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Parametrization study for 316L additive manufacturing using Direct Energy Deposition (DED)

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Mestrado Integrado em Engenharia Mecânica

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October 22, 2020

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Abstract

Direct Energy Deposition (DED) is an Additive Manufacturing (AM) process where a high power energy source is used to scan and locally melt a substrate onto which metal powder is blown and consolidated into. This process is repeated layer by layer creating solid and dense near net shape metallic parts with a high deposition rate, being suitable for the manufacturing of large, complex geometries.

The DED process is currently limited by the difficulty in understanding how the processing parameters affect the end product's properties and dimensional accuracy, and as such this dissertation work attempts to break down the phenomena at play to better understand the underlying relationships that exist in DED and improve this promising process.

Several preliminary single line clads were deposited with 316L stainless steel in order to find a working set of parameters for 3D depositions, from which tensile and microhardness specimens were machined and tested.

The developed set of processing parameters yielded near net shape parts with mechanical properties surpassing those of wrought 316L.

Resumo

Direct Energy Deposition (DED) é um processo de fabrico aditivo onde uma fonte de energia de elevada potência varre um substrato, fundindo-o localmente. Um jato de pós metálicos é direccionado para esse banho de fusão onde as partículas fundem e ficam depositadas. Este varrimento é repetido camada a camada para criar objectos tridimensionais densos e *near net shape* (próximos da forma final), tendo capacidade para gerar geometrias complexas com facilidade e peças de grandes dimensões tirando partido das elevadas taxas de deposição possíveis.

Actualmente, o processo DED está limitado pela falta de conhecimento relativamente aos efeitos concretos no produto final de cada um dos seus parâmetros de processamento, sendo que este trabalho visa desconstruir os processos físicos em jogo no DED para melhor o controlar.

Vários ensaios preliminares foram executados, com a deposição de linhas únicas de aço inóx 316L fazendo variar os parâmetros de processo entre elas, sendo que de seguida o conjunto de parâmetros mais promissor foi usado para depositar paredes tridimensionais de onde foram retirados provetes para análise metalográfica, ensaios de tração e de dureza. Foi ainda utilizado um software de controlo de malha fechada como forma de melhorar a consistência e qualidade do material depositado.

O resultado final foi um conjunto de parâmetros que, juntamente com a técnica de controlo de malha fechada utilizada, deu origem a peças densas, *near net shape*, com propriedades mecânicas superiores às do 316L convencional.

Acknowledgements

I would like to start by thanking INEGI for the opportunity to develop this project in an industry leading environment, learning about and developing cutting edge technologies. I'd like to thank Dr. Isaac Ferreira in particular for his support as my co-supervisor, always available to help with honest and valuable advice.

To Prof. Dr. Rui Jorge Neto, as my supervisor for this dissertation and teacher of numerous classes during the last 5 years studying at FEUP, I express my deepest gratitude for sparking my passion for these manufacturing technologies and for guiding this dissertation.

I'd also like to acknowledge Dr. André Ferreira for his valuable contributions to this work.

To my parents and my brother, who deserve all the appreciation I can give for the support they have given me before and during these difficult times.

Finally, I'd like to thank the friends who I have shared these great years with. Without them this adventure would have lost meaning, and I certainly wouldn't have remained sane during quarantine.

This work was elaborated within the scope of the TOOLING4G project. Project No. POCI-01-0247-FEDER-024516 financed by Programa Operacional Competitividade e Internacionalização e Programa Operacional Regional de Lisboa, PORTUGAL 2020, through the Fundo Europeu de Desenvolvimento Regional (FEDER).

Pedro Manuel Sousa

“All we have to decide is what to do with the time that is given us.”

J.R.R. Tolkien

Contents

1	Introduction	1
1.1	Objectives	2
1.2	Document structure	3
1.3	Work plan	4
2	State of the art	5
2.1	Additive Manufacturing	5
2.2	Direct Energy Deposition	9
2.2.1	Laser properties	10
2.2.2	Powders and powder feed system	12
2.3	Selective Laser Melting	15
2.3.1	Laser characteristics	16
2.3.2	Metallic Powders for SLM	17
2.4	Microstructure in metal AM	19
2.5	Printing Parameters for DED	22
2.5.1	Clad geometry and properties prediction	25
2.6	DED process control	27
3	Experimental	29
3.1	Materials and Methods	29
3.1.1	Materials	29
3.1.2	DED Setup	30
3.1.3	Optical Microscopy	34
3.1.4	Tensile tests	34
3.1.5	Microhardness Measurements	36
3.2	Experimental trials	37
3.2.1	Single line depositions	37
3.2.2	3D Depositions	43
3.2.3	Microstructure analysis	54
3.2.4	Microtensile specimens and tests	57
3.2.5	Microhardness measurements	61
4	Conclusions	63
4.1	Goals	63
4.2	Future work	64
5	Appendix	73

List of Figures

1.1	Direct Energy Deposition process with a coaxial nozzle [4]	2
1.2	Dissertation work plan in the form of a Gantt chart	4
2.1	FDM process schematic and part example	6
(a)	FDM process schematic [10]	6
(b)	FDM built nylon part [11]	6
2.2	SLA process schematic and part example, note the absence of visible layer lines	6
(a)	SLA process schematic [12]	6
(b)	SLA built part [13]	6
2.3	Binder jetting process schematic and part example	7
(a)	Binder Jetting process schematic [12]	7
(b)	Binder Jetting built part, stainless steel infiltrated with bronze [16]	7
2.4	BPE and BMD example material and parts	8
(a)	BPE build metal parts [9]	8
(b)	Bound metal rod for BMD [8]	8
2.5	DED process schematic	9
2.6	Spectral reflectivity values for steel, gold, aluminum and copper [22]	11
2.7	Laser beam profile representation at waist	11
2.8	Comparison between 3 nozzle and coaxial nozzle setup [29]	13
2.9	Overlapping laser intensity and powder particle distributions, adapted from [22]	13
2.10	Powder Bed Fusion cross section	15
2.11	Lasers scanning the powder bed in a Renishaw machine [33]	16
2.12	Schaeffler diagram with 316L and 316L-Si marked, adapted from [40]	19
2.13	316L microstructure examples, as-cast and annealed, etched with glyceresia and Kalling's no. 2 reagent respectively [42]	20
(a)	As-cast	20
(b)	Annealed at 954°C	20
2.14	Example microstructure from a DED built part, adapted from [43]	21
2.15	Conduction, Transition and Keyhole Mode weld cross sections [50]	22
2.16	Clad geometry parameters	25
3.1	Typical Morphology of MetcoClad 316L-SI Powder [63]	29
3.2	COAX12V6 Laser Cladding Head by itself and mounted on the KUKA robotic arm [65]	31
(a)		31
(b)		31
3.3	Coherent HighLight FL 3000 laser system used	32
3.4	Medicoat AG powder feeders used	32

3.5	EMaQs system, mounted on the cladding head, and LompocPro 8 software interface	33
(a)		33
(b)		33
3.6	DED setup used	34
3.7	ASTM E606-92 standard dimension ratios for flat sheet specimen with a rectangular cross section [71]	35
3.8	Microtensile specimen dimensions	35
(a)		35
3.9	Struers® Duramin microhardness tester used	36
(a)		36
3.10	Taguchi factors and levels considered	37
3.11	Trial 1 clads	39
(a)		39
(b)		39
3.12	Trial 2 clads	40
(a)		40
(b)		40
3.13	Optical microscopy image of condition 7 clad	41
3.14	Main effects plot for the second Taguchi matrix	42
(a)		42
(b)		42
(c)		42
3.15	Clad height considerations for layer height	44
(a)		45
(b)		45
(c)		45
(d)		45
(e)		45
(f)		45
3.17	Side view of the built cylinders, in chronological order from left to right	46
3.18	20mm height wall deposition results, in chronological order, side and transverse view	47
(a)		47
(b)		47
(c)		47
(d)		47
(e)		47
(f)		47
(g)		47
(h)		47
3.19	Temperature and power modulation graphs during the deposition of the first two wall clads	49
(a)		49
(b)		49
3.20	Temperature and power modulation graph during the deposition of the first 40mm wall clad (wall #6)	51
3.21	40mm height wall deposition results, in chronological order, side and transverse view	52

(a)	52
(b)	52
(c)	52
(d)	52
(e)	52
(f)	52
3.22 3 line wall deposition results, with details	53
(a)	53
(b)	53
(c)	53
(d)	53
(e)	53
3.23 3 line walls built	54
3.24 Wall #3 sample before and after being machined and set in resin for microstructure and microhardness analysis	54
(a)	54
(b)	54
(c)	54
3.25 Microstructure representative of the interest areas present in the sample	55
(a)	55
(b)	55
(c)	55
(d)	55
3.26 Inter-layer interface showing a large inclusion and numerous voids	56
3.27 Feret diameters representation [72]	56
3.28 Microtensile specimens and their placement within the deposited wall	57
(a)	57
3.29 Intermediate machining stages of the specimens	57
(a)	57
(b)	57
3.30 Resulting specimens, 6 machined for the horizontal direction (marked H) and 6 for the vertical/transverse direction (marked V)	58
(a)	58
(b)	58
3.31 Tensile test results for the horizontal and the transverse/vertical direction respectively	59
(a)	59
(b)	59
3.32 Microtensile specimens after being tested	60
(a)	60
3.33 Microhardness values and range along the specimen's height	61
(a)	61

List of Tables

2.1	Typical wavelengths and BPP for CO ₂ , Nd:YAG and Yb fiber lasers, for their fundamental frequencies [22–24, 26]	10
2.2	SLM processing parameters for 316L steel [37]	17
2.3		21
2.4	Processing parameters, resulting energy density and normalised enthalpy for laser based powder DED using 316L	24
3.1	Composition of the 316L-Si powder used [63]	30
3.2	Particle size distribution of the 316L-Si powder used [63]	30
3.3	L9 Taguchi array for trial 1	38
3.4	L9 Taguchi array for trial 2	39
3.5	Single clads trial 2 results	40
3.6	Parameters used for the first set of 3D clads	43
3.7	Height and height error measurements for the test cylinders	46
3.8	E-MAqS closed-loop settings	48
3.9	Process and E-MAqS parameters used and resulting average width and height measurements, for each wall 20mm deposited	50
3.10	Process and E-MAqS parameters used and resulting average width and height measurements, for each 40mm wall deposited	50
3.11	Tensile test results summarised	59

Abbreviations

3D	Three Dimensional
AM	Additive Manufacturing
ASTM	American Society for Testing and Materials
BMD	Bound Metal Deposition
BPE	Bound Powder Extrusion
BPP	Beam Parameter Product
CAD	Computer Aided Design
DED	Direct Energy Deposition
DMLM	Direct Metal Laser Melting
FDM	Fused Deposition Modeling
HAZ	Heat Affected Zone
LCD	Liquid-Crystal Display
LENS	Laser Engineered Net Shaping
MPS	Melt Pool Size
UTS	Ultimate Strength
UV	Ultraviolet
PBF	Powder Bed Fusion
SLA	Stereolithography
SLM	Selective Laser Melting
YS	Yield Strength

Chapter 1

Introduction

Additive manufacturing (AM), more commonly known as 3D printing, describes the process of automatically building a three-dimensional object from a CAD model, usually by means of overlapping layers. It is an evolution of the rapid prototyping concept developed in the 1990s to produce non-working parts mostly for design purposes, and has been cementing itself as a broad family of manufacturing processes capable of shaking the industrial world. ASTM International defines additive manufacturing as "a process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies" [1] and as such serves as an umbrella term for a multitude of dissimilar processes whose only link is their additive nature.

AM provides several advantages over traditional subtractive manufacturing methods and as such has garnered the interest of academia and industry alike: these processes can create infinitely complex parts at no extra cost in a single manufacturing step, allowing for the reduction of assembly part counts. The automatic nature of AM also allows for parts to be produced on demand, reducing lead times and inventory for spare parts [2, 3].

Direct Energy Deposition (DED) in particular is an additive manufacturing process where a source of focused thermal energy (a laser, electron beam or plasma arc) is used to melt added material (in the form of blown powder or wire) in layers, creating three dimensional parts. The most common variant of DED, studied in this dissertation using 316L stainless steel, is the powder fed and laser based direct energy deposition method. As the name implies, powder is blown through a nozzle into a melt pool created by the laser's incidence on the substrate, continuously adding material to the workpiece. Figure 1.1 shows an example of the DED process.



Figure 1.1: Direct Energy Deposition process with a coaxial nozzle [4]

1.1 Objectives

This dissertation is focused on studying and optimising the multiple parameters used to constrain the blown powder laser direct energy deposition process using 316L stainless steel, making use of the increasing number of publications and reviews on not just DED but metal additive manufacturing as a whole and through experimentation.

With literature as a starting point and by understanding the different mass and heat transfer phenomena at play, the experiments were designed in order to better understand how those parameters (such as laser power output, scanning speed, powder feed rate and particle size) impact the built part's microstructure, mechanical properties and geometrical qualities. The general goal is to reach a set of processing parameters capable of creating large scale near net shape parts accurately, with adequate mechanical properties and great repeatability. The following more discrete goals can also be defined:

G1 - Evaluate the discrete influence of each processing parameters in the deposited clads and the DED process efficiency.

G2 - Study the potential benefits of closed-loop control in the DED process.

G3 - Analyse the resulting mechanical properties for the optimal processing parameters found.

G4 - Investigate the existence of anisotropy in the built parts.

1.2 Document structure

This dissertation is divided in 4 distinct chapters, organised as follows:

- Chapter 1 - Introduction: In this current section, chapter 1, an overview of the dissertation is done and the motivation and goals for it are laid out. Furthermore, a work plan is defined.
- Chapter 2 - State of the Art: In this chapter the state of the art regarding additive manufacturing is reviewed, in specific for metals and using directed energy deposition, and the key points concerning the relevant process parameters and their influence are laid out. The predictable effects of the DED processing conditions on the resulting part's microstructure and mechanical properties is analysed and the use of closed loop control techniques is discussed.
- Chapter 3 - Experimental: This chapter begins with an overview of the equipment used throughout the dissertation's experimental component, then the design of experiment (DOE) for the preliminary trials and goes over the experimental work done for this dissertation. Finally, the analysis of the resulting microstructure, microhardness and tensile properties is done.
- Chapter 4 - Conclusions: In chapter 4, conclusions are drawn from the results gathered and the goals set in Chapter 1 are revisited, reviewing if and in what measure they were achieved. Future work is proposed as a continuation of the present dissertation.

1.3 Work plan

A work plan was elaborated as a tool to define time goals and pace the research and experimental work. The plan was to immediately start working on the document, laying down the foundations of the State of the Art review, in Section 2, and defining goals for the dissertation. Then, after the technology and underlying mechanisms were reasonably understood, experimental work was to start by April with time to space for the analysis of the results, elaboration of the document and delivery by the end of June.

However, shortly after the beginning of the dissertation work, the COVID-19 pandemic struck Portugal, limiting access to the installations, forcing a change of the defined plan and eventual delay of the dissertation delivery date. Parallel to this situation there was a number of technical difficulties with the DED system which delayed the start of the experimental leg of this work. The revised plan, in the form of a Gantt chart, can be seen in Figure 1.2.

