well as the regenerative bone repair of implants at a later stage.

Similar modifications can also yield sulfonated PEEK (SPEEK), which can be adhered to other antimicrobial substances to achieve an improved inflammatory environment in implanted soft tissues. The antimicrobial peptide HHC36 has been shown to provide up to 10 days of extended sterilization effect on SPEEK by a simple solvent evaporation method (HSPEEK)[77], which opens further possibilities for minimally invasive bone and joint grafting and repair in the elderly. The flowchart of the synthesizing HSPEEK is shown in Figure 9.

However, the integration of this novel material obtained by modification with AM technology has yet to be further explored, but its promising future in the biomedical field will undoubtedly point to the need for complex structures, making this area highly explorable.

3.2. Polyimide

Polyimide, a material that predates PEEK, is one of the most heat-resistant polymers currently available[78]. It has a rigid aromatic molecular structure and therefore exhibits excellent corrosion and heat resistance, mechanical and electrical properties[79] and can be used at extreme operating temperatures. It is currently of great value in many fields such as space exploration, defense and security, electronics, and electrical appliances.

On the other hand, given its stability at high temperatures, it is difficult to fuse polyimide in conventional processing methods, limiting the value of this material in cutting-edge applications. AM technology allows the polyimide material to be processed with precision, enabling it to be used in more complex scenarios[79].

The precursors of polyimide materials can be used for manufacturing by means of VPP technology due to their ability to conveniently bind photocurable functional groups[80]. In 2017, researchers prepared photosensitive polyimide oligomers into solvent-free photocurable inks and processed them using VPP technology, successfully obtaining products with excellent mechanical properties[81]. In the same period, the printing of insoluble engineering thermoplastic polyimides was also successfully accomplished with the VPP technology using soluble precursor polymers for chemical cross-linking under light induction and post-printing heat treatment[82].

These early studies have demonstrated that the polyimide materials are compatible with AM technology. It also paves the way for the subsequent use of AM technology to improve the functionality as well as the practical value of polyimide and polyimide-based composites. For example, new research has begun to focus on greening the AM process and has incorporated this concept into the research process, resulting in an alternative to the harmful organic solvents required for the AM process for polyimide materials[83]. On the other hand, in response to the low strength, poor thermal stability and high curing shrinkage of photocurable resin materials currently available on the market, new light-curable polyimide inks for the

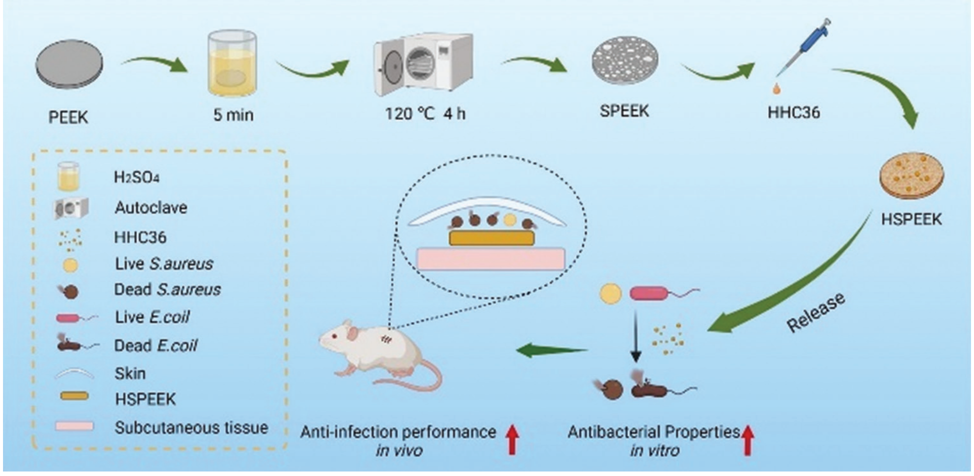

Figure 9. Synthesis of HSPEEK and summary of the experimental procedure[77].

manufacture of polyimide materials using AM technology are being innovated to increase thermal stability and may be used in special applications with higher operating temperatures[84].

Blending polyimide-based composites with CNT is a popular research topic, and researchers are exploring the processing of polyimide-CNT composites in MEX technology after overcoming the problems of inhomogeneous mixing associated with conventional sonication and other methods[85]. After optimizing the process parameters during the process and the filling level of CNT, finished complex structures with exceptional electrical and mechanical properties and anti-electromagnetic shielding were successfully prepared by MEX technology, offering new possibilities for the application of this new composite material in cutting-edge fields such as aerospace[86].

