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Strengthening and Toughening of 3D Printing High Entropy Alloy

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Abstract

By comparing the differences between high-entropy alloys and traditional alloys, this paper highlights the outstanding advantages of high-entropy alloys in modern 3D printing, and introduces the characteristics, defects and corresponding solutions of different types of 3D printing technologies. DED uses a laser, electron beam, or arc to melt the powder or wire form during deposition. SLM is to melt metal powder on a powder bed and use high-energy lasers to print geometrically complex products. EBM uses electron beam to melt metal powder. In addition, the constituent elements, processing conditions and working temperature are also important factors in determining the mechanical properties of high-entropy alloys. By adding or reducing elements and conducting some treatment, the properties of high-entropy alloys such as tensile strength, compressive properties, fracture strength and plastic strain can be found changed.

Keywords: 3D printing technology • High entropy alloy • Strengthening • Toughening

Introduction

High entropy alloy and 3D printing

High entropy alloy, referred to as HEA, is a new material which has attracted considerable attention in material science and engineering. Traditional alloys are dominated by single elements, such as steel, titanium alloy, aluminum alloy and magnesium alloy with Fe, Ti, Al and Mg as main elements respectively. The composition of traditional alloys is to add a small amount of trace elements to the main elements to obtain a new alloy with better properties. While only a small part of its properties can be adjusted. In the past experience, the more metal elements are added, the easier the alloy material is to become brittle [1]. Found that the increase of alloy components will lead to higher mixing entropy. The multi-component high-entropy alloys will form simple high-entropy solid solutions without the formation of various intermetallic compounds and complex phases [2]. High-entropy alloys break this limitation of traditional alloys and boldly use a variety of metal elements as the main elements, but they are not easy to embrittle. In addition, it also has high toughness, strength, high temperature oxidation resistance and corrosion resistance, etc., which has attracted extensive attention of researchers (Figure 1).

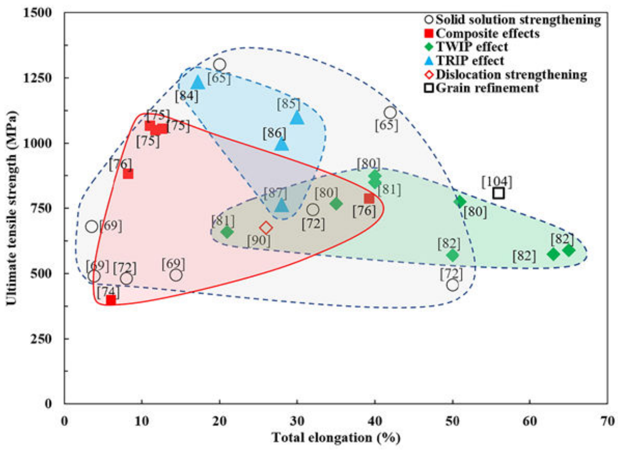

Figure 1. Room-temperature mechanical properties of HEAs classified according to the phases present in the microstructure.

The rapid development of high-entropy alloys prepared by 3D printing in recent years has brought great potential for the fabrication of complex high-entropy alloy products with desirable properties, thus stimulating their applications in industry. Due to the unique advantages of 3D printing technology in printing products with free design and geometric complexity, 3D printing of high-entropy alloys has attracted more and more attention from academia and industry. For printing high-entropy alloy products, DED (currently the most popular printing process for high-entropy

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alloy products) and PBF (laser powder bed fusion technology) processes have been extensively explored. During these processes, a focused high-energy beam interacts with the powder, creating a melt pool that rapidly melts and solidifies. This rapid solidification is beneficial to avoid element segregation, which usually occurs in linear defects (dislocations) or surface defects (stacking defects, grain boundaries, phase boundaries, etc.) and prevent the formation of brittle intermetallic compounds, improving the mechanical properties of the product [3].

Due to their excellent properties, high-entropy alloys are considered as a new type of structural material that has the potential to replace traditional alloys.

Materials and Methods

High entropy alloy 3D printing technology

DED: DED is the most popular printing process of high-entropy alloy products. It uses laser, electron beam or arc to melt powder or wire during deposition [4,5]. The process parameters of DED, including laser power, scanning speed, powder flow rate and hatch space, play a vital role in the performance of printed products [6]. The optimized process parameters produce continuous and completely melted monorail, so as to ensure the high quality of printed products. When preparing high-entropy alloy products with different element powders in situ, process optimization is the key. When melting at least four element powders, it is likely to produce discontinuous and porous melt pools with uneven element distribution [7]. Therefore, it is usually necessary to remelt each solidification pool to reduce this disadvantage and achieve uniform element distribution. The cooling rate of DED depends on the applied laser power and scanning speed, and has an important impact on the phase and crystal characteristics of printed high-entropy alloy products. The increase of laser power reduces the cooling rate and provides enough time for phase transition.

