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Processing Routes of HEAs, Prospects and Challenges: A Review

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Abstract

Characteristic Properties of high entropy alloys (HEAs) are affected by the processing route. Hence, the functionality and efficiency of HEAs are partly dependent on their fabrication technique. Therefore, this study was aimed at reviewing the available production routes of HEAs and their strengths and weaknesses in order to propose the most suitable techniques to be adopted. The resource materials were sourced from Scopus database and google scholar website of articles published in the last five years, laying more emphasis on the most recently published works. In the study, it was concluded induction melting is the best liquid processing route. More so, non-conventional sintering like spark plasma sintering is much better than conventional sintering. In additive manufacturing, 3D printing and laser cladding equip their products with enviable properties. If precision and purity are required, laser cladding should be adopted, while 3D printing is used when customization, rapid prototyping and high design freedom are of essence.

Keywords:
High entropy alloys, powder metallurgy, conventional sintering, spark plasma sintering, laser cladding, 3D printing

1. Introduction

The idea of introducing some elements into a principal element in an alloy system was to induce some properties that monolithic materials do not have. Take for instance, development of titanium alloy (Ti-6Al-4V) was necessitated because monolithic Ti is susceptible to high temperature oxidation. Hence, reinforcement of monolithic materials is undertaken to improve some properties in the substrate (matrix) or introduce new ones in the system. So, it is from this background that high entropy alloys (HEAs) were conceived and invented. HEAs are typical metallic alloys comprising of 5 elements or more combined in equiatomic or near-equiatomic composition (Figure 1). Each element in the alloy possesses an atomic concentration of 5-35%. The entropy of formation is very high, and this attribute contributes to their exceptional characteristics. Their enthalpy of formation is generally small in comparison with that of conventional alloys, and this contributes to their metastable structural configuration. Hitherto, scholars were saddled with alloy's challenges, such as of high temperature oxidation, high temperature creep and high temperature loss of strength which undermined their applications in aerospace, automotive, electrical and structural systems. Hence, the zeal to develop a more robust material via research and innovation led to the discovery of this all-important alloy. Recall that conventional alloys like Ni, Ti and Al alloys exhibited depreciation of strength when used at temperatures between 350–650 °C. That made their performances above these temperatures to be below average, and this contributed to failure of some aerospace, automotive and structural components (Yeh et al. 2015, Sims et al.2017, Bush and Brice 2012, Troparevsky et al. 2015, Fergachi et al. 2019, Zeng et al.2020).

The invention of HEAs dates back to early 2000s when Cantor and Yeh independently published articles that first mentioned the name "high entropy alloys" (Cantor et al., 2004, Yeh et al. 2004, Ujah et al. 2023). Since then, research on HEAs has doubled as the years passed by because of their unique properties. The special characteristics of HEAs have been attributed to some principal effects existing in them due to the configurational entropy of multiple elements that form them. Those effects include high entropy, lattice distortion, sluggish diffusion and cocktail effects. They all synergize to induce solid solution strengthening, grain refinement, dislocation slip blockade, and overall property improvement (Cantor et al. 2004, Yeh et al. 2004). Nonetheless, one of the principal effects as seen above is the cocktail effect which proposes unpredictable properties. It is as a result of this postulation, and that production technique contributes to their properties that the conceptualization of this study came to be. So, the importance of this research is that sound knowledge of the various processing techniques of HEAs will expose probable properties exhibited by the different fabrication methods. Meanwhile, monolithic alloys like, Ti-based alloys, Cr-based alloys and Al-based alloys are susceptible to high temperature oxidation, poor strength at elevated temperatures and poor creep resistance. Hence, most applications of these alloys in aerospace, automotive, marine and military devices are being replaced with HEAs (Ujah et al. 2024). Contribution of this study to body of knowledge is exposing the strengths and weaknesses of production techniques of HEAs. Through this study, best technique to be used for various materials used in different applications will be provided.