In the future, research into improving the accuracy and stability of polyimide materials in AM technology will continue, and this important polymer material and its complexes will play an even more significant role in future developments.

4. High entropy alloys and its composites for additive manufacturing

It has always been a research focus to investigate new alloys, which are commonly employed in AM technology and are essential materials for human society. There is an increasing demand for metal components with high-performance and ultra-complex structures, but traditional manufacturing techniques are limited by their metal processing capabilities. Thus, this highlights the need for rapid commercialization of new alloys and innovation of manufacturing technologies.

In the field of metals, the study of HEAs and their processing methods is currently the focus of research. This unique type of alloy is gradually becoming the most dominant material in future, and its unique value compels researchers to devote greater attention to it; therefore, this section will focus on this unique metal concept, the current status of development and application of HEAs, the composites involved, and the integration of HEAs with AM technology. This section summarizes the work of researchers in recent years and analyzes future directions of research and development.

4.1. Concept and current development

The concept of alloys has been around for a long time and since the discovery of metal materials, the process and method of adding different elements to pure metals to improve metal properties have been explored by generations of researchers.

The concept of HEAs was first introduced by Yeh *et al.* in 2004[87], and its original definition requires that it comprises at least five principal elements and that the atomic concentration of each principal element is between 5% and 35%. Subsequent researchers have expanded the concept of HEAs[88] to also include alloys containing three or four principal elements. Although the concept of HEAs has been promulgated for less than two decades, this concept has already disrupted the traditional framework of alloy research and has grown into a major research hotspot in metallic materials science.

The “entropy” in the “high entropy alloy” refers to the constitutive entropy of the alloy and according to Equation I, an increase in entropy at high temperatures will result in a significant decrease in Gibbs free energy, which, in turn, will show the stability of the phases in the material[89].

$$G=H-TS \qquad (I)$$

where H is the enthalpy, T is the absolute temperature, and S is the entropy.

Compared to conventional alloys, HEAs have several outstanding core effects such as diffusion hysteresis effect and cocktail effect. These effects associated with solid solution strengthening can lead to a variety of unique properties, including microstructural stability and oxidation resistance. Different studies have been carried out to investigate the mechanical, electrical, and other properties of HEAs[90], and new HEAs are also constantly developed for use in a variety of applications and in special advanced fields[91]. HEAs can also be used as metallic materials under extremely high or low temperature operating conditions.

In their application as refractory materials, HEAs can maintain structural stability at high temperatures due to lattice distortion, high entropy effects, and diffusion hysteresis[92,93]. Since 2010, few research groups have successfully produced NbMoTaW and VNbMoTaW that can withstand temperatures of up to 1600°C[92,94]. Since then, through the incessant innovations and breakthroughs made by the researchers, the practical value of HEAs in the field of refractory materials has been increased by reducing their density and improving many of their properties, including ductility[93,95,96]. In the field of cryogenic applications, research into HEAs has also yielded impressive results. A recent study on CrCoNi-based medium- and high-entropy alloys demonstrated that CrCoNi with excellent damage tolerance exhibits high fracture toughness at temperatures as low as 20 K and shows a deformation structure different from that at higher temperatures[97].

This unique property is due to the synergistic effect of multiple microstructures such as dislocation slip and twin structure, and the performance of HEAs beyond that of other conventional materials once again demonstrates the extraordinary potential of HEAs for future high-performance alloys[97].

4.2. Integration with additive manufacturing

As HEAs are promising materials in many applications, studies have been conducted to investigate their processing by AM technology. As a metallic material, the suitability of HEAs as a raw material for AM technology has been relatively well studied, and subsequent research has begun to investigate the effect of the different parameters applied in the processing by different AM technologies on the performance of the final product.

In the case of the CoCrFeNi alloy system, for example, which is the most frequently used system to study the fabrication of bulk HEAs using AM technology[98], the influence of process parameters such as scanning speed and laser power on the final microstructure of the finished product has been investigated in both the PBF and DED techniques[99,100]. Further attempts have been made to optimize the temperature gradients and cooling rates during material fabrication by means of improved scanning strategies or the use of ultrasonic assistanc[99].

