

RESEARCH ON HIGH-ENTROPY ALLOYS PRODUCED FROM 3D-PRINTED METAL POWDERS

Gabriela-Victoria Mnerie^{1,2}, Nicușor-Alin Sîrbu³, Emilia Dobrin^{4,5}, Lavinia-Ileana Sîrbu⁶, Denis-Andrei Predu⁷

Abstract: High-entropy alloys (HEAs) have gained significant attention in recent years due to their unique combination of mechanical strength, thermal stability, and corrosion resistance, making them ideal candidates for advanced industrial applications such as aerospace, medical, and energy sectors. In parallel, additive manufacturing (AM), particularly 3D printing using metal powders, has emerged as a promising technique for fabricating complex geometries with tailored material properties.

This paper aims to investigate the feasibility of manufacturing high-entropy metallic materials using metal powders and 3D printing technologies. The study will thoroughly analyse:

- The process of obtaining HEA powders with suitable properties for 3D printing.
- The performance of the powders during the additive manufacturing process.
- The mechanical, microstructural, and functional properties of the produced materials.

The paper contributes to the advancement of knowledge regarding the interaction between HEA compositions and additive processes, providing solutions to current challenges in this field.

Key words: high-entropy alloys, additive manufacturing, 3D printing, metal powders, microstructure, mechanical properties, selective laser melting

1. INTRODUCTION

High Entropy Alloys (HEAs) are a relatively new class of advanced materials, characterized by solid solutions of multiple chemical elements in similar atomic proportions (usually at least four or five main elements). These materials offer remarkable properties such as high mechanical strength, thermal stability, corrosion and wear resistance, and structural stability under extreme conditions. Due to these characteristics, HEAs are promising candidates for applications in critical fields such as aerospace, energy, mechanical engineering, and medicine [1].

At the same time, additive manufacturing, commonly known as 3D printing, has become a cutting-edge technology for creating complex, customized, and functional components. 3D printing with metal powders, using technologies such as Selective Laser Melting (SLM), Directed Energy Deposition (DED), or Electron Beam Melting (EBM), allows for the fabrication of parts directly from digital models, reducing waste and providing geometric flexibility [2].

The combination of HEA alloys and additive manufacturing technologies represents a major innovation opportunity. This allows for the creation of high-performance components, tailored to extreme requirements, opening new perspectives for high-tech industrial applications [3].

The integration of High Entropy Alloys into 3D printing processes is of major research interest due to the technical challenges associated with it. The properties of metal powders, such as morphology, particle size, and flowability, significantly influence the success of the additive manufacturing process. Additionally, processing parameters such as laser power, scanning speed, and

¹Researcher, PhD, Eng., National R&D Institute for Welding and Material Testing (ISIM Timisoara), 30, Mihai Viteazu Blv., 300222 Timisoara, Romania, gmnerie@isim.ro

²Associate professor, PhD Eng., Faculty of Engineering, Ioan Slavici University, 144, Aurel Paunescu Podeanu Str., 300763 Timisoara, Romania, gabriela.mnerie@yahoo.com

³Senior Researcher, PhD Eng., National R&D Institute for Welding and Material Testing (ISIM Timisoara), 30, Mihai Viteazu Blv., 300222 Timisoara, Romania, asirbu@isim.ro

⁴Researcher, PhD Student, Eng., National R&D Institute for Welding and Material Testing (ISIM Timisoara), 30, Mihai Viteazu Blv., 300222 Timisoara, Romania, edobrin@isim.ro

⁵PhD Student, Eng., Politehnica University Timișoara, 2 Piața Victoriei, 300006, Timisoara, Romania, emilia.dobrin@student.upt.ro

⁶Eng., National R&D Institute for Welding and Material Testing (ISIM Timisoara), 30, Mihai Viteazu Blv., 300222 Timisoara, Romania, lbuzatu@isim.ro

⁷Student, National R&D Institute for Welding and Material Testing (ISIM Timisoara), 30, Mihai Viteazu Blv., 300222 Timisoara, Romania, dpredu@isim.ro

layer thickness play a crucial role in determining the microstructure and mechanical properties of the fabricated parts [4].

This topic is relevant not only from an academic standpoint but also from an industrial one, with the potential to contribute to the development of innovative components for applications in extreme environments or in cases where material performance is essential.