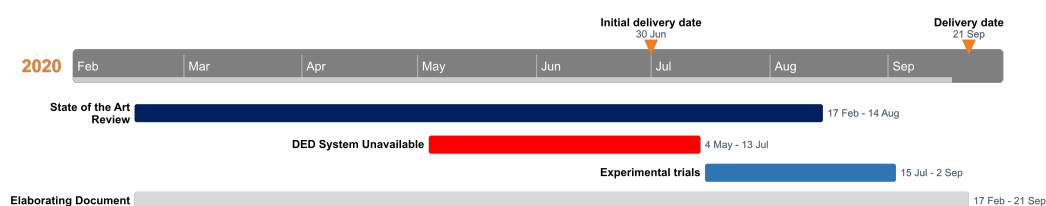


Figure 1.2: Dissertation work plan in the form of a Gantt chart

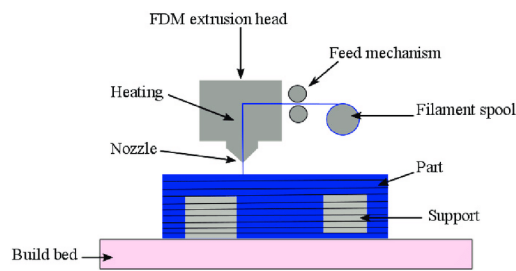
Chapter 2

State of the art

2.1 Additive Manufacturing

There is an additive manufacturing process for nearly every material imaginable, from plastics to metals, ceramics and even composites of these, and each one has its own use case. ASTM classifies these processes in 7 different categories: VAT Photopolymerisation, Material Jetting, Binder Jetting, Material Extrusion, Powder Bed Fusion, Sheet Lamination and Directed Energy Deposition [5].

Of these process categories, material extrusion is the most widespread by far in the form of the Fused Deposition Modeling (FDM) systems, available for a wide range of prices and applications, using thermoplastic filaments, melted and extruded through a small nozzle, to deposit molten plastic in layers, creating parts [6, 7]. Depending on the polymers used these parts can be merely ornamental (with PLA, a low performance thermoplastic) but can also be functional when engineering polymers are employed (PC, Nylons and even PEI and PEEK in specialised high temperature machines). There have also been developed filaments for FDM machines which incorporate chopped glass or carbon fibers and even nano particles, further improving their performance, and even systems which automatically lay continuous fibers in between plastic layers, thus expanding the use cases of the FDM process [8, 9]. Figure 2.1 shows a schematic of the FDM process along with a functional nylon part built using that same process.



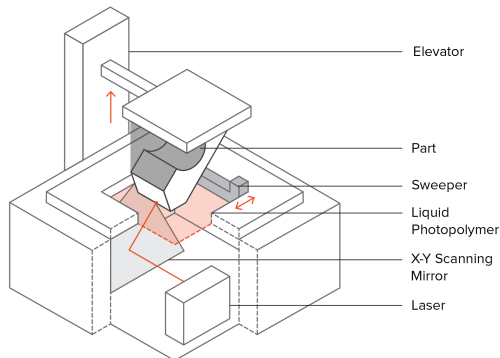
(a) FDM process schematic [10]



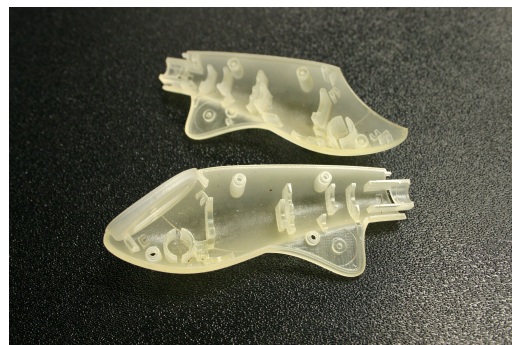
(b) FDM built nylon part [11]

Figure 2.1: FDM process schematic and part example

The VAT Photopolymerisation processes, next to FDM in terms of prevalence, use either a scanning laser (designated as stereolithography, SLA), an UV bulb masked by an LCD (designated as digital light processing, DLP) or more recently simply one high contrast UV LCD (also DLP) to cure resin in layers. These systems can produce highly detailed parts, although with weak mechanical properties and high processing times. The photosensitive resins necessary for the process are generally brittle, expensive and a great limiting factor for the mechanical properties of the produced parts. Consequently, SLA is mainly used for ornamental parts or for producing casts. [6, 7] The following Figure 2.2 shows a schematic of the SLA process along with a part built using it.



(a) SLA process schematic [12]



(b) SLA built part [13]

Figure 2.2: SLA process schematic and part example, note the absence of visible layer lines

These two AM processes, although widespread, are only capable of processing polymers and thus have limited applicability, being usually only used for fixtures, prototypes and small lightly loaded parts. While some aforementioned FDM systems are capable of using engineering polymers and even incorporate fibres in them to build mechanically sound parts, there are applications where additively manufactured metal parts are required, thus calling for the analysis of other AM

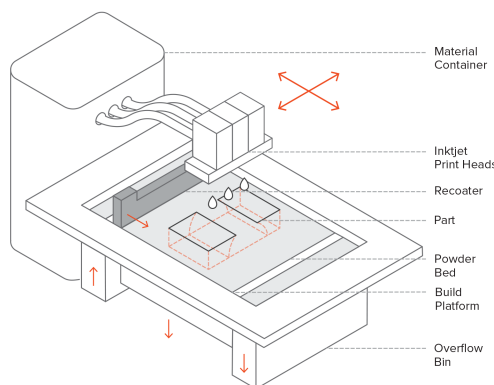
process categories:

- Powder Bed Fusion (PBF)
- Direct Energy Deposition (DED)
- Binder Jetting
- Material Extrusion:
 - Bound Powder Extrusion (BPE, designed by Markforged [9])
 - Bound Metal Deposition (BMD, designed by Desktop Metal [8])

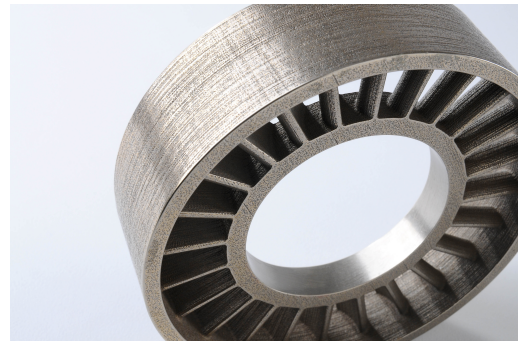
While SLM and DED result in net-shape and near net-shape metal parts, respectively, both Binder Jetting and BPE yield green parts that need to undergo some post-processing steps, be it a debinding, sintering or infiltration process before becoming fully dense metallic parts. For this reason SLM and DED are referred to as direct-to-metal AM processes.

The binder jetting process starts off by depositing a thin layer of metal powder over it's print bed. As the process' name implies, a print head then deposits a polymeric binder in the area of the part's cross section, forming a new layer of bound metal powder, and this cycle is repeated until the part is completed. The resulting green part must then be subject to either infiltration or sintering. For sintering, the green parts are placed in an oven at a high temperature (usually <90% of the metal's melting point) burning the binder present and allowing for necks to form between the powder particles present, strengthening and densifying it. However, this also results in heavy shrinkage, occasional distortion and a hot isostatic pressing step is nonetheless necessary to obtain fully dense parts[14, 15] .

For infiltration the part is placed in an oven at high temperatures, burning off the polymeric binder and allowing for low melting point materials to be infiltrated into the porous part, achieving full density with minimal shrinkage and distortions at the cost of the lower mechanical properties offered by the infiltrated metal[14, 15]. Figure 2.3 shows a schematic of the binder jetting process and a sample part of such process.



(a) Binder Jetting process schematic [12]



(b) Binder Jetting built part, stainless steel infiltrated with bronze [16]

Figure 2.3: Binder jetting process schematic and part example

The bound powder extrusion (BPE, developed by Markforged) and bound metal deposition (BMD, developed by Desktop Metal) processes are both material extrusion processes, building parts by extruding layer upon layer of material, but creating a green part like in binder jetting. A debinding and sintering process is then required to remove that previously used binder and densify the parts. While BPE uses bound metal powder rods, BMD extrudes filament much like an FDM machine [8, 9]. Figure 2.4 shows a number of turbo impellers built using BPE, in *a*, and a bound metal rod for the BMD process, in *b*.



(a) BPE build metal parts [9]



(b) Bound metal rod for BMD [8]

Figure 2.4: BPE and BMD example material and parts

The focus of this work is on direct energy deposition, and as such that process will be described in detail below in parallel with selective laser melting as they have much in common such as the high energy density heat sources and localised melt pools. [2]

2.2 Direct Energy Deposition

DED, also known as laser cladding or Laser Engineered Net Shaping (LENS, Sandia National Lab's proprietary designation), is an additive manufacturing process where metal powder is fed through a nozzle and injected directly into a melt pool created by a laser's incidence on the substrate or previously deposited layer. This process can be employed to produce near net shape parts and repair worn high value parts using a wide range of metals and even ceramics, from stainless steels to Inconel, titanium alloys or tungsten, among several others [17–19]. Figure 2.5 shows a schematic of the DED process.

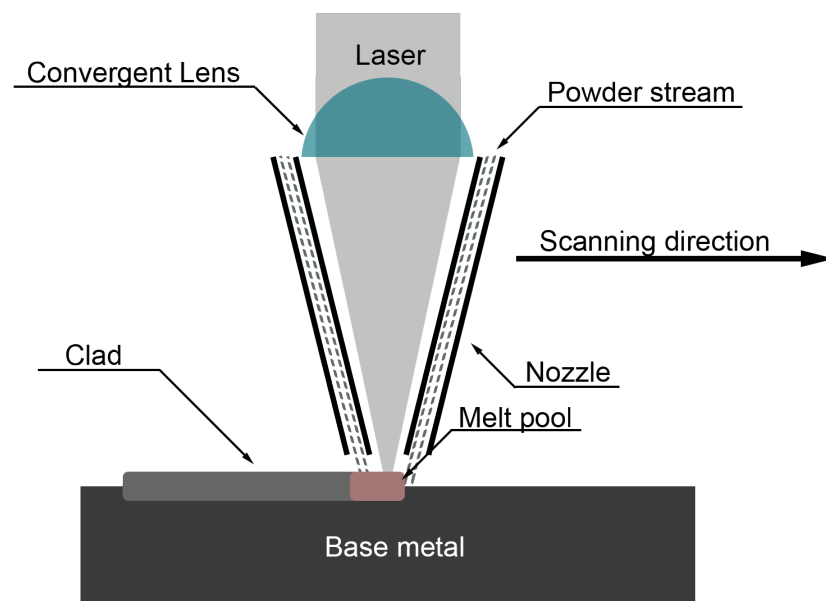


Figure 2.5: DED process schematic

A laser beam, previously aligned in a collimator at the laser source, is focused by a series of optical lenses (represented by a simplified convergent lens in Figure 2.5) into a spot which irradiates the substrate or previously built layer. This high-power laser (from 100 to as much as 5000 Watts) creates a moving melt pool onto which metal powder is blown and melted into. This powder is carried by an inert gas (Nitrogen or Argon) which doubles as protection for the melt pool, preventing oxidation effects in the clad. Some machines also allow for the whole build volume to be replaced by the inert gas, significantly improving not only the part's yield and ultimate strength but also elongation by inhibiting the formation of large oxides formed during the process [17, 20].

The blown powder particles, directed at the melt pool by either a coaxial nozzle or multiple discrete ones, are heated up by the laser and consequently trapped in the melt pool, where they fuse with the molten substrate. Part of the blown powder, however, strikes the area adjacent to the melt pool and is lost, resulting in both an energetic and material loss of efficiency. [19] Furthermore, the

use of blown powder allows for changing alloy concentrations on the fly, resulting in functionally graded parts [17, 21].

This deposition method allows for high volumetric build speeds and build areas, even for complex parts, while obtaining superior mechanical properties when compared to traditional manufacturing techniques, owing to the microstructure created due to the rapid cooling and directional thermal gradient inherent to the process. [20]

2.2.1 Laser properties

There are several relevant types of lasers, generally classified based on the physical state of its active material. They are:

- Gas lasers
- Fiber lasers
- Solid-state lasers
- Excimer lasers
- Semiconductor lasers
- Liquid dye lasers

Amongst them, it is possible to obtain outputs from a few milliWatts to some Megawatts, for continuous wave (CW) lasers, but even higher for pulsed lasers, in wavelengths that range from around 1mm to 1nm [22]. Several factors need to be considered when choosing a laser source for DED, most notably: power output, in order to provide sufficient energy density; adequate wavelength to minimise reflection losses of efficiency; beam quality, to ensure beam focusability and energy distribution [22–24].

The prime candidates for use in laser cladding have been CO₂ (gas) and Nd:YAG (solid state) lasers, but more recently advancements in fiber laser technology, allowing for higher power output, have cemented their place as lasers capable of heavy material processing [22, 25].

These 3 types of laser have previously been used in material processing, be it laser cladding, welding and cutting, and provide sufficient power density for DED. Regarding wavelength, CO₂ lasers' wavelength is an order of magnitude higher than that of Nd:YAG and Yb fiber lasers, as can be seen in table 2.1. Because a steel's reflectivity increases for higher wavelengths (shown in Figure 2.6), the higher wavelength of CO₂ lasers results in a greater amount of wasted energy when they are employed in metal processing, compared to Yb fiber or Nd:YAG lasers [17].