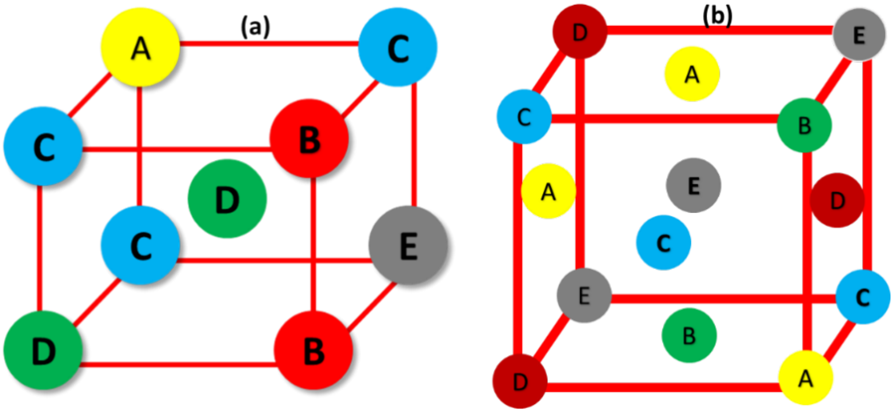

Figure 1. Schematic Diagram of (a) Body-Centered Cubic HEA, (b) Face-Centered Cubic HEA

1.1 Objectives

The objectives of this research include: (a) to investigate the various processing techniques employed in the development of HEAs, (b) to identify the strengths and weaknesses of those techniques.

2. Literature Review

In this section, various processing techniques of HEAs was presented. Figure 2 shows a summary of the processes.

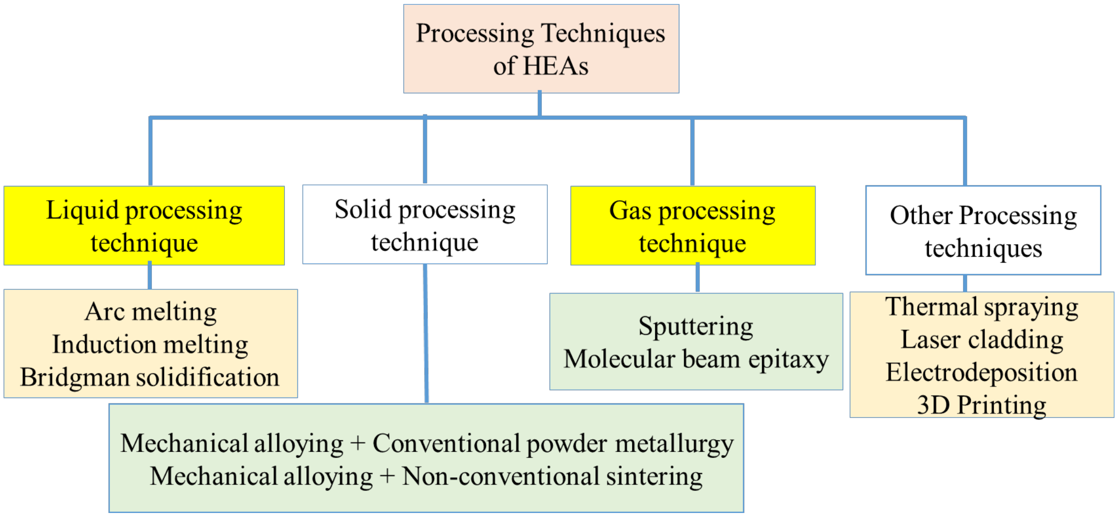

Figure 2. Summary of Processing Techniques of HEAs

2.1. Liquid Phase Processing Route

2.1.1. Arc Melting

Arc melting technique is one of the liquid-phase processing methods used in the fabrication of HEAs which comprises of melting as-received elemental powders with an electric arc under high temperature. The electric arc (EA) is a release of high electric current in between 2 electrodes (Figure 3). The current produces a high temperature at that site that the arc is generated which heats the metal precursors to a molten pool (Zhang and Xing, 2020). The heating is sustained until the whole materials are melted and homogenously mixed. The molten metal precursor blend is cast into a mold constructed in a desired shape. To avert oxidation of the alloy, the process is conducted in an inert environment.