The main goal of this paper is to explore the potential uses of high-entropy metallic materials made from metal powders through 3D printing technology [5]. The research focuses on characterizing the metal powders used, analysing the additive manufacturing process, and evaluating the resulting parts in terms of microstructure and mechanical properties.

This research contributes to understanding how high-entropy alloys can be effectively integrated into additive manufacturing processes, a topic that has been insufficiently explored to date. The originality of the paper lies in [6]:

- Studying the interactions between the properties of HEA metal powders and 3D printing parameters.
- Correlating the microstructure of the fabricated materials with their mechanical and functional properties.
- Designing practical solutions for applying HEAs in critical industrial fields.

2. MATERIALS AND METHODS

2.1. High Entropy Alloys (HEAs)

High entropy alloys (HEAs) (figure 1) are metal alloys composed of five or more principal elements, each having an atomic concentration ranging from 5% to 35%. Unlike conventional alloys, which are dominated by a single principal element, HEAs are based on the high entropy effect to stabilize simple solid phases (e.g., face-centred cubic - FCC, body-centred cubic - BCC). This unique composition leads to a distinct set of properties that set HEAs apart from traditional alloys, such as enhanced mechanical strength, thermal stability, corrosion resistance, and wear resistance [7]. These characteristics make HEAs particularly attractive for applications in industries such as aerospace, energy, and medical devices, where materials must perform under extreme conditions.

The high entropy effect arises from the mixing of several elements in nearly equal proportions, which results in a significant increase in configurational entropy. This increase in entropy helps to stabilize high-temperature phases, reducing the tendency of the alloy to form a single dominant phase. The resulting alloys can exhibit a wide range of microstructures and properties depending on the specific elemental composition and processing conditions [8].

HEAs are typically classified into three major groups based on their crystal structure: FCC-based, BCC-based, and mixtures of FCC and BCC phases. The crystal structure plays a crucial role in determining the mechanical properties, such as strength, ductility, and hardness, as well as the thermal and corrosion resistance of the alloy.

The properties of HEA alloys result from several key effects [9]:

- High entropy effect: The mixing of multiple elements in an alloy promotes the formation of a stable solid solution, reducing the tendency for phase separation.

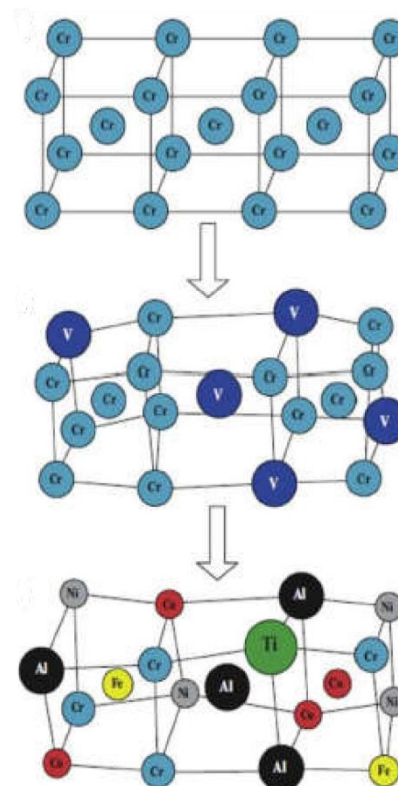


Figure 1 – HEAs structure, [7]

- Mixing effect: The combination of different elements influences the electronic structure and mechanical properties.
- Slow diffusion effect: Atom diffusion is slower in HEAs, providing them with thermal stability.
- Lattice distortion effect: Atomic size differences between elements cause distortions that contribute to mechanical properties.

2.1.1. Compositions and representative examples

High entropy alloys synthesized in the induction furnace were chemically analysed to determine the percentage of chemical elements [10]. The obtained data are presented in Table 1.