SLM: SLM is a kind of PBF printing process, which is characterized by melting metal powder on the powder bed and printing geometrically complex products with high-energy laser [8-15]. The main difference between DED and SLM lies in the conveying mode of powder. In SLM, high-entropy alloy powder is diffused on the building platform through the recoating machine (blade or roller), while in DED; high-entropy alloy powder is blown out by nozzle. SLM is introduced into the printing of high-entropy alloy products because it can produce complex parts with excellent mechanical properties. The printability of SLM to high-entropy alloy powder can be evaluated by single track, single-layer and multi-layer research.

Sputtering refers to the ejection of molten metal from the molten pool, which is a representative phenomenon in the SLM process. The metal evaporation caused by high energy input produces a recoil force to overcome the surface tension, which is opposite to the compression effect of the recoil force, resulting in sputtering [16]. Sputtering is harmful to the mechanical properties of SLM products and may lead to their failure. Sputtering can be reduced by increasing laser scanning speed or reducing laser power. However, the optimization process is ineffective in improving the mechanical properties of products by minimizing laser energy and reducing sputtering [17]. In addition, it is easy to form oxide layer on the droplet during sputtering, which expands the quality and size of sputtered particles [18]. Therefore, when designing SLM high-entropy alloy, the recoil force and sputtering can be reduced by decrease the proportion of volatile elements, and the volatile elements can be compensated to maintain the chemical composition of printed high-entropy alloy products and avoiding the use of high affinity oxidizing elements.

EBM: EBM uses electron beam to melt metal powder [19]. Due to the special operating environment of electron beam, EBM needs to manufacture components in a high vacuum environment of 10-4 mbar or more, providing an ideal non-pollution environment for manufacturing. Its working principle is similar to SLM, but its working conditions are different. Before scanning and melting, the high-entropy alloy powder is pretreated in the powder bed by electron beam up to 1100℃, which is a unique procedure in EBM to reduce the thermal gradient of printed products. Compared with SLM, EBM's lower cooling rate (10^3~$10^5 s^{-1}$) and smaller temperature gradient help to reduce residual stress, deformation and cracking tendency [20,21].

In-situ lloying of at least four premixed elemental powders to produce high-entropy alloy products using EBM and SLM techniques is challenging. During the powder diffusion process, the distribution of element powder in each printed layer will be non-uniform, and the rapid solidification of the molten pool will inhibit sufficient convection and diffusion of elements. This will lead to chemical in homogeneities in the layers and thus cause microstructural in homogeneities of the printed product. Research on high-entropy alloy products $Al_{0.5}CrMoNbTa_{0.5}$ prepared by EBM *in-situ* alloying of mixed element powders showed that TaNbMo-based and (TaMoNbCr)Al solid solutions were formed. However, the microstructure uniformity and mechanical properties of high-entropy alloys still need to be studied for a comprehensive understanding [22]. In addition, a recent study investigated the SLM of $Al_{0.26}CoFeMnNi$ and $Al_{0.26}CoFeMnNiC_{0.12}$ high entropy alloy products using mixed element powders, showing that the use of high energy densities facilitates an enlarged melt pool and uniform elemental distribution inside [23]. However, these studies are still limited and further exploration is required to validate the effectiveness of printing high-entropy alloy products *via* SLM and EBM by *in-situ* alloying methods (Figures 2 and 3).

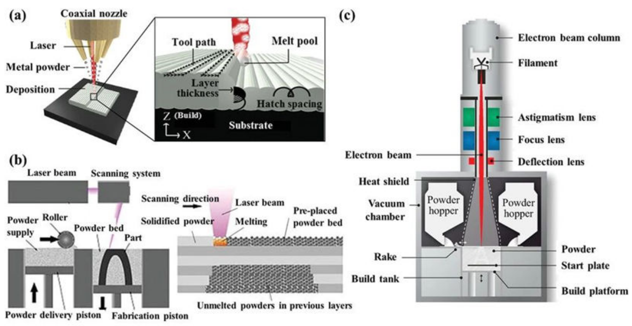

Figure 2. Schematic of 3D printed high-entropy alloy products: a) DED, b) SLM, and c) EBM (Reproduced with permission.