Table 2.1: Typical wavelengths and BPP for CO₂, Nd:YAG and Yb fiber lasers, for their fundamental frequencies [22–24, 26]

Laser type	Wavelength [μm]	BPP [$\text{mm} \cdot \text{mrad}$]
CO ₂	10,64	4-12
Nd:YAG	1,030–1,100	25-45 ¹ / >12 ²
Yb fiber	1,064	2-10

¹ Lamp-pumped

² Diode-pumped

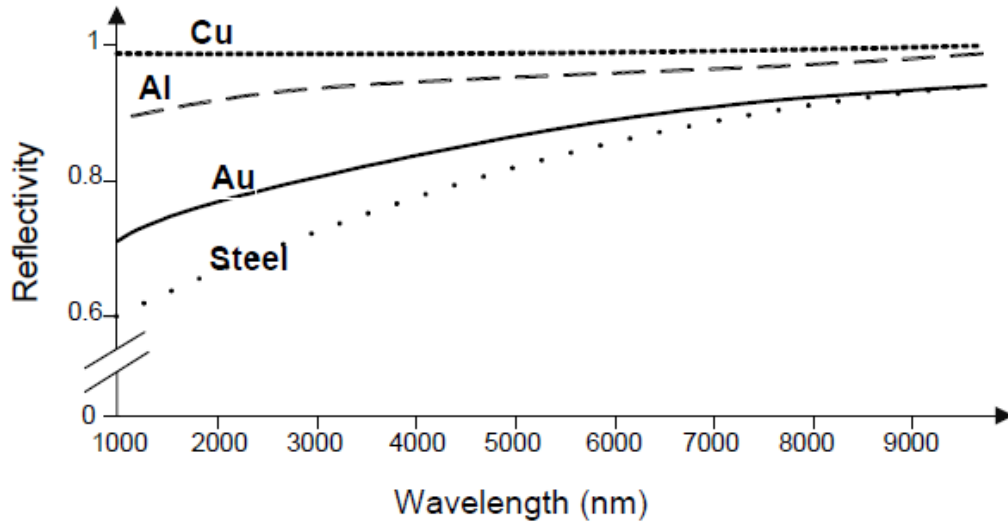


Figure 2.6: Spectral reflectivity values for steel, gold, aluminum and copper [22]

Beam quality can be evaluated using the beam parameter product (BPP) which is the product of the laser beam's divergence half angle $\frac{\theta}{2}$ and radius r_0 at it's waist, represented in Figure 2.7, where a lower BPP value implies the laser beam is of a higher quality and can be focused to a smaller spot size [22, 23].

$$BPP = \frac{r_0 \cdot \theta}{2} \quad (2.1)$$

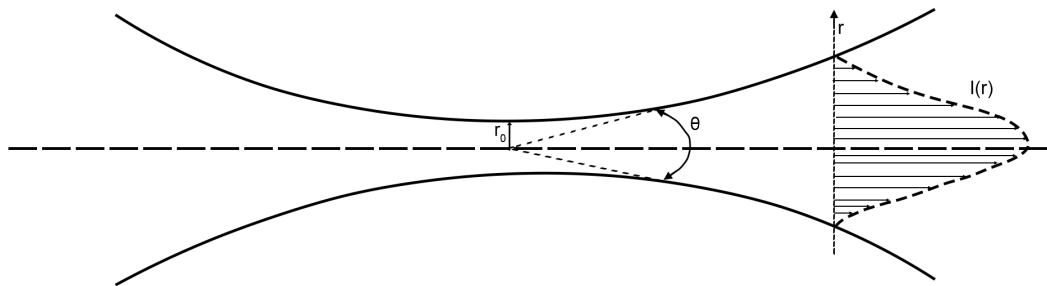


Figure 2.7: Laser beam profile representation at waist

A Gaussian beam describes the ideal output of a laser, with a gaussian intensity profile, and as such has the lowest possible value for BPP given by [22]:

$$BPP = \frac{\lambda}{\pi} \quad (2.2)$$

CO2 laser's beam quality is superior to that of solid state Nd:YAG lasers, as seen in Table

2.1, but still falls short of the more recent fiber lasers. The low BPP attained with Yb fiber lasers allows for spot sizes smaller than $50\mu m$ and for a higher distance between the focusing lenses and the workpiece, improving access to more difficult areas of the workpiece. [24] Consequently, CO₂ lasers are being phased out due to both their low wall-plug efficiency and high wavelength compared to both Nd:YAG and Yb fiber lasers, which results in a great amount of wasted power during the process, and Yb fiber lasers are the leading laser technology for this process [17, 22, 27].

2.2.2 Powders and powder feed system

The quality and characteristics of the powders used in DED greatly affect the outcome of the process and as such must be carefully controlled in order to obtain satisfactory results. These characteristics include the powder particle's shape (sphericity), size distribution (PSD), morphology and naturally it's composition [2]. All of these characteristics also impact the flowability which is an important performance factor for the powder.

In general, a more uniform size distribution leads to a more homogeneous melting which in turn results in better inter-layer properties, better mechanical properties and surface finish. Furthermore, powders with a higher sphericity and lower porosity lead to fabricated parts with lower porosity. Powders with these characteristics (finer and uniform PSD, good sphericity and surface quality) tend to be more expensive compared to coarser powders due to the cost and low yield of the processes used to produce them, so choosing a powder feedstock is always a balance of cost and desired performance [2, 3]. The powders wasted during the process can be reused to reduce costs but always taking into account that the reused powder will be more irregular than the unused powder and can yield worse results, mainly due to the higher amount of oxides present [28].

In DED the metallic powders are transported by the carrier gas, usually Argon, which provides the initial velocity up to the nozzle and injects the powder into the melt pool. Often this gas also doubles as protection gas for the melt pool [20].

The powder can be fed into the melt pool by a coaxial or multi jet nozzle, which along with the nozzle's angle and powder characteristics affects the deposition efficiency. The nozzles are angled so that they intersect the laser at it's focal point [17].

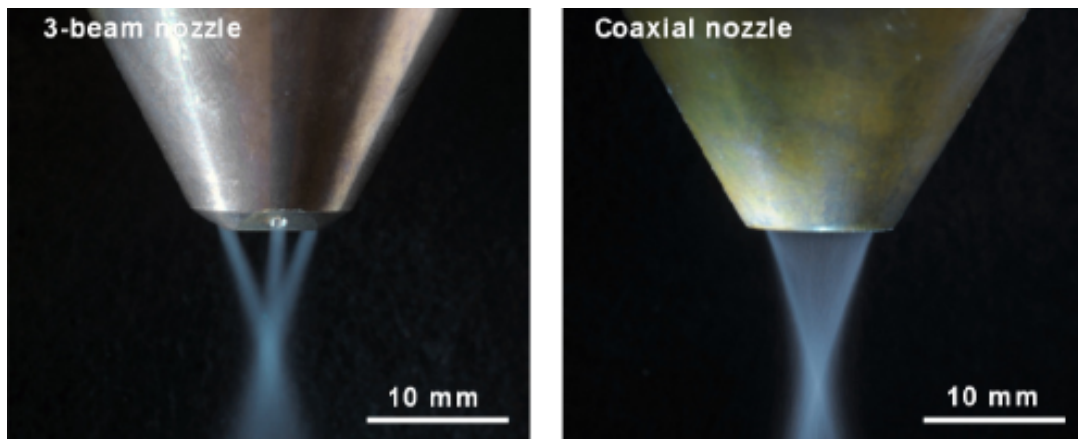


Figure 2.8: Comparison between 3 nozzle and coaxial nozzle setup [29]

Like the laser beam, the powder streams also follow a Gaussian particle distribution. As is shown in Figure ??, in order to deliver a more uniform powder flow to the laser beam's spot some powder must be wasted [22].

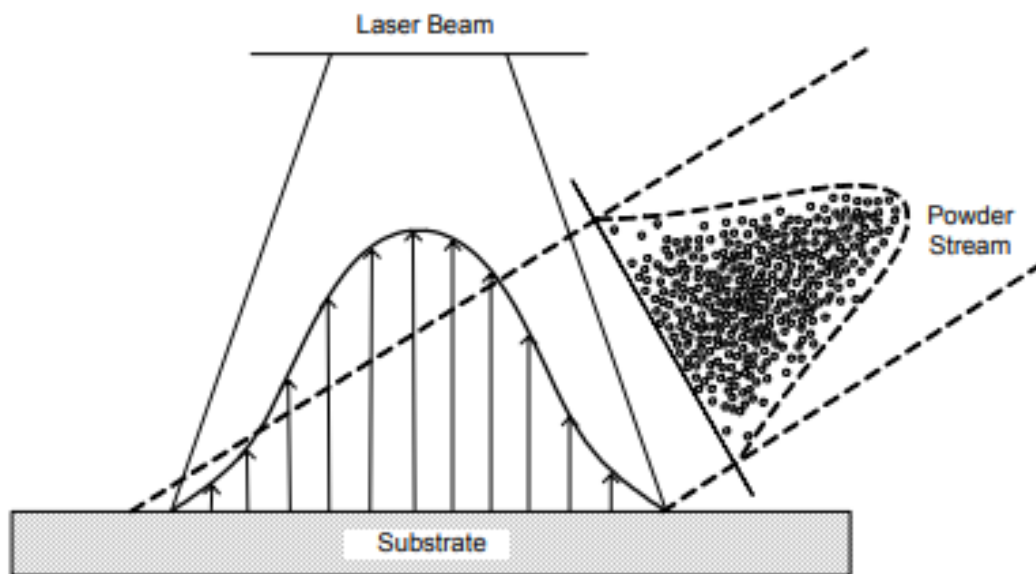


Figure 2.9: Overlapping laser intensity and powder particle distributions, adapted from [22]

The powder stream also influences the power transmitted to the melt pool, as some of the powder blown is heated up by the laser but doesn't hit the melt pool and therefore is wasted. This effect can attenuate the laser power by up to 8% depending on the powder feed rate and velocity, as predicted by Ansari et al. [30]. This wasted powder can be quantified with the catchment efficiency (or powder efficiency) of the process, η . [30] This catchment efficiency has been reported to be as low as 14% in some cases, as reported by Unocic and Dupont [31], but more recently values

around 50% have been demonstrated with conventional nozzle designs [\[32\]](#).

2.3 Selective Laser Melting

SLM, also known as Direct Metal Laser Melting (DMLM) or Laser Powder Bed Fusion (PBF-L), is a subcategory of the Powder Bed Fusion processes which uses a high power laser to scan and melt powdered metal layer by layer, creating fully dense parts.

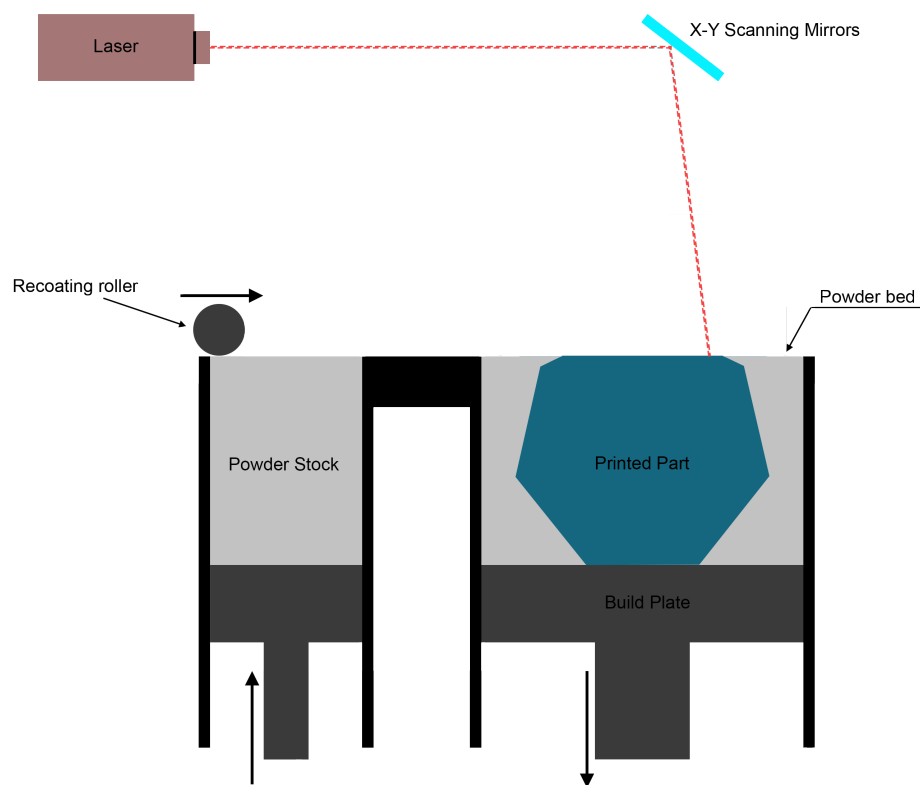


Figure 2.10: Powder Bed Fusion cross section

A high energy density power source (a laser or an electron beam) scans a metal powder substrate, locally fusing the metal and creating a solid cross section of the part being produced. A roller or blade then spreads another layer of powdered metal on top of the previously sintered one and the process is repeated. [2]

The ability to obtain extremely complex and near net shape parts directly from the CAD along with low surface roughness and great mechanical properties makes SLM extremely attractive for industries where high specific performance parts are required but high volume production isn't necessary, such as the aerospace and medical industries. [2]



Figure 2.11: Lasers scanning the powder bed in a Renishaw machine [33]

Due to the high temperatures involved in the process and high surface area of the powders, the build chamber atmosphere must be controlled to prevent contamination of the melt pool and reactions with the oxygen, carbon oxides and humidity present in a non-controlled atmosphere. Furthermore, when using an electron beam for the process, the build chamber must be under vacuum. [34] The typical gases used for this effect are argon and nitrogen which are inert for most alloys used, but for some materials, such as Ti-6Al-4V, argon is the only option because of the alloy's affinity to oxygen and nitrogen. It was found that 316L stainless steel, however, can tolerate up to 1000PPM O_2 with only marginal changes to its mechanical properties making it a robust material for this kind of processing. [35] [34]

Of the direct-to-metal AM processes, SLM provides the best dimensional accuracy (0.5 - 1 mm)[2] and arguably the best surface roughness(7 - 25 μm Ra) [36] [2] but falls short on building speed and size relative to DED.

It is relevant to analyse SLM as it shares many processing conditions with DED: both processes use similar materials, in this case 316L, which require a certain energy input to be fabricated. That energy input, in the form of a laser beam for both SML and DED, interacts with the metallic powders in an identical fashion, making the parallel between the two processes possible to better define suitable working conditions for 316L.

2.3.1 Laser characteristics

Similarly to DED, there are several laser types available with sufficient power and characteristics for the SLM process but the most common are solid state Nd:YAG and Ytterbium fibre lasers.

As was compiled by Yakout et al. [37], reported process parameters and systems used for SLM production of 316L steel are shown in table 2.2.

Table 2.2: SLM processing parameters for 316L steel [37]

SLM machine	$P[W]$	$v[mm/s]$	$h[mm]$	$d_s[mm]$	$E[J/mm^2]$	$E_v[J/mm^3]$
Concept Laser M2 ¹	150–400	500–1800	0.030	0.150	1.59	28–74
HRPM-IIA ²	190	800	0.050	0.150	1.58	32
SLM Solutions 125 ²	50–100	300–1250	0.030	0.070–0.140	0.92	32–80
Concept Laser M1 ¹	104	175–800	0.038	0.130	1.64	33–107
Concept Laser M3 ¹	100	175–380	0.060	0.126	2.86	35–76
EOS M280 ²	150	1200	0.030	0.100	1.25	42
Concept Laser M2 ¹	200	1600	0.050	0.060	2.08	42
SLM-Realizer 100 ²	50	100–300	0.050	0.080	3.13	42–125
Renishaw AM250 ²	200	750	0.050	0.110	2.42	48
Concept Laser M3 ¹	105	380	0.03	0.125	2.21	55–111
SLM-Realizer 250 ²	100–200	200–220	0.050	0.124	5.76	81–150
Phenix PM100 ²	50	120	0.040	0.120	3.47	87
in-house dev. SLM ¹	100	300	0.030	0.112–0.125	2.22	89–99
SLM Solutions 250 ²	380	625–3000	0.050	0.025–0.120	2.8	99–109
SISMA MYSINT100 ²	100–150	700	0.020	0.050–0.070	2.98	102–214
DiMetal-100 ²	200	400	0.040	0.080	6.25	156
HRPM-IIA ²	180	900	0.020	0.060	3.33	167

¹ Nd:YAG solid state laser² Yb-fiber laser

The energy density (E) values reported in table 2.2 range from 0.92 to 6.25 J/mm^2 , an order of magnitude below the values found in literature for DED. This difference can be explained by the much thinner layers built in SLM which require less energy for fusion to take place. When the values of volumetric energy density (E_v) are compared, taking into account that layer thickness, the values for DED and SLM are comparable (25 - 60 and 28 - 214, respectively). Still, this wide range of values shows how dependent the processing parameters are on the machine used.

2.3.2 Metallic Powders for SLM

For the purpose of SLM there are several powder properties and characteristics that can be analysed in order to improve the process' performance and end results. The powder's geometric characterisation can be done through it's morphology (sphericity), powder size distribution (PSD) and porosity. These characteristics are the main contributors to the powder's rheological behaviour.[38] Spherical particles improve both powder packing characteristics and flowability. [38]. [36] found that the absence of finer particles requires a slower scanning speed (leading to lower productivity) to obtain fully dense parts and that the PSD should be taken into account in order to enhance printing parameters and produce optimal results. Furthermore, the powder's PSD has a

great effect not just in the produced part's density but also in its surface quality and mechanical properties, and a wider PSD improves powder packing but reduces flowability because of the finer particles present. Coarse particles, however, improve flowability but reduce surface finish.[39]
[38]

2.4 Microstructure in metal AM

In the laser based AM processes the workpiece is subjected to repeated thermal cycles, where every given volume of it is first heated above melting temperature and then rapidly cools down and solidifies, before being re-heated several times by the deposition of the following layers. This complex thermal history leads to difficult to predict but potentially favourable and fine microstructures [2, 3].