Table 1 – Nominal chemical composition and the composition determined by chemical analysis of the synthesized alloys, [10]

	% gr					
	Ti	Fe	Mn	Nb	Ta	Zr
<i>FeTa_{0,5}Nb_{0,5}Ti_{1,5}Zr_{0,5} – $\rho = 7,61 \text{ g/cm}^3$</i>						
Nominal	23,14	18,02	-	14,97	29,17	14,7
Analysis	22,0	22,0	-	16,1	18,0	13,0
<i>FeMnNb_{0,5}TiZr_{0,5} – $\rho = 6,65 \text{ g/cm}^3$</i>						
Nominal	19,09	22,28	21,92	18,53	-	18,18
Analysis	16,8	20,4	21,0	20,7	-	15,0
<i>FeTa_{0,5}Nb_{0,5}TiZr_{0,5} – $\rho = 8,06 \text{ g/cm}^3$</i>						
Nominal	16,73	19,51	-	16,22	31,62	15,92
Analysis	17,0	22,1	-	14,7	23,0	13,5
<i>FeTa_{0,5}Nb_{0,5}Ti_{1,5}Zr_{0,5} remelted – $\rho = 7,61 \text{ g/cm}^3$</i>						
Nominal	23,14	18,02	-	14,97	29,17	14,7
Analysis	19,2	18,42	-	15,8	25,0	12,4
<i>FeTa_{0,5}Nb_{0,5}Ti_{1,5}Zr_{0,5} heat treated</i>						
Analysis	24,0	19,0	-	16,9	15,8	15,5
<i>FeTa_{0,5}Nb_{0,5}TiZr_{0,5} heat treated</i>						
Analysis	16,4	18,7	-	11,2	30,0	14,0
<i>FeMnNb_{0,5}TiZr_{0,5} heat treated</i>						
Analysis	20,0	16,0	-	16,1	25,0	13,0

Figure 2 shows samples of high entropy alloys with biocompatible properties, synthesized in the induction furnace and embedded for optical characterization [10].

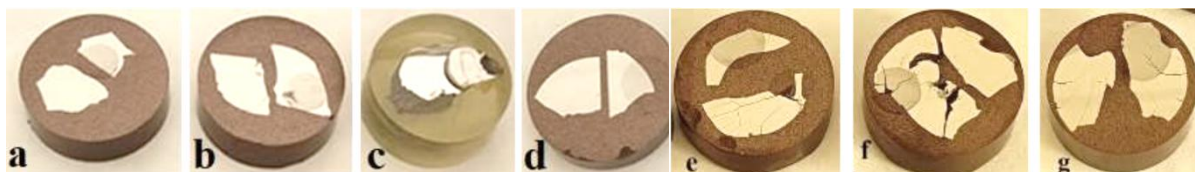


Figure 2 – High entropy alloys with biocompatible properties embedded for optical characterization, [10]

- a) - $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$; b) - $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{TiZr}_{0,5}$; c) - $\text{FeMnNb}_{0,5}\text{TiZr}_{0,5}$;
d) - $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ remelted; e) - $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ heat treated;
f) - $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{TiZr}_{0,5}$ heat treated; g) - $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ remelted, heat treated

Figures 3 and 4 show the micrographs of the $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ and $\text{FeMnNb}_{0,5}\text{TiZr}_{0,5}$ alloys.

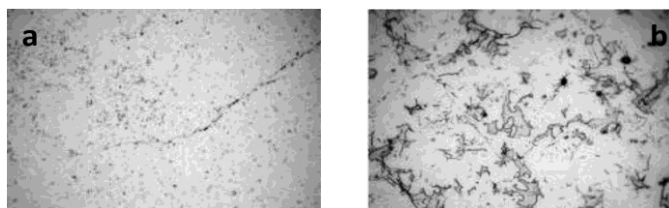


Figure 3 – Micrographs of the $\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ alloy [10]

- a) - unattached sample, 200x; b) - etched sample, immersed in oil, 900x

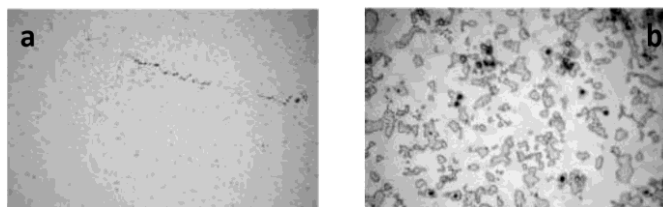


Figure 4 – Alloy micrographs $\text{FeMnNb}_{0,5}\text{TiZr}_{0,5}$, [10]
a) - unattached sample, 200x; b) - etched sample, immersed in oil, 900x

To analyse the behaviour of high entropy alloys with biocompatible properties, Vickers microhardness was determined by performing three indentations, after which an average value was calculated and recorded in Table 2 [11], [12].