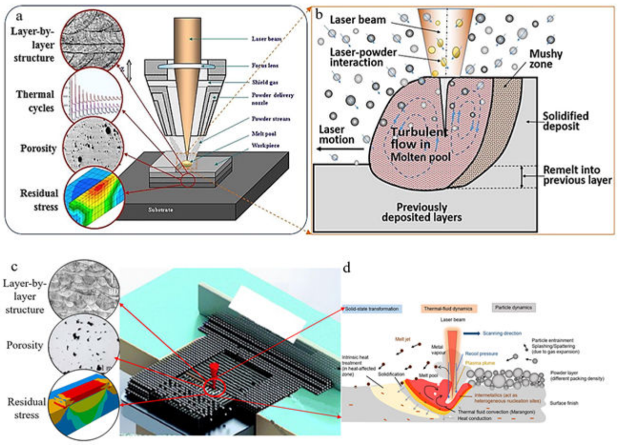

Figure 3. Schematic illustrations and microstructure properties of a component produced by a, b) DED and c, d) PBF methods. Reproduced with permission from elsevier.

Changes of strength and toughness of high-entropy alloys under different processing conditions

Constituent elements, processing conditions and working temperature are important factors that determine the mechanical properties of 3D printing high-entropy alloy products. Adding additional elements to the existing high-entropy alloy can refine its microstructure and promote the formation of precipitates, so as to achieve the strengthening effect [24]. Zhou et al. proved that adding carbon can improve the tensile strength of CoCrFeNi high-entropy alloy products printed by SLM [25]. Wu et al. further explained that this strengthening mechanism is the combination of dislocation network strengthening and nano carbide strengthening [26]. Luo, et al. developed Co free AlCrCuFeNi high-entropy alloy product through SLM and studied the anisotropy of its compressive properties [27]. The fracture strength and ductility of high-entropy alloy perpendicular to the construction direction are higher than those in parallel arrangement, which is due to the anisotropy of crystal characteristics. In the vertical direction, the grains grow preferentially along the <100> direction, which helps to improve the ductility. The rich copper nano precipitates at the grain boundary leads to the increase of fracture strength. In terms of process conditions, the optimization of energy density increases the relative density of printed high-entropy alloy products, changes the crystal phase direction, reduces the grain size and improves the mechanical properties [28-32]. In addition, high-entropy alloy treatment usually improves the mechanical properties of high-entropy alloy products by eliminating various defects and releasing residual stress in DED and SLM printed high-entropy alloy products. Annealing can promote recrystallization and effectively reduce residual stress. In the study of Zhang, et al. the fracture strength and plastic strain of AlCoCuFeNi high-entropy alloy products printed by SLM increased, but the compressive yield strength decreased by 47% after annealing [33]. Similar yield strength reductions were observed in annealed SLM printed CoCrFeNi and DED printed CoCrFeMnNi products [34,35]. High-entropy alloy treatment can also lead to grain coarsening of printed high-entropy alloy products. The effects of grain coarsening and residual stress relaxation on the tensile properties of high-entropy alloy products offset each other. Hot Isostatic Pressing (HIP) promotes the densification of high-entropy alloy and increases the coarsening of grains and precipitates. Joseph, et al. found that HIP induced microstructural coarsening, promoted chemical homogenization, and improved certain mechanical properties of 3D printed $A1_{0.3}$CoCrFeNi high-entropy alloy products [36]. The relative density of the printed product is 99.4%, which increases to 99.5%after HIP treatment. The results show that the number of macropores larger than 5 μm in the hydrothermal glue is less and the density slightly increases. However, due to the brittle fracture of the σ phase under tension-compression conditions and the coarsening of hard BCC at the grain boundaries, other mechanical properties of the $Al_{0.85}CoCrFeNi_{8}$ high-entropy alloy product were affected. The coarse grain boundary particles produced during the HIP process of high-entropy alloys are the main cause of their ductility loss. The coarse grain boundary particles produced during the HIP process of high-entropy alloys are the main cause of their ductility loss. Similarly, Li, et al. obtained an 8% increase in tensile strength but a 49% decrease in elongation for SLM-printed CoCrFeMnNi high-entropy alloy product [37].

3D printed high-entropy alloy products responded similarly to HIP and annealing heat treatments as conventionally fabricated products. For example, the HIP-treated DED-printed AlCoCrFeNi high-entropy alloy products exhibited grain coarsening, chemical homogenization, and relief of residual stress, similar to the HIP-treated cast AlCoCrFeNi products [38,39]. Both hot forming and annealing treatments lead to the precipitation of σ phase in printed high-entropy alloy products, and the precipitation of σ phase in cast products after heat treatment has also been reported. Solution treatment is beneficial to enhance the strength of printed high-entropy alloy products. The effects of water quenching and air cooling on the mechanical properties of SLM and EBM printed $Co_{1.5}CrFeNi_{1.5}Ti_{0.5}Mo_{0.1}$ products were studied by Fujieda, et al. (Figure 4).

