

Parametric study on the L-DED process parameters for the production of Fe to Cu Functionally Graded Additive Materials

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The processing of Iron-based alloy (Fe) - Copper-based alloy (Cu) multi-material is a current research challenge, yet valuable for various engineering applications including tooling, aerospace, power generation, etc. Laser Direct Energy Deposition (LDED) is an innovative manufacturing technique employed in this study to create such Fe-Cu functionally graded materials (FGMs).

Fe-Cu hybrid components are gaining prominence due to their synergistic properties, combining the high thermal conductivity of copper with the corrosion resistance and mechanical strength and hardness of stainless steel. This unique combination enhances their functionality and broadens their applicability across various industrial sectors, providing improved performance and durability.

However, direct deposition of copper on stainless steel, such as 316L SS, is difficult due to considerable differences in physical, chemical, and thermo-mechanical properties. Cu and Fe have poor solubility and significant differences in thermophysical properties making the LDED of Fe-Cu challenging.

The implementation of a functionally graded material (FGM) approach is one way to deal with this problem. The current study tackles the fabrication of Fe-Cu FGM using the Cu compositional grading approach which enables to print gradient with a smooth transition between pure Fe to pure Cu with a Cu increase of 25% in each section. In addition to a trial of continuous gradient with a Cu increase of 1% in each layer.

The thesis starts with a parametric study to evaluate the process parameters (laser power, scan speed, etc.) that enable the printing of homogeneous mixtures with different compositions of Fe-Cu. The optimal process parameters were used to print gradient Fe to Cu thin walls in discrete sections of blended compositions. Ultimately, implementing the optimal process parameters allowed the extension to 3D components, successfully demonstrating the technology's capability to build a 3D demonstrator component and tensile test coupons.

Keywords : Additive Manufacturing, Directed Energy Deposition, Functionally Graded Material, Build-plate Preheating.

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Acronyms

DED	Directed Energy Deposition
AM	Additive Manufacturing
LDED	Laser Directed Energy Deposition
FGM	Functionally Graded Material
MMC	Multi-material component
Cu_xSS_{100-x}	Cu-SS blended compositions with x varying from 25% to 75%
GDxCu	x % graded Fe-Cu FGM
Fe	Iron-based alloy, 316L SS
Cu	Copper-based alloy, CuCr1Zr
EDS	Energy-dispersive X-ray spectroscopy
WAAM	Wire and Arc Additive Manufacturing
VUB	Vrije Universiteit Brussel
SLM	Selective Laser Melting
LENS	Laser Engineered Net Shaping
SEM	Scanning Electron Microscope
T-WAAM	Twin Wire and Arc Additive Manufacturing
SLD	Selective Laser Deposition
L-PBF	Laser Powder Bed Fusion
HSLA-steel	High-Strength Low-Alloy steel
PFR	Powder Feed Rate
CuAl	Copper Aluminum Alloy
HIP	Hot Isostatic Pressing

Greek symbols

v_{scan}	scan speed	mm/min
t_l	layer thickness	mm
LP	laser power	W
t_{wait}	interlayer wait time	s
α	thermal diffusivity	m ² /s
β	mass transfer coefficient	m/s
R_c	Cooling rate	K/s
ϵ	emissivity	—
UTS	Ultimate Tensile Strength	MPa
η	efficiency	%
γ	heat capacity ratio	—
λ	thermal conduction coefficient	W/mK
π	pressure ratio	—
ρ	mass density	kg/m ³
V	volume	m ³
E	volumetric laser energy density	J/mm ³
d_{hatch}	hatch spacing	mm

There has been an increase in interest in the design and production of multi-material components (MMCs) in recent years because of the benefits they offer, including various material properties (mechanical, thermal, and electrical), weight optimization, and cost reduction.

MMCs can change the conventional manufacturing paradigm by fully realizing the unique strengths of each material. Traditionally, a part is usually manufactured of a single material, necessitating the compromise of certain qualities.

The hybrid structure of Copper-based alloy (Cu) and Iron-based alloy (Fe) is highly appealing because the final product can benefit from both the superior mechanical strength, resistance to corrosion of Fe, and the excellent thermal and electrical conductivity of Cu. Because of this, the Fe-Cu parts are widely desired in industries like nuclear, power generation, automotive, electrical, and electronic, etc.

However, due to the substantial differences in physical, chemical, and thermomechanical characteristics of Fe and Cu, direct joining of these dissimilar materials is challenging.

1.1 CONTEXT

Processing Functionally Graded Additive Materials consisting of a Fe and Cu-based alloy is the focus of the current thesis. In addition to destructive testing and metallurgical evaluation, these Fe-Cu-based mixtures' processability will be assessed under various processing scenarios.

Starting with a review of the literature, the thesis work will focus on the processing of Fe-Cu-based multimaterials and the L-DED manufacturing process (as well as its parameters).

This thesis starts by investigating the optimal L-DED process parameters for producing homogenous thin walls from various Fe-Cu pre-blended compositions. Furthermore, the study applies these adjusted parameters to the production of 3D components, demonstrating the technology's ability to create 3D structures and tensile test coupons. The findings highlight the specific process needs for different compositions, as well as the relevance of pre-heating and microstructural characterization in producing high-quality additive manufacturing built components.

1.2 OBJECTIVES

The thesis seeks to optimize process parameters to successfully manufacture mechanical test coupons such as dog bones with 99 % density and no cracks. This optimization entails determining the best combination of laser power, scan speed, powder flow rate, and other relevant factors to ensure high-quality component production. To achieve this objective the following sub-objectives should be fulfilled:

1. **Processability Investigation:** to provide information on the processability of Fe-Cu-based mixtures of varying compositions. This entails examining how different Fe and

Cu composition ratios affect the L-DED process, with a focus on material compatibility, deposition quality, and any potential challenges.

2. **Material Property Analysis:** to retrieve material properties for this novel material created by the L-DED process. Characterizing mechanical properties such as hardness.
3. Implement the necessary modifications to the L-DED processing research platform to enable the successful processing of 2D and 3D Fe-Cu FGM components.
4. The ultimate objective is to demonstrate the technology by a demonstrator component.

1.3 ADDITIVE MANUFACTURING (AM)

"Additive manufacturing" refers to technologies that create three-dimensional objects in a very thin layer (approximately $300\mu\text{m}$) at a time. Each successive layer of melted or partially melted material bonds to the previous layer. Objects are digitally defined by Computer-Aided Design (CAD) software, which generates ".Stl" files that "slice" the object into ultra-thin layers. This data directs the path of a nozzle or print head as it deposits material precisely on the previous layer. Alternatively, a laser or electron beam selectively melts or partially melts a powdered material bed. Materials fuse to form a three-dimensional object as they cool or cure.

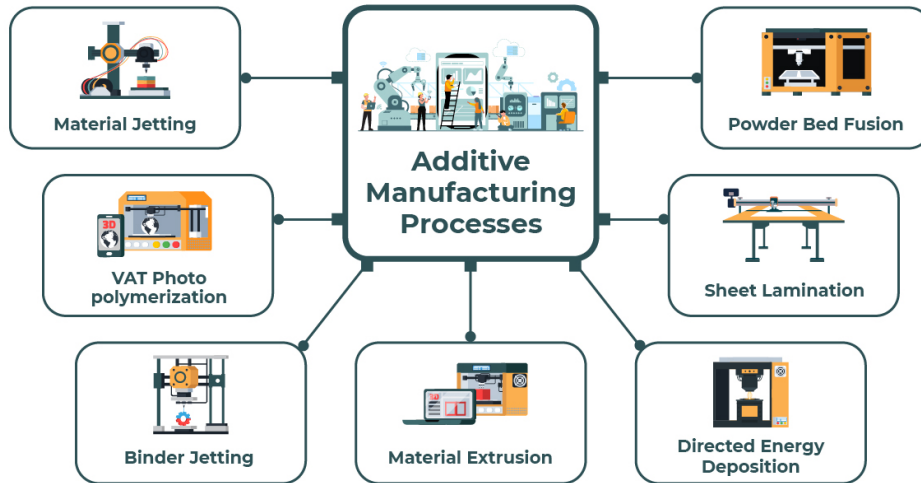


Figure 1.1: Additive Manufacturing Processes. [Kambale, 2021]

In contrast to traditional manufacturing, AM constructs objects layer by layer rather than removing excess from bulk objects through milling or other processes. Although there are numerous AM processes, many manufacturers concentrate on the seven most accessible types of AM as illustrated in Figure 1.1. The following are the primary types of additive manufacturing for metals:

- **Binder Jetting** is a technique that combines liquid binding agents with powdered material to deposit alternating layers of construction materials, bonding agent, and powder spreader to create a 3D object.
- **Powder Bed Fusion** uses a laser or an electron beam to melt powdered material and fuse it into a 3D object.

- **Sheet Lamination** is the process of stacking and laminating sheets of material to create tangible items. Sheet lamination is a broad term that encompasses processes such as ultra-sonic additive manufacturing, laminated object manufacturing, and selective deposition lamination.
- **Directed Energy Deposition** uses thermal energy to melt and fuse material layers to form a three-dimensional object. There exist two types of feedstocks, powder and wire (WAAM). LDED is the most suggested AM technique for producing metallic FGM components at a higher build rate [Paul CP, 2018]. L-DED can be used in creating FGM by either introducing individual streams of various powder or wire at the appropriate rate into the melt pool or by introducing streams of pre-blended powder or wire of various compositions into the melt pool. Several studies have been conducted to show that LDED is a feasible method for creating FGM through the combination of different materials [Yadav et al., 2023, Zhou et al., 2021, X. Zhang et al., 2021]. L-DED is preferable due to its wide range of materials, a great degree of design freedom, and ability to do in-situ repairs, it has become a highly effective method for creating complex and large-scale components.

1.3.1 DIRECTED ENERGY DEPOSITION

Directed Energy Deposition (DED) is a viable metal additive manufacturing process with applications in various high-value industries, including aerospace and automotive. A high-power laser beam is used in this process to melt a metal workpiece locally as illustrated in Figure 1.2. A nozzle blows fine metallic powder into the melt pool thanks to an inert carrier gas. The powder interacts and mixes with the molten substrate, and the resulting material addition solidifies as the nozzle moves to form a track [Snijers et al., 2021].

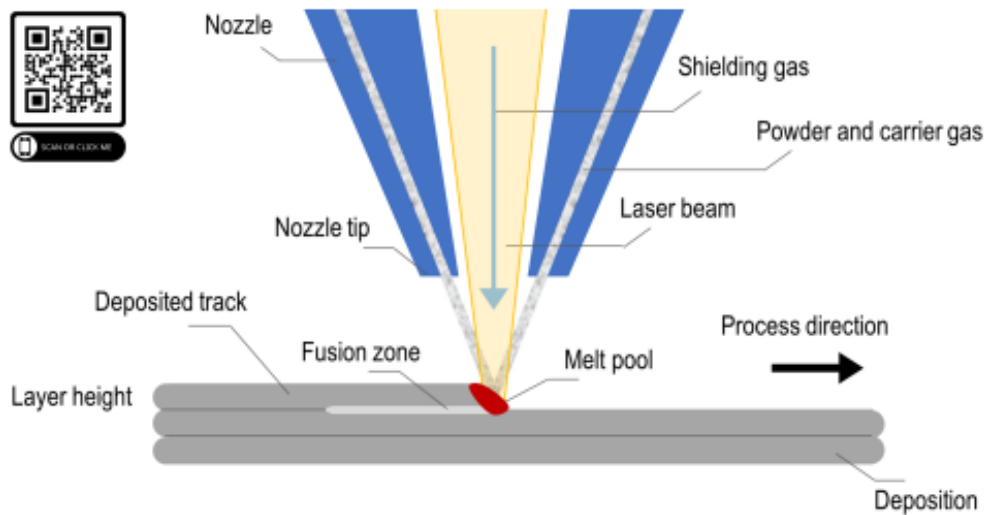


Figure 1.2: Schematic diagram of Laser Directed Energy Deposition process.[Jardon, 2022]

A shielding gas is further blown surrounding the melt pool to prevent it from oxidizing. The printing head moves in three directions, stacking tracks and eventually creating a part layer by layer.

A critical process parameter in AM is the volumetric laser energy density (E) which relies on

most of the process parameters in the following Equation 1.1 :

$$E = \frac{LP}{v_{scan} \times d_{hatch} \times t_l} \quad (1.1)$$

Where,

E : Volumetric laser energy density (J/mm³)

LP : Laser power (W)

v_{scan} : Laser scan speed (mm/s)

d_{hatch} : Hatch spacing (mm)

t_l : Layer thickness (mm)

The volumetric laser energy density is critical since it determines the quality and properties of the finished product. Proper energy density ensures that material layers fuse optimally, resulting in strong interlayer bonds and fewer flaws. It has a direct impact on the mechanical characteristics, microstructure, and overall integrity of the fabricated product. Manufacturers may optimize energy density by modifying factors such as laser power, scan speed, hatch spacing, and layer thickness, resulting in high-quality, reliable, and precise components for a wide range of applications.

A laser power scheme is a systematic approach to regulate and control the laser's power output to optimize the deposition process. To ensure that there is enough energy to melt the feedstock material without producing problems from excessive heat, this entails regulating the laser power levels across multiple layers. The laser power scheme depends on the alloy characteristics, 316L SS needs a decreasing LP scheme, starting with a higher LP and then gradually decreasing the LP until the steady LP to avoid heat accumulation during the process. Copper has another behavior and researchers such as [Yadav, 2020], found that the optimal scheme for processing Cu is the increasing LP.

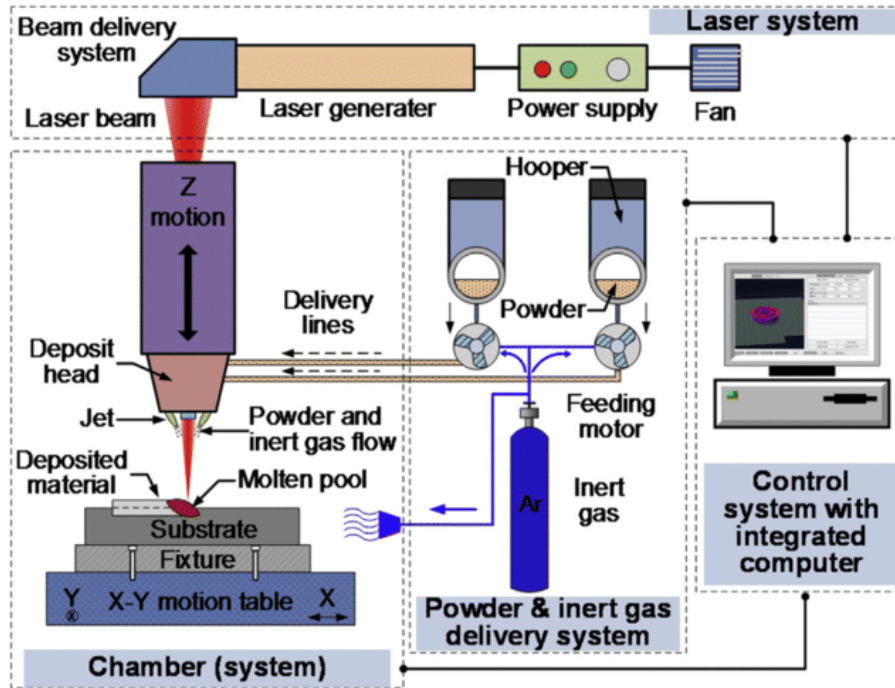


Figure 1.3: Set-up description for L-DED process [Morsi, 2019].

1.4 FUNCTIONALLY GRADED MATERIALS (FGM)

Functionally Graded Materials (FGMs) are a sophisticated class of materials having spatially changing characteristics tailored to specific areas throughout a part. These materials have distinct properties that set them apart from their constituents as represented in Figure 1.4. In the tool and die industry, FGMs are used to improve productivity and heat control. This is accomplished by depositing materials with excellent thermal conductivity onto steel substrates, demonstrating one of the many applications for FGMs.

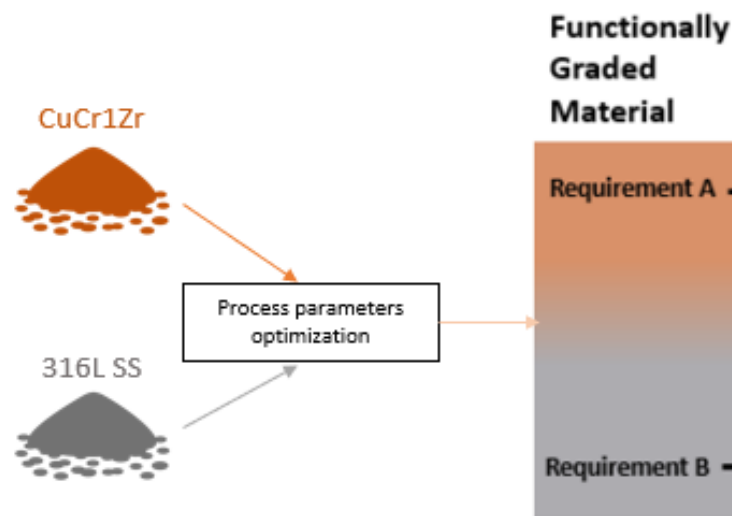


Figure 1.4: Representation of Fe-Cu based alloys FGM



Figure 1.5: Fe-Cu FGM nozzle using Selective Laser Deposition (SLD) [Sher, 2023]

FGMs are intended to conform to functionality in extreme environmental conditions by utilizing multi-material properties in a single component. By providing a diffused interface, and mitigating the issue of premature failure caused by a sharp interface caused by a direct joint of dissimilar material [Yadav et al., 2023].

Figure 1.5 represents an FGM nozzle produced using SLD which is a technology found by Aerosint. This technology enables the selective deposition of multiple powders to create a single layer with at least two materials, serving as an alternative to traditional single-material roller or blade re-coaters in powder bed processes. It applies to various additive manufacturing techniques, including Laser Powder Bed Fusion (L-PBF), Binder Jetting, and Pressure Assisted Sintering.

Powder Metallurgy, Vapor deposition, Centrifugal Casting, and other conventional FGM fabrication techniques are used but have shape design limitations. LDED is the preferred technology for FGM fabrication as it can be used to construct FGM by feeding streams of pre-blended powder of different compositions into the melt pool or by feeding individual streams of different powder into the melt pool at the desired rate.

Since FGM fabrication by transitioning from one material to another necessitates gradual modification of the powder chemical composition, a part can be divided into sections, and each section can be processed with different chemical composition and processing parameters, starting from one material to another one by a step grading percentage.

Graded-x-Copper is denoted by the acronym GDxCu, where "GD" stands for graded, "x" for grading percentage, and "Cu" for copper. The term refers to a composite material whose composition is gradually changed by incorporating copper in different proportions with other materials. The material's qualities are improved by the grading, enabling customized mechanical and thermal properties that are advantageous in applications needing both high thermal conductivity and structural stability. An example of FGM is illustrated in Figure 1.6 which represents the GD25Cu. A joint melt pool forms, with both materials and mixtures distributed via two main mechanisms: capillary convection (Marangoni effect) and diffusion through the moving phase interface [Makarenko et al., 2022]

FGM defects may arise from various causes during the additive manufacturing process. Common defects include porosity, microcracks, and delamination, all of which can substantially impact the material's mechanical qualities and performance. Inadequate laser power or scan speed frequently causes porosity, partially melting the powder. Thermal stress from quick cooling and heating cycles can cause microcracks to occur. Delamination, or layer separation,

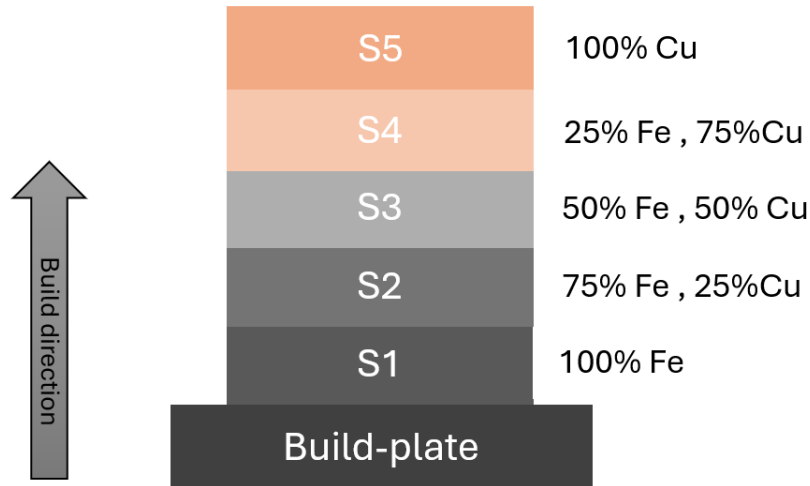


Figure 1.6: Representation of a Fe-Cu gradient of 25% copper increase (GD25Cu)

can occur when there is insufficient bonding between layers, which is commonly caused by improper hatch spacing or layer thickness [El-Galy et al., 2019]. Furthermore, FGMs have intrinsic material incompatibilities, such as discrepancies in thermal expansion coefficients between dissimilar materials, which can exacerbate these problems. Understanding and regulating these parameters is critical to reducing faults and improving the quality of FGM products.

Stress can be generated by the direct contact of two different materials. The FGM theory suggests that when there are gradual transitions between these two materials, the stress can be reduced [Snyers et al., 2021]. A characteristic of FGM is a gradual change in composition and structure with volume, leading to matching modifications in the material's characteristics.

Researchers investigated the additive production of copper and Fe-Cu functionally graded materials (FGMs) with techniques such as laser direct energy deposition (LDED), Selective Laser (SLM), and twin-wire arc additive manufacturing (T-WAAM). They identified the optimal process parameters for Cu deposition and addressed bonding issues in Fe-Cu FGMs. To minimize microstructural porosity in additive manufacturing, many solutions were proposed by researchers to improve deposition quality and mechanical qualities.

2.1 PROCESSIBILITY OF COPPER

2.1.1 CHALLENGES OF COPPER PROCESSING USING FIBER LASER

Copper absorbs part of the laser light that strikes its surface and reflects some of it. To facilitate welding, the absorbed part is transformed into heat. Light's wavelength determines the percentage of light that is absorbed. The infrared spectrum is where copper shines brightest, whereas the blue and green wavelengths are where copper is least reflective. Numerous researches are being done on green and blue lasers to create copper laser welding solutions. [Veilleux, 2024]

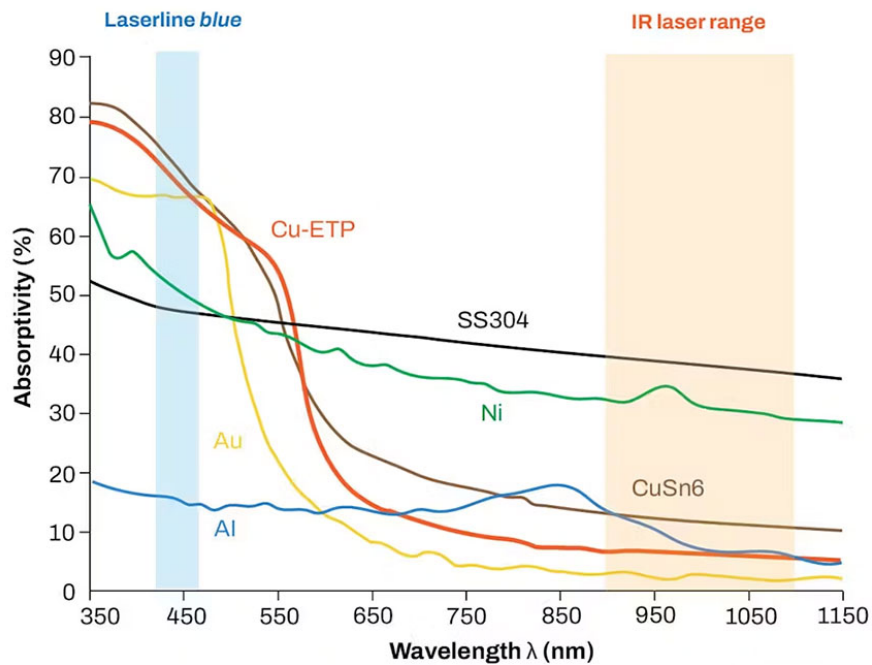


Figure 2.1: The absorption spectrum of pure copper (Cu-ETP), bronze (CuSn6, a strengthened copper alloy), and other metals [Veilleux, 2024]

Figure 2.1 illustrates that each laser type has typical absorbtivity for different materials, as only around 5% of copper can be absorbed by fiber lasers' near-infrared light, which is emitted at a wavelength of roughly 1,070 nm. Alternatively, green lasers have a much higher absorptivity of

35–40%; these lasers run at about 532 nm. Copper has the highest absorptivity (around 60%) in blue lasers, which have a wavelength of about 450 nm. It is evident from this variation in absorptivity across different laser types how difficult and to process Copper using fiber lasers.