Table 2 – Vickers microhardness values for high entropy alloys, [10]

Alloy	Vickers microhardness values
$\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$	802,8 HV
$\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{TiZr}_{0,5}$	898,2 HV
$\text{FeMnNb}_{0,5}\text{TiZr}_{0,5}$	802,9 HV
$\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ remelted	699,0 HV
$\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ heat treated	910,4 HV
$\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{TiZr}_{0,5}$ heat treated	782,2 HV
$\text{FeTa}_{0,5}\text{Nb}_{0,5}\text{Ti}_{1,5}\text{Zr}_{0,5}$ remelted, heat treated	729,4 HV

2.1.2. Characteristic properties

HEAs have attracted considerable interest in recent years due to their unique combination of physical, chemical, and mechanical properties, which set them apart from conventional materials. HEAs are typically composed of four or more principal elements in nearly equal proportions, which provides them with exceptional characteristics resulting from complex atomic-level interactions [13].

HEAs exhibit a wide range of remarkable properties:

- High mechanical strength and ductility at low temperatures: HEAs demonstrate an impressive combination of tensile strength and ductility, even at extremely low temperatures. This property makes them suitable for use in cryogenic environments, where conventional materials may become brittle. Their homogeneous microstructure and complex deformation mechanisms contribute to these superior characteristics [14];
- Structural stability at high temperatures: Unlike traditional alloys, HEAs exhibit remarkable thermal stability, maintaining their mechanical properties even at elevated temperatures. This characteristic is due to their unique solid-phase configuration, which minimizes diffusion and grain growth, thus preventing structural degradation.
- Exceptional resistance to corrosion and wear: HEAs have superior resistance to corrosion and wear due to their diverse chemical composition and the formation of protective surface films. This makes them ideal for applications in aggressive environments such as chemical, maritime, or medical sectors, where conventional materials may suffer rapid degradation [15].

Due to these outstanding properties, high entropy alloys are used in a variety of demanding applications, including [14]:

- Aerospace industry, for components requiring a combination of low weight, high mechanical strength, and thermal stability.
- Gas turbines, where thermal stability and wear resistance are essential for the efficiency and lifespan of components.
- Chemical equipment, exposed to aggressive substances and extreme temperatures.
- Biomedical devices, due to their excellent biocompatibility and corrosion resistance in biological fluids.

2.2. Additive manufacturing technologies (3D Printing)

Additive Manufacturing (AM) represents an innovative process involving the layer-by-layer deposition of material to build parts directly from a digital model [16]. This technology enables the production of complex components with optimized geometry and excellent mechanical and functional properties that cannot be achieved through conventional methods.

3D printing with metal powders is widely used in industries such as aerospace, automotive, medical, and energy due to its significant advantages, including material waste reduction, design freedom, and the ability to manufacture parts with customized features.

Frequently used technologies include:

- **Selective Laser Melting (SLM):** Using a laser to selectively melt the powder, ensuring near-complete density (figure 5) [17]:
 - Uses a high-power laser to selectively melt the metal powder layer by layer, ensuring near-complete density of the manufactured components.
 - Advantages: High precision, excellent mechanical properties, ability to manufacture complex geometries.
 - Applications: Aerospace industry, medical implants, injection moulds.

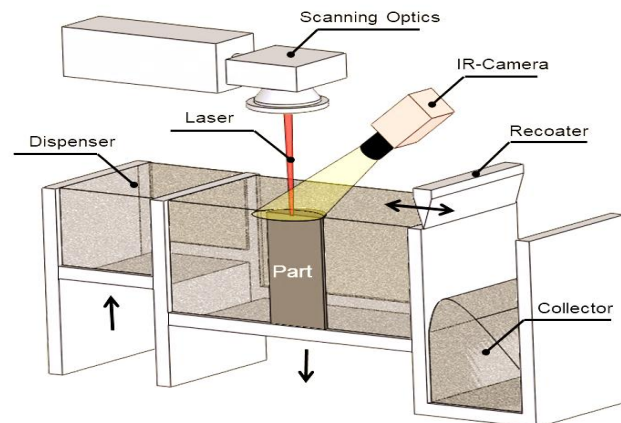


Figure 5 – SLM Technology, [17]

- **Electron Beam Melting (EBM):** The electron beam generates energy to melt metal powders (figure 6) [18]:
 - Uses an electron beam generated in a vacuum to melt layers of metal powder, providing good structural homogeneity.
 - Advantages: Higher processing speed reduced residual stresses, suitable for refractory alloys.
 - Applications: Biomedical industry (implants), industrial endurance applications.