Evidently, the processing parameters such as the laser power or scanning speed play a major role in defining the temperature gradients during the build, but the part's geometry and heat conduction properties of the substrate and deposited metal will also have a relevant influence.

Regarding 316L specifically, being an austenitic stainless steel, it's microstructure is predominantly comprised of austenite (γ -phase iron).

The Schaeffler diagram is a widely accepted tool to roughly predict the constitution of stainless steel weld deposits, and as such could be applied to the present study. This diagram, shown in Figure 2.12, attempts to predict the predominant phases by weighing the ferrite forming elements (Cr, Si, Mo, Nb, W) against the austenite forming elements (Ni, C, Mn, Cu, N) [40].

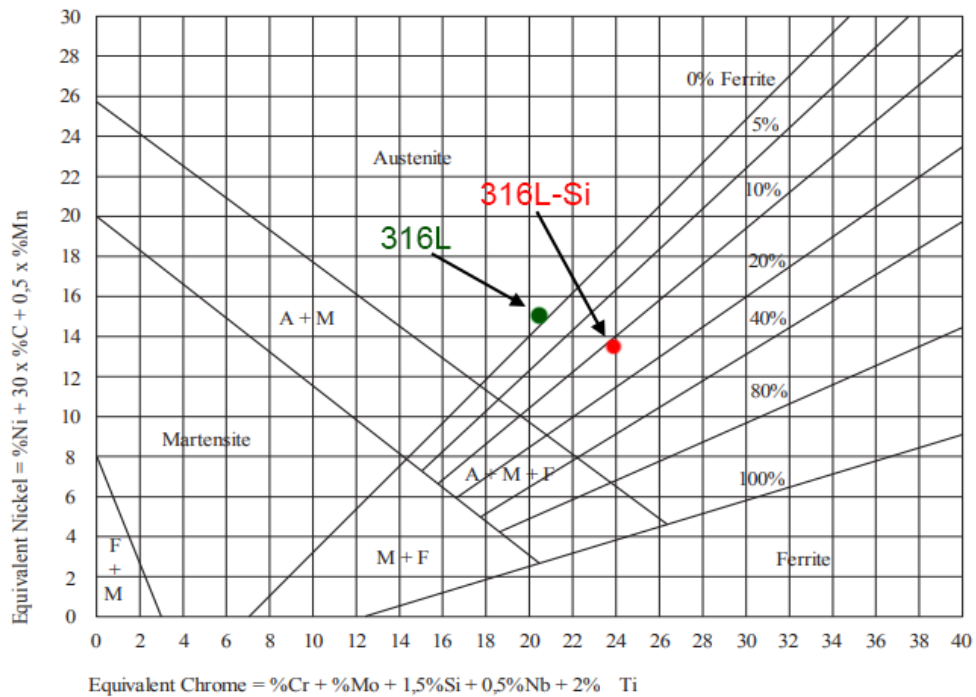


Figure 2.12: Schaeffler diagram with 316L and 316L-Si marked, adapted from [40]

The inputs for this diagram are the equivalent chrome and nickel content, calculated as follows:

$$Eq. Chrome = \%Cr + \%Mo + 1,5 \cdot \%Si + 0,5 \cdot \%Nb + 2 \cdot \%Ti \quad (2.3)$$

$$Eq. Nickel = \%Ni + 30 \cdot \%C + 0.5 \cdot \%Mn \quad (2.4)$$

For 316L-Si the calculated values are: Eq. Chrome = 22,95 and Eq. Nickel = 13,4, inside the austenitic stainless steel range, thus having austenite as it's primary phase with roughly 10% δ -ferrite present. The more standard 316L, however, has Eq. Chrome = 20,63 and Eq. Nickel = 14,9 leading to a lower amount of ferrite present at equilibrium compared to the Si modified steel.

As can be seen in Figure 2.13 the microstructure of both 316L samples shown is predominantly austenitic, but the as-cast sample shows a greater amount of delta ferrite due to segregation during solidification: the austenite to δ -ferrite transformation occurs only at a narrow band of high temperatures (between 1392°C and 1493°C [41]) so the moderately high cooling rate of the casting process somewhat prevents that transformation.

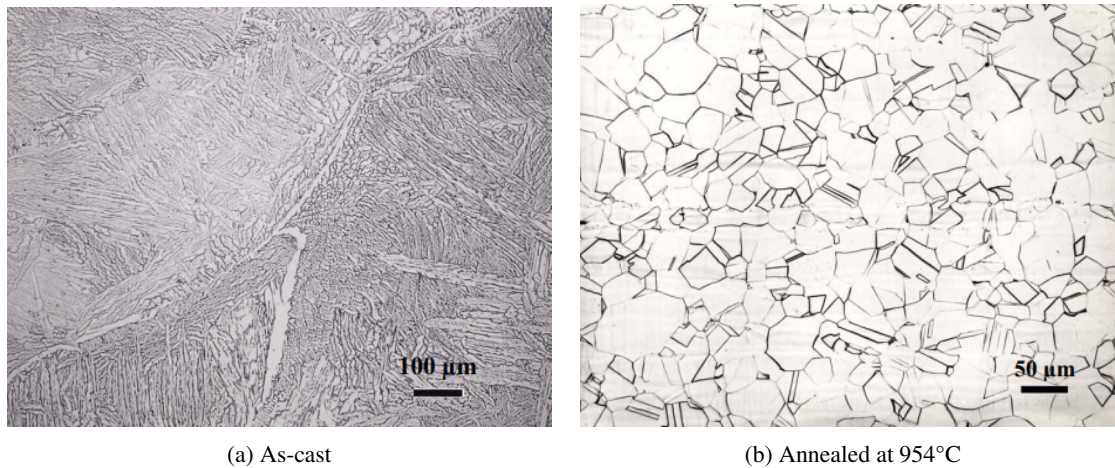


Figure 2.13: 316L microstructure examples, as-cast and annealed, etched with glyceresia and Kalling's no. 2 reagent respectively [42]

The present work, however, focuses on the use of 316L-Si, a modified version of 316L used primarily for welding with added silicon to improve the wettability of the melt pool and weld quality. Furthermore, and as mentioned before, silicon acts as a ferrite stabiliser, increasing the prevalence of delta ferrite at room temperature and thus preventing the occurrence of hot cracking.

The quick localised heating and cooling generates elevated temperature gradients which, in the case of 316L steel, give origin to long columnar austenitic grains (in the gradient's direction) surrounded by δ -ferrite. [20]

This peculiar microstructure can lead to improved mechanical properties, with yield strengths up to 66% higher than the wrought material [28]. In Table 2.3 a comparison is done between the mechanical properties of wrought and DED built 316L, noting that when two numbers are shown (x/y) it represents the value for the horizontal direction followed by the transverse direction.

Table 2.3

	HV	YS [MPa]	UTS [MPa]	ϵ [%]	Ref.
DED	245 $\pm$ 25	425 $\pm$ 20	470 $\pm$ 40	18 $\pm$ 4	[43]
	289	576/479	776/703	33/46	[44]
	-	469	649	23	[28]
	-	530 $\pm$ 5	670 $\pm$ 6	34 $\pm$ 1	[20]
	-	558/352	639/536	24/46	[45]
Wrought	152	280 $\pm$ 25	450	30	[28, 44]
As Cast	-	262	552	55	[28]

In the following Figure 2.14 4 different microscopy images of DED built parts are shown. Note the layer interfaces.

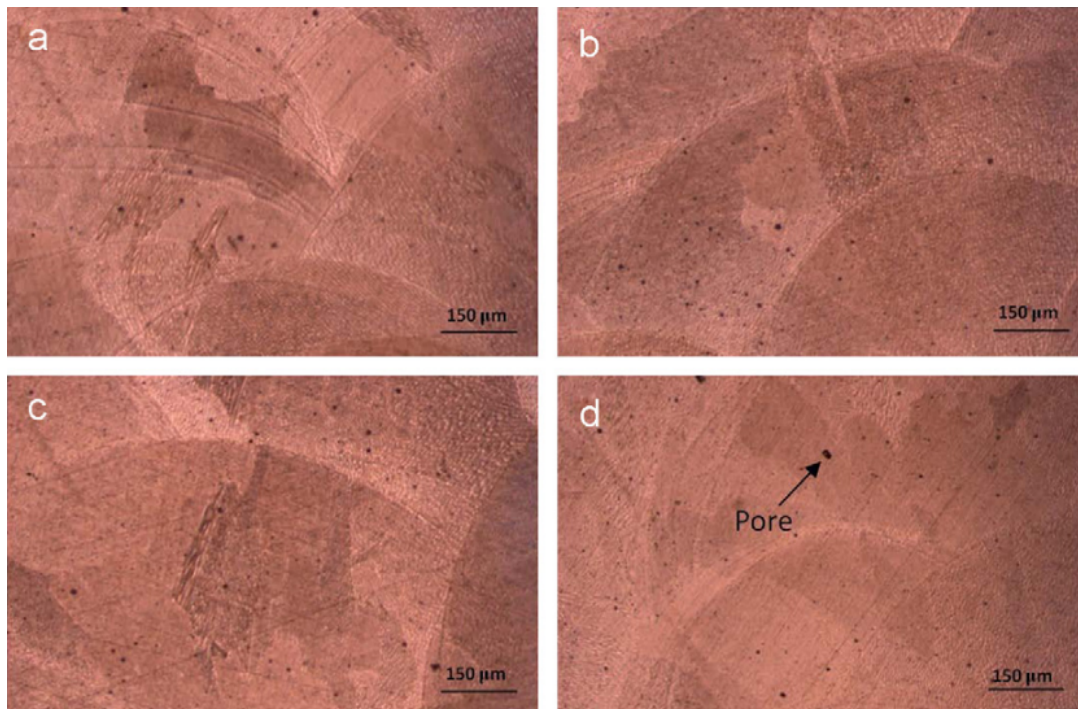


Figure 2.14: Example microstructure from a DED built part, adapted from [43]

2.5 Printing Parameters for DED

The most important printing parameters for DED are the laser power (P), spot size (D), scanning speed (v) and powder feedrate (f). These define first and foremost the heat input into the process which can be analysed as the laser energy density (E), a parameter which has been successfully used to predict the result of the DED process. [46] [47] [3]

It can be calculated as follows:

$$E = \frac{P}{v \cdot d_{spot}} \quad (2.5)$$

If the energy input is too high and causes the melt pool to reach a high enough temperature, material may be vaporised from the melt pool with enough force to push the molten metal aside and form a cavity. If it is too low, lack of fusion may occur leading to bad inter-layer bonding or even failed clads [48, 49]. The transition from conduction-mode melting to keyhole-mode melting must be avoided in the DED process as it leads to the appearance of voids in the clads and shape defects.

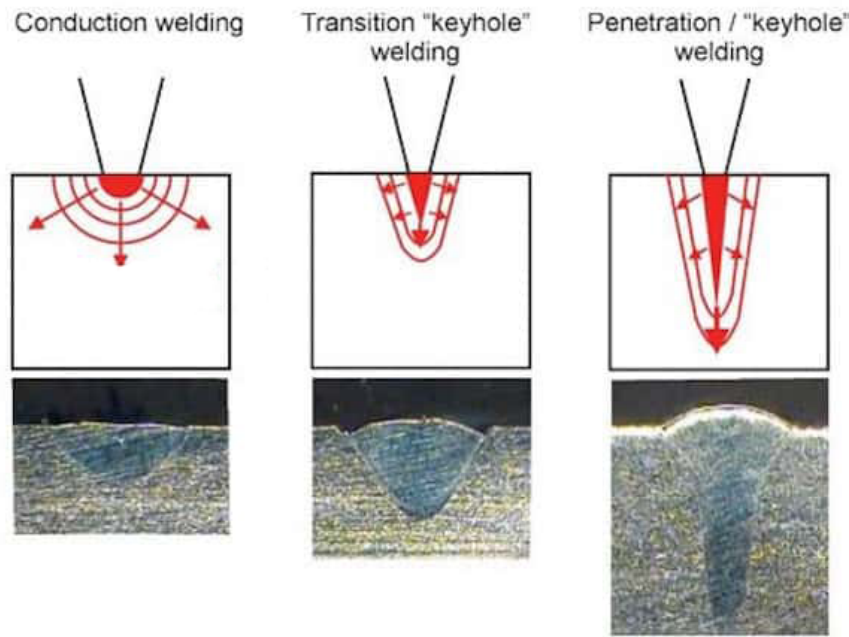


Figure 2.15: Conduction, Transition and Keyhole Mode weld cross sections [50]

The point at which this transition happens can be predicted with the analysis of the normalised enthalpy (h^*). First proposed by Hann et al. [46] as an analytical approach to predict laser weld geometry and properties, the concept has since been applied to laser AM processes [47, 51–53].

The enthalpy, ΔH , can be defined as the energy absorbed during the laser interaction time by the heat diffusion volume, where that volume can be assumed to be a cylinder for one dimensional

heat flow:

$$V_{diffusion} = \pi \cdot d_{spot}^2 \cdot \delta \quad (2.6)$$

In these conditions, the heat diffusion depth (the cylinder's height, δ) can be calculated as:

$$\delta = \sqrt{\alpha \cdot t_i}, \text{ with } t_i = \frac{d_{spot}}{v} \text{ being the interaction time} \quad (2.7)$$

ΔH can then be calculated as

$$\Delta H = \frac{E_{input}}{V_{diffusion}} = \frac{\mu \cdot P \cdot t_i}{\pi \cdot d_{spot}^2 \cdot \delta} = \frac{4 \cdot \mu \cdot P}{\pi \cdot \sqrt{\alpha \cdot v \cdot d^3}} \quad (2.8)$$

The normalised enthalpy is then defined as $h^* = \frac{\Delta H}{h_s}$, where h_s is the enthalpy at melting for a given metal being processed, calculated as:

$$h_s = \rho \cdot c_p \cdot (T_m - T_0) \quad (2.9)$$

Hann, Iammi, and Folkes [46] found that the melt pool becomes unstable if the enthalpy is greater than the enthalpy of vaporisation (roughly $h^* > 10$ for most metallic alloys) or lower than the enthalpy at melting ($h^* < 1$). This defines a normalised enthalpy interval where the process should be stable in conduction-mode melting [46, 52].

The scanning speed must also be analysed closely along with the spot size as it defines the interaction time between the laser and the substrate, t_i , as mentioned before. It has been found that this parameter can impact the porosity of the built part independently of the specific energy density [54]. It also determines the productivity of the AM process, along with the clad width and height. Although the clad width and height cannot be directly imposed, they can be predicted and controlled by means of the energy input and powder feedrate.

The powder feedrate, f , determines the rate at which powder is fed into the gas stream and blown into the melt pool. It plays a major role in deciding the final part's density and the clad's geometry as well.