Surface preparation and pre-heating are two pre- and post-processing methods that can help control copper's high reflectivity and thermal conductivity. Uniform deposition is ensured by optimizing the quality of the powder and the feed rate, and oxidation is prevented by utilizing a shielding gas or inert atmosphere. Systems for closed-loop control and real-time monitoring allow for dynamic modifications to preserve process stability.

2.1.2 PROCESS WINDOW OF COPPER

[Yadav, 2020] conducted a thorough analysis of the L-DED processing of bulk Cu structures. Using a 2 kW fiber laser-based Directed Energy Deposition (DED) system, featuring a 5-axis manipulator within a glovebox, twin feeder channels, a coaxial nozzle, and computer numerical control. Argon gas is utilized as both a carrier and shielding gas. The system maintains a constant argon flow rate of 5 liters per minute and a beam spot diameter of 3 mm on the substrate surface for the deposition of pure copper. Through single-track experiments, the lowest and maximum Laser Powers (LP) that can be employed for Cu deposition was discovered after a systematic investigation of the L-DED of bulk Cu structures. A minimum LP of 600 W is necessary for melting Cu. A powder feed rate of 4 g/min and an LP of 1800 W was found to be maximum as porosities started to form and cracks appeared. Process parameters are therefore chosen for single-track analysis within the LP range mentioned above.

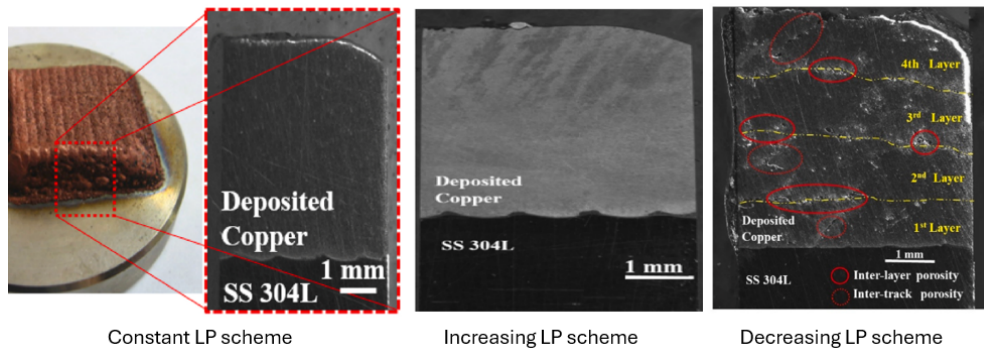


Figure 2.2: Effect of Laser Power (LP) schemes on porosity and crack density: Constant LP, increasing LP and decreasing LP [Yadav, 2020]

Three distinct schemes—the Constant LP scheme (1500W), the Decreasing LP scheme (1500W to 600W), and the increasing LP scheme (600W to 1500W)—were examined in the same research as illustrated in Figure 2.2. It was found that the increasing LP, strategy between two subsequent layers results in bulk deposits with no interlayer porosity and a relative density of almost 99.9%. This scheme would be beneficial to reduce interlayer porosities while L-DED of graded thin walls 4.2.1.

The study of [Zardoshtian et al., 2023] investigated the multiple-factor relationship between sample properties and process factors using the Laser Directed Energy Deposition with Powder Feed (LDED-PF) method for Cu processing. To create near-net-shape multilayer cuboid geometries that are free of defects, the goal was to optimize critical process parameters and create the optimal processing window. A processability map was produced which is shown in Figure 2.3, by analyzing the effects of different parameter combinations, namely power LP and feed/velocity (F/V), on defects such as balling, evaporation, and lack of fusion (LOF) porosity.

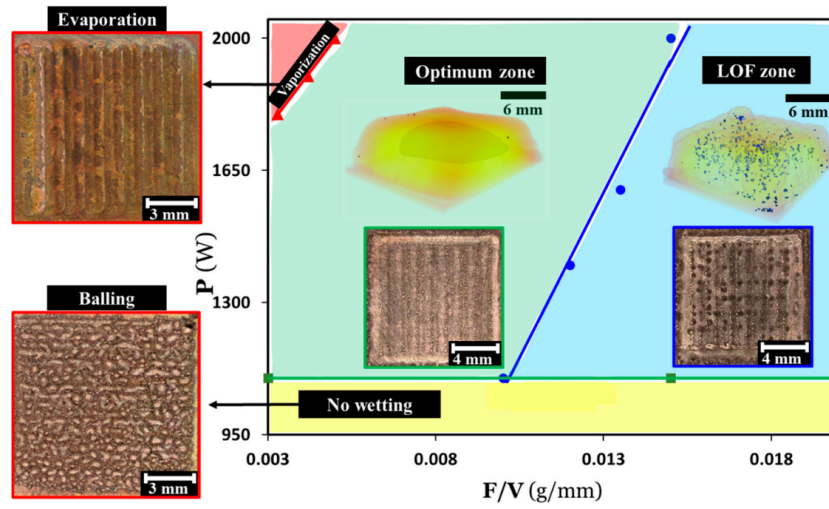


Figure 2.3: L-DED processability map of CuCrZr alloy [Zardoshtian et al., 2023]

The green color designates the optimal processing window, in which only a small amount of gas porosity defects can exist.

A minimum of 1100 W of laser power is needed to properly wet the substrate and create a stable melt pool; otherwise, the balling problem appears. A laser power of 1500 W, a powder feed rate of 4 g/min, a scanning speed of 4.5 mm/s, and an overlap ratio of 0.4 are the optimal process parameters for single-layer multitrack deposition. The model's predictions and the observed results agree quite well, with 95% confidence intervals around the bottom and upper bounds.

2.2 PROCESSIBILITY OF FE-CU FGMS IN LITERATURE

The literature regarding Fe-Cu FGMS is limited due to the challenging bonding between these dissimilar materials. [Yadav et al., 2023] used the increasing LP Scheme (III) for LDED of GD20Cu, and it was very beneficial in decreasing the interlayer porosities and microcracks as shown in Figure 2.4. Various Fe-Cu gradients were fabricated (GD100Cu, GD50Cu, and GD20Cu), and LDED of GD100Cu and GD50Cu produced defect-free depositions. Meanwhile, Cracking predominates in the solidification of the GD20Cu structure at higher laser powers, whereas inter-layer porosity development predominates in the Cu-rich areas at lower laser powers. However, a greater degree of crack density and porosity is produced when laser power is increased in conjunction with interlayer delay, the specified compositional dependant parameters for GD20Cu are summarised in Table 2.1.

S. no	Compositions	Observations
1	SS 304L	Even deposit and defect-free deposit
2	Cu20SS80	The irregular and burned texture with crack is observed above 1200 W, whereas 1000 W yields non-uniformity in the built track. Thus, laser power 800 W is selected for bulk depositions. Tracks deposited at 800 W yield higher surface irregularity. Surface uniformity and regularity are satisfactory for a track built at 1000 W at 0.5 m/min.

3	Cu40SS60	Tracks deposited at 800 W yield irregular tracks. 1200 W yields regular deposit however it leads to an uneven distribution of Cu and cracks in the deposited track. 1000 W at 0.3 m/min yields satisfactory track depositions.
4	Cu50SS50	Tracks deposited at 800 W yield irregular tracks. 1200 W yields regular deposit however it leads to an uneven distribution of Cu and cracks in the deposited track. 1000 W at 0.3 m/min yields satisfactory track depositions.
5	Cu60SS40	Irregular tracks are observed at 800 W. Higher laser power i.e. 1200 W yields an uneven distribution of Cu and cracks at the surface of LDED built track. 1000 W at 0.3 m/min yields satisfactory track depositions.
6	Cu80SS20	Irregular tracks are observed at 800 W and 1000 W. Track deposited at 1200 W at 0.5 m/min yields satisfactory deposition.
7	Cu	Even deposit and defect-free deposit with increasing laser power strategy.

Table 2.1: L-DED built track of GD20Cu blended compositions optimal process parameters [Yadav et al., 2023]

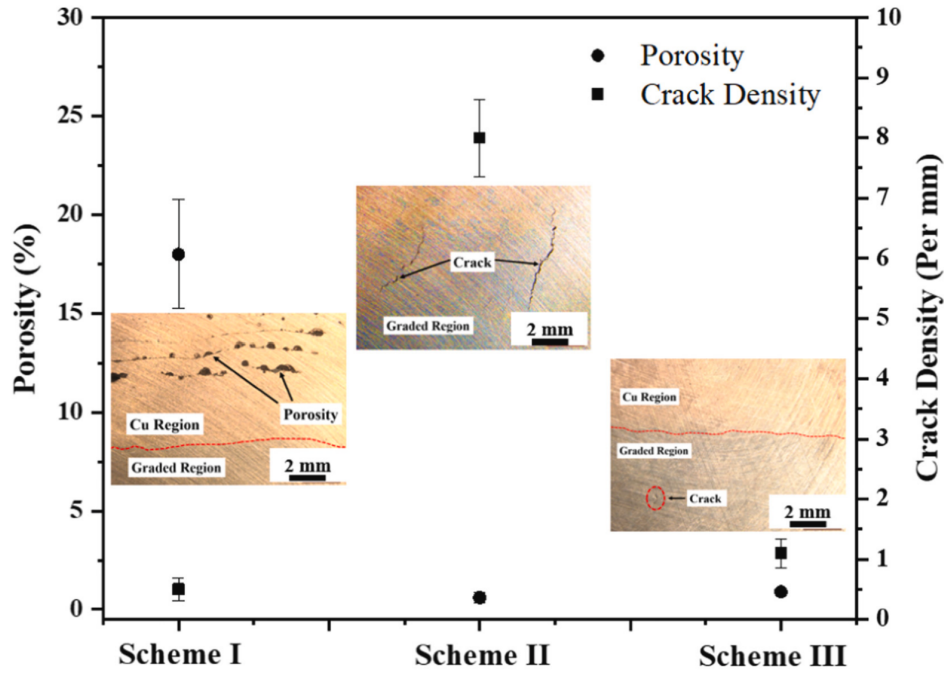


Figure 2.4: Effect of LP schemes on porosity and crack density: Scheme I (Constant LP), Scheme II (Decreasing LP) and Scheme III (Increasing LP) [Yadav et al., 2023]

Even though interlayer porosity was solved by increasing LP and interlayer delay, still one can notice that the microcracks are still problematic for high laser power.

Tensile tests presented in Figure 2.5 reveal that GD100Cu and GD50Cu evaluated UTS values are greater than the GD20Cu. The Cu region experiences failure in the tensile specimen of the LDED-built GD100Cu and GD50Cu, suggesting defect-free deposition at the interface and graded region. On the other hand, the lower Cu portion of the GD20Cu tensile specimen fails, suggesting the presence of a microcrack in this area.

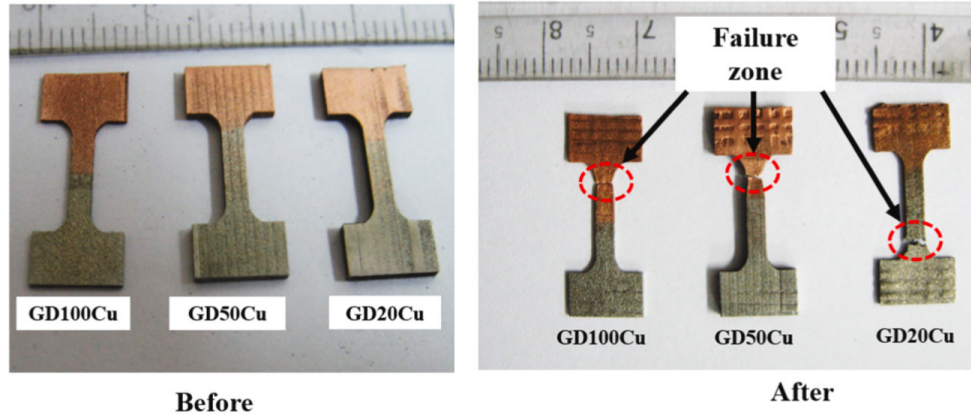


Figure 2.5: representation of Test coupons (GD100Cu , GD50Cu and GD20Cu) before and after Tensile test [Yadav et al., 2023]

[X. Zhang et al., 2021] attempted to use LDED technology to create well-bonded Fe-Cu hybrid components. To solve the problems in Fe-Cu direct junctions, the nickel-based alloy Deloro 22 (D22) is inserted between Cu and Fe. Defect-free interfaces are obtained at the Cu-D22 and D22-Fe transition zones by using this method. Figure 2.6 represents DED-fabricated Fe-D22-Cu FGM using this method.

By performing tensile tests, the CuD22SS samples cracked at the Cu zone, while the interface remained crack-free, suggesting that the bonding strength exceeded the Cu's UTS.

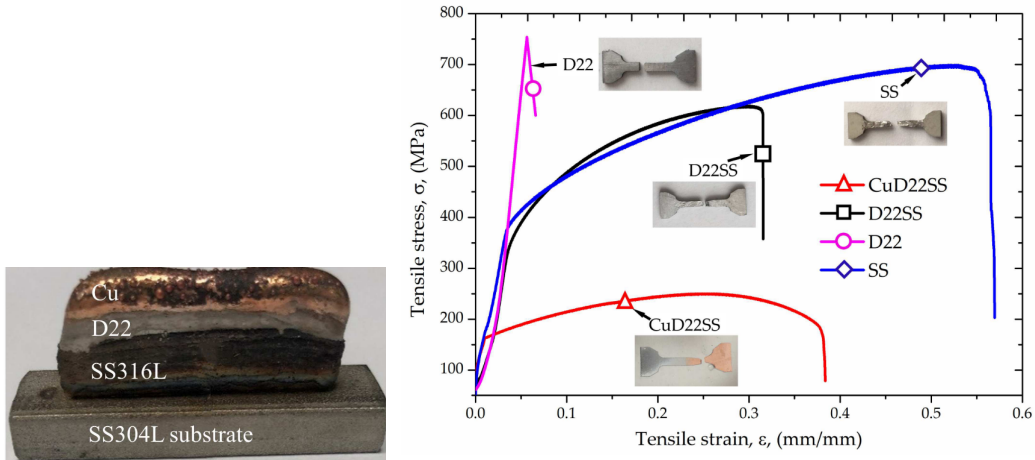


Figure 2.6: DED-fabricated Fe-D22-Cu FGM and representation of tensile tests results [X. Zhang et al., 2021]

2.3 FE-Cu FGM PRODUCED BY TWIN-WIRE AND ARC ADDITIVE MANUFACTURING (T-WAAM)

Tiago A. [Tiago, 2022] has examined the use of twin-wire and arc additive manufacturing (T-WAAM) to fabricate a functionally graded material "High-Strength Low-Alloy" steel (HSLA-steel) to copper-aluminum alloy. A gradient of electrical conductivity, composition, hardness, and electrical impedance was found in the interface region. Despite the sudden transition between these two materials, a sizable interface region (12 mm) was produced by the subsequent remelting and heating of previously deposited layers when a new one was added. This fact illustrates the possibility of using WAAM to fabricate FGM.

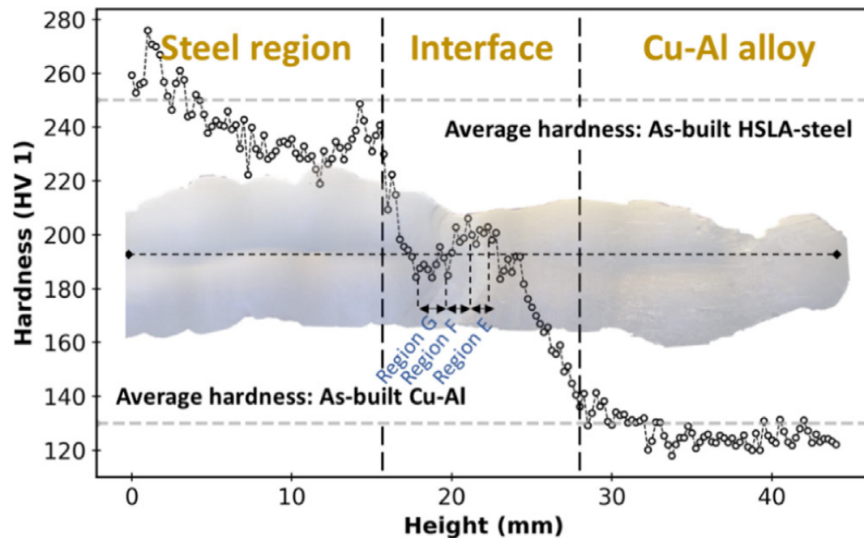


Figure 2.7: The microhardness plot as the function of distance on the sample cross-section of Fe-Cu FGM using T-WAAM.[Tiago, 2022]

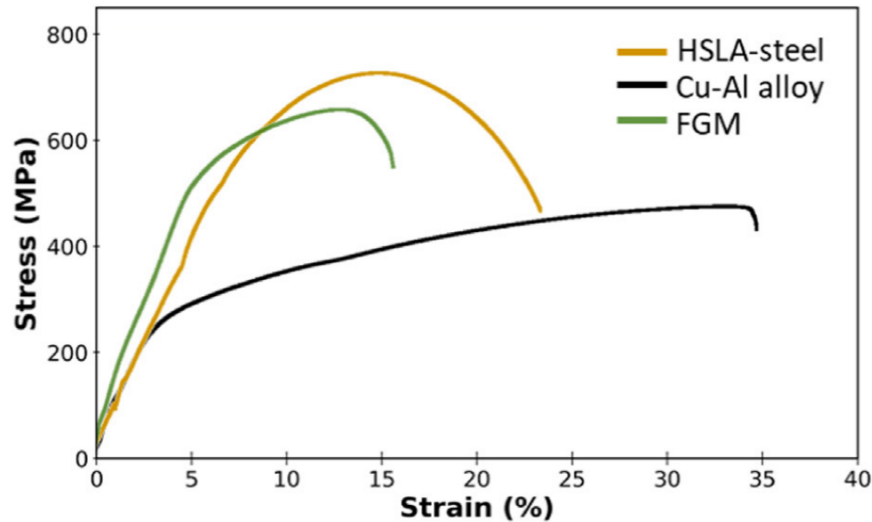


Figure 2.8: Representative stress-strain curves of the HSLA-steel and Cu-Al alloy parts and their dissimilar multi-metallic part. [Tiago, 2022]

The hardness measurements along the height of the CuAl/HSLA-steel section are shown in Figure 2.7. A linear decrease of hardness by increasing the percentage of Cu-based alloy was observed as the hardness decreased from 250 HV (100% Fe) to 120 HV (100% Cu) and 180 HV in the interface.

The FGM part was able to attain a tensile strength of 690 MPa and an elongation at fracture of 16.6% as shown in Figure 2.8. This indicates that the interface strength is higher than the copper-only zone. The fracture happened in the Cu region close to the interface.

2.4 COMPARISON BETWEEN L-DED AND SELECTIVE LASER MELTING (SLM)

A wide range of metal additive manufacturing techniques, such as powder-bed fusion (e.g., selective laser melting; SLM); electron beam melting (EBM); directed energy deposition (e.g., Laser Engineered Net Shaping; LENS; Laser Metal Deposition; LMD)) have been used to fabricate FGM structures.

According to a literature study based on the publications [Wei et al., 2019, Yadav et al., 2023], every FGM part created using the multiple material LENS technique is a vertical composition gradient FGM, meaning it is created between two deposition layers rather than inside one, making it not fully three-dimensional.

In theory, any LENS system with a multiple powder mixing capability should be able to print horizontal FGM structures. Even though SLM has higher print resolution compared to L-DED, printing small horizontal FGM components proved to be challenging in practice, because it would need an unacceptable amount of time and material waste to change the powder mixing composition proportion regularly, which would also require cleaning the powder-supplying system of all leftover mixed powder.

In this paper [Wei et al., 2019], a selective laser melting system is coupled with a 6-channel ultrasonic selective powder delivery system. Using this type of exclusive AM technology, 316L/Cu10Sn samples for horizontal FGM and 3D FGM were successfully produced.

As illustrated in Figure 2.9, the interesting phenomenon is that the hardness plots at the gradient zone did not smoothly transit from 316L to Cu10Sn, following the decreasing trend of composition changing. Rather, when the low hardness Cu10Sn was added to 25% of the combination, the hardness decreased from 202.5 HV to 174.11 HV. After that, the hardness began to rise and reached its highest value in the region containing 50% 316L and 50% Cu10Sn, 213.24 HV. After that, the hardness dropped and in the pure Cu10Sn region, it achieved a minimum value of 148.11 HV. Compared to Figure 2.10 which illustrates the Micro-hardness variation across L-DED built Fe-Cu FGM. A linear decrease of Hardness by increasing the percentage of Cu-based alloy was observed as the hardness decreased from 225 HV (100% Fe) to 90 HV (100% Cu).

SLM provides high-precision 3D prints but it's limited when it comes to large dimension parts. In addition to the challenge of dealing with multi-materials.

2.5 DEFECTS IN LDED PRODUCTS

2.5.1 MICROSTRUCTURAL POROSITY

The presence of random void flaws significantly degrades the mechanical and physical properties of AM materials. Three crucial areas of the structure of additively manufactured parts contain the pores that resulted from AM processing: the bulk material, the spaces between deposited layers, and the interface region in the case of multi-materials. The interlayer pores significantly impact the bond strength. When the load is applied in the transverse direction normal to the printed layers, they are crucial to the mechanical performance of printed structures.