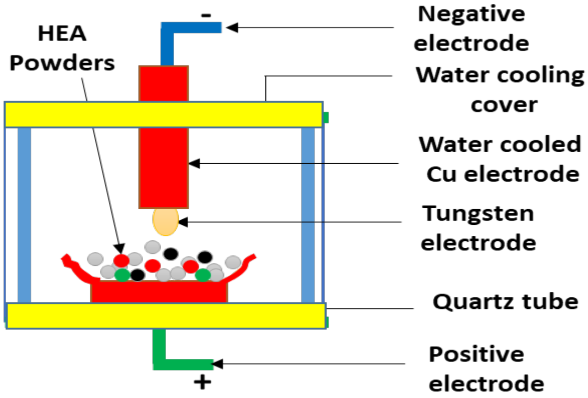

Figure 3. Schematic Diagram of Arc Melting

2.1.2. Induction Melting

Induction melting method is used in melting and casting of HEAs, just like arc melting. Though in this method, an alternating current (AC) is transmitted via a helix of wire to generate a magnetic field. Then, an eddy current is induced by the magnetic field which melts the elemental HEA powder placed in a crucible on top of the coiled wire. The main advantages of induction melting are its fastness and efficiency in comparison to arc melting (Salifu and Olubambi, 2023). It is more friendly to the environment as the heat generated is not as large as that of the arc melting, though it is costlier. This method is more controllable, hence, the microstructure generated by this method is more homogenous (Laplanche et al. 2015).

2.1.3. Bridgman solidification technique

Bridgman solidification method can be used in processing single-crystal or polycrystalline HEAs from a metal melt pool. This method is done by inserting a seed crystal of the required material into a crucible that is filled with a molten material (Derby and Yeckel, 2015). Then, heating of the crucible is followed by application of temperature gradients across the length of the crucible. After heating, the set-up is allowed to cool. During cooling, the seed crystal is pulled upwards in order for the molten material to be sucked into the seed crystal by capillary pressure. The result will be the formation of a unidirectional single-crystal or polycrystalline alloy. This technique is exceptionally utilized for fabricating alloys with a particular grain configuration or alignment; a columnar grain structure, for instance, can be made to have a unique grain direction parallel to the axis of the crystal. It controls the grain growth orientation via manipulating the thermal flow route, temperature slope, and drawing speed of the solid-liquid interface at the event of solidification, in order to enhance uniform cooling gradient and the properties of the alloy (Wang et al., 2019).

2.2. Solid Phase Processing Route

2.2.1. Mechanical alloying with conventional powder metallurgy

Mechanical alloying (MA) consists of a process whereby elemental powders in a vial are blended together through the application of force in a high energy ball mill. Particles of the powders immersed in the ball mill vail are fractured by the applied force into smaller fragments. The broken pieces keep colliding with each other at a high speed inducing their fusioning. As the crushing and alloying continue, the particles get reduced until the intended particle size and structure are attained. This method is employed in producing alloys and composites that can hardly be produced using traditional melting and casting. It is useful in increasing the strength and hardness of alloys, besides reducing their particle sizes (Varalakshmi et al. 2010, Alshataif et al. 2020). Meanwhile, conventional powder metallurgy or sintering involves pressing and sintering of powder particles. This is employed after MA so as to enhance the fusion of the materials. Hence, sintering increases the strength, density and properties of alloyed materials. Recall that MA can only break down the particles of the elemental powders, fuse them partially, but sintering completes the bonding. MA usually leaves the alloyed materials with low densification, high porosity and low strength. To increase the density and strength, and reduce porosity, conventional sintering is employed [139]. This is actually achieved via pressing the bulk material with hydraulic press and heating to the required temperature regime. But prior to the pressing, binding agents and lubricants are added to the ball-milled powders to enhance bonding of particles. This is then followed by heating the bulk mass to an elevated temperature inside a heating furnace (Figure 4), to perpetuate solidification and densification of the bulk mass.