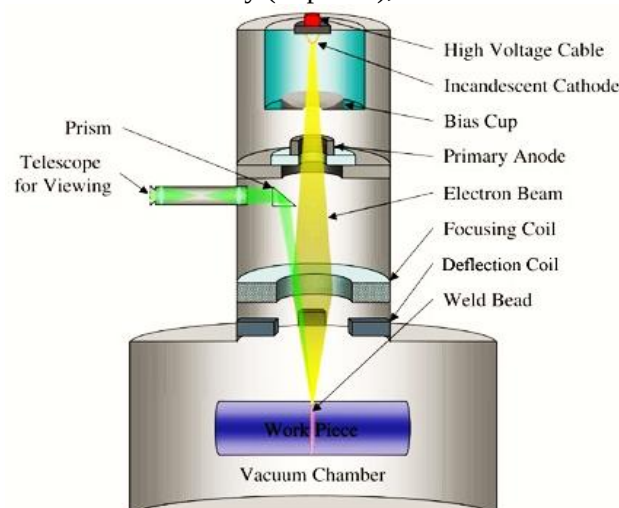


Figure 6 – EBM Technology, [18]

- Directed Energy Deposition (DED): An energy beam (laser or plasma) simultaneously melts the powder and substrate, making it ideal for repairs and large surfaces (figure 7) [19]:
 - Uses an energy beam (laser or plasma) to simultaneously melt the metal powder and substrate, making it ideal for repairs, additional layering, and manufacturing large components.
 - Advantages: High versatility, efficient material use, the ability to repair or reinforce existing structures.
 - Applications: Aerospace, energy, and automotive industries.

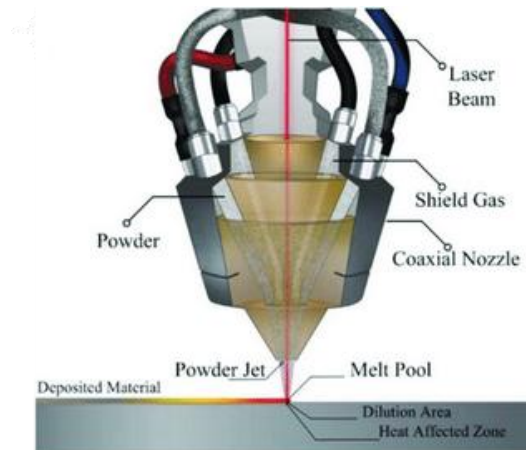


Figure 7 – DED Technology, [19]

The advantages of HEAs are:

- High geometric freedom.
- Reduction of material waste.
- Possibility of manufacturing customized parts.

The challenges of HEAs are:

- Control of porosity and defects (e.g., cracks, inhomogeneities).
- Difficulty in processing HEAs due to high melting points and compositional variations.
- The need for optimal processing parameters to avoid phase segregation.

2.3. Interaction between HEAs and 3D printing technologies

HEAs represent both a challenge and an opportunity for obtaining high-performance components [20]. The integration of these innovative alloys into 3D printing technologies enables the production of complex structures with superior mechanical, thermal, and chemical properties.

2.3.1. Influence of the printing process on the microstructure

The additive manufacturing process parameters, such as cooling rate, scanning speed, and beam power, play a crucial role in the formation of the HEA microstructure. These parameters influence grain size, phase distribution, and the homogeneity of the manufactured material [21]. For example:

- High cooling rate favours the formation of amorphous or nanostructured phases, giving the material superior mechanical properties but also a higher susceptibility to cracking [22];
- The preheating temperature of the platform helps reduce residual stresses and prevent cracking, ensuring uniform solidification and minimizing the risk of deformation.
- Scanning speed and deposition strategy influence the formation of crystal texture, impacting the mechanical anisotropy of the manufactured part [23].