Figure 2.11 represents the different types of microstructural pores in additive manufacturing parts. Entrapped gas pores are spherical pores that are caused by the presence of oxygen in the printing chamber. The primary cause of incomplete melting-induced porosity is inadequate

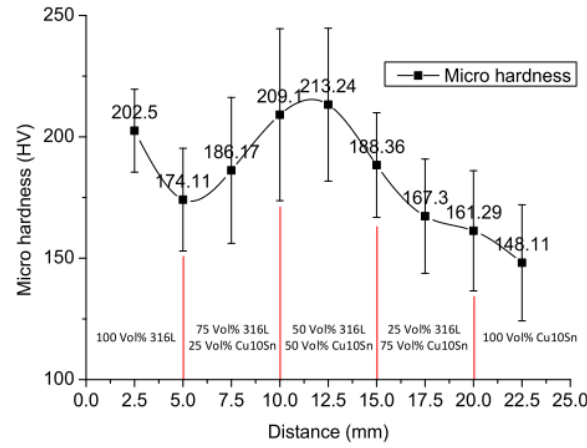


Figure 2.9: The microhardness plot as the function of distance on the sample cross-section of 316L SS - Cu10Sn FGM using SLM.[Wei et al., 2019]

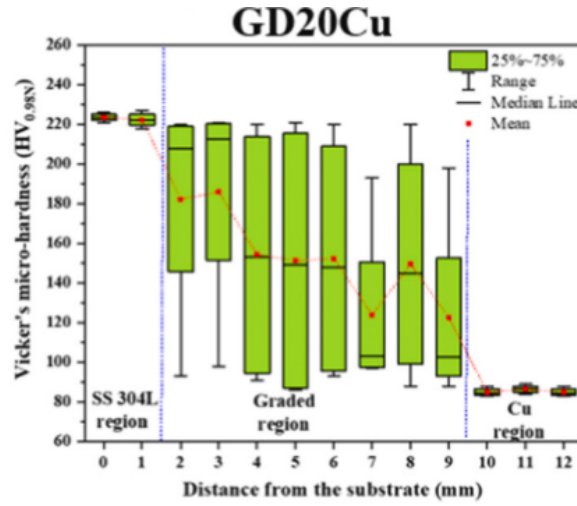


Figure 2.10: Micro-hardness variation across LDED built Fe-Cu [Yadav et al., 2023]

energy input during the fabrication process. Due to the lack of energy, the powder bed partially melts, causing holes and pores in the hardened substance. Inadequate configuration of process parameters, such as laser power, scan speed, layer thickness, and hatch spacing, is one of the factors causing this problem. To minimize flaws like porosity, these parameters must be tuned to enable proper melting and bonding of the material [Dixit and S. Liu, 2022]. Lack of fusion with unmelted particles inside large irregular pores is mainly caused by low energy density. Finally, cracks are a result of rapid melting and solidification.[Sola and Nouri, 2019]

2.5.2 CRACKS

In LDED, metallic materials may melt and solidify quickly due to high local energy input. The cooling rate during the operation can reach $10^7 - 10^8$ K/s, resulting in significant residual thermal stress in the direct energy deposited product. Cracks occur and propagate because of high temperatures and residual stress. Stainless steel-based alloys can crack during LDED processing because of the low heat conductivity and high thermal expansion coefficient. The susceptibility of fracture formation can be minimized by preheating the substrate or performing post-processing stress reduction procedures.[Majumdar et al., 2021]

In the scope of this thesis is intended to solve this problem using build plate preheating as

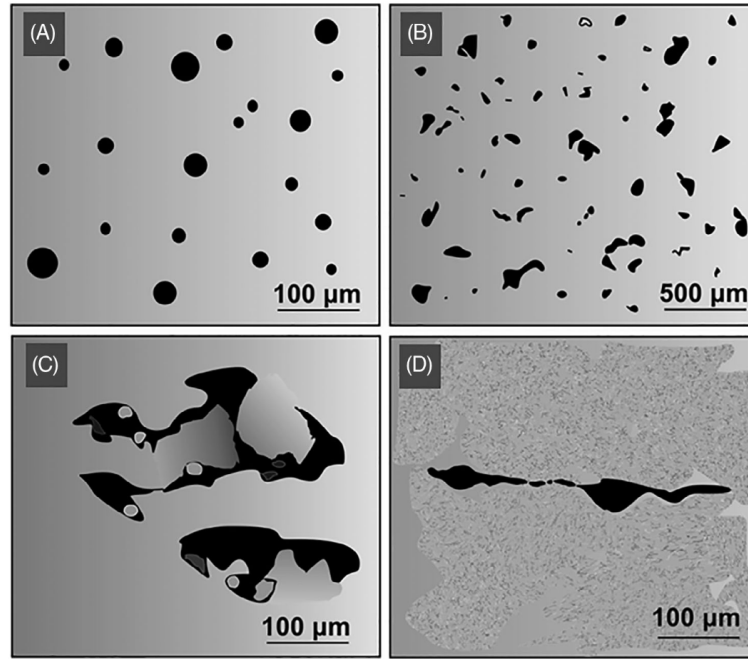


Figure 2.11: Microstructural porosity types in AM : A (entrapped gas pores); B (incomplete melting-induced porosity); C (lack of fusion with unmelted particles inside large irregular pores); D (cracks) [Sola and Nouri, 2019]

it reduces the temperature difference between the substrate and the material, reduces stresses and strains in the finished part, and prevents warping and separation from the substrate [M. Liu et al., 2021] thus preventing the cracks due to residual stresses. The deposition process of metallic powder is especially highly susceptible to cracking due to the rapid heating and cooling rates caused by a high-energy laser. It was demonstrated through experimental research [Shim et al., 2017] that substrate preheating using induction heating can result in a slower cooling rate.

2.5.3 OXIDATION

In the context of LDED processes, oxidation is defined as the production of oxide layers on the surface of the deposited material as a result of oxygen exposure during the deposition process. This oxidation can occur when the laser heats the material and it comes into contact with oxygen in the surrounding atmosphere.

Oxidation can have a variety of effects on the quality and attributes of deposited materials. It may cause changes in material qualities, such as decreased strength or increased brittleness, as well as a reduction in the overall integrity of the deposited portion. Furthermore, oxide coatings may interfere with following processing steps or layer bonding in multilayer depositions.

Controlling oxidation is critical in LDED to provide the appropriate material characteristics and part quality. To reduce oxygen exposure during deposition, process factors like as laser power or shielding gas rate may need to be adjusted. Furthermore, post-processing techniques such as heat treatment or surface finishing can be used to remove or minimize oxide coatings and restore the material's characteristics for the outside surface. However, if the oxidation is inside the bulk structure it's not recoverable, and a process parameters optimization analysis should be done to avoid this issue.

2.6 OPTIMISATION OF MICROSTRUCTURAL POROSITY IN ADDITIVE MANUFACTURING

2.6.1 PROCESSING STEPS TO OVERCOME POROSITY

Intersection porosity facilitates the rapid separation of printed layers in response to a transverse tensile force on the graded material. This problem can be solved [Al-Maharma et al., 2020] by changing the printing scheme to one layer where possible, for example, a 0/90 woven structure is printed into one layer to improve stress distribution. Increasing the temperature of the chamber using buildplate pre-heating or introducing an intersection material [X. Zhang et al., 2021] would help to solve this issue.

Figure 2.12 highlights the most current solutions and shows how they improve the mechanical properties of materials processed by AM. These solutions can be used to cure any kind of air pore.

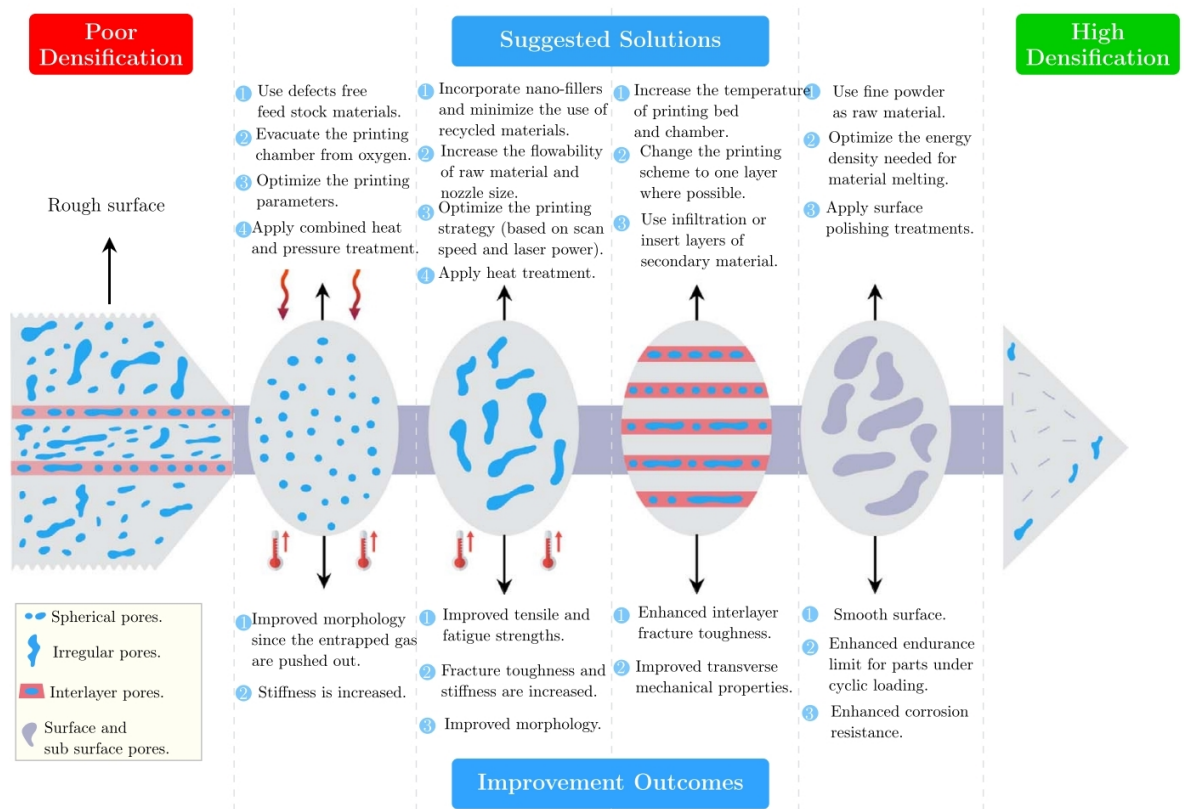


Figure 2.12: Optimizing the mechanical performance of AM processed materials.[Al-Maharma et al., 2020]

Powder production also affects porosity. The spherical shape provides the solution to the porosity problem [Snyers et al., 2021] thanks to its excellent flowability, increased packing density, and enhanced melting properties. Spherical particles flow more consistently, resulting in uniform powder dispersion and fewer gaps. They also achieve increased packing density, resulting in fewer interstitial voids. Furthermore, their homogeneous surface area-to-volume ratio enables consistent melting and solidification, while their smooth surfaces reduce oxidation, resulting in fewer inclusions. These parameters work together to increase the density and quality of the final deposited layers, effectively lowering porosity in LDED processes.

The paper [El-Wardany et al., 2018] suggests various strategies that the writers employed to

overcome porosity during Cu L-DED. The outcomes of the technologies created show how crucial it is to treat copper powder to make powder flow more easily during deposition. Moreover, the treated powder exhibits a greater resistance to surface oxidation, resulting in a significant decrease in porosity development and an enhancement in the quality of the copper deposits.

2.6.2 POST-PROCESSING STEPS TO OVERCOME POROSITY

Several approaches have been studied to reduce porosity by [W. Zhang et al., 2024]. One of them is Hot Isostatic Pressing (HIP), in which the microstructure of AM components can be homogenized while simultaneously densifying and releasing tension in the matrix by synergistically applying high pressure and temperature. This is commonly regarded as the best post-treatment method for improving the fatigue performance of DED parts and fixing process-related flaws. Process optimization, quality, and composition control of the powder feedstock can contribute highly to overcoming porosity.

Materials and Methods

3.1 MATERIALS AND SUBSTRATE

Experiments are conducted with commercially available 316L SS (Carpenter Additive (Powder-Range)) and CuZr1Cr powder (Eckart, Amnovis, and KU LEUVEN). Both materials' powders are spherical and semi-spherical as illustrated in Table 3.1, with particle sizes ranging from 15-45 μm . Table 3.2 presents the composition of both alloys. As a substrate for LDED deposition, a hot rolled and annealed 316L SS circular solid disc is used.

Table 3.1: Powders specification

Powder	Particle diameter (μm)	Production
CuZr1Cr	15-45 (Eckart), 45-100 (Amnovis)	Spherical (Eckart), gas-atomized (Amnovis)
316L SS	15-45 (PowerRange)	Near-spherical gas-atomized

Table 3.2: Composition of CuZr1Cr and 316L SS used for LDED [METALCOR, [n.d.](#)]

	CuZr1Cr	316L SS
Ni	-	12.5-15%
Cr	0.5-1.2 %	17-19%
Si	0.10%	1%
Fe	$\leq 0.08\%$	Rest
C	-	$\leq 0.03\%$
Mo	-	2.5-3%
Mn	-	2%
P	-	0.045%
S	-	0.015%
Cu	Rest	-
Zr	0.03-0.3%	-
N	-	≤ 0.11

As was highlighted in the literature review, the significance of treating Cu powder is to facilitate easier powder flow during deposition. Furthermore, the gas-atomized spherical powder particles exhibit greater resistance to surface oxidation, resulting in a decreased formation of porosity and an enhancement in the quality of the Cu and Fe-based alloy deposits [Susan et al., 2006][El-Wardany et al., 2018][Snyers et al., 2021].

Moreover, powders with greater particle size ratios may result in less deposit porosity than powders with smaller ratios [Susan et al., 2006], the rationale behind the usage of various particle sizes is to examine how particle size affects porosity.

Table 3.6 provides a summary of the powder specifications. It emphasizes that Fe-based alloys have a lower melting point than Cu-based alloys, which makes it difficult to deposit this mixture because the first will solidify more quickly and result in a less mixed bulk structure. Problems

Table 3.3: Physical and Thermal Properties of 316L SS and Cu [X. Zhang et al., 2021]

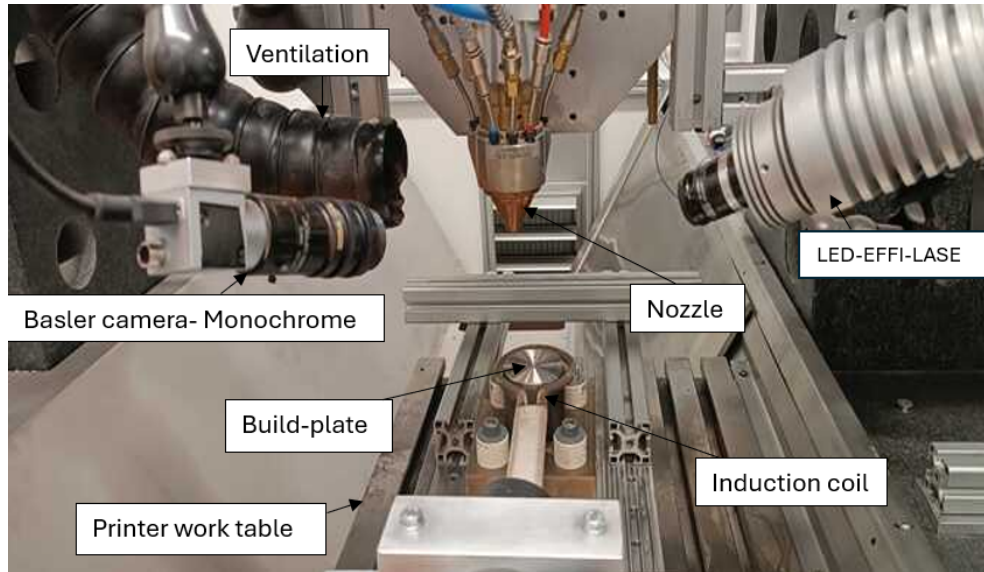
	SS316L	Cu
Density (g/cm ³)	7.99	8.96
Melting point (°C)	1375–1400	1085
Thermal conductivity (W/(m K))	13.6	400
Thermal expansion coefficient ($\times 10^{-6}$ /°C)	27.5	17

with flowability can arise from the small difference in density, but these can be resolved by optimizing the flow of the carrier gas.

3.2 EXPERIMENTAL SET-UP

3.2.1 THE MiCLAD RESEARCH PLATFORM AT THE START OF THE MSc THESIS

The MiCLAD machine manufactured the test specimens, developed at the Vrije Universiteit Brussel (VUB) in Belgium as presented in Figure 3.1, which enables controlled laser metal deposition, as well as various in-situ optical measurement systems. During the construction process, these integrated sensors collect data on deposition geometry and temperature. It contains a fiber laser (SPI redPOWER® QUBE) operating at a wavelength of 1064 nm, with a spot size of 2.5 mm and a maximum average power of 2 kW, in addition to a monochrome camera (Basler) which visualizes the cladding nozzle and melt-pool for operational reasons.

**Figure 3.1:** Set-up description for L-DED process on MiCLAD machine.

A coaxial continuous feed nozzle (Fraunhofer ILT COAX-S40) conducted the metallic powder in the deposition head with a 3.5 g/min powder feeder rate. The local shielding is done using high-purity Argon to protect the materials from oxidation around the melt-pool. Argon (Ar) flow was used as the carrier and shielding gas, and a flow range of [5-10 L/min]. Ar is a popular choice due to its inert nature. It effectively shields the melt pool from oxygen and nitrogen.

To realize 3D object deposition, the work table can move along three linear axes X-Y-Z. A CNC controller integrates and controls all of these systems.

Induction Pre-heating of the build plate set-up is illustrated in Figure 3.1, where a circular

induction coil is fixed around a cylindrical build plate to pre-heat it before the LDED process, to avoid high-temperature gradients. Figure 3.3 illustrates the effect of heat input on residual stresses as two types of welding processes (conventional and preheated) were compared and the preheated one results in less residual stresses. The build plate pre-heating was a subject of two MA1 projects, where last year G.De Muynck, implemented the open loop temperature control and he used a thermocouple to measure the maximum temperature at each setpoint. Meanwhile this year Y.Bouguezine implemented the closed-loop control using a pyrometer which is a safer approach since there will be no contact between the pyrometer and the buildplate.

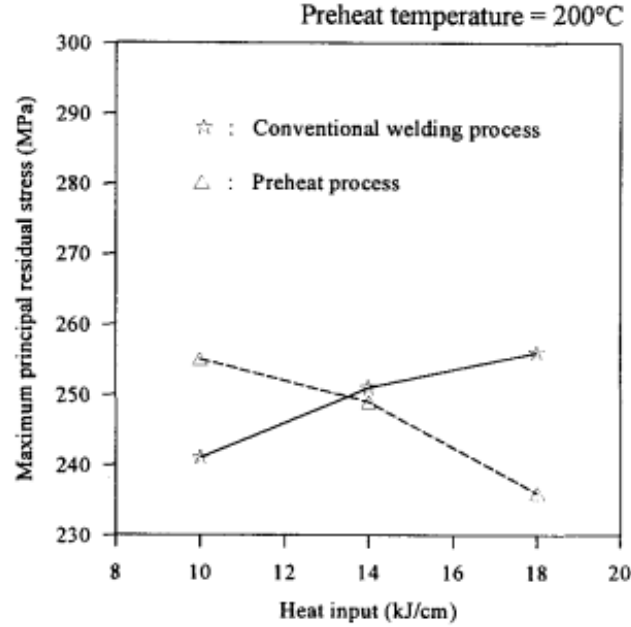


Figure 3.2: The effect of heat input on residual stresses Y.Bouguezine MA1 project

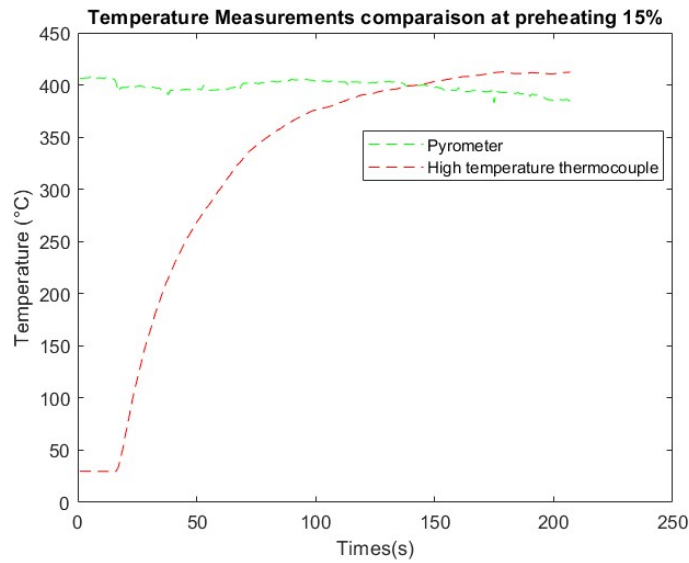


Figure 3.3: A comparison between Pyrometer and thermocouple for builplate temperature measurements Y.Bouguezine MA1 project

3.2.2 SECONDARY HOPPER IMPLEMENTATION TO THE MiCLAD RESEARCH PLATFORM FOR DUAL POWDER SUPPLY

The powder feeding system is a critical part of the L-DED process. MiCLAD machine uses "Medicoat" powder feeders which are sophisticated devices designed to precisely deliver powder materials in a variety of industrial applications, including thermal spraying and additive manufacturing. These feeders provide precise and consistent powder flow, which is essential for producing high-quality coatings and deposits. They are outfitted with sophisticated control systems that govern the feed rate, resulting in uniform distribution and less material waste. Medicoat powder feeders are well-known for their dependability, user-friendliness, and flexibility to various types of powders, making them a vital component in operations requiring precise material application and high-performance criteria. These feeders use vibration and frequency control to improve powder consistency and precision. The vibration mechanism is built into the hopper or feed system to avoid powder agglomeration and provide smooth, consistent dispensing. The powder flow rate can be precisely controlled by altering the vibration frequency.

There are a few major limitations on the MiCLAD machine that limit its application versatility and efficiency in applications involving FGMs. Within the framework of this thesis, the MiCLAD machine's main drawback is that it was able to use only a single -hopper since this is the first attempt to process multimaterials. The first test campaign (see paragraph 4.1) was performed using this single hopper and the different blended compositions were manually pre-mixed. It was noticed that in a single-hopper system, changing materials is a costly and potentially contaminated process. The hopper and feed lines must be thoroughly cleaned after every material change, which increases downtime and lowers output overall.

During the deposition process, the machine is unable to dynamically blend various powders to create customized material compositions. This restricts the ability to accurately change the material's properties to meet the needs of particular applications or to optimize the material for particular performance attributes.

To improve the MiCLAD machine's functionality, a secondary hopper should be added in light of these limitations as shown in Figure 3.4. A secondary hopper would make it possible to use multiple materials at once, greatly increasing the machine's versatility and enabling the construction of multi-material structures and functionally graded materials. This ability is crucial for optimizing material qualities for particular uses, which lowers material composition variation time and improves performance. The implementation of a secondary hopper would allow for the instantaneous blending of various powders, optimum material composition, and reliable quality.

3.2.2.1 Powder mixing for FGM fabrication

Since FGM fabrication by transitioning from one material to another necessitates gradual modification of the powder chemical composition, I chose to divide the part into sections, and each section with different chemical composition, starting from 100% Fe to 100% Cu by a step of 25% grading percentage, this choice was based on literature as researchers attempted different grading percentages and mainly it was concluded that to overcome the challenge of Fe-Cu bonding, the least the grading percentage the better the deposition quality, so 1% grading percentage would be the optimal FGM, but it's costly and it will be very challenging to analyze the optimal parameters for 100 section. For the LDED of an FGM part, automatic control of the powder feed rate is required as well, then for GD25Cu, the feeder should be able to vary the PFR for each of the 5 sections.

Because of this hardware modification that greatly influenced this thesis work, I decided to refer to the single-hopper configuration as Phase I, while the superior dual-hopper layout is called



Figure 3.4: Medicoat Twin powder feeders

Phase II in this thesis. During Phase I, mixing was done manually and powder was placed in Hopper I for each blended composition. During phase II, Hopper II was employed to store Cu powder and Hopper I for Fe powder.

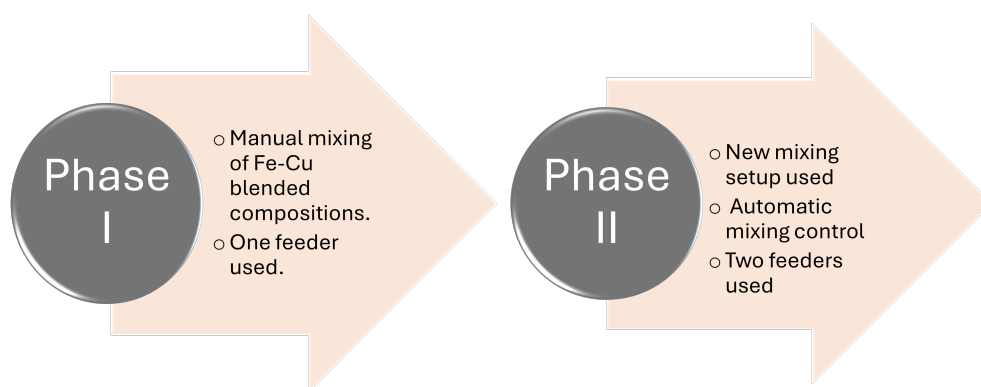


Figure 3.5: Feeders implementation workflow throughout the thesis

The mixing of powders was an issue because when both feeders were connected using a Y link (Setup 1), there was backflow due to a lack of synchronization, as well as leaks, so another configuration was done by mixing the powders on top of the nozzle (setup 2) to avoid problems

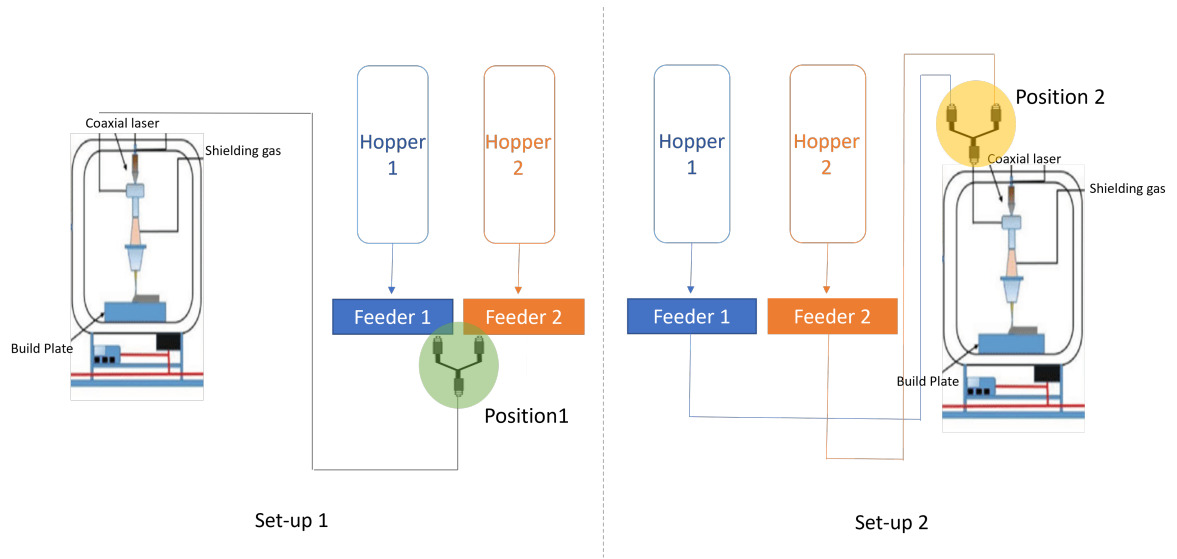


Figure 3.6: Powders Pre-mixing configurations

related to dissimilar flowability of powders as shown in Figures 3.6 and 3.7. Previously, only Feeder I was controlled by the MiCLAD machine's CNC controller, so the set points for Feeder II were manually changed during the print. However, Feeder II is integrated into the CNC controller and can now be automatically controlled during the print, thanks to my supervisors Michaël Hinderdael and Zoé Jardon.