For another frequently used alloy system in research, AlxCoCrFeNi, a new study published is equally exciting[98,101]. This study, using the LPBF technique, succeeded in preparing a high-strength alloy with a layered microstructure consisting of body-centered cubic and face-centered cubic nanospheres layer by layer from AlCoCrFeNi alloys, making it stronger and more ductile than other alloys[101].

In addition, two popular research directions in the field of HEAs processing by AM technology are *in situ* alloying and *in situ* strengthening. *In situ* alloying is the direct *in situ* synthesis of HEAs by AM technology without pre-alloying. Through such studies, it has been found that the AM technique allows the costly pre-alloying process to be bypassed in favor of the direct preparation of HEAs from different pure metal powders, with acceptable densities and excellent mechanical properties[102]. *In situ* strengthening, on the other hand, refers to the generation of strengthening particles directly in the process through *in situ* chemical reactions between different elements or compounds during processing. In this way, a more homogeneous and detailed particle distribution, which is indicative of a better strengthening performance, can be obtained, as compared with the particle distribution obtained with conventional methods[103,104]. The emergence of these two research directions means that both AM technology and HEAs materials are refining each other, which is beneficial to devising a manufacturing method of greater value.

4.3. Processing of high entropy alloys composites and future directions

The HEA composites can be prepared by two methods: (i) The addition of other substances to the HEAs matrix to form metal matrix composites, and (ii) the addition of HEAs particles to a conventional alloy matrix to form metal-metal composites[105,106]. Both of these categories have been studied specifically for manufacturing with the application of AM technology, and it has been verified that the composites possess, for example, improved tensile and friction resistance properties[107].

In the future, as more unique properties of HEAs are explored and exploited, the synergy between HEAs and AM technology will become even stronger, and it is expected that this material will play a valuable role in more fields and serve the practical needs of cutting-edge fields better with the help of AM technology.

5. Standards in additive manufacturing

As AM technology flourishes, standards are needed for all aspects of the development, use, and refinement of the varied materials that are applied to this technology. Industry standards are the foundation for development, the beacon that inspires researchers to make breakthroughs and the guide for those trying to make the most out of this revolutionary technology. This section will therefore focus on the internationally recognized material standards currently used for AM technology, with a view to informing research in this area.

It should be noted that an early effort to formulate standards in the field of AM technology was undertaken by the American Society of Automotive Engineering (SAE) in the USA, which launch a standard on laser-deposited titanium products in 2002[108]. This standard is no longer valid and has been reaffirmed by the SAE as AMS4999A in 2016[109]. This has been updated with regard to the requirements for raw materials.

In 2008, the ASTM International started to engage became in this important endeavor and in 2009, the ASTM Committee F42 on Additive Manufacturing Technology was formed and divided into eight technical subcommittees to oversee the development of standards for different parts of the AM technology field[110]. Of the 22 standard headings currently listed on the F42 website, the more important ones include F42.01 Test Methods, F42.04 Design and F42.05 Materials and Processes[111]. In turn, F42.05 includes a dozen standards that define the requirements and characterization of polymer and metal powder raw materials for specific types of processing[112,113], performance specifications for specific products[114-117], as well as specific procedures and specifications for different processes[118]. On the other hand, ISO also formally established Committee TC261 in 2011 to work specifically on the standardization issues throughout the entire process of additive manufacturing[119]. In its 1st year since inception, it started a partnership with the F42 committee and has been working on the definition of terms, coordinate system definitions, data formats, etc., in the following years.

In 2021, ASTM and ISO have again jointly published the harmonized standard ASTM ISO/ASTM52903-1-20 for raw materials for MEX technology, including unfilled, filled, and reinforced plastic materials suitable for processing into parts and taking into account the influence of components that may contain flame retardants or stabilizers[120].

The European Union and China have also started to formulate their own AM standards. For example, the European Union launched the SASAM (Support Action for Standardization in Additive Manufacturing) project in 2015[121] and China established the TC562 National Technical Committee for Additive Manufacturing Standardization in 2016[122]. They have also started to develop standards in a number of key areas of AM technology, some of which are related to the requirements for raw materials.

As can be understood from the above history of development, the most influential standards in the field of AM technology are still those developed by ASTM and ISO, and they have developed some standards for constraints and characterization of metals as well as polymeric raw materials. Table 1 provides a summary of information on these material standards that are still in force.