Now that the main imposable parameters have been discussed it is important to review more specifically what practical research has been done to understand these parameters:

Pinkerton and Li [55] used a pulsed CO2 laser to deposit thin walled 316L specimens using different pulse frequencies, from 8.3 Hz to a continuous beam, finding that a higher pulse frequency yielded denser depositions, but decreased their hardness. A fine cellular structure with directional growth at the layer boundaries was found for all conditions tested.

Majumdar et al. [54] made use of a CW diode laser to deposit stacked 316L clads, varying the applied laser power, scanning speed and powder feed rate, arriving at a set of optimum working conditions for low porosity depositions. Manjaiah, Hascoët, and Rauch [56] conducted a similar study, studying the effects of the aforementioned processing parameters on the single clad geometry. An optimal set of processing parameters was then derived and defect free hollow parts were built using those parameters.

Ma et al. [43] varied the laser power, scanning speed and powder feed rate for multiple 316L cuboid depositions, finding that lower speeds yielded denser parts while higher speeds yielded increased hardness but bad shape quality. Scanning strategy was also evaluated, concluding that a scanning direction change every layer reduced the appearance of over-deposition at the part's edges. For the optimal processing parameters found, the mechanical properties measured through tensile testing were close to those of wrought 316L, with improved yield strength but lower elongation.

Ziętala et al. [44] studied the mechanical properties, anisotropy, microstructure and corrosion resistance of DED built dense 316L cubes. The microstructure obtained was composed of fine elongated austenite grains, oriented along the direction of greater heat dissipation, with intercellular delta ferrite and sigma phase present. Microhardness measurements were taken, resulting in comparable or slightly higher hardness than commercial annealed 316L. Results from tensile tests done on specimens machined in the horizontal and transverse direction yielded improved values compared to wrought 316L and in line with 316L manufactured through other laser based AM processes for both direction. Anisotropy was found, with the strength values for the horizontal direction being greater than the transverse direction, and the inverse relationship was found for the elongation values. The corrosion resistance found was similar to that of conventionally manufactured 316L.

Saboori et al. [28] studied the influence of using recycled metallic powders in DED, and found that while the average yield and ultimate strength was similar for both cases, the elongation obtained when recycled powders were used was much lower and the strength values had more variation.

Aversa et al. [20] studied the effects of the build atmosphere on the microstructure and mechanical properties of DED built 316L parts. In one case, nitrogen was used as a complete build atmosphere, and in the other used solely as shield gas, delivered directly to the melt pool. The mechanical properties of the parts built using a full N₂ build chamber were found to be higher relative to when a shield gas was used, owing to the finer oxides present.

The optimal processing parameters found by these authors are compiled in the following Table 2.4.

Table 2.4: Processing parameters, resulting energy density and normalised enthalpy for laser based powder DED using 316L

P [W]	v [mm/s]	f [g/s]	d _{spot} [mm]	E [J/mm ²]	h*	Ref.
450	4	0.14	1.7	66.18	4.3	[55]
300	5	0.136	3	20	1.09	[54]
1650	23.3	0.53	1	70.82	14.5	[43]
400	15	-	0.45	59.26	14.5	[44]
1200	35	0.45	3.4	10.08	1.37	[56]

This analysis of these reported parameters can begin by looking at the specific energy density, E , that is a good starting reference for the rest of the parameters. For 316L, optimal E values shown in Table 2.4 range from 10 to 70 J/mm².

This wide range of values for energy density is likely due to a number of factors: first and foremost, with DED still being a novel AM process with such complex physical phenomena at play, the distinct manufacturers' equipment will not all produce the same end result for the same set of given processing parameters. Regarding the processing conditions themselves, the variations can be explained by the use of distinct target layer heights and due to the fact that the E value doesn't take into account the type and wavelength of the laser used: using a CO₂ laser at 10,64 μ m means only 40% of it's output energy is used towards the process itself with the rest being wasted because of the several factors discussed previously while an Nd:YAG laser at 1,030 μ m utilises 50% of it's output [19].

Regarding the normalised enthalpy, the distribution reported is even more spread out, with values outside the previously considered upper limit of 10 for a stable process. This indicates that the normalised enthalpy may not be a good predictor for the DED process.

2.5.1 Clad geometry and properties prediction

Attempts have been made to accurately predict the geometry and properties of single clads making use of analytical, FEM and statistical analysis.

The most important properties of the single clad geometry are described in Figure 2.16:

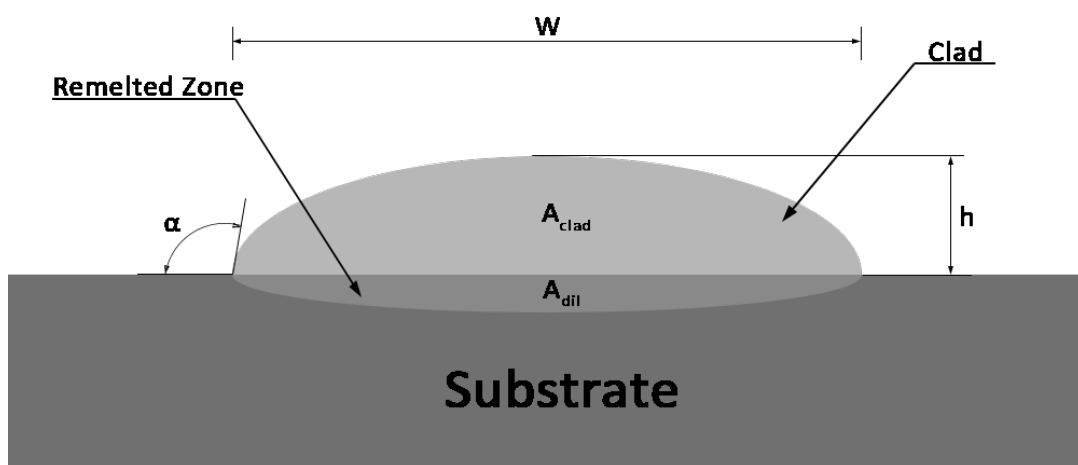


Figure 2.16: Clad geometry parameters

The dilution factor, D_f , can be calculated as $D_f = \frac{A_{dil}}{A_{clad} + A_{dil}}$

Ansari et al. [30] developed an extensive analytical model of the DED process based on Rosenthal's equation to predict clad geometry and dilution, having validated the model with single-track depositions of Ti-5553 (Ti-5Al-5V-5Mo-3Cr). Great accuracy ($> 80\%$) was obtained in the neighbourhood of the optimal processing parameters obtained with the analytical model.

Costa et al. [57] proposed a phenomenological method to obtain a processing zone for DED based on a multi-variable statistical analysis of single track clads of Stellite 6 (Co-30Cr-5W). Relations between the clad geometry and complex processing parameters (Φ_i) were found, which revealed the influence of the different parameters. The complex parameters with the best fit were:

$$\Phi_1 = \sqrt{\frac{P \cdot f}{v^2}} \quad (2.10)$$

$$\Phi_2 = \frac{P \cdot v}{f} \quad (2.11)$$

$$\Phi_3 = \frac{v}{f} \quad (2.12)$$

Having then obtained the following linear relations through regression analysis, for Stellite 6:

$$h[mm] = -0.24 + 6.33 \cdot \Phi_1 \quad (2.13)$$

$$D_F[\%] = -12.69 + 0.37 \cdot \Phi_2 \quad (2.14)$$

$$\alpha[^\circ] = 29.22 + 2.59 \cdot \Phi_3 \quad (2.15)$$

These simple mathematical formulas can be used as a starting point for clad geometry prediction and clearly show a linear dependency between the complex process parameters proposed and the clad geometry properties studied [57].

2.6 DED process control

The control of the build conditions is essential to obtain consistent results across different complex part geometries and processing constraints, and as such, research has been conducted in order to attempt to closely monitor and impose those relevant conditions. They are defined as the thermal conditions necessary for a part to be built, where consistent build conditions are closely related to a consistent melt pool size and temperature [58–60]. Therefore, having a stable melt pool is necessary for a well controlled process.

Vasinonta et al. [59] developed a control approach based on processing maps (for both thin wall and bulky structures), relating dimensionless variables (normalised melt pool length, normalised substrate height and normalised temperature) based on the analytical solution of the Rosenthal equation for a scanning point source of heat [59, 61], as follows:

$$T(x, y, z) = T_0 + \frac{P_{laser}}{2 \cdot \pi \cdot R} \cdot e^{\frac{-v \cdot (x+R)}{2 \cdot \alpha}} \quad (2.16)$$

Where T_0 is the ambient temperature, k and α are the average thermal conductivity and diffusivity of powder and substrate metal, and $R = \sqrt{x^2 + y^2 + z^2}$ is the distance from the point of interest to the heat source.

Closed loop control, however, is desirable as it reduces the tuning and testing required in order to obtain a stable and repeatable process, and external disturbances' effects are reduced.[58] It is necessary to integrate a sensing system in the DED process to implement closed-loop control, and for that purpose thermal imaging cameras are widely used, allowing for both melt pool geometry and temperature measurements. Furthermore, they can be mounted coaxially to the laser fibre in order to obtain overhead images of the melt pool. Having those infrared images of the melt pool allows for measurement of its temperature distribution and melt pool size and, for example, adjust the laser power or velocity accordingly to maintain a given target temperature. [58]

Closed-loop control has been demonstrated successfully in DED, especially in blown powder and laser setups, although literature is still scarce. Nonetheless, advances have been made more recently in this regard: Gibson et al. [58] and Baraldo et al. [62] found that closed loop control by modulating laser power for a target MPS yielded more consistent results for thin walled geometries when compared to open loop control. Furthermore, it was observed that the calibration of the thermal imaging camera for exact temperature and MPS measurements was not necessary as relative measurements are adequate as long as they're consistent throughout the build.

Chapter 3

Experimental

3.1 Materials and Methods

3.1.1 Materials

The powder used for the cladding trials was Oerlikon MetcoClad 316L-Si, a gas atomised 316L-Si powder designed for laser cladding and AM applications. It's composition can be seen in table 3.1, and it's nominal particle size distribution range in Table 3.2 [63]. In Figure 3.1 a representative image of the morphology found in this powder is shown.

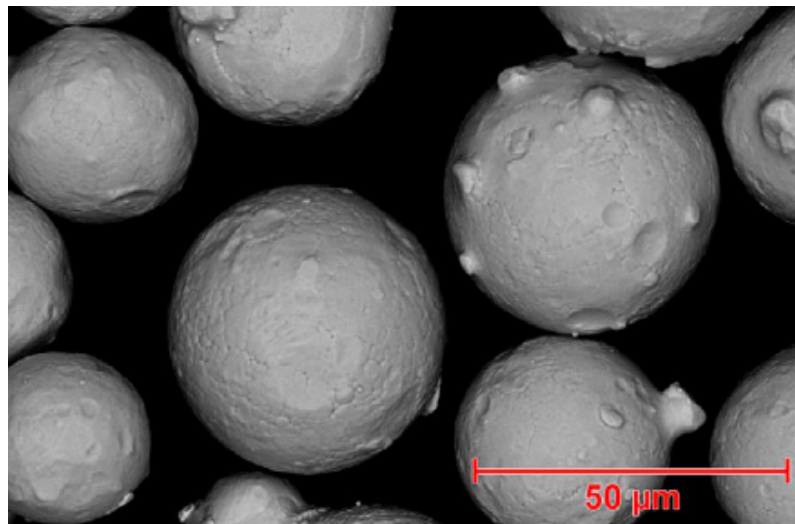


Figure 3.1: Typical Morphology of MetcoClad 316L-SI Powder [63]

Similarly to 316L, 316L-Si possesses excellent corrosion and pitting resistance, and for that reason it is widely used in marine, food and medical applications. The added silicon present in 316L-Si, however, acts as a fluxing agent, improving the clad quality and, as mentioned before, promotes the fixation of delta ferrite thus preventing the occurrence of hot cracking in the clad

parts. [63, 64].

Table 3.1: Composition of the 316L-Si powder used [63]

	Fe	Ni	Cr	Mo	Si	Mn	C
MetcoClad 316L-Si	Balance	12.0	17.0	2.5	2.3	1.0	0.03

Table 3.2: Particle size distribution of the 316L-Si powder used [63]

Nominal Range [μm]	Particle Size Distribution		
	+106 [μm]	-106 +44 [μm]	-44 [μm]
-106 +44	5	90	5

The substrate used in all of the depositions was a 20mm hot rolled plate of CK45 medium carbon steel (AISI 1045).

3.1.2 DED Setup

The DED setup used for the experimental procedures consisted of a KUKA (Germany) robotic arm equipped with a Fraunhofer IWS (Germany) COAX12V6 Laser Cladding Head, a cladding unit built for metallic components repair, surface protection deposition and AM. This cladding head is equipped with 4 discrete nozzles for powder delivery and allows for compensation of the powder delivery in each nozzle to obtain a homogeneous powder flow at the focal point, independently of the build direction. Furthermore, the whole nozzle assembly is water cooled which allows for continuous operation at up to 6KW of laser power, being compatible with Nd:YAG, fiber, disk(solid-state) or diode lasers [65]. As the cladding head allows for the use of shield gas, Argon will be used in the present work. In Figure 3.2 the cladding head is shown by itself and mounted on the robotic arm used.

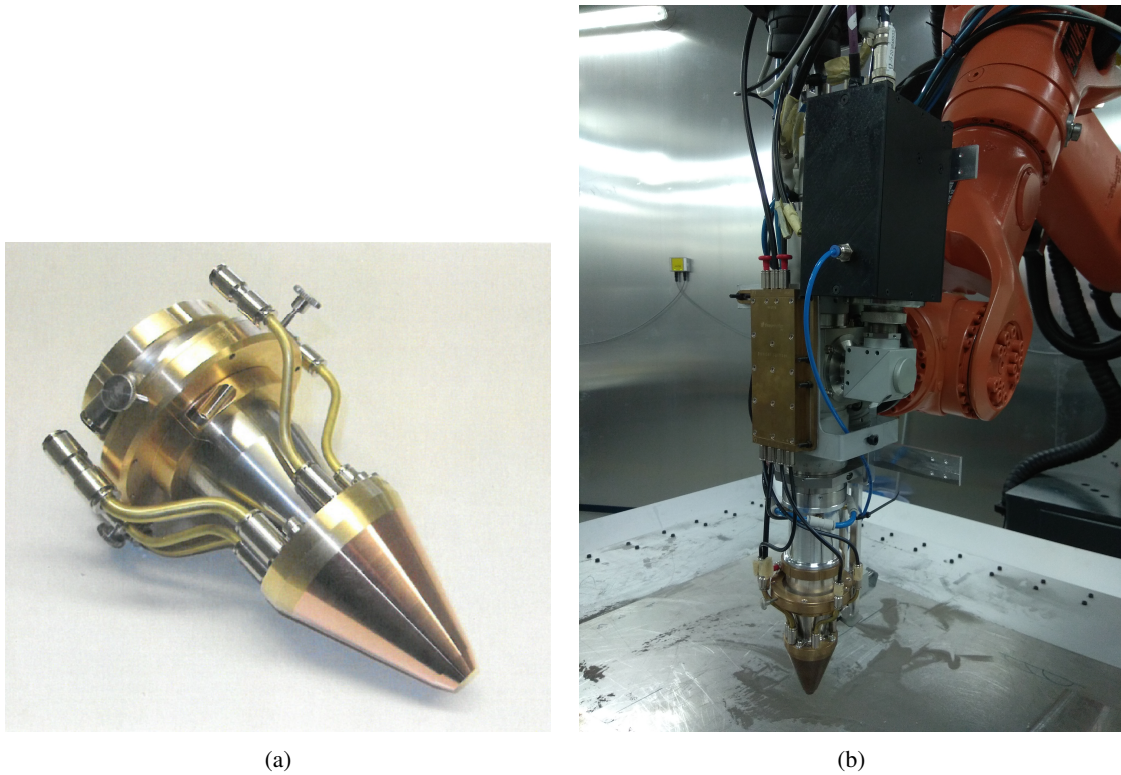


Figure 3.2: COAX12V6 Laser Cladding Head by itself and mounted on the KUKA robotic arm [65]

The laser system used is a Coherent HighLight FL 3000, a fiber laser capable of delivering up to 3KW with an accuracy of $\pm 1\%$ at 1070 ± 10 nm. Being a fiber laser, it is able to output a laser beam with an excellent BPP of 3.0 to $10.0 \text{ mm} \cdot \text{rad}$, depending on other factors [66].