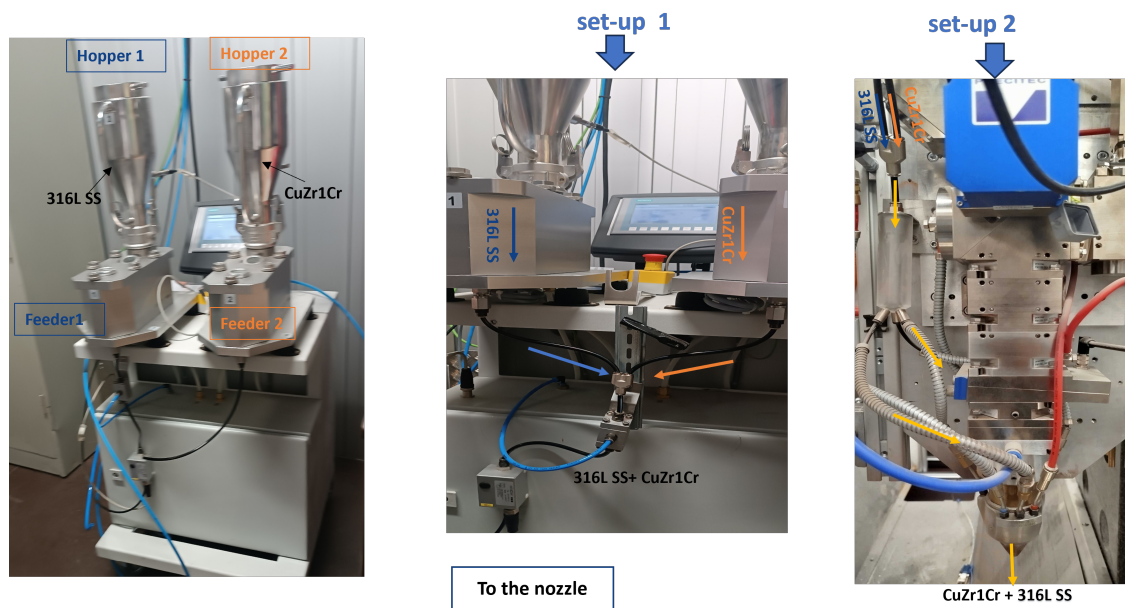


Figure 3.7: Feeders set-up 1 and 2

3.2.2.2 Powder flow rate calibration for two hoppers

The first step of the powder flow rate calibration was to record the powder weight versus time and it was done via the **Kern Balance Connection** which enables the easy incorporation of weight measurements into Excel documents. By reducing manual entry errors and simplifying data management, this tool improved data accuracy and efficiency during the experimental phase.

To select the optimal feeder frequency with a narrow flow rate range as the nozzle has a maximum PFR set-point designed of 3.5g/min. Knowing that, I want this to correspond to the 100% amplitude, to gain maximum control over the smaller flow rates, reason why a thorough monitoring and frequency adjustment at 100% mass flow rate are carried out during the powders flow rate calibration process. The CuCr1Zr (Eckart, 15-45 μ m) calibration curves at different feeder frequencies are shown in Figure 3.8, which demonstrates that the powder mass flow rate is maximal at low frequencies and vice versa. The optimal frequency for feeder 2 is 124 Hz which delivers at 100% set point a powder flow rate of 5.78 g/min which is the closest to the nozzle set point.

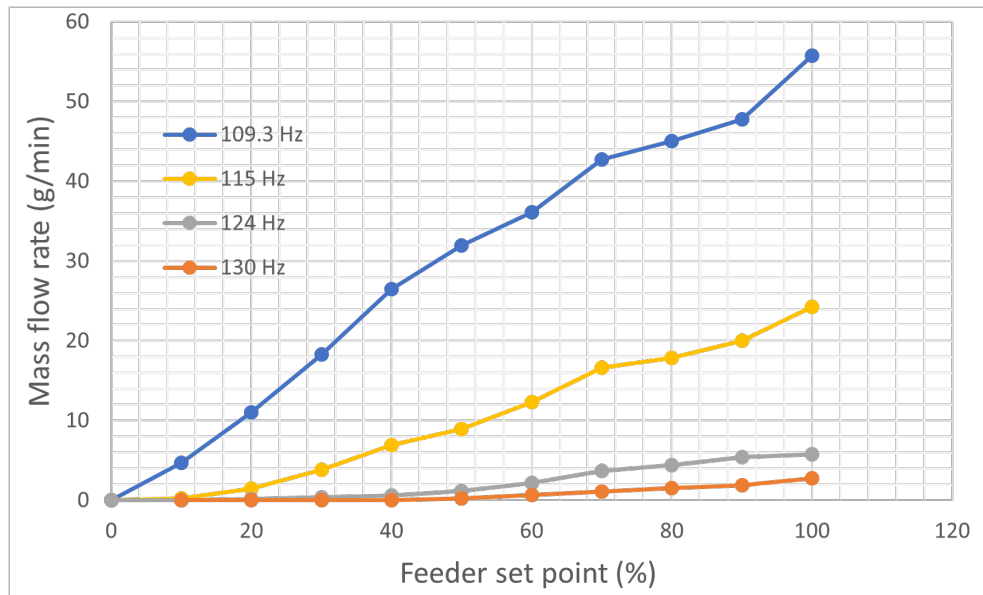


Figure 3.8: Mass flow rate calibration for CuCr1Zr (15-45 μ m) at various feeder vibration's frequencies

The first set of tests was done using only one feeder, and the Fe-Cu blended compositions were mixed manually and added separately to the feeder. To make the process more automated, the second feeder should be used, a mass flow rate calibration was done concerning the feed rate percentage of Cu because of the dissimilarity of size and shape of powders. Figure 3.9 represents the calibration curves for both feeders. This function gives us a direct relationship between the wanted output powder flow rate and the input feed rate.

3.2.2.3 Recalibration after changing carrier gas flow meter and higher powder particle size

In Medicoat feeders, argon (Ar) and helium (He) valves are used to control the flow of these gases. Argon provides an inert atmosphere that inhibits oxidation and contamination, resulting in high-quality coatings, while helium's specific features, such as low density and thermal conductivity, improve coating precision and efficiency. The use of argon and helium in Medicoat feeders takes advantage of the strengths of both gases, resulting in excellent coating quality and process efficiency. Since in the lab, only Ar is used as the inert gas, there was a need to replace the He valve with an Ar valve to guarantee the accuracy of flow control.

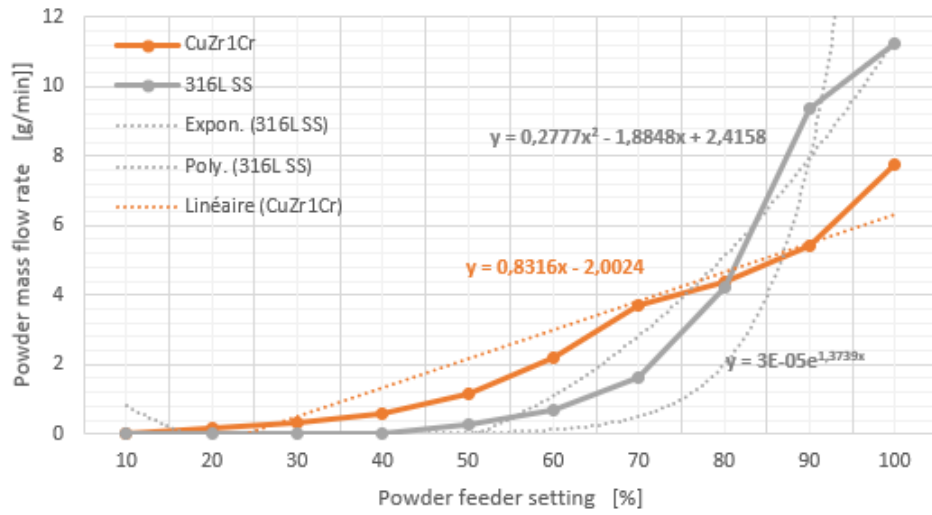


Figure 3.9: Mass flow rate calibration for 316L SS (45-100 μm) and CuCr1Zr (15-45 μm)

To ensure the accuracy and dependability of the gas flow measurements going forward, a recalibration procedure was carried out after the replacement of the carrier gas flow meter. A preliminary test was conducted on the recently installed flow meter to evaluate its accuracy in comparison to standard calibration criteria. If differences were found during verification, modifications were performed to bring the flow meter readings into line with the anticipated values. Figure 3.10 a slight error difference was noticed by introducing the new gas flow meter but this last calibration was considered more precise and it will be the one used for the future tests.

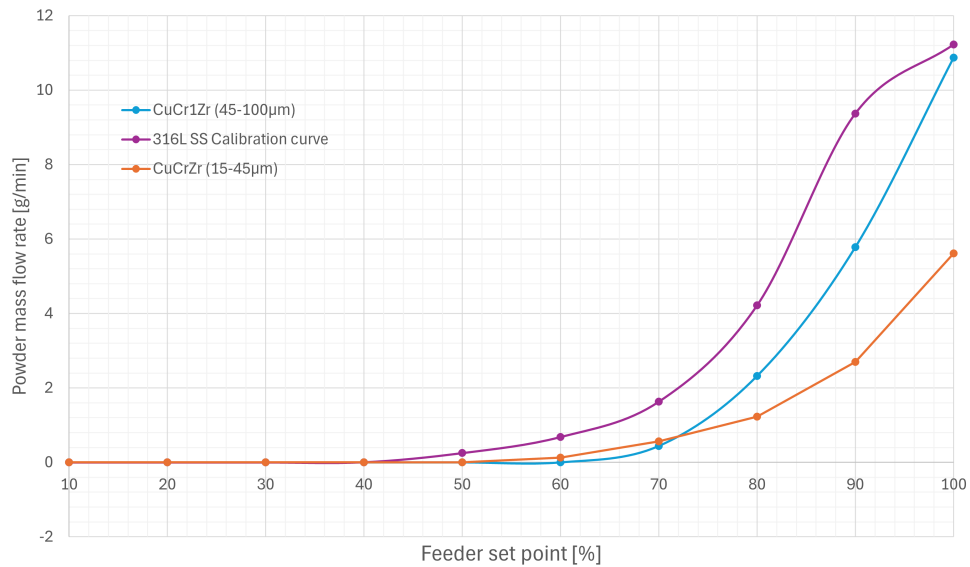


Figure 3.10: Mass flow rate calibration for 316L SS (45-100 μm), CuCr1Zr (45-100 μm) and CuCr1Zr (15-45 μm)

Increasing the powder particle size generally increases the powder feed rate due to enhanced flowability, since larger particles experience less inter-particle friction and cohesion. This produces a more stable and consistent feed rate. Figure 3.10 illustrates that larger particles (45-100 μm) of CuCr1Zr tend to increase the powder feed rate by 42% because volumetric feeders dis-

pense a higher mass of powder per unit of time when particle size is larger, compared to the smaller particles which pack more tightly and flow less freely. Additionally, larger particles are less likely to cause nozzle clogging than small powders, which can quickly bridge and block channels.

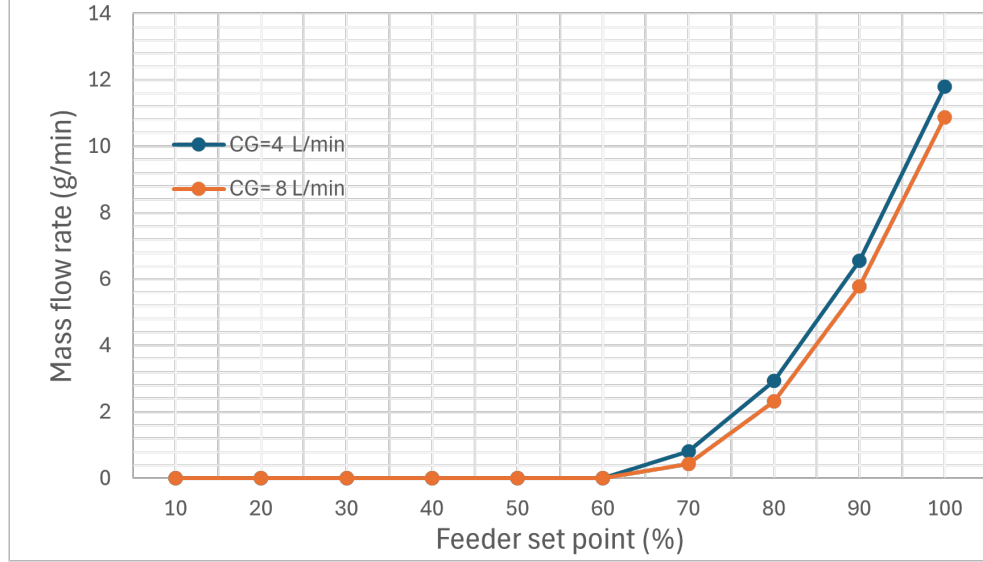


Figure 3.11: Carrier gas flow rate effect on powder feed rate for CuCr1Zr (45-100 μm)

The carrier gas flow rate has a slight effect on the powder feed rate. Figure 3.11 shows that if we double the CG the powder feed rate decreases by $\pm 10\%$. As the CG increases, there will be a powder loss at the outlet of the nozzle due to the high gas flow rate.

3.2.2.4 Linearisation of the powder calibration curve

The powder feed rate is a critical parameter in determining the build rate in the LDED process and obtaining the exact composition as desired. However, if the powder feed rate exceeds the powder's melting rate, a produced product with significant roughness or cratering may result. On the other side, if it is set too low, it may result in a slower build rate and the presence of unmelted particles on the created surface [Majumdar et al., 2021]. As a result, achieving an optimal feed rate is critical to producing a defect-free product with a high build rate.

To establish set points for the powder feeder using calibration curves and linear interpolation, we started by conducting calibration tests to measure the mass flow rates at various set points. The next step is plotting the calibration curve for CuCr1Zr (Eckart CuCr1Zr (15-45 μm)) as illustrated in Figure 3.12. For a desired mass flow rate, one can identify two calibration points that bracket this value. Finally, the linear interpolation formula 3.1 should be applied:

$$S = S_0 + \frac{(M - M_0)(S_1 - S_0)}{M_1 - M_0} \quad (3.1)$$

where S is the set-point, and S_0 , M_0 and S_1 , M_1 are the known set points and mass flow rates. This method ensures precise control of the powder feeder for accurate additive manufacturing.

The same linearization approach was used when the Cu powder changed, Table 3.5, presents the updated set points when using the new powder (Amnovis CuCr1Zr (45-100 μm)) as one can observe that the increase in particle size affects the feeder set-point. Whenever a new powder

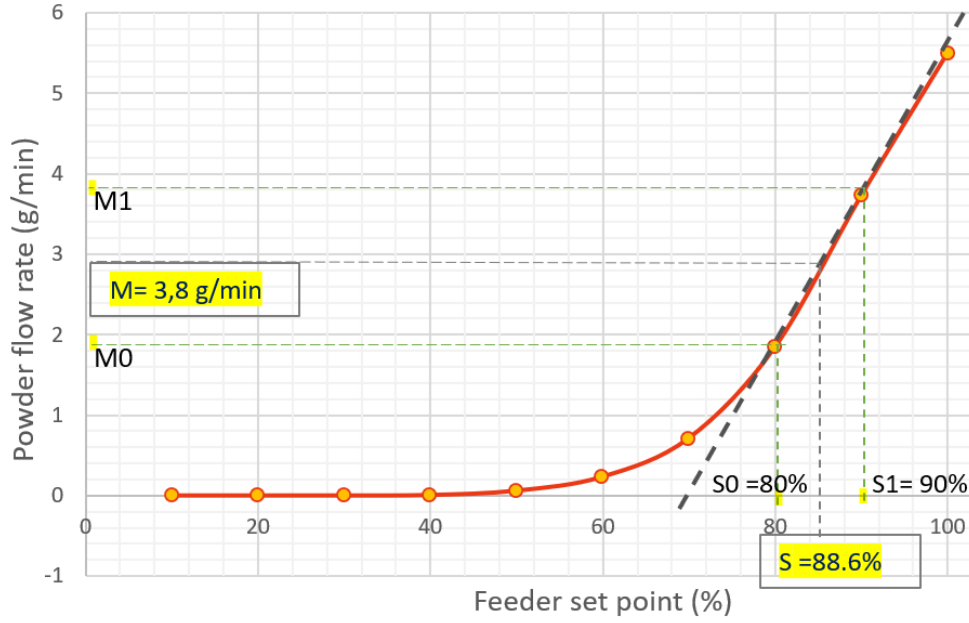


Figure 3.12: Linear interpolation using the first calibration curve of CuCr1Zr

Table 3.4: Powder feeders set points for different blended compositions using the first calibration (for Eckart CuCr1Zr (15-45 μ m))

Fe		Cu		Feeder 1	Feeder 2
(%)	(g/min)	(%)	(g/min)	(%)	(%)
100%	3.5	0%	0.0	77.4	0.0
75%	2.6	25%	0.9	73.9	71.4
50%	1.7	50%	1.7	70.4	79.4
25%	0.9	75%	2.6	62.0	83.6
0%	0	100%	3.5	0.0	88.6

with a different supplier and different particle size will be used, a new calibration is obligatory to be done to avoid excess and too low PFR.

Table 3.5: Actual powder feeders set-points for different blended compositions using the last calibration (for Amnovis CuCr1Zr (45-100 μ m))

Fe		Cu		Feeder 1	Feeder 2
(%)	(g/min)	(%)	(g/min)	(%)	(%)
100%	3.5	0%	0.0	77.2	0.0
75%	2.6	25%	0.9	73.8	70
50%	1.7	50%	1.7	70.4	76
25%	0.9	75%	2.6	62.0	79
0%	0	100%	3.5	0.0	82

3.3 MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION

3.3.1 SAMPLE PREPARATION

LDDED deposited bulk structures of individual Fe-Cu blended compositions' thin walls are sectioned for metallography examination. Epoxy resin is used to mount the extracted samples. Following that, samples are polished and ground with abrasive paper ranging in grit from 180

to 1200 grit, then cloth polished. Table 3.6 presents the polishing specifications used for the microscopic analysis of all the samples in the scope of this thesis.

Table 3.6: Polishing Specifications

Surface	Abrasive/Size	Speed (rpm)	Time (Min.)
Abrasive paper	200mm P180 to P1200 grit	150	Until plane
cloth	9 μm Monocrystalline Diamond Suspension	150	4
cloth	3 μm Monocrystalline Diamond Suspension	150	4
cloth	1 μm Monocrystalline Diamond Suspension	150	4

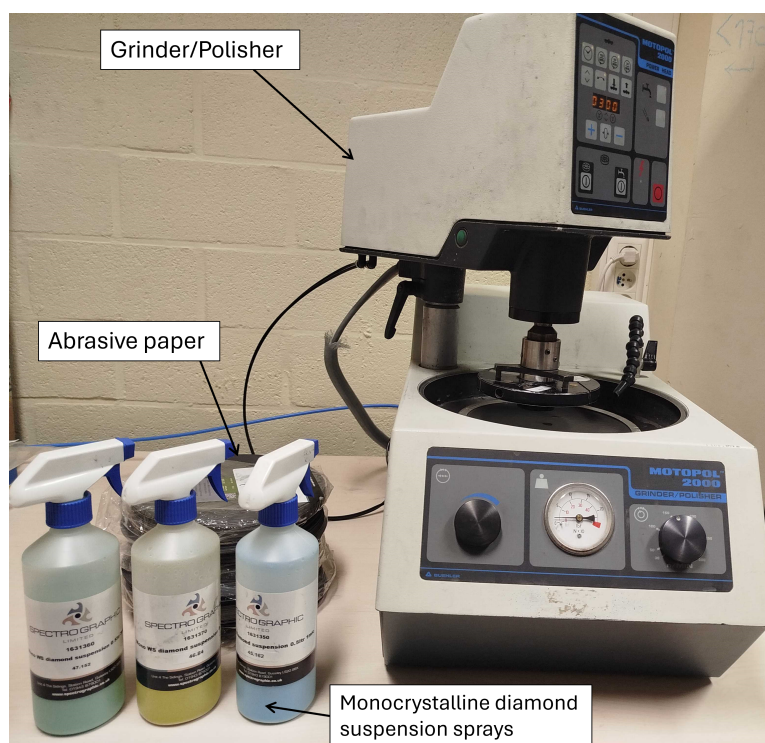


Figure 3.13: Polishing set-up

3.3.2 ETCHING AGENTS

For chemical composition contrast, Fe is etched using "Villela Reagent", a solution composed of picric acid (1 g), hydrochloric acid (5 ml), and ethyl alcohol (95 ml). Often used to depict the grain boundaries of Fe as it allows inspection of the scanning strategy, dilution, track dimension, layer height, and, on a smaller scale, the grain borders, which have a different composition and thus react differently to the etchant.

When the percentage of Cu is lower, the employed etchant (Villela) is the most suited. It is not advised for pure Cu since it makes the sample extremely bright, making inspection challenging. Figure 3.14 illustrates the effect of etching of a 100% Cu sample, concluding that etching is useful to better visualize the sample especially when there are water spots on the surface of the sample. However, the etching time is very important for Cu samples as if it's too high the brightness of the sample will increase and it will be challenging to analyze it microscopically.

Three types of etchants (COPPER ETCH? ASTM E407 and KLEMMS) were tested to check which one is more suited for Cu regions. Copper Etchant would be used for the graded walls to visualize the section's transition as illustrated in Figure 3.15.