However, as stated in ASTM's February 2022 article on materials suitable for AM in construction, there are

Table 1. Summary information on ASTM standards for raw materials in the AM sector

Standard no.	Applicable material	Applicable technology	Date of update
ASTM F2924-14 (2021)	Ti-6Al-4V	Full-melt powder bed fusion	October 20, 2021
ASTM F3001-14 (2021)	Ti-6Al-4V (extra low interstitial)	Full-melt powder bed fusion	October 22, 2021
ASTM F3049-14 (2021)	Metal powders	Powder-based additive manufacturing processes	October 25, 2021
ASTM F3055-14a (2021)	Nickel alloy (UNS N07718)	Powder bed fusion	October 29, 2021
ASTM F3056-14 (2021)	Nickel alloy (UNS N06625)	Powder bed fusion	October 29, 2021
ASTM F3091/F3091M-14 (2021)	Plastic materials	Powder bed fusion	October 29, 2021
ASTM F3184-16	Stainless steel alloy (UNS S31603)	Powder bed fusion	October 10, 2017
ASTM F3213-17	Co-28-Cr-6-Mo	Powder bed fusion	January 11, 2018
ASTM ISO/ASTM52901-16	General raw materials	Not specified	April 03, 2017
ASTM ISO/ASTM52903-1-20	Plastic materials	Material extrusion	March 09, 2021
ASTM ISO/ASTM52925-22	Polymers	Laser-based powder bed fusion	April 11, 2022

no standards regarding raw materials for many of AM's new applications, such as building houses, to meet the requirements of these applications[123].

6. Conclusions and future development

This paper introduces the seven major AM methods and their respective working principles, the types of raw materials that are compatible with the different technologies, and the advantages and disadvantages of each technology. This is followed by a description of the emerging materials used in AM technology, namely, PEEK, polyimide and HEAs, and the development of and adaptation to AM technology involving these composites. Finally, we present the international standards in the field of AM, focusing on their requirements for raw materials used in AM technology and categorizing them by time and geography.

The summary and outlook for the above are as follows:

(i) The AM technology can be divided into several subcategories driven by different processing principles. These technologies are mainly applicable to the manufacture of polymers as well as metallic materials and have emerged to correspond to application scenarios and targeted machining of component features. It is worth noting that while popular technologies such as DED and PBF are attractive options among the researchers and industries in recent years, the sub-technologies still possess the potential and value in their respective application areas and neither one of them will be phased out.

(ii) PEEK is a popular, new material for use with AM technology and has exceptional performance. The disadvantages that it exhibits with conventional processing methods are no longer present in AM technology. Research into PEEK and its composites is ongoing, and there is still much to be explored in this regard. Similarly, the integration of polyimide materials (including the composites in which they constitute) with AM technology has also passed the feasibility stage. In the next few years, research on employing these two materials in printing will move towards increasing precision, controlling defect, allowing full control of the AM process, and thus further improving the print quality.

(iii) HEAs are currently a type of popular material for researchers to experiment with until the next revolutionary alloy is introduced in the future. Since they are metallic in nature, HEAs possess several advantages in combination with AM technology. The concepts of *in situ* alloying and *in situ* strengthening adopted in the field of AM technology confer this class of alloys even more possibilities, and it is expected that more harmonious integration of these two fields will happen in the future.

(iv) The AM technology sector has been the focus of international associations since 2002, and a series of standards have been released, which have been enriched and improved since 2008 due to the rapid development of the AM technology. However, as AM technology becomes a more prevailing technique, the issue of standards for raw materials for specific industries needs to be addressed and this will be a key issue in future.

In the future, the deeper integration of AM technology with more materials will continue to accelerate, and the application of the technology will continue to expand towards more complex and refined directions. For example, by combining it with machine learning, it will greatly accelerate the optimization of the generation of different structures for different metals with different process parameters under the PBF technique[124,125]. A variety of models, including decision trees, have been demonstrated to learn and predict the performance of products printed with PBF technology[126]. We also look forward to the good news in this area. In other areas, electronic additive manufacturing is also an emerging research direction. This direction, which requires higher printing accuracy, has also been shown to be realized using MJT technology[127-129]. It is believed that as the AM technology starts to influence more application scenarios, the incentives of interdisciplinary integration will speed up the entry of this processing tool into a virtuous cycle to benefit different application aspects in a more effective manner.

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