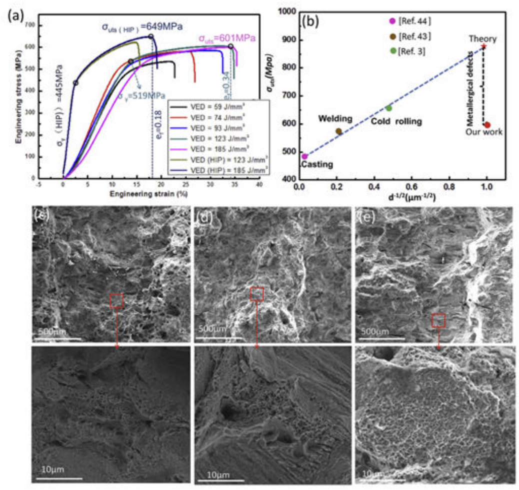

Figure 4. a) Tensile engineering stress-strain curves of the SLM and SLM-HIP CoCrFeMnNi alloy. b) Grain size dependence of ultimate tensile strength (suts) [44] and fracture morphologies c) 185 J/mm^3, d) 123 J/mm^3, e) SLM-HIP 185 J/mm^3 (Reproduced with permission).

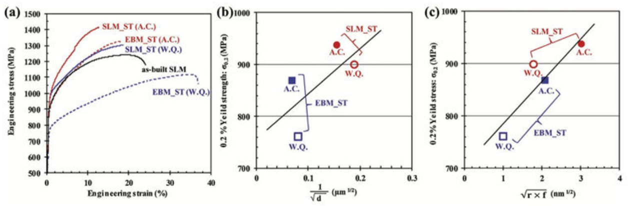

Figure 5. The comparison of mechanical properties of $Co_{1.5}CrFeNi_{1.5}Ti_{0.5}Mo_{0.1}$ high-entropy alloy products printed by SLM and EBM with the solution treatments of both water quenching and air cooling: a) tensile stress-strain curves, b) relationship between yield strength and grain size (d: average grain diameter), and c) dependence of size and volume fraction of ordered particles on yield strength (f: volume fraction of ordered particles; r: radius of ordered particles). a–c) Reproduced with permission. Copyright 2019, Elsevier.

The yield strength of solution treated printed high-entropy alloy products largely depends on the precipitation morphology of ordered particles, and ordered particles have a weak blocking effect on dislocation movement , which both help to improve the ultimate tensile strength of the product. The interaction mechanism between dislocation and deformation induced twinning contributes to the continuous accumulation of dislocations and ensures the high work hardening rate and good tensile properties of high-entropy alloy at low temperature Figures 5 and 6.

Chew, et al. evaluated the tensile properties of DED-printed CoCrFeMnNi high-entropy alloy products at different temperatures of -130,0, and 25°C [40].

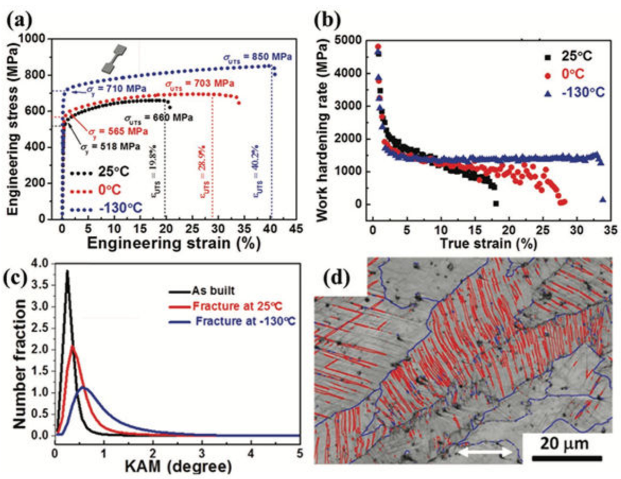

Figure 6. a) Tensile stress–strain curves of DED-printed CoCrFeMnNi high-entropy alloy products at 25, 0, and -130°C, respectively; b) strain hardening curves deformed at 25,0, and -130°C, respectively; c) kernel average misorientation distributions, and d) grain boundary map showing the deformation twins at -130°C; d) The blue and red lines represent the high-angle grain boundaries and deformation twins, respectively. The white arrow indicates the tensile axis. a–d) Reproduced with permission.