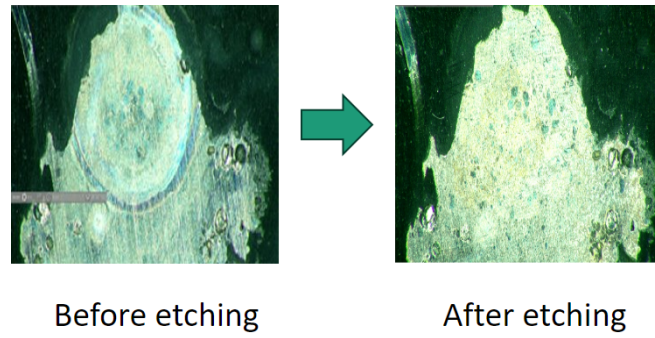


Figure 3.14: Etchant effect on 100% Cu test samples (Used etchant is Villela)

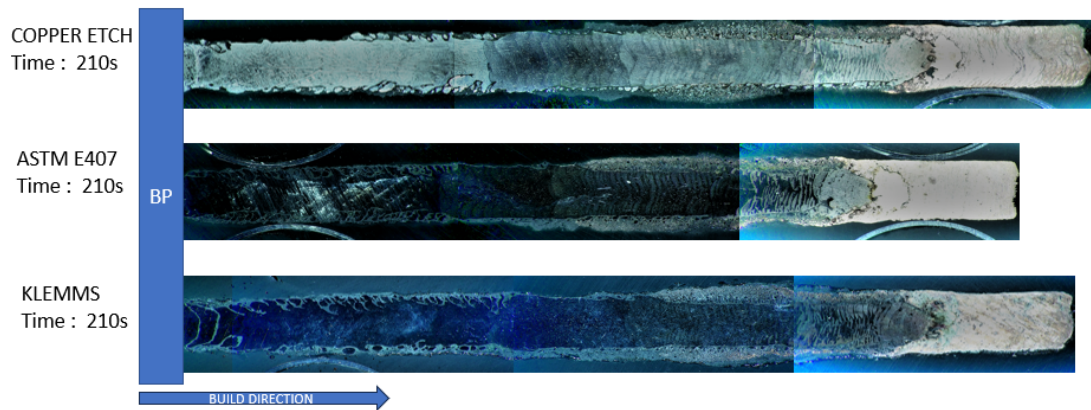


Figure 3.15: Etchant effect on illustrating the transient regions of a graded thin wall (Copper Etchant)

3.3.3 DETERMINATION OF THE POROSITY PERCENTAGE BASED ON MICROGRAPHS

Using several cross-sectional images of the sample, the relative area of the pore compared to the image area is determined, and the build quality examination and relative density are investigated.

The software Image-J is used to analyze relative density. To calculate the porosity percentage in ImageJ, first one should transform the grayscale image of the porous material to binary by using thresholding, which highlights the pores in the binary image. The area of the identified pores is then measured using the Analyze Particles tool, with parameters adjusted to eliminate noise and precisely assess pore characteristics. The porosity % is calculated by dividing the entire area of the discovered pores by the total area of the image, then multiplying the result by 100. This procedure makes it possible to precisely quantify porosity, which is essential for scientific study in several disciplines, such as materials science, and biomedical engineering.

3.3.4 MICROHARDNESS MEASUREMENTS

In materials science and engineering, hardness measurement is a fundamental characteristic that provides important insights into the mechanical properties and performance of materials. Hardness measurements provide a material's resistance to permanent deformation and are useful in determining whether or not it is appropriate for use in structural parts or cutting tools, for example. Hardness data helps with material selection, quality assurance, and manufacturing process optimization so that goods fulfill durability requirements and desired performance criteria. Furthermore, studies of hardness frequently show correlations with other mechanical

attributes such as wear resistance, and tensile strength, offering important insights for material characterization and design optimization.

Microhardness is measured along the grading direction with a dwell period of 10 seconds using the Duramin 5655100 which measures hardness by first securely placing the test sample on the machine's anvil.

LDED-constructed samples are subjected to the single-cycle diamond indentation at a preload of 4,903 N. After configuring the settings, the machine precisely lowers the indenter into the sample surface, resulting in an indentation.

Following the indentation procedure, the Duramin 5655100 uses an optical system to determine the size of the indentation made by the indenter. The machine's software uses these measurements to calculate the material's hardness. This approach offers precise and dependable hardness testing.

3.3.5 EDS AND SEM ANALYSIS

The elemental distribution, microstructural analyses, and fractography are carried out with a FEG-SEM coupled to an energy-dispersive spectroscopy (EDS) device (by **KU LEUVEN**).

Energy Dispersive X-ray Spectroscopy (EDS) and Scanning Electron Microscopy (SEM) are essential techniques in material science for assessing sample composition and microstructure at high resolution. SEM produces precise images of materials' surface morphology at nanoscale resolution, revealing features such as grain boundaries, fractures, and surface roughness.

EDS, when combined with SEM, enables elemental analysis by identifying distinctive X-rays generated when the sample is blasted with electrons. This technique enables researchers to determine the chemical composition of specific regions inside the sample, hence determining the distribution and concentration of elements. Furthermore, EDS can accurately estimate elemental composition, which aids in phase identification and gradient mapping.

SEM and EDS work together to give a full understanding of materials' structural and chemical properties, allowing for research in domains such as metallurgy, nanotechnology, and biomaterials.

The work proceeds systematically throughout three main sections. Section 4.1 examines homogeneous thin walls with different Fe-Cu compositions. To find the best processing window for each composition based on metallurgical analysis, this investigation entails using Laser Direct Energy Deposition (L-DED) to deposit thin walls of various compositions and methodically adjust process parameters. This investigation is expanded to in-situ mixed gradient thin walls (Section 4.2), where the emphasis is on forming gradient structures by the in-situ mixing of Fe and Cu during deposition. Here, the work investigates parameter optimization to minimize defects such as intersection porosity and produce smooth transitions between compositions. In section 4.3, the study results in the fabrication of a three-dimensional demonstrator component of Functionally Graded Material (FGM), addressing the difficulties associated with printing intricate geometries and evaluating the mechanical attributes of the printed parts using tensile testing. Through methodical testing and analysis, the research endeavors to enhance the comprehension of processing Fe-Cu FGM for diverse applications and optimize L-DED parameters.

4.1 PROCESSING OF HOMOGENEOUS THIN WALLS WITH VARIOUS PRE-BLENDED FE-CU MIXTURES

LDED process parameters govern engineering components' build quality and properties. As a result, the thin walls of various blended compositions are deposited using L-DED to determine the best set of process parameters for each GD25Cu composition. Following that, bulk structures of various blended compositions are constructed using L-DED to ensure the viability of identified parameters for multi-layer depositions. This section summarizes the process window for L-DED of Fe-Cu FGM by thin walls and bulk depositions at various Fe-Cu pre-blended compositions, the mixture of those various compositions was manually (Phase I).

Thin walls of various Fe-Cu pre-blended compositions ($Fe_{100-x}Cu_x$, x varies as 25, 50, 75, and 100) are laid in the preliminary investigation at varying laser power, scan speed as presented in Table 4.1. The quality of the deposition process was continuously monitored and process parameters were systematically optimized to improve the process. We can get the optimal settings for any composition by looking at these variations. This is an important phase since the microstructure and mechanical characteristics of the final deposits are greatly influenced by the processing conditions.

Table 4.1: Experimental details of LDED of Fe-Cu blended compositions

Fe-Cu Composition	LP_{start} [W]	LP_{end} [W]	v_{scan} [mm/min]	t_{wait} [s]	Preheating
$Fe_{75}Cu_{25}$	800,1000	375,450,500,600,800	900	0	off, on
$Fe_{50}Cu_{50}$	1000,1100,1200	375,400,450	900	0,1,2	on
$Fe_{25}Cu_{75}$	1200	375,500,650,800,950	900,1200	0,1,2	on
Cu_{100}	1200	400,500,600	1200,1800	0,1,2	on

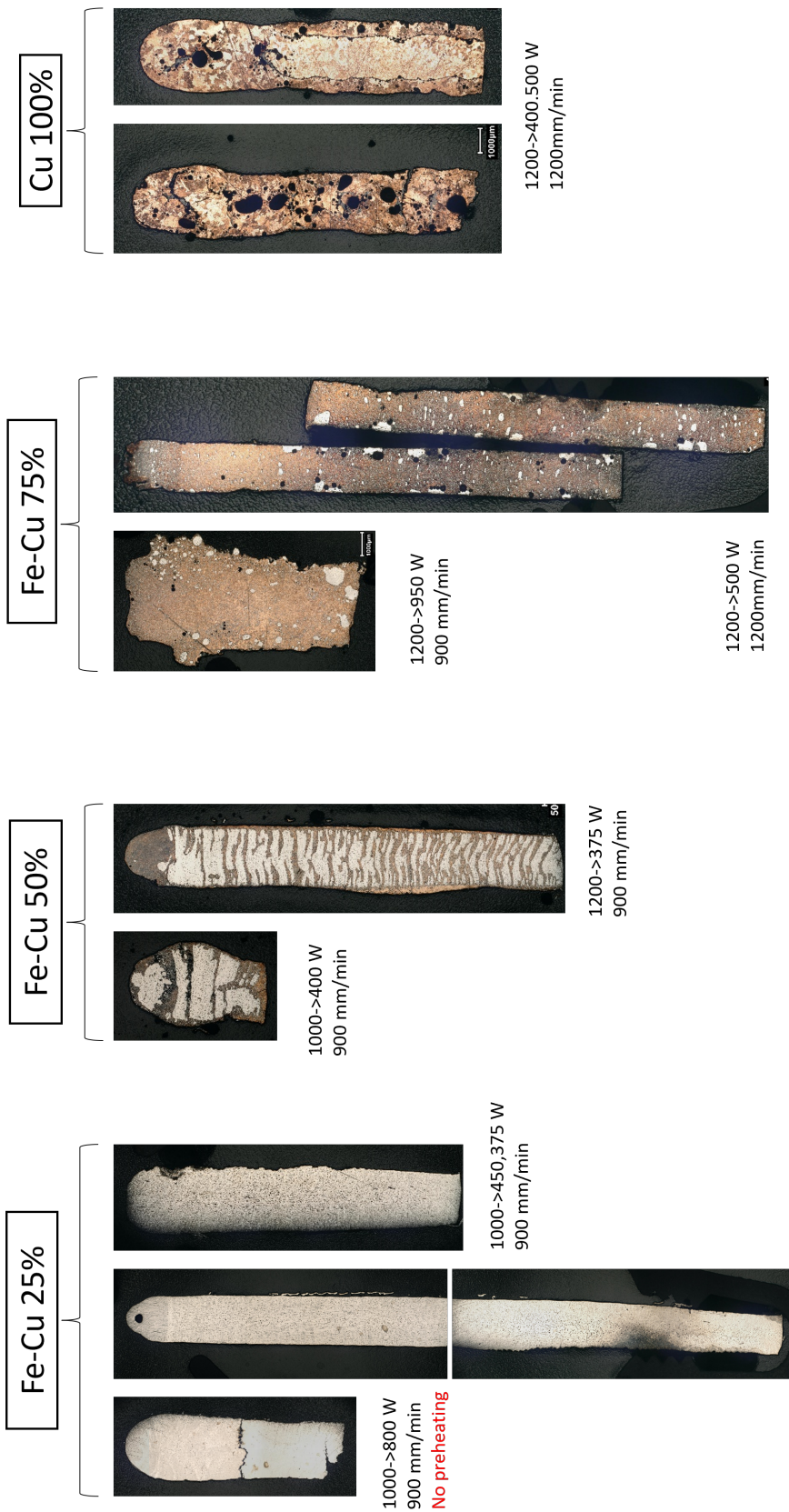


Figure 4.1: Representative cross-sectional view of Thin walls LDED Fe-Cu blended compositions (Microscopic images done by **KU LEUVEN**)

4.1.1 BUILDPLATE PRE-HEATING EFFECT ON CRACKS

For samples with blended composition $Fe_{75}Cu_{25}$, some cracks occurred in a sample when the build plate was not pre-heated as illustrated in Figure 4.1; however, when induction was applied, the sample remained porosity-free and crack-free. All the advantages of using for using buildplate preheating are already mentioned in 3. For all the test campaigns in the scope of this thesis, the pre-heating will be used.

4.1.2 MICROSTRUCTURE ANALYSIS FOR FE-CU BLENDED COMPOSITIONS

The microstructure analysis of the Fe-Cu pre-blended compositions will be discussed in this section. In f-Figure 4.2, the evolution from $Fe_{75}Cu_{25}$ to Cu_{100} was illustrated micrographically and in Table 4.3 summarises the selected optimal parameters for each Fe-Cu pre-blended composition.

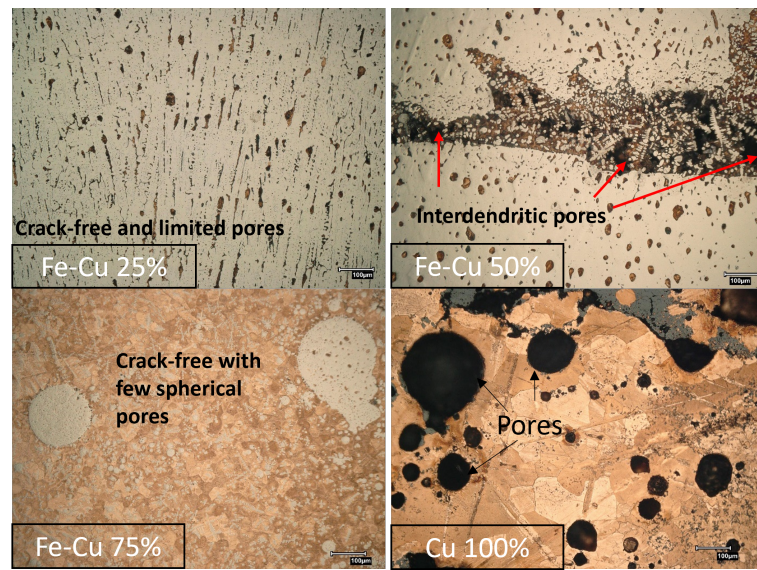


Figure 4.2: Optical micrograph of a cross-sectional view of LDED built thin walls presenting microstructural evolution from $Fe_{75}Cu_{25}$ to Cu_{100} (Microscopic images done by KU LEUVEN)

4.1.2.1 Observations for $Fe_{75}Cu_{25}$

Low Cu concentration compositions, such as $Fe_{75}Cu_{25}$, result in dominant columnar growth with dendritic morphology. This is due to faster solidification and cooling during the LDED process [Yadav et al., 2023]. In addition, phase-separated Cu particles limit grain growth and provide multiple nucleation sites, resulting in a finer microstructure as shown in Figure 4.2.

4.1.2.2 Observations for $Fe_{50}Cu_{50}$

The bulk structure along the build direction is depicted by the zebra-like pattern, which is caused by the different densities of Cu and Fe. In [Zhou et al., 2021], came across the same phenomenon of non-homogeneous material composition issue. This is caused by an increase in Cu concentration, which lowers the cooling rate, limits grain growth, and encourages heterogeneous nucleation, leading to the formation of fine cellular dendritic Fe grains in the Cu-rich zone of GD25Cu. Contrary to what is observed at lower Cu concentrations, increasing Cu concentration causes cellular morphology.

4.1.2.3 Observations for $Fe_{25}Cu_{75}$

Porosity begins to show up as entrapped gas pores (small circular pores) in $Fe_{25}Cu_{75}$, the size of the pores increases when increasing the Cu content, as well as Key holes caused by high energy deposit. Figure 4.3 illustrates the effect of v_{scan} on the mixing of Fe and Cu during deposition, as the v_{scan} increases the mixing of Fe-Cu is better but one can still see the zebra patterns.

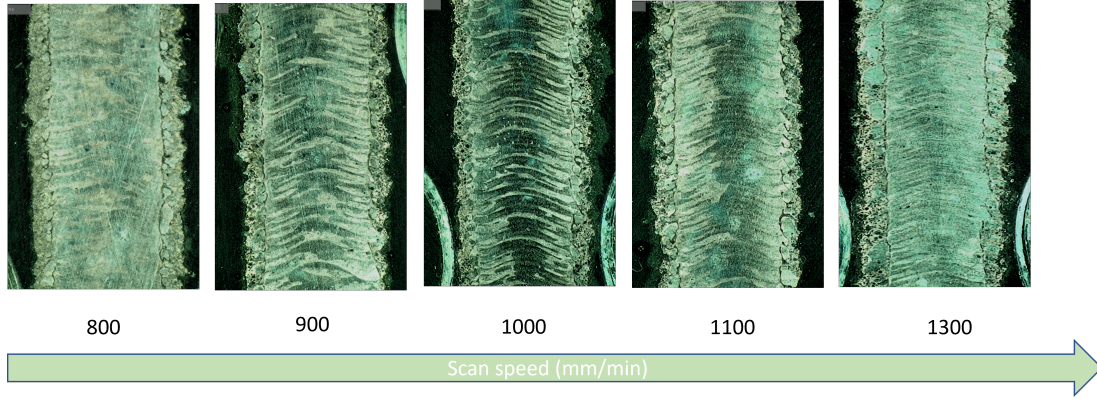


Figure 4.3: Effect of v_{scan} on the mixing of Cu-rich regions and Fe-rich regions in $Fe_{25}Cu_{75}$ blended composition

4.1.2.4 Observations for Cu_{100}

Figure 4.2 clearly shows the presence of porosity in Cu 100%, indicated by small dark spots. Porosity occurs when bubbles of gas (argon) are trapped in the bead after solidification [El-Wardany et al., 2018].

4.1.3 OPTIMISATION OF PROCESS PARAMETERS FOR LDED $Fe_{25}Cu_{75}$ THIN WALLS

A test campaign was conducted to inspect the effect of LP and v_{scan} on pores. This test campaign was the first test in phase II. Figure 4.5 presents the sweet spot (green region) as the process window for $Fe_{25}Cu_{75}$ to get poreless bulk structures.

At high v_{scan} small gas pores appear since at high v_{scan} the energy density is low and results in this type of pores.

Sample [-]	Bulk area [px]	Pores area [px]	Porosity percentage [%]
682	496045	1898	0.383%
683	336312	36	0.011%
684	843129	1553	0.184%
685	1083803	825	0.076%
686	1355276	1374	0.101%
687	627422	1535	0.245%

Table 4.2: Porosity percentage for $Fe_{25}Cu_{75}$ samples deduced using ImageJ post-processing

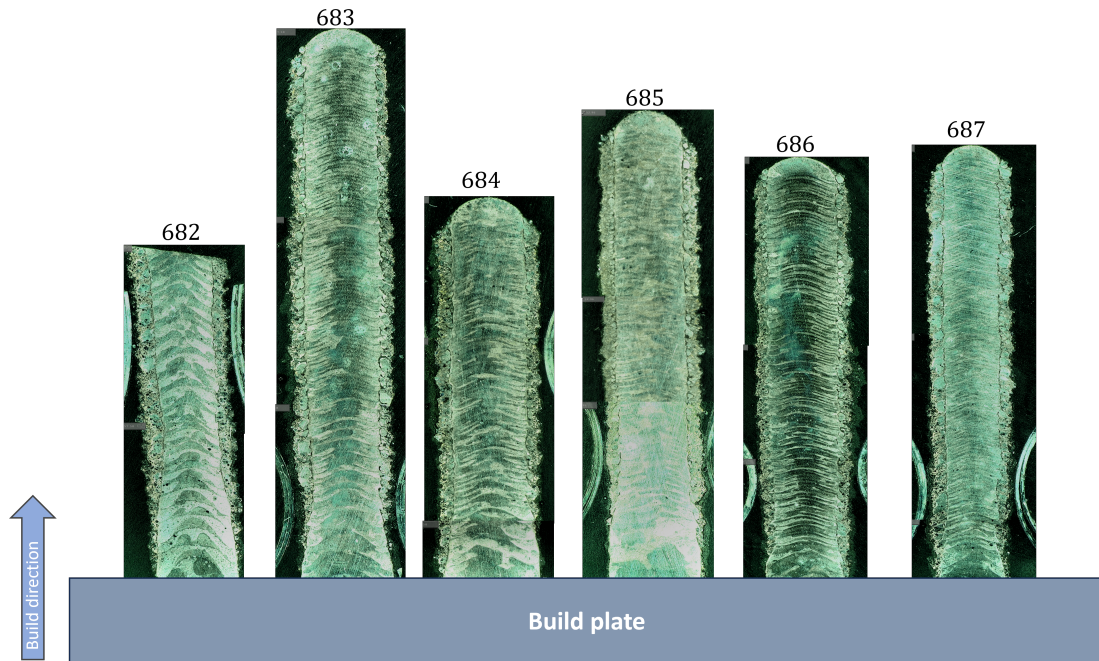


Figure 4.4: L-DED built thin wall of $Fe_{25}Cu_{75}$ pre-blended composition

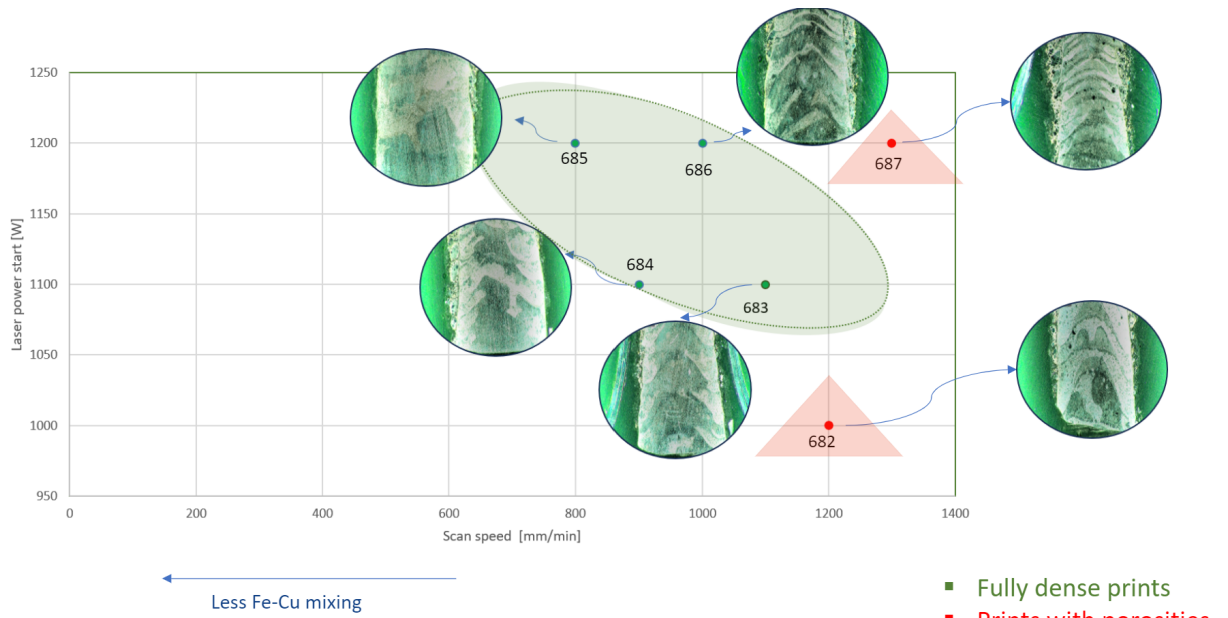


Figure 4.5: The effect of LP and v_{scan} on porosity for $Fe_{25}Cu_{75}$

4.1.4 OPTIMAL PROCESS PARAMETERS FOR EACH BLENDED MIXTURE

The optimal parameters for various Fe-Cu pre-blended compositions are summarized in Table 4.3; for $Fe_{75}Cu_{25}$ and $Fe_{50}Cu_{50}$, no pores or fractures were observed. Additionally, the presence of pores in $Fe_{25}Cu_{75}$ was resolved, and 4.1.3 determined the process window. Printing Cu_{100} was difficult, and there was a significant percentage of pores.

Table 4.3: Selected optimal parameters for L-DED built Fe-Cu blended compositions

Composition	LP (W)	v_{scan} (mm/min)	Observations
$Fe_{75}Cu_{25}$	1000 $\rightarrow$ 375	900	Crack-free and dense parts
$Fe_{50}Cu_{50}$	1200 $\rightarrow$ 375	900	Crack-free and limited pores
$Fe_{25}Cu_{75}$	1000 $\rightarrow$ 500	1200	Crack-free and limited pores
Cu_{100}	1200 $\rightarrow$ 500	1200	Pores defects are prevailing

4.2 LDED PROCESSING OF IN-SITU MIXED GD25Cu GRADIENT THIN WALLS

Optimal parameters selected from the examination of various Fe-Cu blended compositions (4.1.4) were needed to print a continuously graded Fe-Cu thin wall. Using these process parameters, a thin wall consisting of five sections is to be constructed as a first attempt, as shown in Figure 4.6.