Figure 3.3: Coherent HighLight FL 3000 laser system used

The power feed system was comprised of two independent Medicoat AG Disk Feeder systems, which offer stable and pulsation free powder flow from 0.5 to 100 g/min, carried by an inert gas of choice [67].

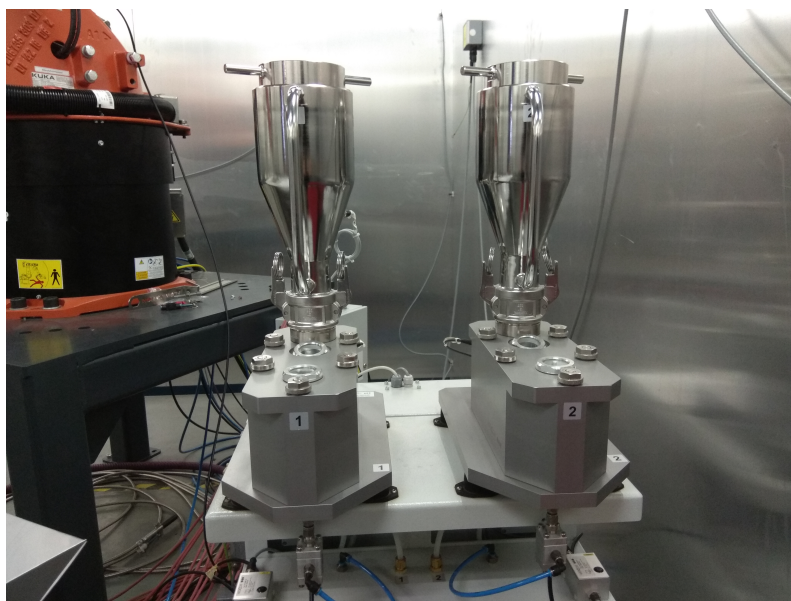
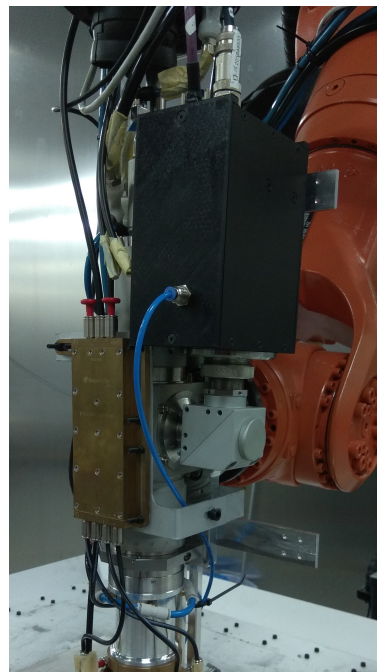
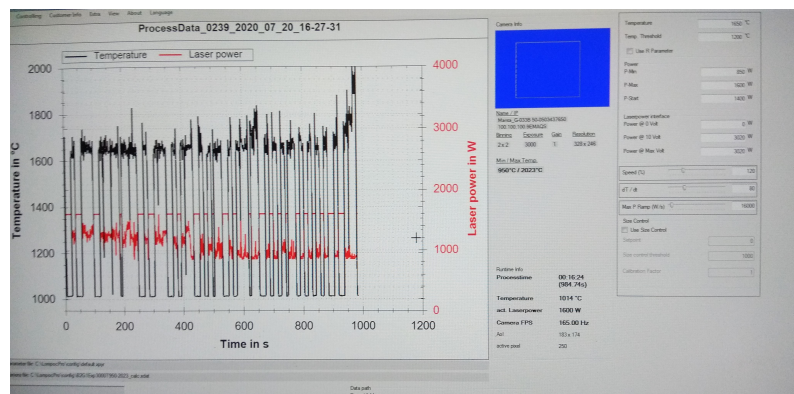


Figure 3.4: Medicoat AG powder feeders used

The cladding head was equipped with an E-MAqS Temperature Measuring system which, coupled with the LompocPro 8 software, allows for the closed-loop control of the DED process. The E-MAqS system uses a thermal sensing camera to measure the melt pool temperature distribution and feeds those values to the control software, LompocPro 8, which uses it to modulate the applied laser power, according to a number of given parameters, maintaining a target melt pool temperature [68, 69]. The main parameters to be set are the Target Temperature (the target temperature for the melt pool), Minimum and Maximum Power (the lower and upper thresholds for the applied laser power modulation) and Starting Power (the initial power value for every deposition step). The E-MAqS system's hardware and LompocPro's interface can be seen in Figure 3.5.



(a)



(b)

Figure 3.5: EMAqS system, mounted on the cladding head, and LompocPro 8 software interface

A complete view of the DED system can be seen in the following Figure 3.6.



Figure 3.6: DED setup used

3.1.3 Optical Microscopy

For the purpose of microstructural analysis by optical microscopy the samples were polished up to a $1\ \mu\text{g}$ diamond suspension and then etched with Kalling's No.2 reagent.

Kalling's No.2 reagent, also known as waterless Kalling's, is an etchant used for 300 and 400 series stainless steels or Ni-Cu alloys and superalloys which quickly attacks ferrite, slightly attacks austenite and does not interact with carbides [70].

In this work the specimens analysed were always composed of a 316L-Si deposition onto a CK45 substrate. With CK45 being a carbon steel care was taken as to only allow for the etchant to interact with it briefly, as it is more heavily attacked by the reagent than the stainless steel.

The optical microscope used was an Olympus[®] PMG 3 (Japan) inverted microscope.

3.1.4 Tensile tests

The tensile tests were carried out in a Shimadzu[®] EZ-LX Universal Tensile Tester (Japan) with a 5KN load cell, at 5 mm/min. The extension values were measured using a Shimadzu TRViewX Digital Video Extensometer, which tracks two horizontal stripes drawn in the specimens using a video camera.

The design of the tensile specimens was based on the geometry specified in ASTM E606-92 [71] but, due to the size constraints derived from the small deposition samples available, had to

3.1.5 Microhardness Measurements

The microhardness measurements were done using a Struer[®] Duramin microhardness tester, pictured in Figure 3.9, using a Vickers indenter at 300 grams of force for 15 seconds. The sample was swept 5 times, taking 9 measurements at 0.2 mm intervals with each sweep: 3 of the substrate, 3 of the HAZ and 3 of the clad material.



(a)

Figure 3.9: Struers[®] Duramin microhardness tester used

3.2 Experimental trials

3.2.1 Single line depositions

3.2.1.1 Preliminary tests 1

In order to find a preliminary working range for the 316L-Si powder with the DED setup available a series of single line clads were done on a 20mm CK45 steel plate. The experiment was based on an L9 Taguchi array.

The chosen factors for the experiment were the laser power, scanning speed and powder feed rate. Both the laser power and scanning speed were chosen because they directly influence the power input and therefore the cladding quality and geometry. The power feed rate was chosen because it plays an important role in the clad height and porosity characteristics.

The levels for these factor were based on the values available in literature for 316L in similar setups and chosen in order to sweep energy density values from 10 to 60 J/mm^2 , which is representative of the diverse values used in literature and should provide information regarding the lower limit and optimal value of power input in order to obtain melting.

To achieve that energy range the Taguchi factors and levels shown in Figure 3.10 were considered:

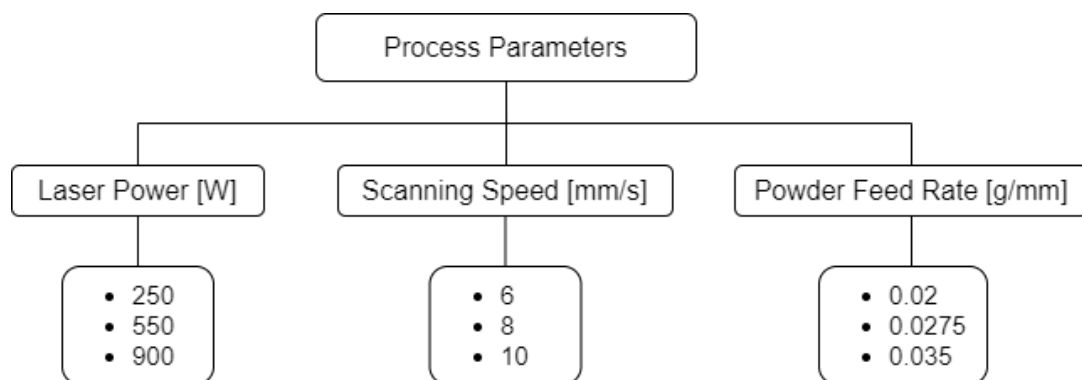


Figure 3.10: Taguchi factors and levels considered

From which it is possible to create the Taguchi array presented in table 3.3:

Table 3.3: L9 Taguchi array for trial 1

Condition	Laser power [W]	Scan speed [mm/s]	Powder feed [g/mm]	E [J/mm^2] ¹	h* ¹
1	300	6	0.02	20.0	1.31
2	300	8	0.0275	15.0	1.14
3	300	10	0.035	12.0	1.02
4	550	6	0.0275	36.7	2.41
5	550	8	0.035	27.5	2.09
6	550	10	0.02	22.0	1.87
7	900	6	0.035	60.0	3.94
8	900	8	0.02	45.0	3.41
9	900	10	0.0275	36.0	3.05

¹ Calculated for $d_{spot} = 2,5\text{mm}$, not a factor

The powder feed values were determined as a linear powder feed rate, in g/mm units, as it more consistent from a physical standpoint: to achieve a certain clad area enough powder must be supplied on a spatial basis and not a temporal one.

The shield gas flow rate was maintained at 25 l/min, the carrier gas flow rate at 5l/min and the spot size at $d_{spot} = 2,5\text{mm}$ for all conditions. It should be noted that the gas flow rates vary greatly between different DED systems due to the different cladding heads, powder delivery systems and nozzle geometries used. In this case, the values were chosen as they have previously been used with success with this setup.

In this first series of trials 9 clads were deposited, each 50mm in length, 10mm apart from each other and the substrate was kept at ambient temperature in order to obtain baseline values.

3.2.1.2 Results

For conditions 1 to 3, which employed 300W of laser power and energy densities ranging from 12 to $20 J/mm^2$, no clad was achieved. For conditions 4 to 6, with 550W of laser power, discontinuous clads were achieved with little to no penetration. Finally, with the highest laser power of 900W, successful clads were obtained for conditions 7 and 8, with energy densities of 60 and $45 J/mm^2$ respectively, but not for 9, with an energy density of $36 J/mm^2$.

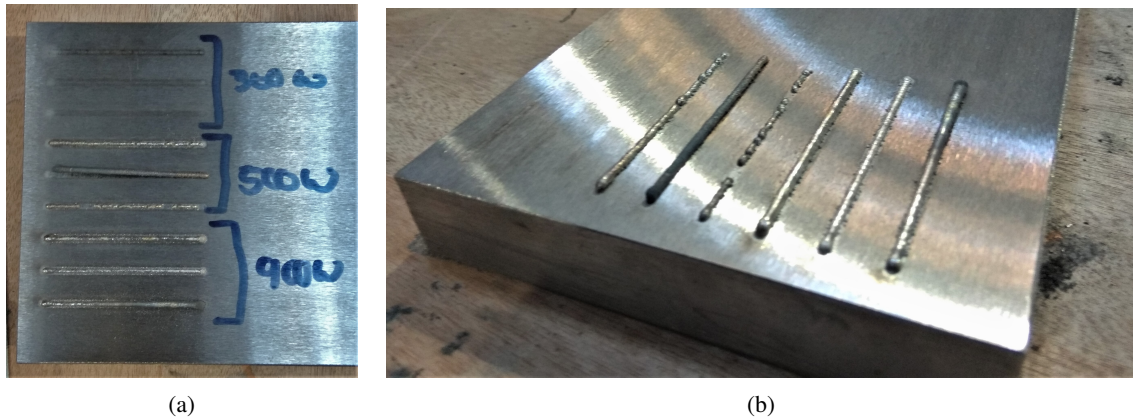


Figure 3.11: Trial 1 clads

Successful clads were obtained above $E=45 \text{ J/mm}^2$ and $h^* = 3.41$. Having only obtained passable results for those power input conditions, a new set trials was done, with updated power values as the initial range considered proved to be too low.

3.2.1.3 Preliminary tests 2

This second set of clads used the same scanning speed and powder feed rate values as in trial 1, but with the laser power levels being 1000, 1250 and 1500W in order to scan energy densities up to 100 J/mm^2 . This resulted in the following L9 Taguchi array in table 3.4:

Table 3.4: L9 Taguchi array for trial 2

Condition	Laser power [W]	Scan speed [mm/s]	Powder feed [g/mm]	$E [\text{J/mm}^2]^1$	h^*^1
1	1000	6	0.0200	66.7	4.38
2	1000	8	0.0275	50.0	3.79
3	1000	10	0.0350	40.0	3.39
4	1250	6	0.0275	83.3	5.48
5	1250	8	0.0350	62.5	4.74
6	1250	10	0.0200	50.0	4.24
7	1500	6	0.0350	100.0	6.57
8	1500	8	0.0200	75.0	5.69
9	1500	10	0.0275	60.0	5.09

¹ Calculated for $d_{spot} = 2,5\text{mm}$, not a factor

This range of energy input values results in an overlap with the higher range of energy input values tested in trial 1 but extends it further up to 100 J/mm^2 .

As before, the protective gas flow rate was maintained at 25 l/min, the carrier gas flow rate at 5l/min and the spot size at $d_{spot} = 2,5\text{mm}$ for all conditions.

The 9 clads were also deposited with 50mm in length, 10mm apart from each other and the substrate was kept at ambient temperature.

3.2.1.4 Results

As seen in Figure 3.12, the depositions were successful with the exception of conditions 1 and 3. Condition 1 was the first clad produced and because of it suffered from lack of powder, due to a startup delay in the powder feeding system. The condition 3 clad, with the lowest power input of 40 J/mm^2 , detached from the surface after being deposited due to lack of fusion.