Wang et al. used the laser powder bed additive manufacturing technology, using the classic five-element high-entropy alloy system (CoCrFeMnNi) and Fe-based metal glass as printing raw materials, successfully realized bimetallic printing, and the printed materials perfectly inherited the advantages of the two materials. The wide hardness values range and high upper limits (hardness values from ~251 HV to ~1210 HV) and excellent corrosion resistance (from ~0.967 mm/year to ~0.765 mm/year), perfectly giving HEA a good combination of strength and ductility. Metal glass of different thicknesses is printed on a printed high-entropy alloy, and the hardness value of the combined material corresponding to different thickness parameters is measured (to assess corrosion resistance), and the metal glass layer thickness is expressed as T 0 (no MG layer / MG-free), T 300 (300 μm thick MG layer), T 600 (600 μm thick MG layer) and T 900 (900 μm thick MG layer) (Figure 7).

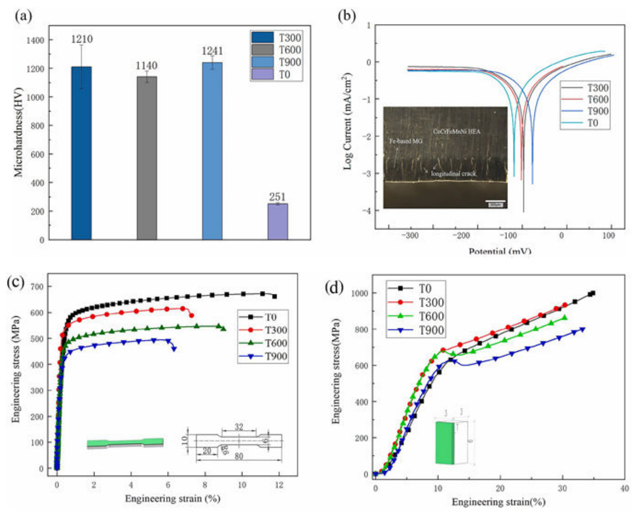

Figure 7. a) Microhardness b) Corrosion resistance c) Tensile and d) Compressive properties of the as-printed bimetals.

Discussion

At present, the strength and toughness of multicomponent high entropy alloys are not high, and the plastic deformation ability is poor. In order to solve this problem, the microstructure and mechanical properties of multi-element high-entropy alloy were studied, and the microstructure and strengthening mechanism of the alloy were studied. At present, the multi-component high-entropy alloy developed only has compressive properties, but not tensile properties at room temperature, which limits their wide application in real life [41-47]. In G-MV3 alloy system, with the increase of Mo element, the change trend of room temperature compressive properties is different from that of G-MV10. Because there is no precipitation of the second phase, the yield strength increases continuously, the plastic deformation decreases, and there is no turning point. Among them, M3V3 alloy has the best comprehensive properties. XRD phase analysis and SEM structure observation show that there is only one BCC phase in G-MV3 alloy system. Due to the addition of Mo element with high bulk modulus, the yield strength of the alloy is improved. In multicomponent alloy system, V element can enhance the alloy, but in order to ensure good plastic deformation ability, V content needs to be reduced. In addition, according to the experimental results of Dai et al., with the increase of Zr content, the fracture strength of multi principal component high-entropy alloy first increases and then decreases. When it reaches a certain fixed value, the comprehensive mechanical properties of the alloy are the best. The work hardening after yield makes the alloy have high strength and good plastic deformation ability. The alloy can fully meet people's requirements for the properties of metal materials, and can be further applied to relevant industries to promote the further development of machining industry.

Conclusion

In recent years, high entropy alloys have been favored by many researchers because of their excellent properties, and are expected to replace the position of traditional alloys in related industries, so as to further promote the development of related industries. In the processing of high entropy alloy, many factors will affect its mechanical properties. Adding C element and reducing Co element can increase the fracture strength and improve the ductility. The optimization of energy density will also increase the relative density and mechanical properties of high-entropy alloy products. After high-entropy alloy treatment, the plastic strain increases, but the compressive yield strength decreases. Hot isostatic pressing promotes the densification of high-entropy alloy and increases the coarsening of grains and precipitates. At the same time, the product produces brittle fracture and ductility loss. The yield strength of the alloy can be improved by increasing the Mo element and reducing the content of V element and adding appropriate amount of Zr element. Therefore, under the condition of reasonable control of the environment and its own element influencing factors, available high-entropy alloys can be manufactured according to different needs as raw materials for 3D printing to promote the development and progress of related industries.

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Author Contributions

S. C. Cao and A. Guo designed the study. Z. Lin and wrote the main draft of the paper. W. Xiong and S. Zhan organized figures.

Conflicts of Interest

The authors declare no conflict of interest.

Data availability statements

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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