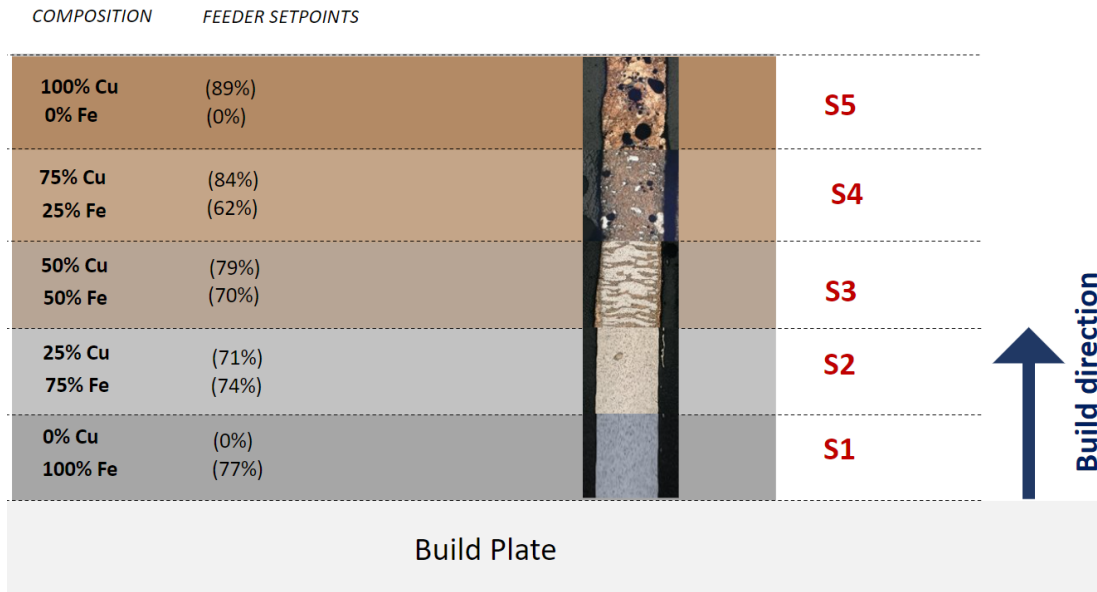


Figure 4.6: GD25Cu thin wall based on optimal parameters for the different Fe-Cu blended compositions [by Zoé Jardon]

4.2.1 ESTABLISHING THE PROCESS WINDOW FOR THE L-DED OF GD25Cu THIN WALLS

Figure 4.7 illustrates the optimization of the various process parameters, as the parameters were adjusted to allow for further linearization of the process parameters.

Through the process parameter evolution plots from S_1 to S_5 , one can observe that l_t is dropping, however, there is an off-peak at S_4 that can be reduced to align with the gradient decline. Constant increases are observed along the thin wall in both v_{scan} and LP.

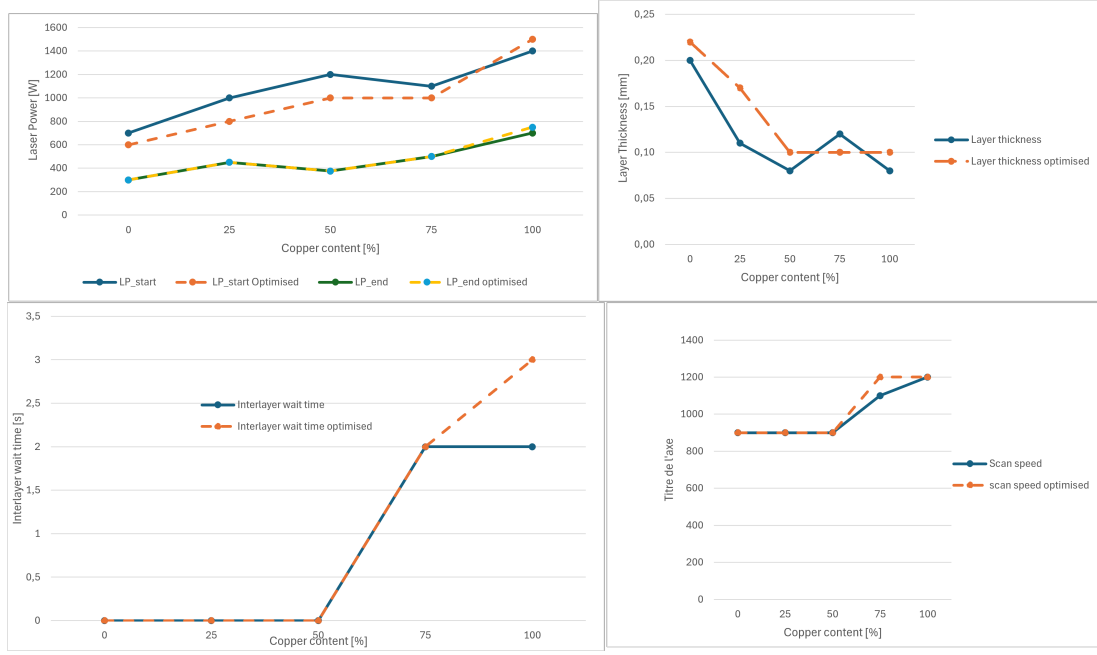


Figure 4.7: Optimisation of process parameters

The interlayer delay in L-DED is used to enable adequate cooling, bonding, and stress relief between successive layers of deposited material. The interlayer delay was also optimized, based on experimental results showing that the higher the interlayer delay the more stabilized the deposition process at S_5 which is due to the high thermal conductivity of Cu, this wait time provides more time for previous layers to solidify.

4.2.1.1 Optimisation of intersection porosity for GD25CU thin walls using Eckart Cu powder 15-45 μ m

The interlayer porosity is affected by various process parameters. In Figure ?? two samples of the same process parameters besides the LP_{end} , at higher LP_{end} keyholes appear due to high energy deposit. In Figure 4.9, the interlayer delay and the layer thickness (t_l) were increased, resulting in a better Cu bulk structure (see sample 697), however as the LP_{end} increased further (see sample 699), keyhole porosity appears again.

Figure 4.10 represents the optimization of S4-S5 intersection porosity in the GD25CU thin walls, where the micrograph on the right was the optimized thin wall by increasing the LP_{start} and decreasing the t_l . The S4-S5 bonding is still not good (present of a small percentage of porosity in the intersection).

It was discovered later in 4.3.1, that the intersection porosity in S4-S5 is a lack of bonding which is due to oxidation. A fact that couldn't be detected in prior cross-sectional analyses.

This revelation allows for a more in-depth understanding of the material's behavior. To address this, comprehensive documentation of the oxidation findings is required, followed by a rigorous study to ascertain the extent and reasons. Using this newfound knowledge, modifications can be made to process parameters to increase overall quality.

As discussed in 2.5.3 the oxidation in LDED may be caused by a lack of shielding. The Authors of [Svetlizky et al., 2022] insisted on the importance of controlling the oxygen content in the deposition chamber to a few ppm during the DED process, which can be done by optimizing the shielding gas flow rate.

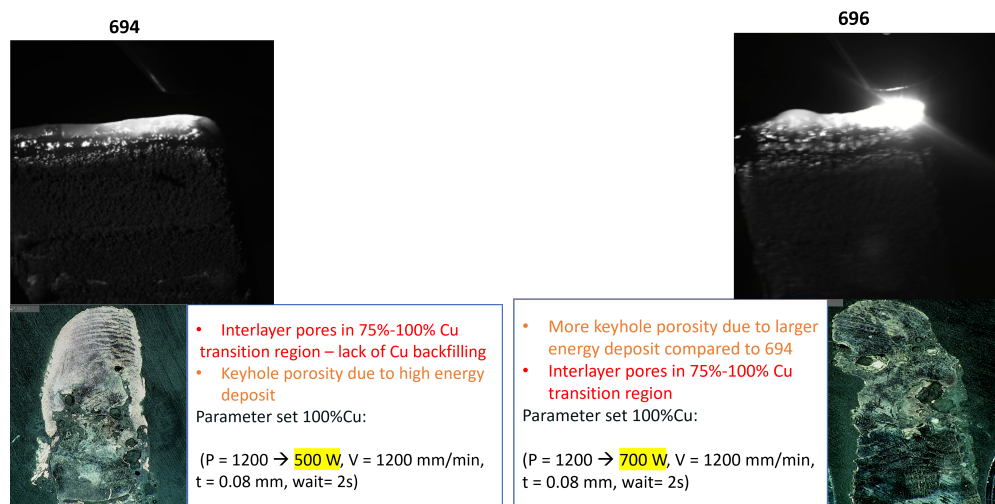
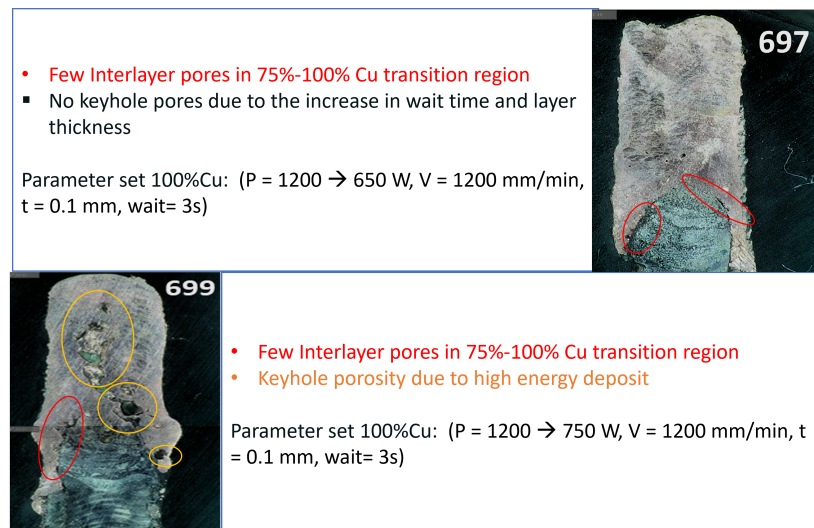
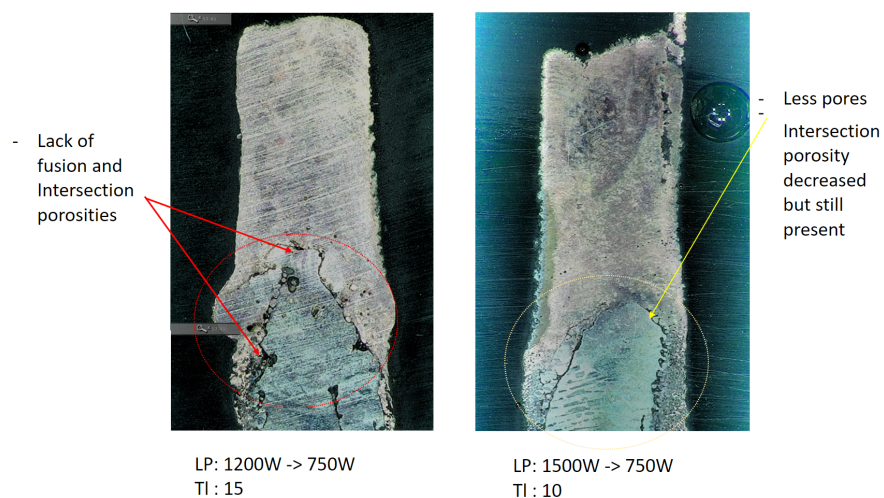
Figure 4.8: LP_{end} effect on porosities for GD25CuFigure 4.9: Optimisation of intersection Porosity by increasing t_l and t_{wait} 

Figure 4.10: Optimisation of intersection Porosity by decreasing transition layers and increasing LP

4.2.1.2 Optimisation of intersection porosity for GD25CU thin walls using Amnovis Cu powder 100 μ m

The optimization of intersection porosity continued after receiving a new Cu powder (Ammovis Cu powder 45-100 μ m), but after trying the L-DED of a similar GD25CU thin wall using the new powder, the results were not similar to what we got before. Figure 4.11 represents two GD25CU thin walls built using the same process parameters but with different Cu powders (different particle sizes and different suppliers), the lower wall (Ammovis Cu powder 45-100 μ m) reveals areas of notable porosity, indicating the lack of fusion pores and gas entrapment pores as well as keyholes. These flaws are frequently caused by improperly adjusted PFR, which results in ineffective material melting and bonding. For this issue, a new calibration was done to update the setpoints (see 3.2.2.3).

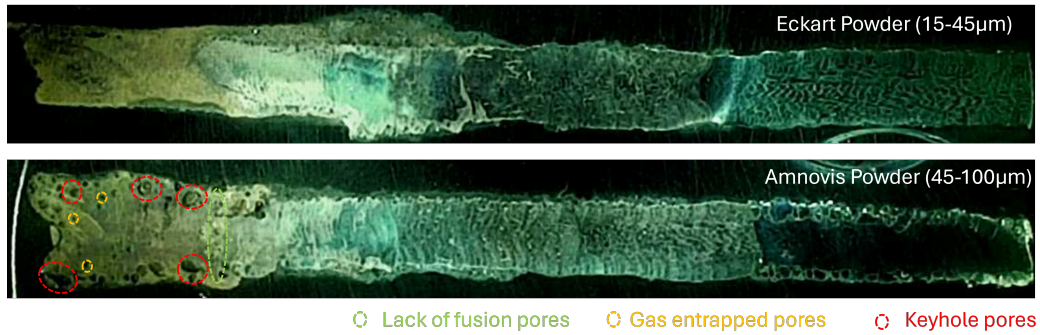


Figure 4.11: Comparison between GD25Cu thin walls using different Cu powder particles: upper wall (Eckart Cu powder 14-45 μ m), lower wall (Ammovis Cu powder 45-100 μ m)

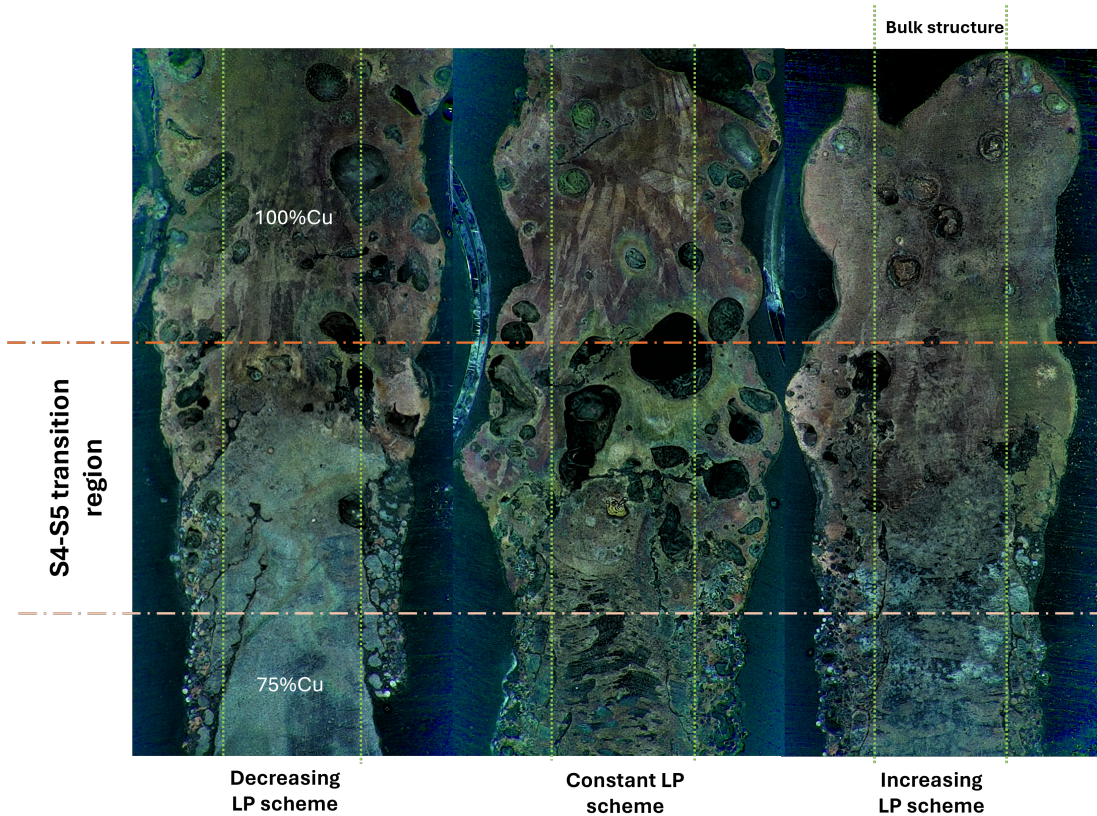


Figure 4.12: Optimisation of intersection Porosity by changing the LP scheme

Intersection porosity in the Cu-rich region was solved by a layer-wise increase in laser power and interlayer delay [Yadav et al., 2023]. Tests were performed to check if this method would be beneficial for decreasing the pores in Cu-rich regions. Figure 4.12 validates the findings of [Yadav et al., 2023], as one can notice that the increasing LP scheme has a smooth S4-S5 transition. However LP_{end} was too high causing keyholes.

4.2.1.3 Effect of shielding gas and carrier gas flow rates combination on oxidation

The process performance and material quality of L-DED are highly influenced by the selection of shielding gas and carrier gas flow rates. To get high-quality L-DED results with minimal oxidation, an effective combination of Argon as the shielding gas and an optimum carrier gas flow rate is required.

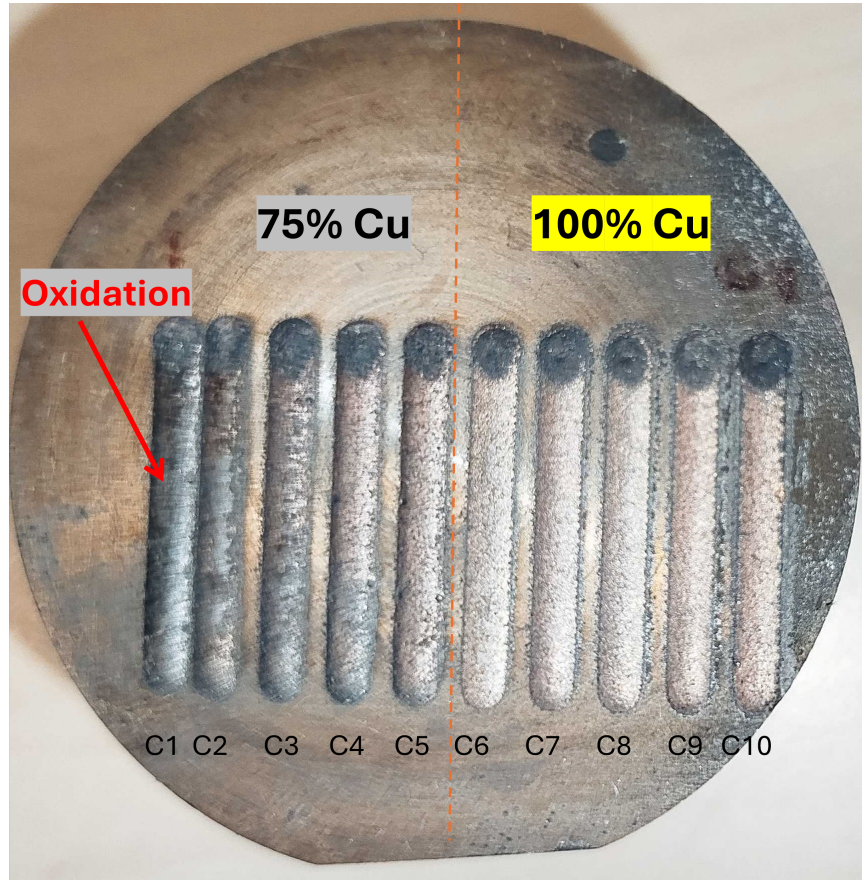


Figure 4.13: Optical micrographs of L-DED 75% Cu and 100% Cu single tracks deposited using different combinations of shielding gas and carrier gas flow rates

A test campaign was conducted to assess the best combination of carrier gas and shielding gas, depositing single tracks of 0.1 mm thickness at different CG-SG combinations for both Fe25Cu75 and Cu100 compositions, as shown in Table 4.4. Oxidation occurs when shielding gas is low (CG=8-SG=5), as shown in Figure 4.13 for 75%Cu, and it was reduced at (CG=8-SG=8,6).

Changing the SG-CG combinations had no effect on single tracks with 100% Cu.

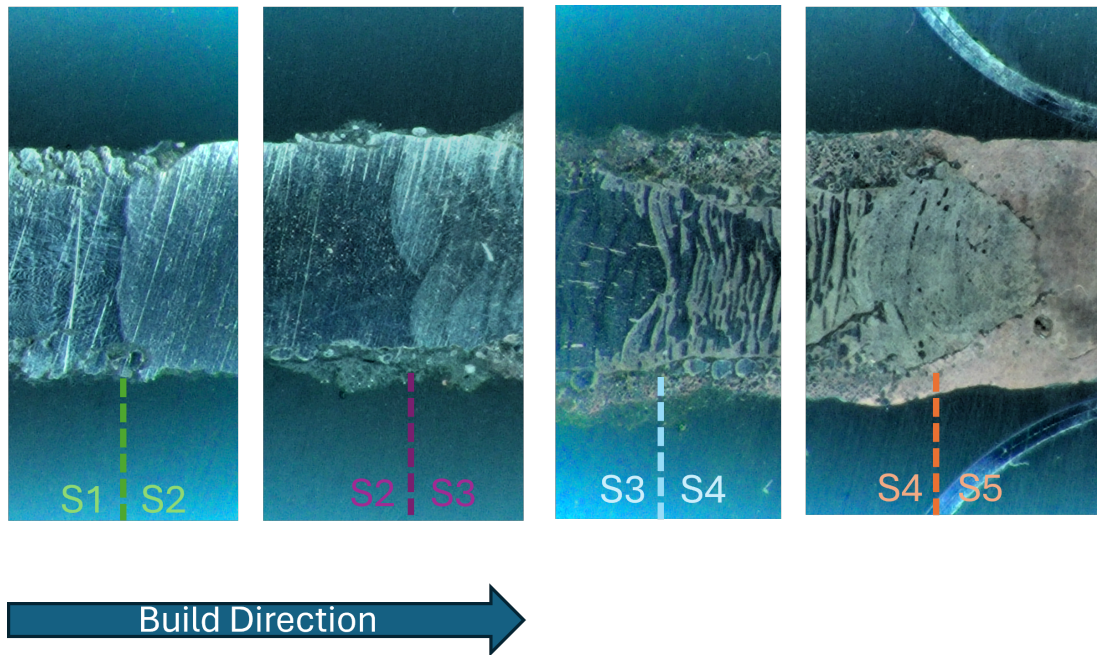
4.2.2 THE OPTIMAL PROCESSING PARAMETERS FOR THE GD25Cu GRADIENT

Table 4.4: Single tracks deposited using different combinations of shielding gas (SG) and carrier gas (CG) flow rates

#	CG (L/min)	SG (L/min)
75% Cu - 25% Fe		
C1	8	5
C2	6	5
C3	5	5
C4	8	8
C5	8	6
100% Cu		
C6	8	5
C7	6	5
C8	5	5
C9	8	8
C10	8	6

4.2.3 MICROSTRUCTURE ANALYSIS FOR GD25Cu THIN WALLS

Microstructure characterizations indicate good metallurgical bonding of individual layers of L-DED deposits and good intersection bonding from S1 to S4 as shown in Figure 4.14, mainly the sections were crack- and pore-free. Figure 4.2.3 represents the cross-sections of L-DED-built GD25Cu thin walls, where it was noticeable that the transition S4 to S5 contains impurities in the form of intersection porosity.