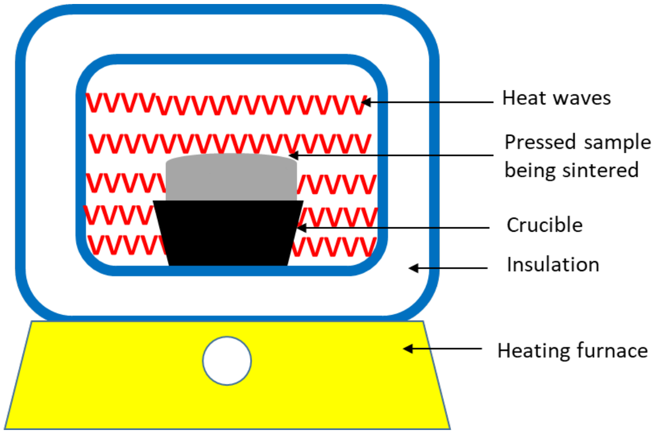

Figure 4. Schematic Diagram of Conventional Sintering Furnace

2.2.2. Non-conventional sintering

Non-conventional sintering is described as a sintering process where heat is not supplied directly to the sample being sintered. Hence, it is a process in which heat in the form of electromagnetic radiation, laser or pulsed current is supplied to a blended powder sample in order to induce inter atomic diffusion that culminates into strong bonds between the powder particles (Cavaliere et al. 2019). This type of heating generates high thermal energy at a very short time, making the non-conventional heating more efficient both in generating superior products and consuming lesser energy. The heating and cooling are completed in a short time, hence, there is no enough time for grain coarsening and formation of detrimental intermetallics (Ujah et al. 2020c). The high heat produced evaporates impurities, which implies that products from this technique are purer with lesser porosity (Rao and Sinha 2020). Report has it that non-conventional heating generates better sinter than conventional heating. One of the most prominent examples of non-conventional sintering is spark plasma sintering (SPS). SPS is a non-conventional sintering technique employed in powder metallurgy which makes use of pulsed direct current (DC) applied concurrently with axial pressure (Figure 5) to consolidate a bulk mass of alloys, compounds or composites. The bulk mass usually possesses enhanced microstructure without pores, zero grain coarsening, inter-locked grain border-lines, cohesive matrix/additive interface, and uniformly dispersed reinforcements (Chu et al.2023).

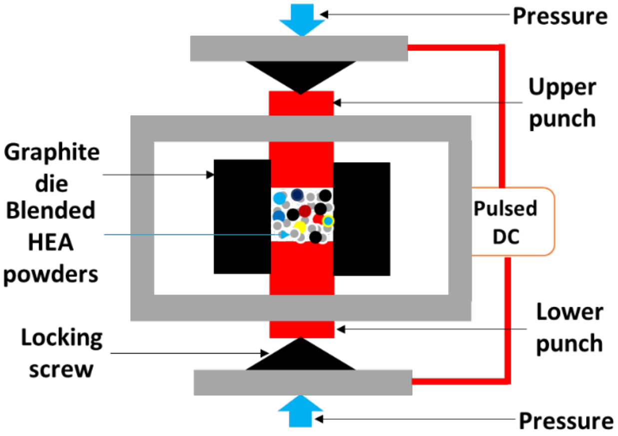

Figure 5. Schematic Diagram of SPS Technique

2.3. Gas Phase Processing Technique

2.3.1. Sputtering

Sputtering is a physical vapour deposition (PVD) process whereby particles of gas or plasma with high kinetic energy are impinged on the surface of a solid material, inducing the release of some particles from the surface (Behrisch 1981), as can be seen in Figure 6. It is usually employed in the deposition of thin films, etching of metals (Baptista et al. 2018), production of optical coatings, semiconductors and nanotech devices (Abid Al Shaybany 2020). Sputtering can be employed in the production of HEAs by depositing multiple elements concurrently on a substrate. In the process, solid solution of elements deposited is formed through the mixing together of impinged particles without segregation of elements. Meanwhile, one of the sources of defects in the sputtering method is the high mechanical energy of the sputtered particles which induces heavy plasticity in the deposited material.