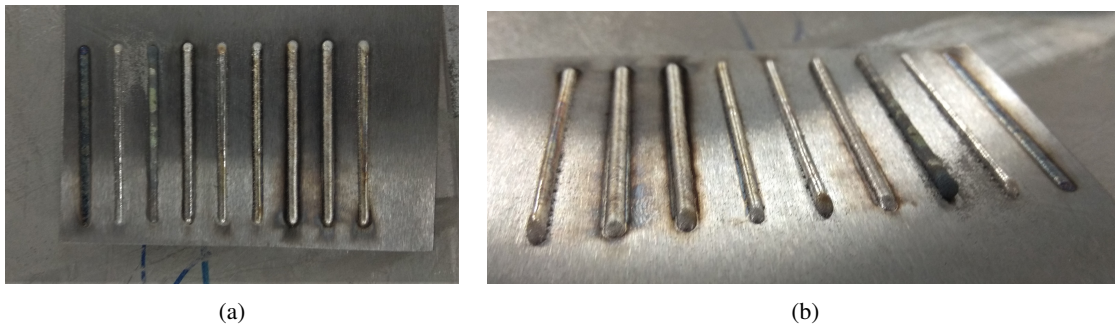


Figure 3.12: Trial 2 clads

The clads were then cut perpendicular to their deposition direction, on a representative area of the clad, in order to analyse their cross section. Images of the cross section were taken at 23x magnification on a Leica optical microscope, and from there the clad geometry was measured and powder deposition efficiency calculated as seen in equation 3.1. These measurements are shown in Table 3.5, where w is the deposition width, h the deposition height and A the cross sectional area of the deposition.

Table 3.5: Single clads trial 2 results

Condition	w [mm]	h [mm]	A [mm ²]	Deposition rate [g/min]	Powder eff. [%]
2	1.55	1.35	1.95	5.55	42
4	2.16	1.48	2.74	7.79	79
5	1.94	1.35	2.10	5.97	36
6	2.03	1.17	2.85	8.10	68
7	2.64	1.49	3.20	9.10	72
8	2.65	1.09	2.23	6.34	66
9	2.10	1.37	2.14	6.09	37

$$\text{powder efficiency} = \frac{f}{A \cdot \rho} \quad (3.1)$$

While condition 4 yielded the highest deposition efficiency, condition 7 resulted in the most consistent clad, with the highest deposition rate, good geometry, better penetration overall and a high 72% powder deposition efficiency. A representative cross section of condition 7 can be seen in figure 3.13. Note a darker semi circle in the substrate, the heat affected zone (HAZ).

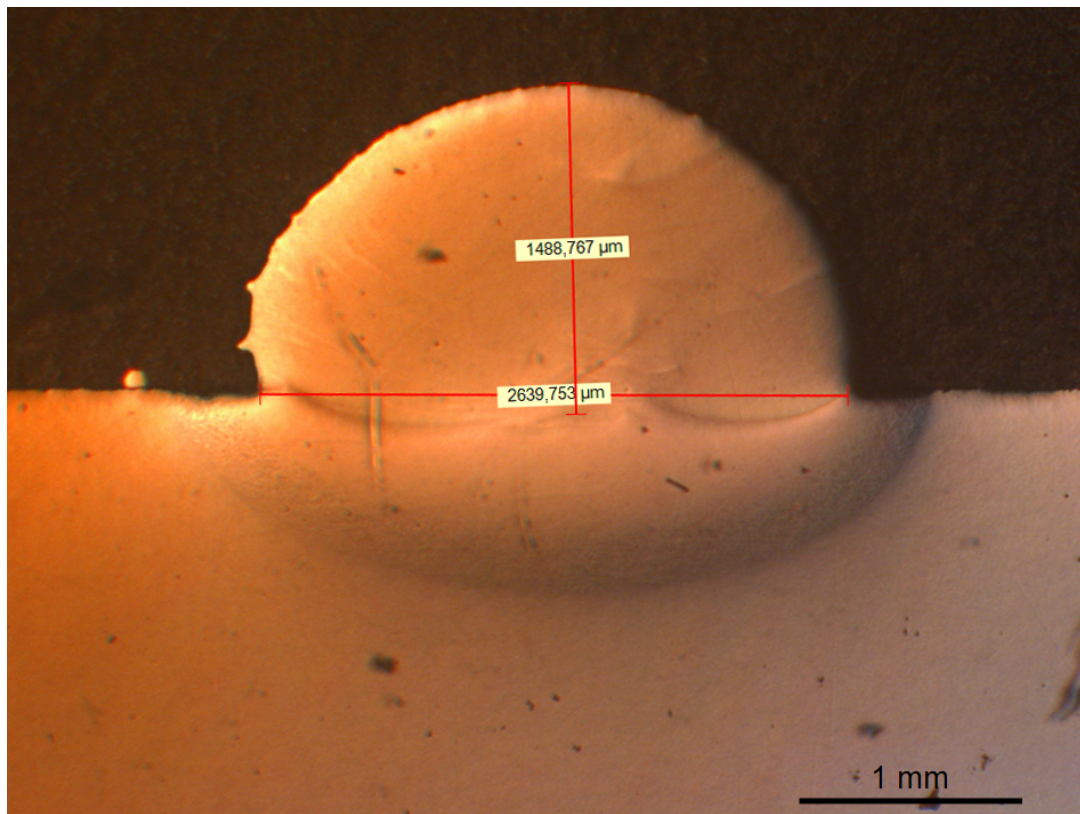
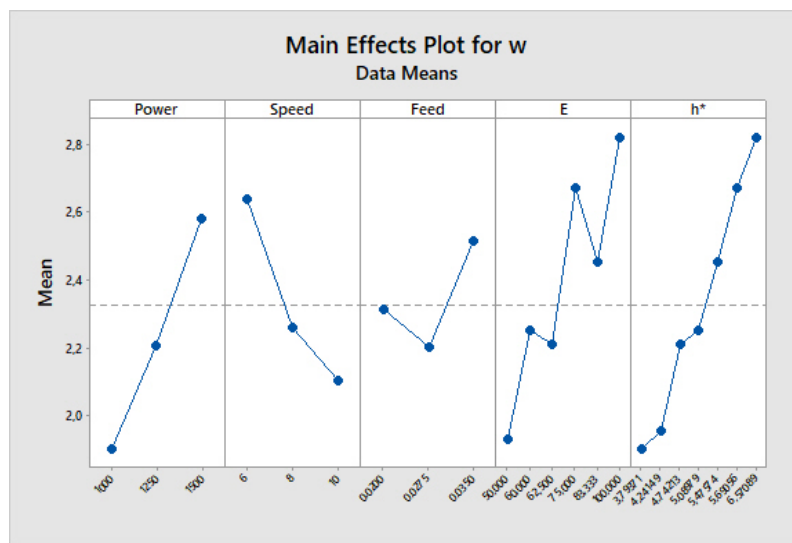
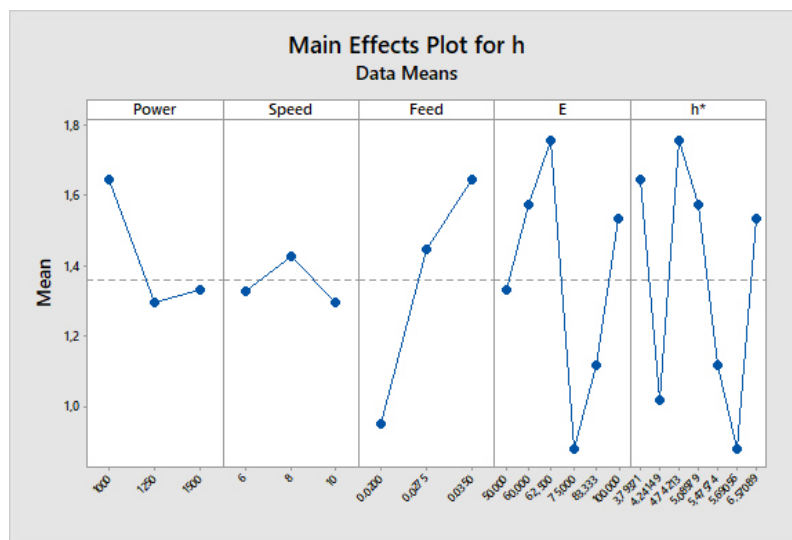


Figure 3.13: Optical microscopy image of condition 7 clad

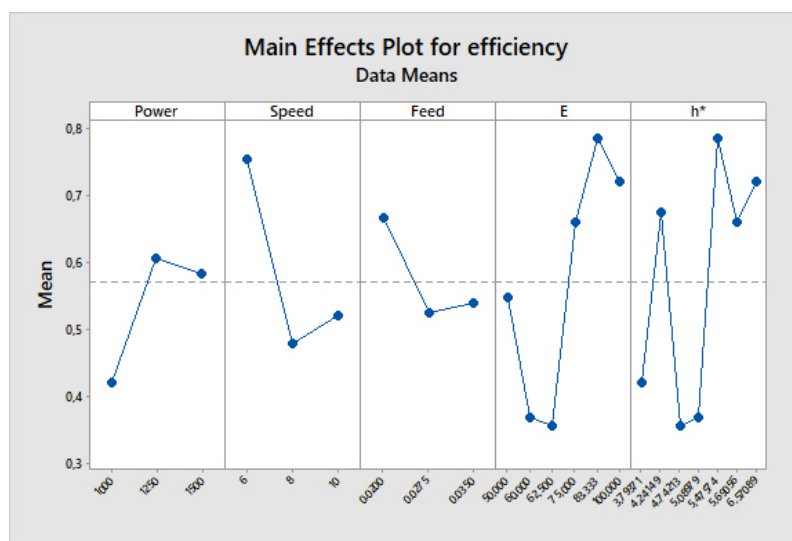
Now that the responses for the Taguchi array have been calculated (clad width, height and efficiency) the main effects plot can be analysed, shown in Figure 3.14.



(a)



(b)



(c)

Figure 3.14: Main effects plot for the second Taguchi matrix

It is clear how the clad width increases as the power input increases (when the laser power, E or h^* increases or the speed decreases). Regarding the clad height, the only factor which induces a clear effect is the powder feed rate, increasing the clad height as it increases. As for the efficiency, the main effects are scattered and a conclusion cannot be drawn from any of the factors.

3.2.2 3D Depositions

Having obtained stable clads and reasonable penetration in the single line clad trials, a set of processing parameters was chosen based on the previous condition 7. The next step is to attempt to deposit a 3 dimensional part in order to assess the applicability of the previously used parameters to create volumes.

The chosen processing parameters are as shown in Table 3.6:

Table 3.6: Parameters used for the first set of 3D clads

Power [P]	Speed [mm/s]	Powder feed [g/min]	$E [J/mm^2]$	h^*
1500	6	12.5	100	6.57

To define the layer height for the 3D depositions two data points were considered: the height obtained for the single line clad (h_C), and a proposed theoretical height of a rectangular clad with identical width and area to the one deposited (h_R), where $A_C = A_R$. This can be more clearly understood by analysing Figure 3.15.

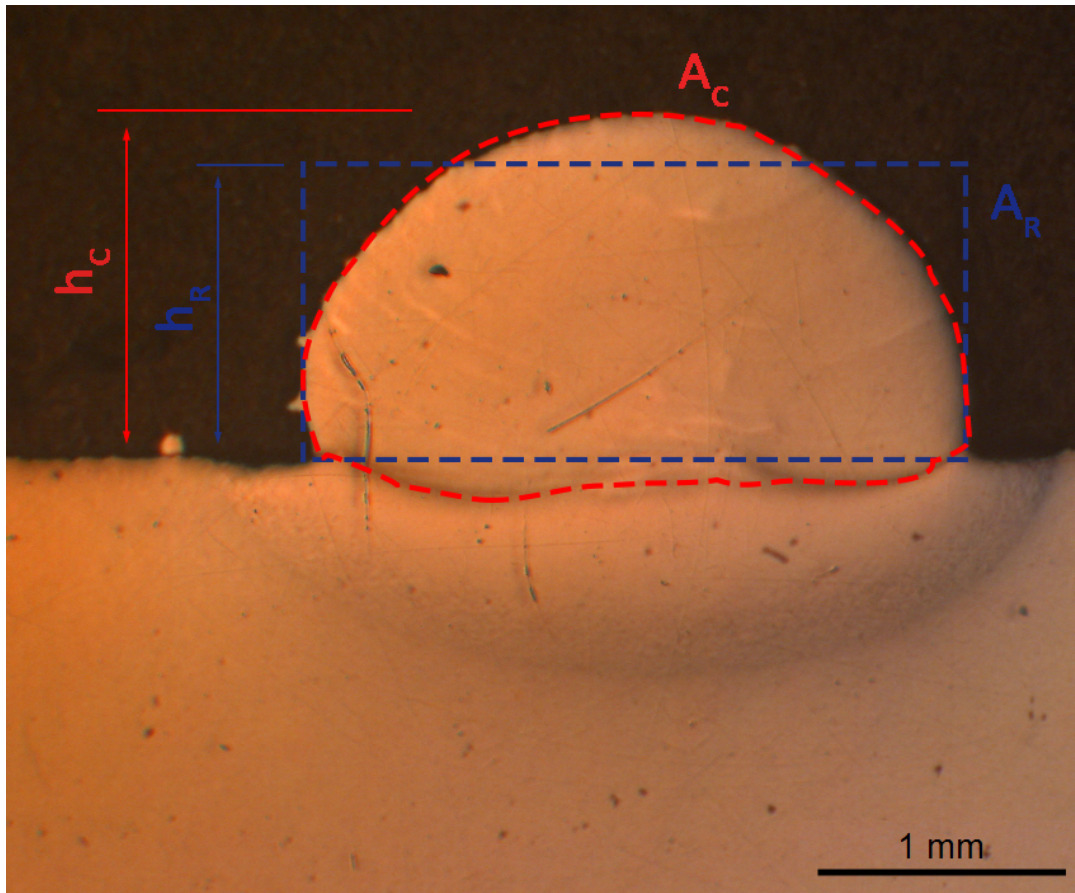


Figure 3.15: Clad height considerations for layer height

The calculation of h_R is based on the approximation that after the following clad is deposited on top of the previous one, the previous clad will flatten while maintaining the same cross sectional area and become close to a rectangle.

This results in $h_C = 1,49\text{mm}$ and $h_R = \frac{A_C}{w} = 1,21\text{mm}$. The optimal value for the layer height should therefore be between 1,21mm and 1,49mm, assuming the powder efficiency remains roughly the same between these two values. The average value of $h = 1,35\text{mm}$ was used as the layer height for the first 3D trial.

3.2.2.1 Cylinder perimeter depositions

The geometry chosen for the first 3D depositions was a hollow cylinder as it allows for the clear analysis of the interactions between the deposited layers and for the validation of the layer height chosen. Furthermore, this smooth geometry is devoid of sharp features which would result in heat concentration areas and therefore inconsistencies in the built layers. In figure 3.16 the toolpaths and final results for the three cylinders built are shown. All three have a diameter of 35mm.