**Figure 4.14:** Representation of sections from 1 to 5 of a GD25Cu thin wall

4.2.4 COMPOSITIONAL VALIDATION OF IN-SITU POWDER MIXING THROUGH ELEMENTAL DISTRIBUTION IN AN LDED PROCESSED Cu-SS FGM

The EDS-based analysis is used to investigate how different Cu and Fe blended compositions affect elemental distribution in the LDED-built Fe-Cu FGM. Figure 4.17 depicts the elemental distribution of major elements such as Fe, Cu, Ni, Zr, C, Si, Mn, Mo, and Cr along the build direction of an LDED-built FGM. This analysis is a validation step for phase II, to check if the

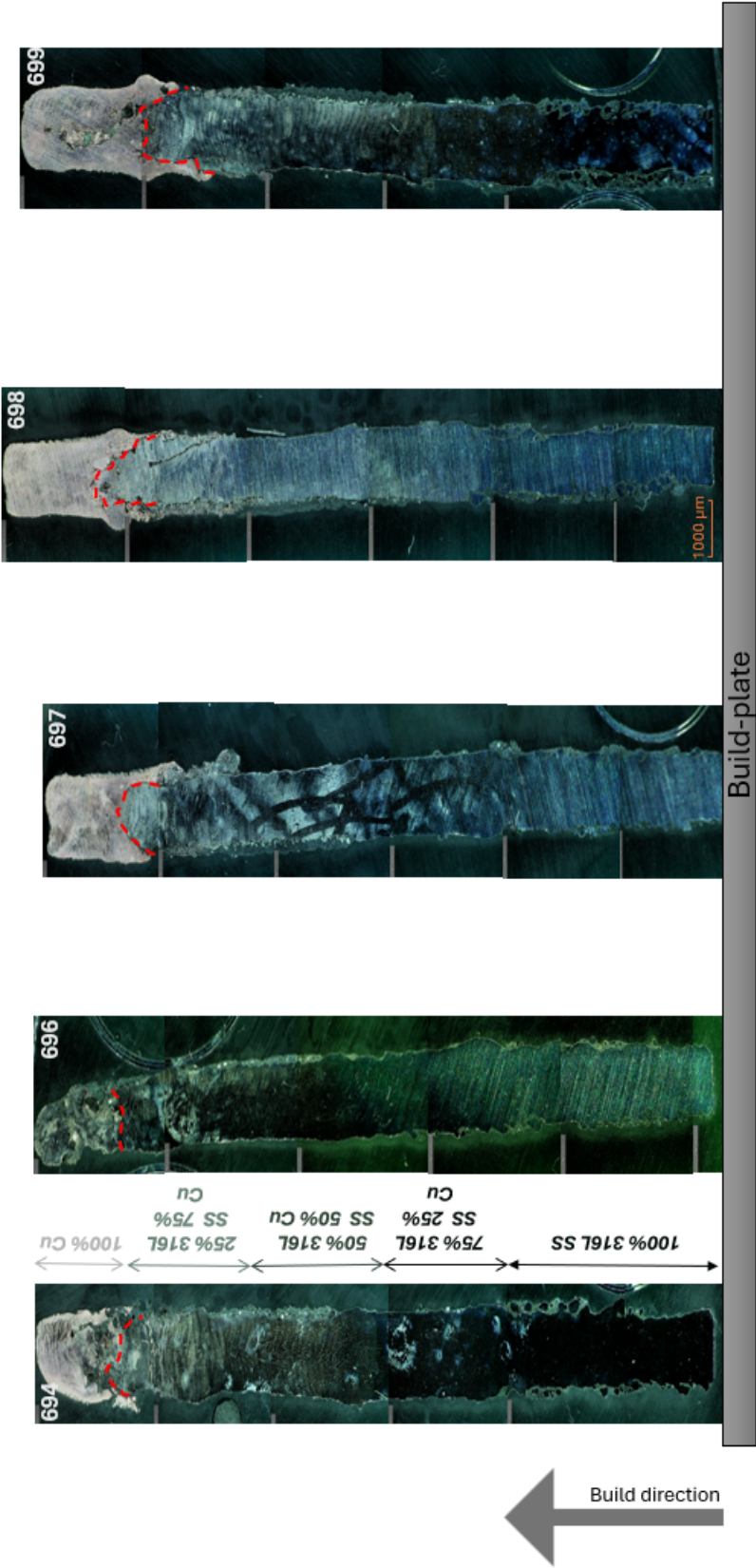


Figure 4.15: Cross-sectional representation of L-DED built GD25Cu thin walls

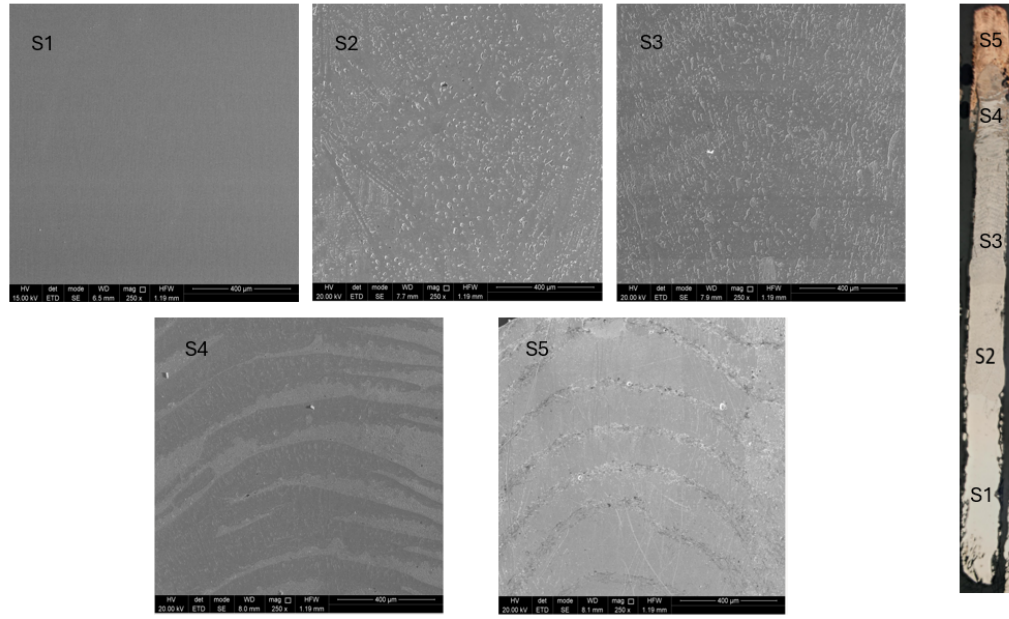


Figure 4.16: SEM of a cross-section of an L-DED built GD25Cu thin wall provided by **KU LEUVEN**

calibration of the powder feeders is correct and what is delivered by the feeders is the actual composition. Figure 4.17 shows a gradual transition in elemental distribution from Fe to Cu in LDED-built GD25Cu. The elemental concentrations of Fe and Cu vary significantly across the graded region of GD25Cu.

Table 4.5: Calculated base alloying element fractions for the different Fe-Cu blended compositions (wt.%)

Alloy	100-316L	25%Cu	50%Cu	75%Cu	100-CuCrZr
Fraction	0.00	0.25	0.50	0.75	1.00
SiK	0.24	0.18	0.12	0.06	0.00
ZrL	1.45	1.51	1.58	1.65	1.72
MoL	1.26	0.95	0.63	0.32	0.00
CrK	17.78	13.69	9.59	5.50	1.41
MnK	1.00	0.75	0.50	0.25	0.00
FeK	66.18	49.64	33.09	16.55	0.00
NiK	12.09	9.07	6.04	3.02	0.00
CuK	0.00	24.22	48.44	72.66	96.88

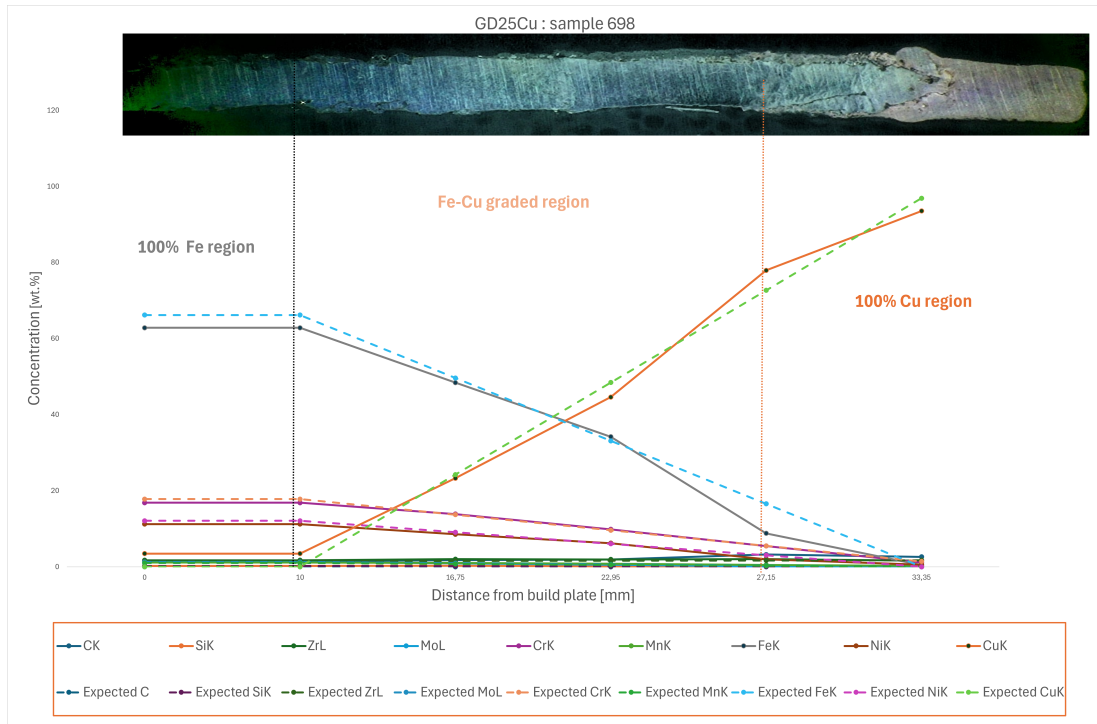
Since the Cu-Fe system is poorly soluble in liquid and solidifies quickly, the non-uniform and random phase-separation Cu-rich and Fe-rich zones can be blamed for this macro-segregation of Cu and Fe.

Another EDS analysis was performed to validate the composition of an inverted thin wall as illustrated in Figure 4.18,

The regions are separated into GD25Fe (15-35 mm) and GD25Cu (0-15 mm), with a clear transition zone at 15 mm. In the GD25Cu region, Cu shows a high weight percentage; in the GD25Fe region, this weight percentage sharply declines and approaches the predicted value of 0%. On the other hand, Fe exhibits a lower percentage in the GD25Cu zone and a higher percentage in the GD25Fe region, ultimately reaching approximately 60%.

Table 4.6: Elemental composition for different Fe-Cu blended compositions (wt.%) for sample 698

Element	100%Fe	75%Fe	50%Fe	25%Fe	100%Cu
SiK	0.23	0.25	0.09	0.06	0.00
ZrL	1.70	2.01	1.92	2.03	1.58
MoL	1.22	0.91	0.55	0.07	0.00
CrK	16.84	13.85	9.83	5.46	1.12
MnK	1.11	1.01	0.77	0.50	0.23
FeK	62.81	48.41	34.17	8.79	0.41
NiK	11.21	8.56	6.20	1.95	0.55
CuK	3.45	23.27	44.60	77.91	93.51

**Figure 4.17:** Compositional analysis for a LDED built GD25Cu thin wall (sample 698)

Additional elements such as Si, Zr, Mo, Cr, Mn, and Ni exhibit different patterns, suggesting diffusion or segregation throughout the transition region. These conclusions are corroborated by the metallographic image, which shows microstructural alterations that match the compositional gradients shown in the EDS data.

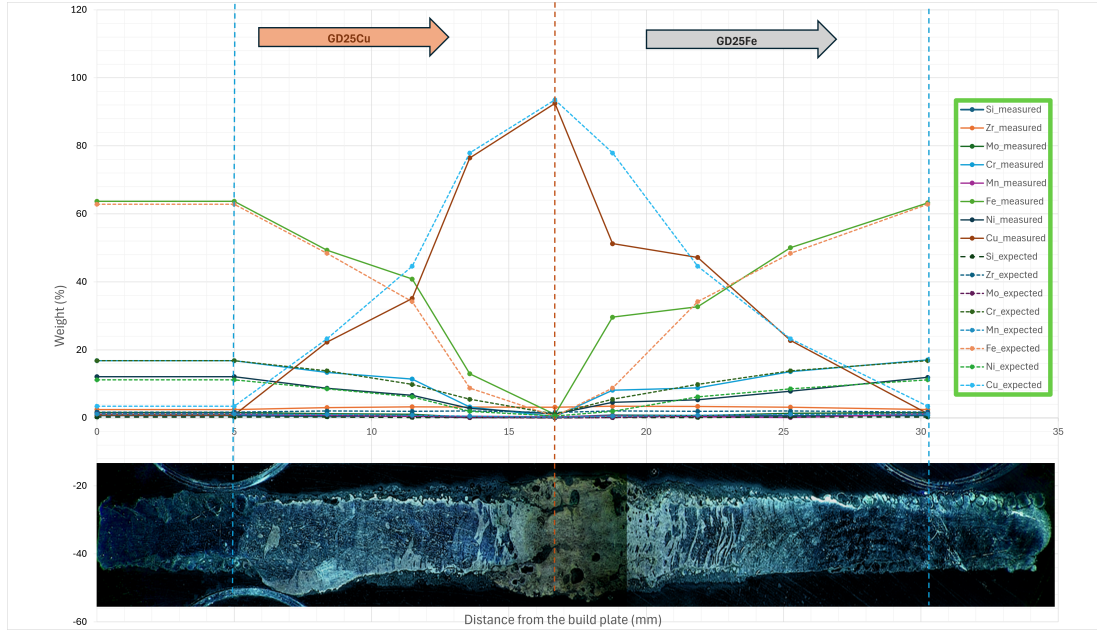


Figure 4.18: Compositional analysis for a LDED built Fe-Cu-Fe inverted thin wall (sample 744)

4.2.5 MICROHARDNESS ACROSS THE GD25Cu GRADIENT

Figure 4.19 shows a plot of the measured micro-hardness fluctuations of the Fe-Cu FGM bulk structures produced by L-DED along the grading direction. The micro-hardness value in bulk GD25Cu exhibits a smooth transition from Fe to Cu; in the Fe, graded, and Cu regions, it varies between 172 HV - 167 HV, 158 HV - 149 HV, and 60 HV, respectively. The micro-hardness plot shows how the gradient in hardness value decreases along the build direction as the proportion of Cu composition increases.

Table 4.7 summarises the hardness measurement for the different samples represented in Figure 4.19. An increase in LP_{start} for 100% Fe results in an increase of hardness of 20 HV (comparison between 717 and 716). Hardness in S5 is not affected significantly by the variation of LP, still, as the LP increases the hardness decreases slightly in 100% Cu.

To validate the results from the hardness test, a comparison of results was performed between our results and those of Yadav et al., 2023 illustrated in Figure 4.20. The difference in hardness for the 100% Fe section is mainly because in Yadav et al., 2023 the LP used in this section is 1000 W, which confirms that as the LP increases in the Fe-rich region the hardness increases as well. Meanwhile, starting from 50% Cu one can notice that the hardness of sample 717 was higher than the one of Yadav et al., 2023. Finally, at 100% Cu the LP used in sample 717 was 1500 W meanwhile in Yadav et al., 2023 they used 800 W which results in higher hardness compared to sample 717, which is a direct indication that in Cu-rich regions the hardness is higher as the LP is lower.

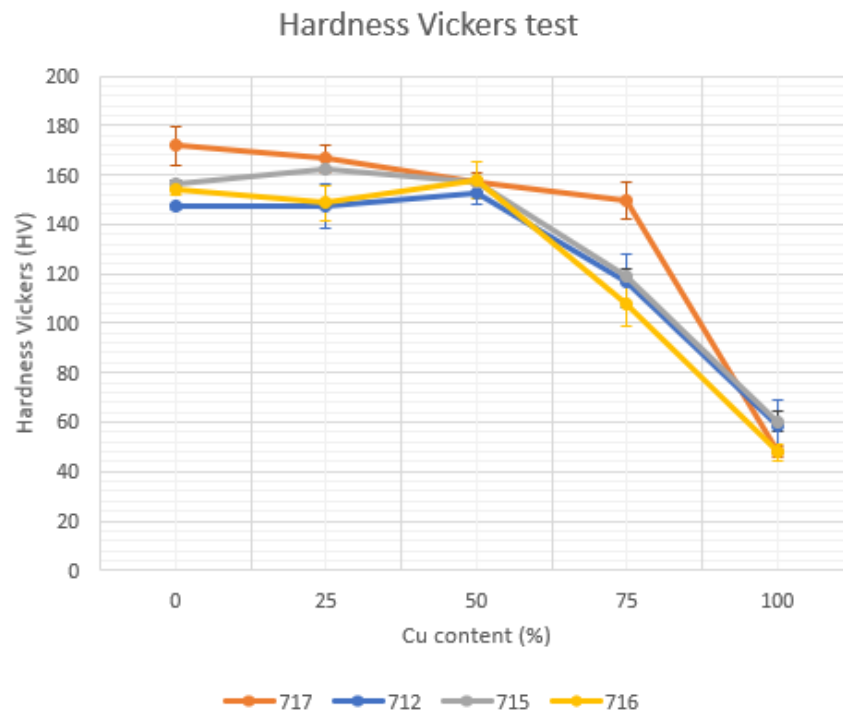


Figure 4.19: Hardness Vickers Test for GD25Cu thin walls

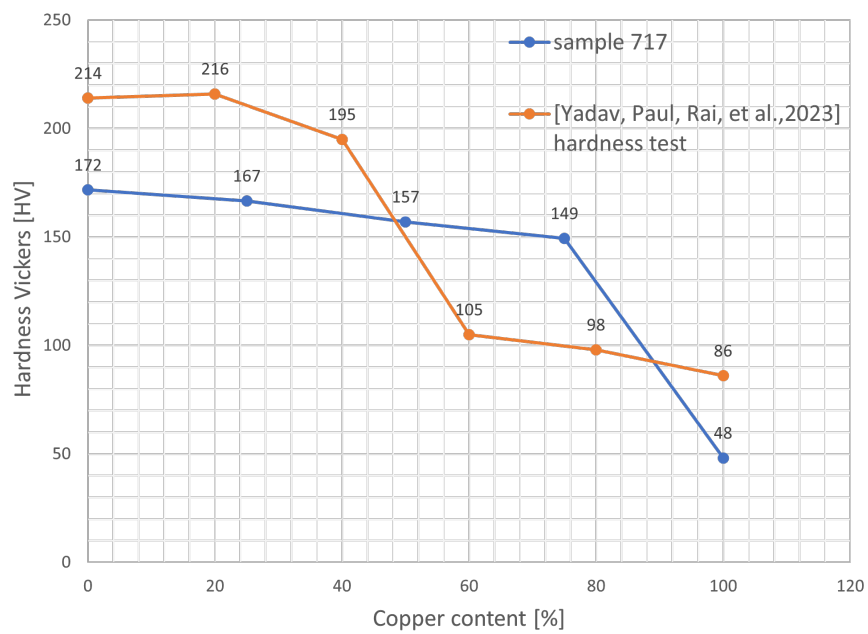


Figure 4.20: Comparison of Hardness Vickers results of GD25Cu built-in AM lab and GD20Cu of [Yadav et al., 2023]

Sample #	Cu Content [%]	LP _{start} [W]	v _{scan} [mm/min]	#1	#2	#3	Average [HV]
717	0	600	300	171	180	164	172
	25	800	450	173	163	164	167
	50	1000	375	159	153	159	157
	75	1000	500	149	157	142	149
	100	1500	750	45.9	48	50.5	48
712	0	350	300	147	148	146	147
	25	800	450	149	138	155	147
	50	1000	375	150	157	150	152
	75	1000	500	126	105	120	117
	100	1200	750	70.9	52.5	51.6	58
715	0	400	300	154	157	157	156
	25	800	450	162	162	162	162
	50	1000	375	158	158	156	157
	75	1000	500	121	121	116	119
	100	1300	750	64.9	58	57.9	60
716	0	400	300	152	156	154	154
	25	800	450	155	141	150	149
	50	1000	375	150	158	165	158
	75	1000	500	116	108	98.5	108
	100	1400	750	49	43.9	50.6	48

Table 4.7: Hardness values for different thin walls with changing LP in S1 and S5

4.3 FABRICATION OF A THREE-DIMENSIONAL FGM DEMONSTRATOR COMPONENT

4.3.1 TENSILE SAMPLES GD25Cu

The first step to fabricate the tensile test coupons is to print a Fe-Cu-Fe FGM "inverted thin wall" (see Figure 4.21) which is a construction with Fe concentrated on the outside and Cu concentrated in the center, with a gradual transition between the two materials. This design takes advantage of the capabilities of both Fe and Cu, merging their different features into a single structure. The production of an inverted thin wall FGM utilizing L-DED entails multiple processes. A comprehensive G-code of the thin-walled structure is constructed, outlining the gradual shift from Fe on the outside to Cu in the core. The MiCLAD machine is then set up and calibrated using Fe and Cu process parameters selected previously for a simple GD25Cu thin wall.

Several important steps are required to transition from printing an inverted thin wall to printing a tensile test coupon. First, a thorough G-code to print 3D model of the tensile test coupon is created, ensuring that it fits standard testing requirements such as those outlined in ASTM standards. This model covers the coupon's size and shape, as well as the gradient transition from Fe to Cu, which specifies how the material composition varies from the edges (Fe) to the center.

Selecting the hatching distance which is the gap between successive laser passes during the deposition is a crucial step in the printing process. To produce the appropriate microstructure and mechanical qualities, as well as to guarantee the best possible bonding between layers, the hatching distance needs to be carefully selected. A hatching distance that is too small might result in excessive heat accumulation and residual strains, whereas a hatching distance that is too wide can cause weak bonding and porosity. To maintain uniform quality throughout the gradient, the hatching distance for the Fe-Cu gradient is modified in response to changes in the composition of the material.

Furthermore, the inherent quick cooling rates in LDED contribute to grain structure refine-

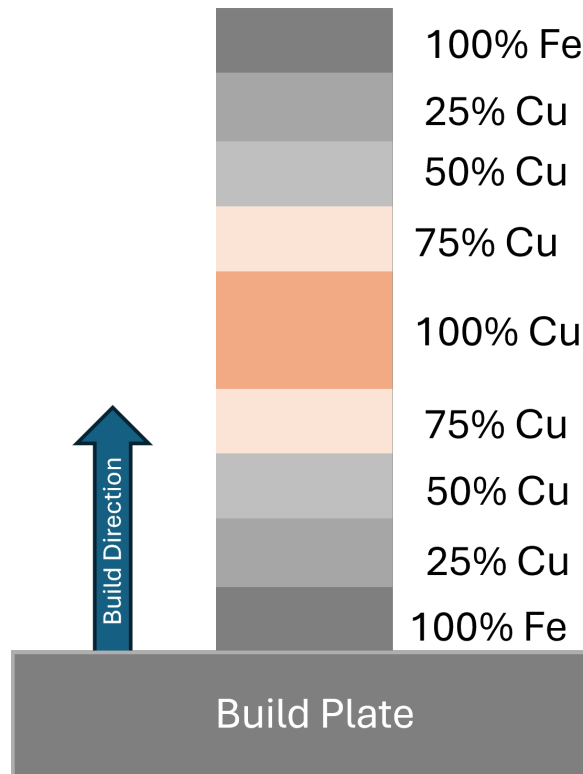


Figure 4.21: Representation of Fe-Cu-Fe inverted thin wall

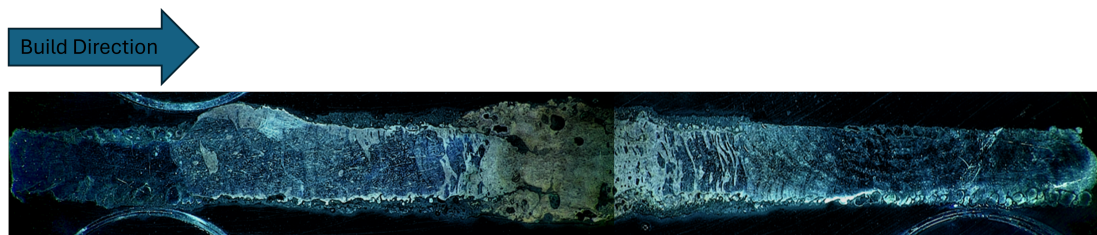


Figure 4.22: L-DED built Fe-Cu-Fe FGM inverted thin wall

ment, which improves the alloy's tensile performance. These characteristics make GD25Cu tensile samples generated by LDED appropriate for high-performance applications requiring outstanding mechanical qualities.