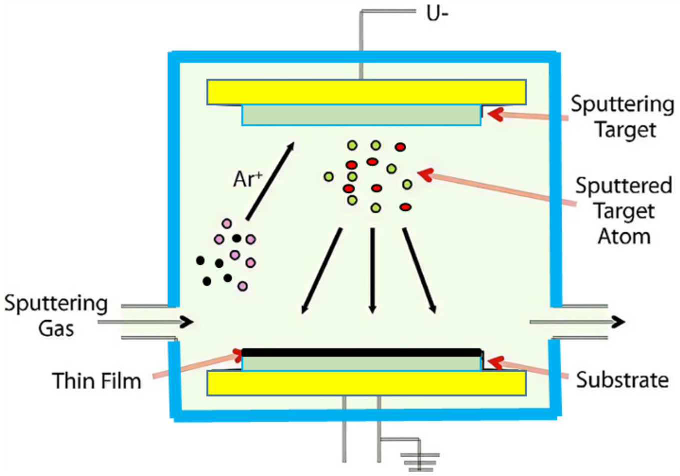

Figure 6. Schematic Diagram of Sputtering Technique

2.3.2. Molecular beam epitaxy

Molecular beam epitaxy (MBE) is a material production process employed for producing thin films of crystalline substances in a vacuum. MBE operation entails channeling rays of atoms towards a substrate that is heated to a high temperature beyond its melting point, so that the beam of atoms is impinged as a single crystal film on the melting substrate (Joyce 1985). This process takes place at a low temperature, hence, defects associated with high temperatures are avoided. This allows accurate tailoring of configuration and weight of the film deposited. MBE process is mostly employed in the production of industrial products like photonics, semiconductors and optical gadgets. The vacuum chamber of MBE is furnished with numerous high-precision mass spectrometers and crystal monitors to regulate the composition and thickness of the film being deposited. As a high-energy beam of atoms is produced from the source and sent to the target substrate, the beam condenses on getting to the melting substrate. The condensed atoms pile up in a strata-by-strata configuration to form a single crystal film. It is this process of single crystal film growth that is called epitaxy (Orton and Foxon 2015). The film formed assumes the same crystal configuration of the substrate. These unique features of MBE make it suitable for the development of intricate configurations like quantum wells and super lattices for semiconductors (Eaglesham 1995).

2.4. Other Processing Techniques

2.4.1. Thermal spraying

Thermal spraying is categorized under "line-of-sight" coating technique. Here, the material used for coating the substrate is poured into the spray gun in the form of powder (Figure 7a), rod or slurry. The feedstock is heated to its melting point and discharged unto the target substrate (Pawlowski 2008). Three forms of thermal spraying method are in existence. They include: heating from burning sources, e.g. "high velocity oxygen fuel spray" (HVOF); heating from electrical plasma or arc, called "atmospheric plasma spray" (APS); and heating from low temperature sources gas expansion, called cold spray (CS) method (Meghwal et al. 2020, Ujah et al. 2020a).

2.4.2. Laser cladding

Laser cladding is a surface modification process which utilizes a laser beam to deposit materials like, metals, alloys, composites, or polymers on the surface of a substrate. Here, a laser is directed onto the substrate (Figure 7b), so that the heat melts material being deposited and the melt pool fuses unto the substrate to form a coating. The technique can be used to improve the wear resistance of a material by depositing wear resistant HEAs through deposition of adequate elements, or to repair damaged parts by depositing protective layer on the machine parts. When comparing laser cladding with thermal spraying, it will be observed that the former has higher accuracy, more controllable and causes lesser damage to the substrate than the latter (Geng et al. 2020, Jiang et al. 2016).