2.3.2. Effects of microstructure on mechanical properties

The microstructure obtained through additive manufacturing significantly influences the mechanical properties of high-entropy alloys. The grain structure, phase distribution, and the presence of any defects contribute to the final mechanical behaviour of the manufactured components [24].

HEAs manufactured through 3D printing may exhibit the following characteristics [25]:

- High mechanical strength, due to fine grains and uniform phase distribution, contributing to increased durability.
- Low toughness, if cracks or segregation occur during the solidification process, which may reduce material performance in demanding applications.
- Controllable porosity, which can be adjusted according to the application, offering a balance between strength and reduced weight.

Optimizing process parameters is important for maximizing the mechanical and functional properties of the manufactured parts.

2.3.3. Applications and specific requirements

3D printing of HEA materials opens opportunities for advanced applications in various industries, thanks to the unique combination of properties these alloys offer. Key areas of use include [26]:

- Aerospace industry – components exposed to extreme thermal stresses, such as engine parts and support structures, due to thermal stability and high mechanical strength.
- Medical industry – manufacturing of customized implants, where HEA properties like biocompatibility and corrosion resistance are essential for the success of applications.
- Chemical industry – manufacturing of corrosion- and wear-resistant equipment for aggressive environments, where durability and structural integrity are critical.

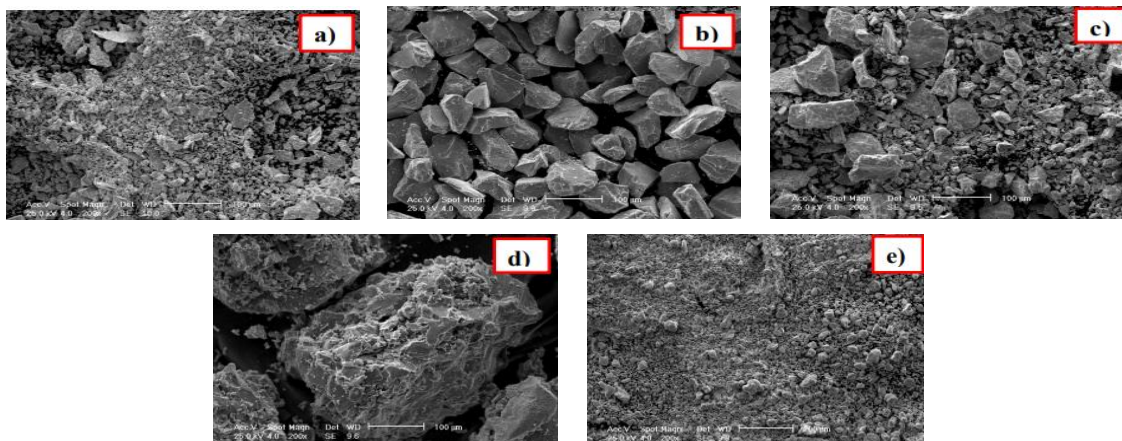
3. RESULTS AND DISCUSSIONS

3.1. Characterization of metal powders

Powder characterization is essential for understanding their behaviour during the deposition process and for optimizing manufacturing parameters.

3.1.1. Particle morphology and size

The morphological and dimensional characterization of metal powders used in additive manufacturing is crucial for determining their quality and processability [27]. The particle shape, size distribution, and surface features significantly influence the behaviour of the powder during deposition and its interaction with the energy source in the 3D printing process.



*Figure 8 – SEM and EDS analyses of elemental powders, [27]
a) Hf, b) Nb, c) Ta, d) Ti, e) Zr*