Figure 4.23 presents the tensile test coupons built using the thin walls optimal process parameters, they revealed important information that we couldn't get using only cross-sectional micrograph analysis, which is the oxidation in the transition S4-S5. This oxidation is due to high energy deposits during the deposition of the first layers, which produce keyholes that enable oxygen to enter the melt pool while printing. A possible solution was to investigate the effect of the shielding gas and carrier gas combinations on the oxidation of single tracks as already discussed in 4.2.1.3.

Unfortunately, it was not possible to run tensile tests since the bonding in S4-S5 was so weak and it should be optimized before trying L-DED of new tensile test coupons.

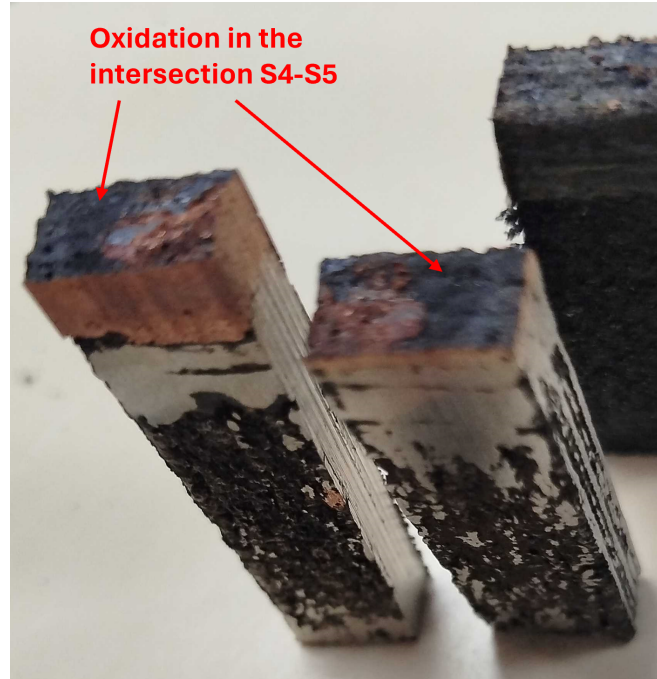


Figure 4.23: Oxidation in the S4-S5 intersection region of a Fe-Cu FGM tensile test coupon

4.3.2 THE GD1Cu DEMONSTRATOR

Printing a 1% Fe-Cu graded cylinder with L-DED requires a precise procedure of gradually transitioning the composition from 100% Fe at the base to a final mixture including 100% Cu with a gradient percentage of 1%. The printing process begins with the application of 100% Fe powder to create the initial layers. As the build develops, the Cu feeder delivers incrementally Cu into the Fe matrix at the same time Fe feeder decreases the powder flow rate respectively to achieve a fine gradient as represented in 4.24.

Figure 4.25 illustrates the change of feeder setpoints as the copper content increases, this data is based on the calibration measurements done in 3.2.2.4. The transition is closely monitored to guarantee uniform distribution, culminating in a top layer composition of 100% Cu.

Throughout the process, parameters such as laser power, scanning speed, and powder feed rates are constantly modified to maintain a stable melt pool and achieve the necessary compositional gradient. Post-processing processes as surface finishing, are used to improve the metallurgical properties of the graded cylinder and attain its final dimensions and surface quality.

Figure 4.27 represents the GD1Cu demonstrator after surface finishing and a cross-section of the cylinder to check the bulk structure quality. Figures 4.28 and 4.29 represent microscopic images for the bulk structure of the demonstrator, which reveals the gradual shift from 100% Fe to 100% Cu without any intersection porosities. The GD1Cu approach was the key to being able to L-DED Fe to Cu smoothly and cancel the defects. On the other hand, there were unexpected cracks in the demonstrator as shown in Figure 4.30, which should be improved in future work.

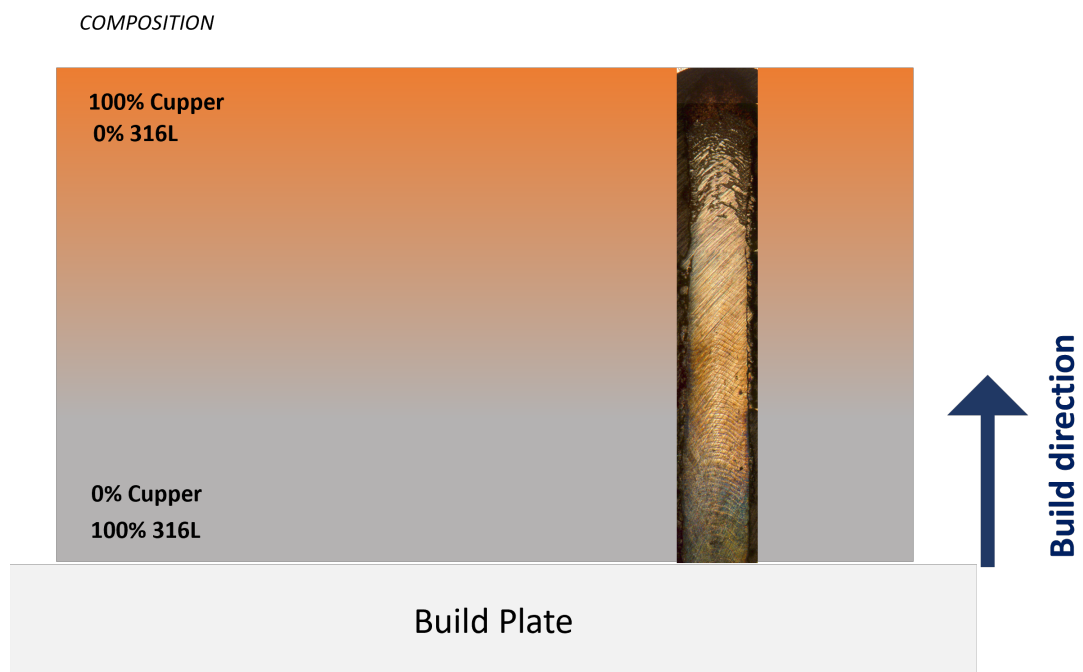


Figure 4.24: Representation of GD1Cu for smooth transition between Fe and Cu

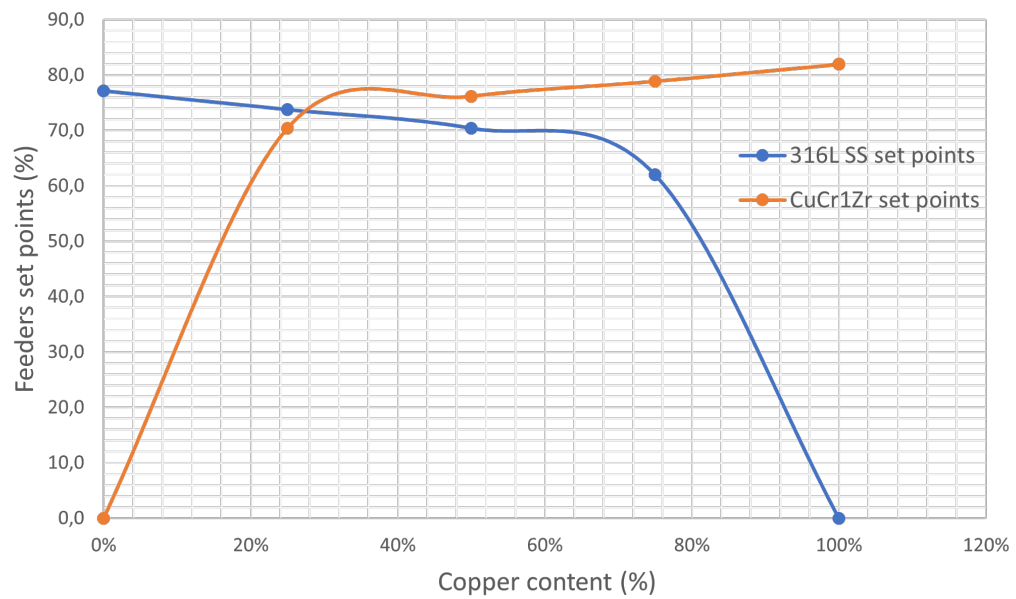


Figure 4.25: Feeders setpoints curve for L-DED of GD1Cu demonstrator

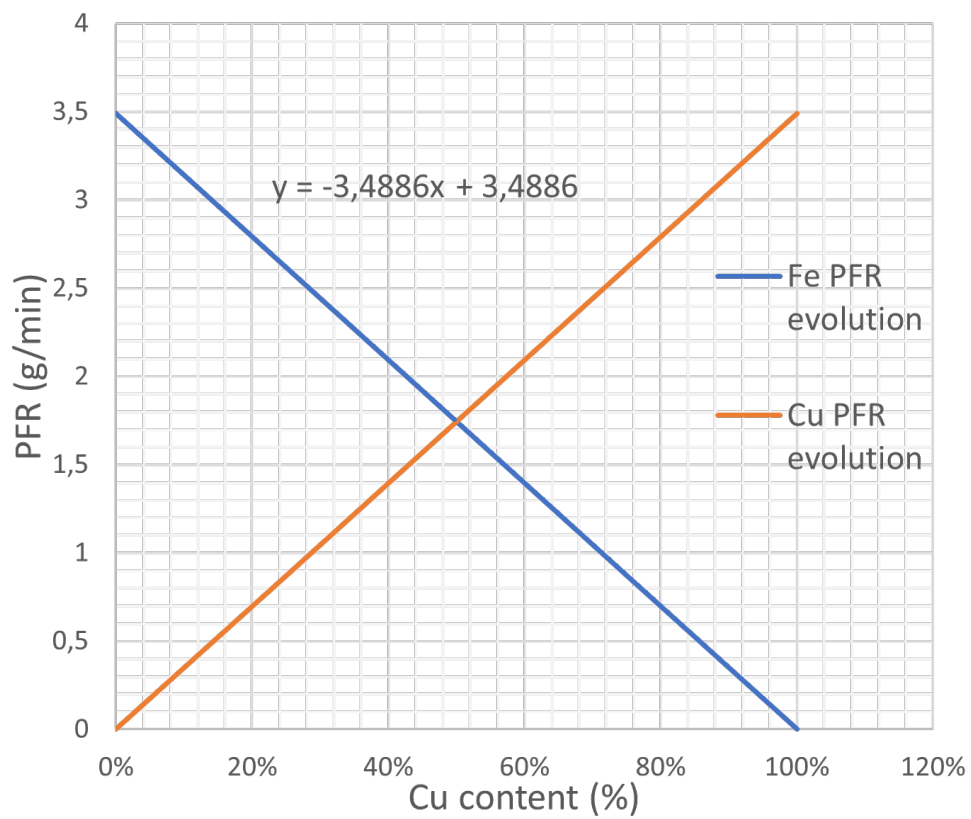


Figure 4.26: Powders evolution curve for L-DED of GD1Cu demonstrator



Figure 4.27: L-DED built GD1Cu 3D demonstrator

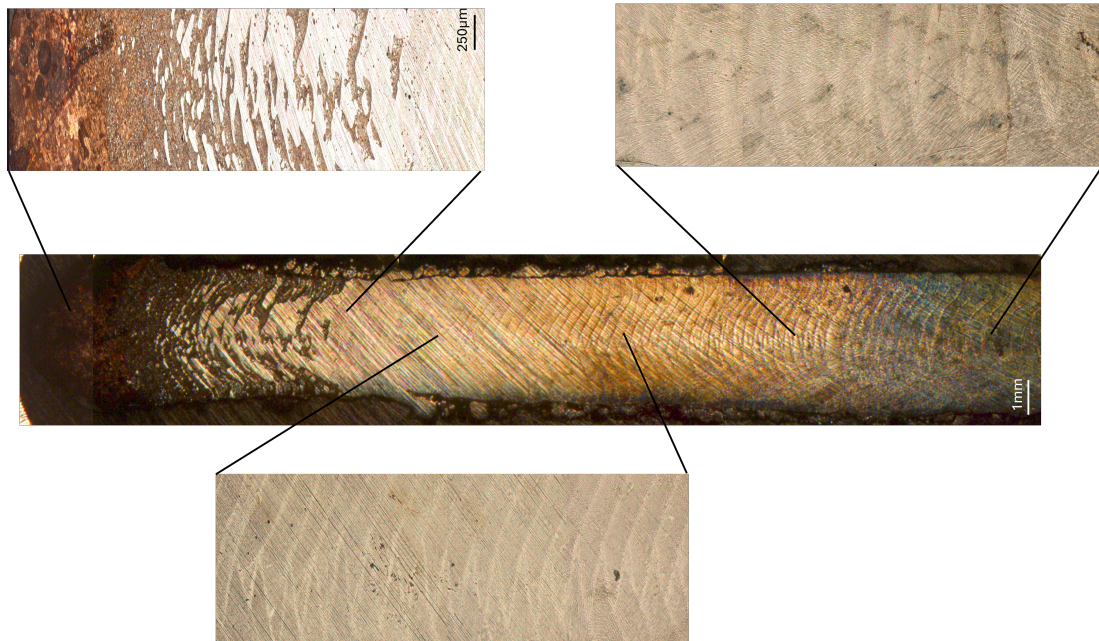


Figure 4.28: Microscopic images for the bulk structure of the GD1Cu demonstrator with a zoom in the build direction, done in **SURF Department**

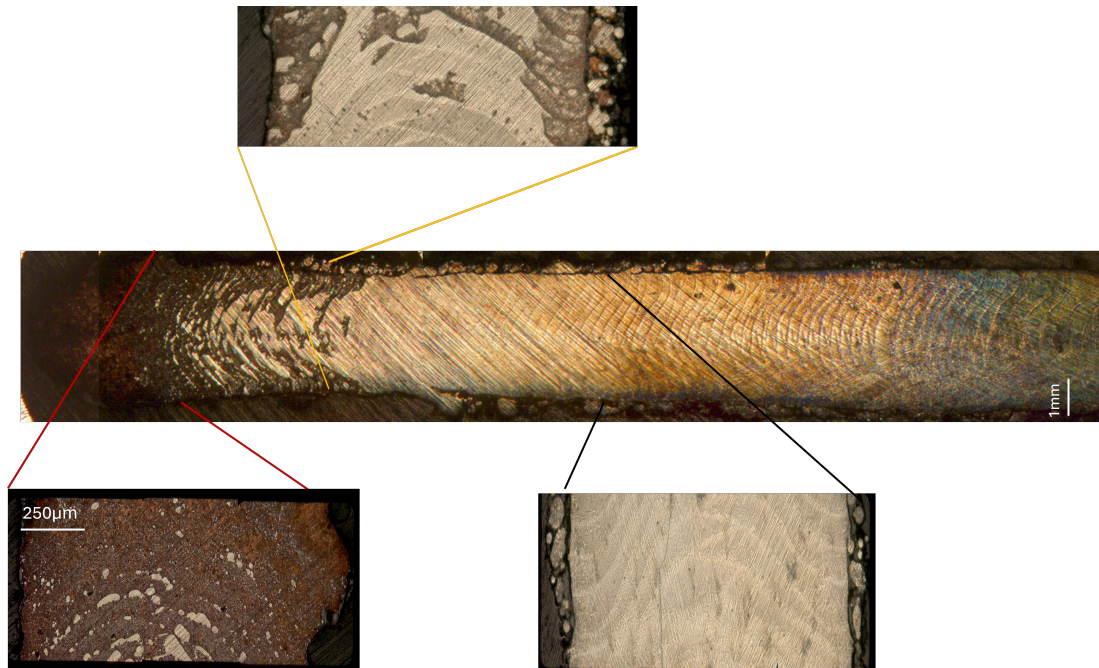


Figure 4.29: Microscopic images for the bulk structure of the GD1Cu demonstrator with a zoom along sections

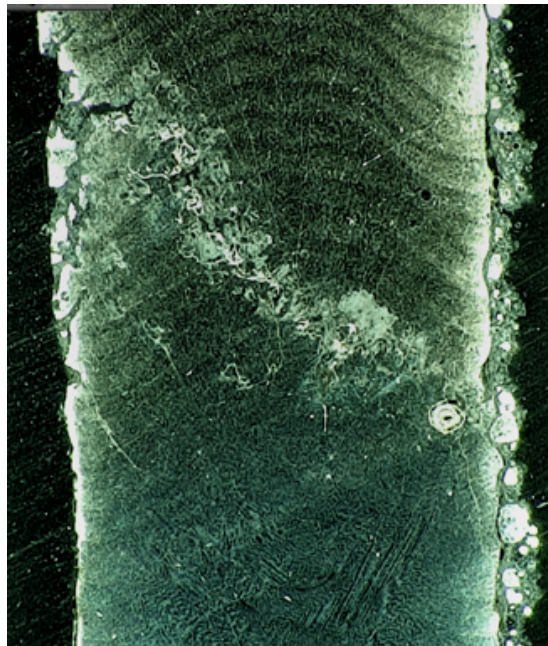


Figure 4.30: Microscopic image of a crack in the GD1Cu demonstrator

This chapter discusses the significant findings from the experimental results on the L-DED processing of homogeneous thin walls with various Fe-Cu pre-blended compositions. The discussion addresses the optimization of process parameters, the impact of build plate pre-heating on crack formation, and the microstructural analysis of the produced samples.

5.1 OPTIMIZATION OF PROCESS PARAMETERS

The initial experiments focused on determining the optimal L-DED process parameters for different Fe-Cu pre-blended compositions. By systematically varying the laser power and scan speed, it was possible to identify suitable conditions for each composition. The results, summarized in Table 4.1, indicate that each composition required a specific set of parameters to achieve high-quality thin walls.

For $Fe_{75}Cu_{25}$, a range of laser powers (800-1000 W) and scan speeds (900 mm/min) were tested. Similarly, $Fe_{50}Cu_{50}$ and $Fe_{25}Cu_{75}$ compositions required higher laser powers (up to 1200 W) with consistent scan speeds. Pure copper (Cu_{100}) demanded the highest laser power settings (up to 1200 W) and faster scan speeds with increased interlay wait time. These variations highlight the need for tailored process parameters to accommodate the differing thermal and material properties of each blend.

5.2 EFFECT OF BUILD PLATE PRE-HEATING ON CRACK FORMATION

The introduction of build plate pre-heating proved crucial in mitigating crack formation in the samples, particularly for the $Fe_{75}Cu_{25}$ composition. Figure 4.1 illustrates that samples built without pre-heating exhibited significant cracks, whereas pre-heated samples remained crack-free. Pre-heating minimizes thermal gradients, thereby reducing thermal stresses and the likelihood of cracking. This finding aligns with existing literature, such as M. Liu et al., 2021, which underscores the importance of pre-heating in additive manufacturing processes to enhance part integrity.

5.3 MICROSTRUCTURAL ANALYSIS

Microstructural analysis, depicted in Figure 4.1 and further detailed in the microscopic images, reveals the evolution of microstructures across different Fe-Cu compositions. For instance, the $Fe_{75}Cu_{25}$ composition showed a homogeneous distribution of phases, whereas higher copper content compositions ($Fe_{25}Cu_{75}$ and Cu_{100}) exhibited more pronounced phase separation and grain boundary formation.

These observations suggest that the copper content significantly influences the microstructure. The microstructural characteristics directly impact the mechanical properties of the deposits, as homogeneity and grain size are critical factors in determining strength and ductility.

5.3.1 MICROHARDNESS ACROSS THE GD25Cu GRADIENT

The microhardness variations along the GD25Cu gradient, as depicted in Figure 4.19, demonstrate a clear and gradual transition from the Fe-rich region to the Cu-rich region. In the Fe region, the hardness values range from 172 HV to 167 HV, while in the graded region, the hardness fluctuates between 158 HV and 149 HV, eventually reaching 60 HV in the Cu region. This trend indicates a smooth decrease in hardness corresponding to the increasing proportion of Cu along the build direction.

The summary of hardness measurements for various samples, as presented in Table 4.7, reveals insightful trends related to the process parameters. Notably, an increase in the laser power (LP_{start}) for 100% Fe results in a hardness increase of 20 HV (comparison between samples 717 and 716). However, the hardness in the Cu region (100% Cu) tends to decrease slightly with an increase in laser power, suggesting an inverse relationship between laser power and hardness in Cu-rich regions.

To further substantiate these findings, a comparison with the results reported by Yadav et al., 2023 was conducted, as illustrated in Figure 4.20. The notable difference in hardness in the 100% Fe section is attributed to the higher laser power (1000 W) used in Yadav et al., 2023, which corroborates the trend that increased laser power enhances hardness in the Fe-rich region. Conversely, in the Cu-rich region, the lower laser power (800 W) used in Yadav et al., 2023 results in higher hardness compared to our sample 717, which used a laser power of 1500 W. This confirms that in Cu-rich regions, the hardness is inversely proportional to the laser power.

Overall, the microhardness measurements and comparisons indicate that both material composition and laser processing parameters significantly influence the hardness profile of the GD25Cu gradient. These insights are crucial for optimizing the manufacturing process to achieve desired mechanical properties in functionally graded materials.

Conclusions

This research provides a comprehensive understanding of the Laser Directed Energy Deposition (L-DED) processing of Fe-Cu pre-blended compositions, emphasizing the critical role of optimizing process parameters to achieve defect-free builds. Pre-heating the build plate effectively prevents cracking and ensures structural integrity. Microstructural analysis reveals important details about phase distribution and grain structure, which are key to modifying the mechanical properties of the final components. These findings support the development of Fe-Cu functionally graded materials (FGMs) in advanced manufacturing.

In conclusion, the thesis highlights the significant interplay between process parameters, material composition, microstructure, and mechanical properties in L-DED manufacturing of FGMs. Fine-tuning laser power and scan speed for each composition is essential for producing high-quality components with tailored mechanical properties. Pre-heating minimizes thermal gradients, reducing cracks and enhancing part integrity.

Microstructural analysis shows phase separation and grain boundary formation, emphasizing the need to control these processes for desired material properties. Microhardness measurements across the GD25Cu gradient illustrate the impact of composition and laser processing on mechanical properties, providing guidelines for optimizing hardness profiles.

Overall, this thesis advances the understanding of process-material-property relationships in L-DED-produced FGMs, paving the way for further research and development in additive manufacturing techniques to produce innovative and high-performance functional materials.

Future work in this research subject holds promising avenues for further exploration and advancement. The following areas represent potential directions for future investigation:

- **Optimization of Process Parameters:** Continued refinement of process parameters, including laser power, scan speed, and powder feed rates, can further enhance the quality and efficiency of L-DED manufacturing. Exploring advanced control strategies, such as closed-loop feedback systems, could enable real-time adjustment of parameters to adapt to variations in material properties and printing conditions.
- **Advanced Thermal Management Techniques:** Investigating novel thermal management techniques, such as localized heating or active cooling systems, could offer improved control over temperature gradients and thermal stresses during printing. These techniques have the potential to minimize defects, enhance part quality, and expand the range of printable materials and geometries.
- **Microstructural Control and Tailoring:** A deeper exploration into the mechanisms governing microstructural evolution in FGMs could enable precise control and tailoring of microstructures to achieve specific mechanical properties. Techniques such as in-situ monitoring of microstructural evolution and advanced post-processing treatments could be explored to optimize material properties and performance. Additionally, Fe-Cu mixing would be enhanced using Ultrasonic-Assisted LDED.
- **Characterization of Mechanical Properties:** Comprehensive characterization of mechanical properties, including tensile, compressive, and fatigue behavior, is essential for validating the performance of L-DED-produced FGMs. Future research could focus on conducting detailed mechanical testing under various loading conditions to assess the strength, ductility, and durability of printed components.
- **Applications and Industrial Implementation:** Exploring practical applications and industrial implementation of L-DED-produced FGMs represents a crucial next step toward realizing the full potential of this technology. Collaborations with industry partners and end-users can provide valuable insights into specific application requirements and facilitate the transition of research findings into real-world applications.

Overall, future work in this field should aim to address the remaining challenges and limitations of L-DED manufacturing while unlocking new opportunities for innovation and application across diverse industries. By continuing to push the boundaries of knowledge and technology, researchers can contribute to the ongoing evolution of additive manufacturing and its transformative impact on modern manufacturing processes.

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