2.4.3. Electrodeposition

Electrodeposition technique is used in the development of HEAs in which alloys are deposited on a surface through the help of an electric current which induces oxidation in the anode and reduction at the cathode immersed in the electrolytic system. In this process, the substance to be deposited is contained in the electrolyte immersed in the electrolytic tank. The tank contains two electrodes that are connected to a power source. When a potential difference is set in the system, oxidation takes place at the anode while the material to be deposited is reduced at the cathode (Sodokin et al. 2023, Li et al. 2024).

2.4.4. 3D Printing

3D printing is a technique which converts a digital 3D model into a physical prototype via additive manufacturing. It should be recalled that additive manufacturing involves joining materials layer by layer to generate a 3D piece of product. The 3D model is divided into tinny layers and transferred into a 3D printer, which creates the wholesome piece of the object layer by layer (Nachal et al., 2019). The fundamental principle of every 3D printer is creating a product, like automotive cylinder (Figure 7c), layer by layer through the addition of precursor materials instead of subtraction of materials as seen in machining. There are various routes whereby 3D printers operate. These routes include: fused deposition modeling (FDM), stereolithography (SLA), selective laser sintering (SLS), direct metal laser sintering (DMLS), and laminated object manufacturing (LOM) (Kafle et al. 2021).

3. Methods

The method used in this study is through sourcing of materials from open literature. The search was based on all works published on HEAs indexed in Scopus database and google scholar in the last five years. Thereafter, the articles obtained were sieved and the relevant ones were selected for the study.

4. Data Collection

From the selected articles, relevant information was sieved out, analyzed and presented in the work as shown.

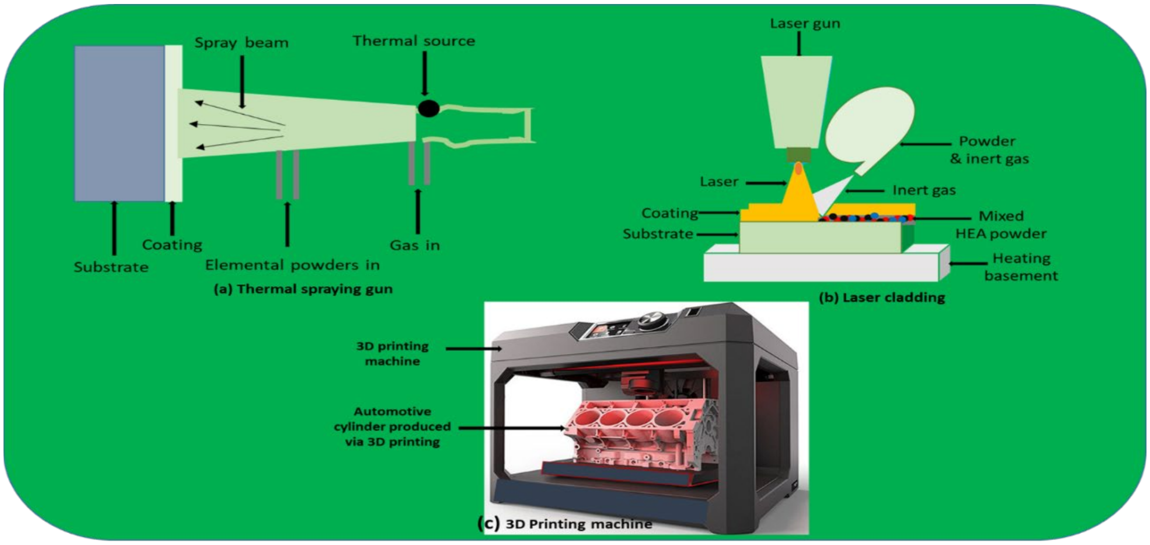

Figure 7. Schematic Diagrams of Further Processing Techniques: (a) Thermal spraying gun, (b) Laser cladding mechanism, (c) 3D Printing mechanism