a). Experimental results

- Morphological Analysis:
 - Powders obtained through gas atomization:
 - Exhibit a uniform spherical morphology with a smooth surface and minimal presence of satellites (smaller adhering particles);
 - High sphericity is an indicator of good flowability and homogeneous distribution during deposition;
 - Show low internal porosity, improving the density of the fabricated parts.
 - Powders obtained through mechanical milling:
 - Exhibit irregular and angular shapes, with fractured particles that may cause agglomeration during the recoating process;
 - The presence of elongated particles and sharp edges may negatively affect powder flowability;
 - Irregular morphology can negatively impact melting behaviour and defect formation in printed parts.
- Particle size distribution:
 - The dimensional distribution of the powders was analysed using laser granulometry (LSA), with the results indicating the following characteristics:
 - Size range: 15-50 μm , considered optimal for Selective Laser Melting (SLM) technology.
 - Median value (d50): 30 μm , suggesting good dimensional uniformity.
 - Fine fraction (below 15 μm) is approximately 10%, which is within acceptable limits to prevent excessive particle accumulation in the air during the deposition process.
 - Coarse fraction (above 50 μm) is minimal (<5%), reducing the risk of clogging in the powder feeding mechanisms.
- Sphericity and aspect ratio:
 - The calculated sphericity values using image analysis:
 - Gas-atomized powders: 0.95, indicating an almost perfect spherical shape.
 - Mechanically milled powders: 0.70, demonstrating a more elongated and irregular shape.
 - Aspect ratio (length/width):
 - Atomized powders: 1.1 (nearly spherical).
 - Milled powders: 1.5–2.0 (irregular shape).

b). Discussions and interpretations

- Influence of particle morphology on the 3D printing process:
 - Spherical powders are preferred for good flowability and uniform packing, resulting in parts with high density and fewer defects.
 - Irregular powders can cause air pockets between particles, promoting the formation of porosity and internal defects in the fabricated parts.
 - Uniform particle shapes contribute to optimal recoating, reducing non-uniformities in each layer deposited during printing.
- Impact of particle size:
 - Smaller sizes contribute to good packing density and interconnection between particles, facilitating complete melting during the laser scanning process.
 - Larger particles can create defects, such as:
 - Lack of fusion between layers.
 - Rough surfaces requiring post-processing.
 - A bimodal distribution (presence of both small and large particles) can improve packing density but must be carefully controlled to avoid segregation during powder handling.

- Considerations for the Manufacturing Process:
 - For SLM technology, the optimal particle distribution should be between 20-45 μm to ensure uniform deposition and efficient laser energy absorption.
 - For DED technology, a higher fraction of particles ($>50 \mu\text{m}$) is preferred for more stable deposition and controlled flow.

3.1.2. Chemical composition

- Results:
 - EDS analysis confirmed the presence of main elements (e.g., Al, Co, Cr, Fe, Ni), with a composition close to the planned theoretical one.
 - Optical Emission Spectroscopy (OES) showed minimal loss of volatile elements (e.g., Al and Ti), under 2% by weight.
- Discussions:
 - The low oxygen content ($<0.05\%$) indicates good powder quality and potential enhanced oxidation resistance of the fabricated parts.
 - Minor compositional differences may affect phase stability in the final material.

3.1.3. Physical properties of powders

a) Results:

- Bulk density and tap density:
 - Measured bulk density: 55% of theoretical density.
 - Tap density (after compaction): 63%, indicating good compactability.
 - Values comply with ASTM standards for metal powders used in additive manufacturing.
- Powder flowability:
 - Hall Flow time: 12 s/50 g, indicating good flowability for SLM technology.
 - Spherical powders exhibited superior flow behaviour compared to those with sharp edges.
- Residual moisture:
 - Karl Fischer measurements indicated a moisture content of less than 0.02%, preventing agglomeration during the deposition process.

b) Discussions:

- Impact of powder density on part quality:
 - Higher apparent density improves powder layer uniformity and reduces the risk of porosity.
 - Higher tap density may facilitate better compaction during recoating.
- Importance of flowability in the manufacturing process:
 - Good flowability allows uniform deposition and prevents particle segregation during recoating.
 - Comparison with powders used in similar applications (e.g., AlSi₁₀Mg, Inconel 625).
- Improvement measures:
 - Powder treatment through plasma spheroidization to improve flowability.
 - Addition of friction-reducing agents to improve powder flow behaviour.

3.2. Behaviour in the 3D printing process

3.2.1. Density of manufactured parts

a). Experimental results

- Computed tomography (CT scan):

- Parts manufactured using optimal process parameters (200 W laser power, 800 mm/s scanning speed) exhibited a density of 99.2%, indicating excellent coalescence of metal particles.
- Using suboptimal parameters (low power of 150 W or increased scanning speed to 1000 mm/s) resulted in a density of 97.5%, with internal porosity measured up to 3%.
- Gravimetric measurements:
 - Density tests showed values of approximately 98.8% compared to the theoretical density of the HEA alloy, confirming the high quality of the parts fabricated by SLM.

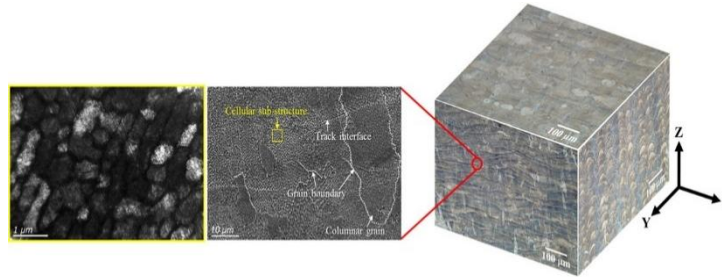


Figure 8 – Inconel 718 Parts Fabricated by SLM, [28]

b). Discussions

- Effect of process parameters:
 - Insufficient laser power can lead to incomplete particle melting, generating porosity between layers and bonding defects.
 - Excessive scanning speed reduces the interaction time with the laser, leading to lack of fusion defects.
 - Optimal laser pass overlap is essential to avoid void formation.
- Influence of density on mechanical properties:
 - High density is essential for achieving superior mechanical properties, including tensile strength and uniform hardness;
 - Porosity affects behaviour under dynamic loading, promoting crack initiation in cyclic loading applications.

3.2.2. Microstructural defects

a). Optical microscopy (OM)

- Defects of uneven porosity, with diameters up to 20 μm , were identified, more frequently occurring in areas with incomplete laser scan overlaps.

b). Scanning electron microscopy (SEM)

- Detailed surface analysis revealed microcracks at the interfaces between layers, caused by rapid cooling and the accumulation of residual stresses.
- Local segregation of elements such as Cr and Al at grain boundaries was observed, indicating uneven solidification.

3.2.3. Microstructure of manufactured parts

a). X-ray diffraction (XRD):

- Dominant FCC and BCC phases specific to the HEA composition used were identified, with a slight presence of secondary intermetallic phases.
- A slight preferential texture was observed in the direction of layer growth.

b). EDS (Energy Dispersive Spectroscopy) analysis:

- The distribution of alloying elements was homogeneous overall, with minor segregations at grain boundaries for Cr and Fe.

c). Transmission electron microscopy (TEM):

- Revealed a fine distribution of intermetallic precipitates in the FCC matrix, contributing to increased hardness.

4. CONCLUSIONS

Characterization of metal powders:

- The HEA powders obtained by gas atomization exhibited an optimal spherical morphology for the 3D printing process, with a uniform particle size distribution (15-50 μm);
- The chemical composition of the powders was in accordance with the theoretical specifications, with minimal loss of volatile elements, ensuring good phase stability.
- The powders demonstrated favourable physical properties, such as high flowability (12 s/50 g) and an apparent density of 55%, which are essential for uniform deposition in the additive manufacturing process.

3D printing process performance:

- The optimal processing parameters (laser power: 200 W, scan speed: 800 mm/s) resulted in parts with a density of 99.2%, confirming good particle coalescence.
- Microstructural analysis revealed a uniform distribution of FCC and BCC phases, demonstrating that the material structure is significantly influenced by printing parameters.
- The identified defects (cracks, porosity) were minimized through optimization of scanning strategies and post-processing heat treatments.

Properties of manufactured parts:

- The fabricated parts exhibited competitive mechanical properties, with a tensile strength of 1100 MPa, hardness of 330 HV, and ductility of 18%, comparable to or superior to conventional alloys.
- Wear tests indicated a friction coefficient of 0.35, confirming the material's resistance to friction and durability in tribological applications.
- Corrosion resistance in aggressive solutions (3.5% NaCl) was high, confirming the potential of HEA for applications in harsh environments.

5. ACKNOWLEDGEMENTS

The paper was developed within the project PN 23.37.01.03 with the title „Research on the development of 3D printed high entropy alloys for the construction of severely wear and vibration stressed components”, (financed by the Ministry of Research, Innovation and Digitization, in the frame of the Nucleu Program of ISIM Timișoara PN 23 37/2023-2026, contract 16N/2023, and of the project „Infrastructure for excellent research in welding”, Acronym INFRATECH, SMIS code 126084

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