5. Results and Discussion

The advantages and disadvantages (strengths and weaknesses) of the various processing techniques discussed is presented in Table 5.1

Table 5.1: Strengths and Weaknesses of Processing Techniques of HEAs (Popescu et al.2021, Aliyu and Srivastava, 2019, Liu et al.2022, Rossnagel, 1999, Simon, 2018, Salifu and Olubambi, 2023, Cieslak et al. 2018, Ujah et al. 2020b)

Type	Advantages	Disadvantages
Arc melting	It is simple and affordable. It is scalable. It can be commercialized It is very fast method.	Product has heterogeneous microstructure which affects the properties. It requires a lot of

		energy. It is not environmentally friendly due to high temperature.
Induction melting	Faster and more efficient than arc melting. It is more environmental friendly.	It is costlier than arc melting. It may cause contamination to its products.
Bridgman solidification	It controls grain growth. Efficient in Czochralski growth for single crystal of semiconductors. Less expensive.	It can produce defects like voids, large columnar grains, inclusions and segregation of elements
Conventional sintering	It is affordable, scalable and very efficiently used in industries for ages	It is slow, requires high temperature, allows grain growth, and expensive.
Non-conventional sintering (SPS)	Produces refined grains, uniform microstructure, purified products, highly densified objects with superior properties. Takes shorter time than conventional sintering and more energy saving.	It is more expensive than conventional sintering. It is not easily scalable, so not commercially viable.
Sputtering	It is eco-friendly. It is scalable so, it is industrially useful, it can deposit very thin film	It cannot deposit on complex geometry, slow operation and costly equipment.
Molecular beam epitaxy	Film deposited by MBE is smoother than film deposited by sputtering. It can handle complex geometries,	It requires very high vacuum, Deposition rate is very low, it exhibits defects like pinhole.
Thermal spraying	It is very fast, versatile, affordable, eco-friendly,	It lacks smooth finishes. It harbors impurities and pores.
Laser cladding	It has small heat affected zone, minimal distortion of the substrates, and high metallurgical bonding. It is used where precision, purity and high efficiency cannot be compromised.	Its equipment is costly. It requires skilled operators. It is not well-suited for large-scale production.
Electrodeposition	It is versatile, cost effective, easily controlled, and can deposit on complex geometry.	Electrodepositing low melting alloys is usually very hard, it is slow, and prone to pores.
3D printing	It has higher design freedom, fast in producing prototypes, very easy to produce customized products, there is a minimal waste.	It is selective in materials that can be used for production, it has size limitations, initial cost of 3D printer is not cheap, lack of strength and durability in finished products.

6. Conclusion

Review of the processing techniques used in the development of HEAs has been completed. The following points were drawn from the study.
Among the liquid phase processing techniques, induction melting is the most environmentally friendly, fastest and most effective. Bridgman solidification is efficient but costly. So, it is only applied for special production of direction-sensitive crystals like semiconductors.

In solid phase processing method, non-conventional sintering is more energy saving, more efficient and faster. But conventional sintering is more scalable, cheaper but consumes higher energy. In terms of their products, non-conventional route produces better products.

In gas phase, sputtering is eco-friendlier but it is a “line-of-sight” process, hence, it cannot handle complex geometries. However, in molecular beam epitaxy, the film deposited is smoother, so, it gives better finishes than sputtering, and can handle more complex geometries.

In further techniques considered, laser cladding is used where precision and purity of products are of essence, like in medical equipment or aerospace components. But for customization of products, 3D printing is the best choice. Meanwhile, when mass production, cheap and versatile products are being considered, electrodeposition should be the choice route.

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