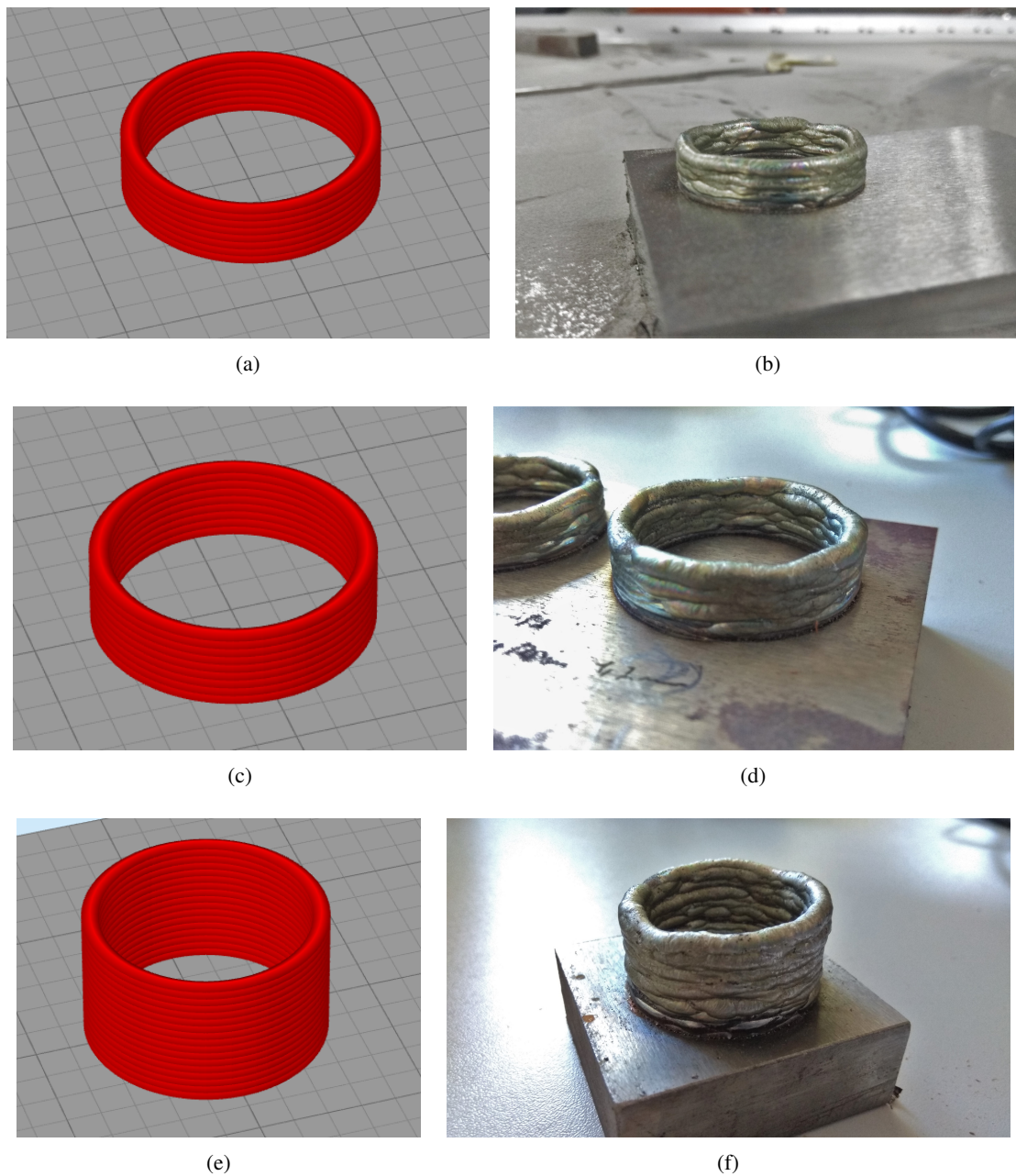


Figure 3.16: Toolpaths (Simplify3D¹) and final result for the test cylinders built

The first build is shown in figure 3.16 (a) and (b). The cylinder, with a target height of 10mm, was comprised of 8 layers at 1.35mm layer height. For the following cylinder, shown in figure 3.16 (c) and (d), in an attempt to find a more stable layer height it was brought down to 1.2mm. This h reduction resulted in a 9 layer build. Having successfully reached the intended height another cylinder was built with a height of 20mm to validate the previous parameters. The toolpath and end result can be seen in figure 3.16 (e) and (f).

¹simplify3d.com

The height measurements for these 3 cylinders are shown in Table 3.7.

Table 3.7: Height and height error measurements for the test cylinders

Build	h [mm]	H target [mm]	H measured [mm]	error/layer [%]
1	1.35	10	8.55	13
2	1.2	10	9.94	0.56
3		20	17.89	10

As can be seen in the previous Table 3.7, the layer height change from 1,35 to 1,2mm yielded a much more accurate build height for the 10mm cylinders but the 20mm cylinder's height still fell short of the target, likely due to accumulated error of the subsequent layers and heat buildup in the part. It is apparent in all three cylinders how the deposition of single stacked clads leads to inconsistent deposition, even when the target height is achieved as seen in figure 3.17.



Figure 3.17: Side view of the built cylinders, in chronological order from left to right

3.2.2.2 Wall depositions

Having obtained reasonable results for the cylindrical geometry, attempts were made to build a vertical wall in order to then extract samples for microstructure analysis and tensile test specimens from it. The geometry chosen was a 80mm long wall comprised of two overlapping clads, with a total height of 20mm at first, to validate the chosen parameters. The overlap between the two clads was set at 30%.

The results of the first wall deposition can be seen in figure 3.18.a and b.

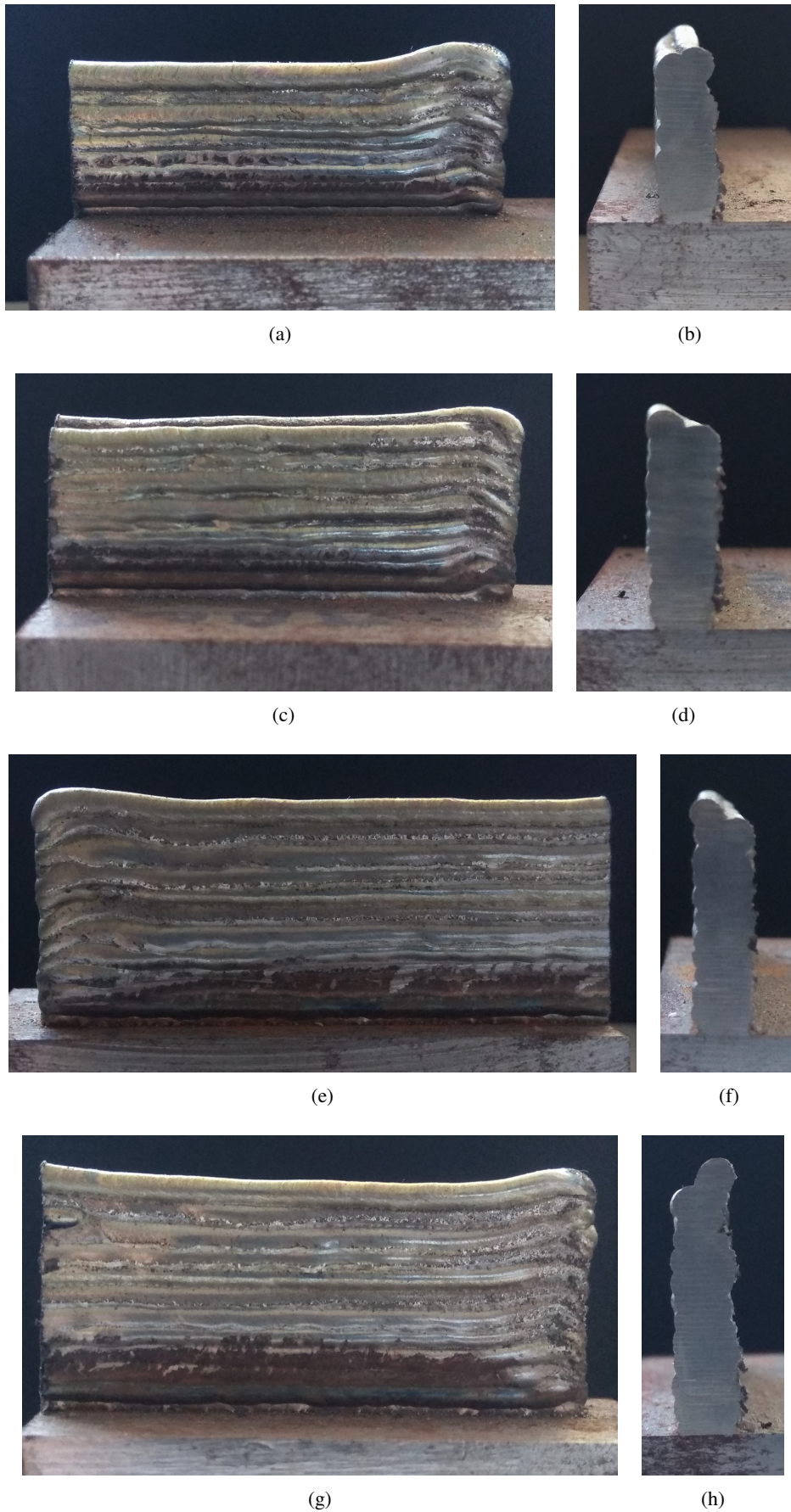


Figure 3.18: 20mm height wall deposition results, in chronological order, side and transverse view

As observed in the previous depositions the layers were still inconsistent and a raised section of over-deposition was created at the ends of the wall, where the cladding head changes direction and therefore increasing the energy and powder input locally, as the laser power and powder feed remain constant. This over-deposition effect has been observed by other authors [43].

Furthermore, deposited layers become more and more uneven as more layers are deposited, possibly due to the temperature of the workpiece increasing during the deposition and thus allowing the melt pool to reach higher temperatures than expected after the first few layers.

As an attempt to reduce these defects the closed-loop control system, E-MAqS, was activated. In Figure 3.18.c and d the result for the same wall geometry but produced with closed-loop control is shown. The temperature and power modulation graph during the deposition for both the first (open-loop) and second (closed-loop) walls is shown in Figure 3.19.a and b respectively, with a linear regression drawn. The melt pool temperature evolution is shown in blue and, for Figure 3.19.b, the power modulation is shown in red. The average melt pool temperature, laser power and layer time is shown at the top of the graphs.

In the first wall deposition the melt pool temperature rises from 1730°C to more than 1800°C by the end of the deposition, indicating that the processing conditions at the start and at the end of the deposition are distinct.

The temperature and power targets for the E-MAqS system can be seen in Table 3.8, where "T target" is the desired melt pool temperature throughout the cladding process, "P min" and "P max" are the lower and upper limits for the laser power modulation and "P start" is the laser power at the start of each layer.

Table 3.8: E-MAqS closed-loop settings

T target [°C]	P min [W]	P max [W]	P start [W]
1700	1150	1600	1400

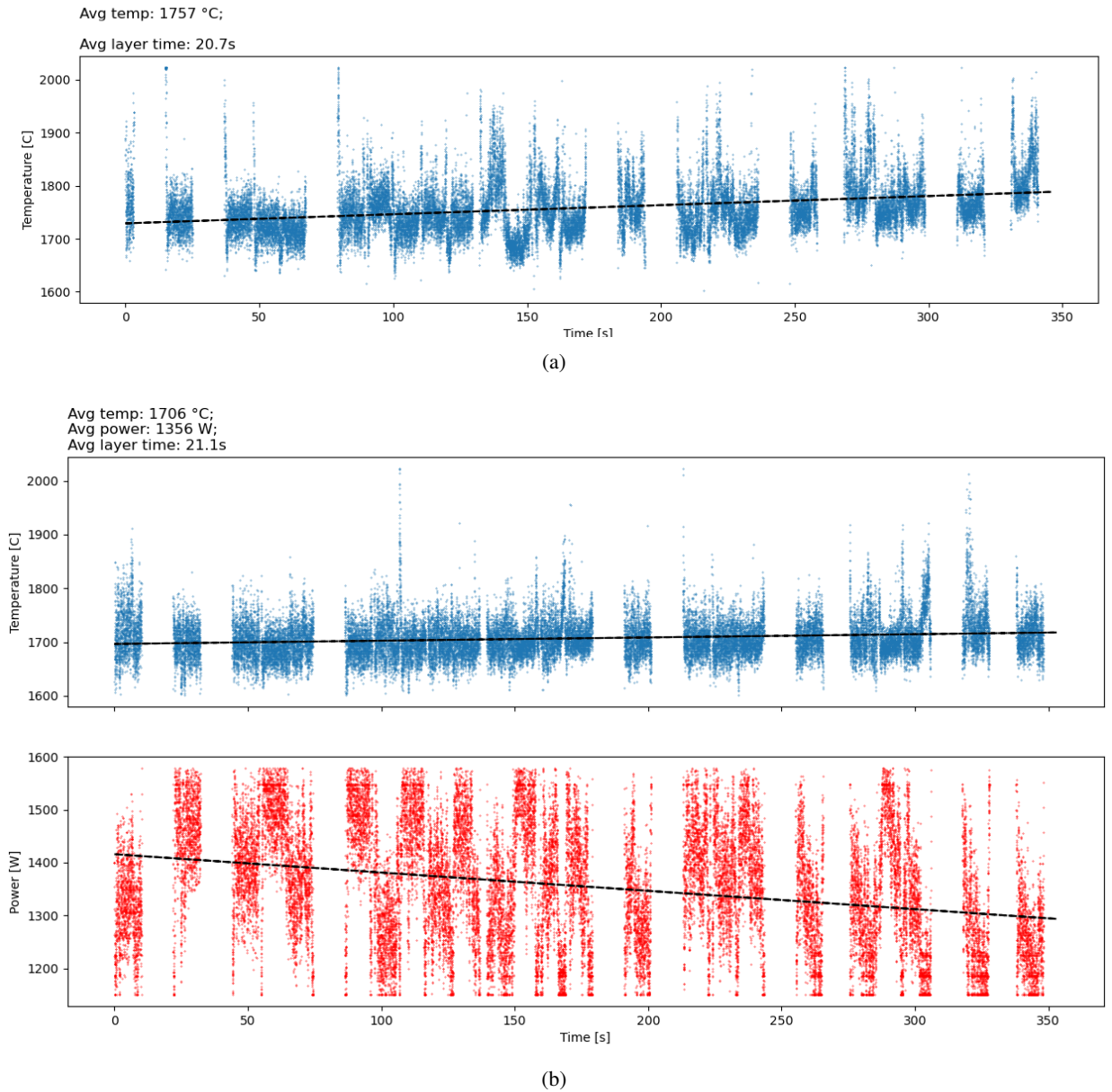


Figure 3.19: Temperature and power modulation graphs during the deposition of the first two wall clads

With the use of the closed-loop control system described above the obtained height of the wall was much closer to the intended 20mm, with more consistent layers throughout the build and the discontinuities as the edges of the wall were reduced.

The two following deposited walls, shown in Figure 3.18.e through h, were built with similar settings to the previous one, making only small adjustments to the E-MAqS parameters. The parameters used and resulting average height and width measurements of all 4 walls can be seen in Table 3.9. The average widths and heights were calculated by taking six measurements along the length of each wall. All of the walls built showed over-deposition at the edges, although the

use of closed-loop control reduced it's impact.

Table 3.9: Process and E-MAqS parameters used and resulting average width and height measurements, for each wall 20mm deposited

Wall#	v[mm/s]	f[g/min]	T target[°C]	Pmin[W]	Pmax[W]	Pstart[W]	h[mm]	w[mm]
1	6	12.5	-	-	-	-	16.60	5.94
2	6	12.5	1700	1150	1600	1400	18.46	5.69
3	6	12.5	1650	750	1600	1400	19.98	4.98
4	6	12.5	1650	650	1600	1250	19.88	5.04

Between these sets of parameters, the ones that yielded better results were the ones used for walls 3 and 4. The drop in "P min" and "P start" from wall 3 to wall 4 did not have a clear measurable impact in the dimensions of the wall, although wall 3 does appear to have more uniform layers. For that reason, the conditions used for that deposition were then reused to attempt to build a similar wall but with a total height of 40mm, suitable to allow for the machining of microtensile specimens for both the horizontal and transverse directions.

The first 40mm wall deposited can be seen in Figure 3.21.a and b. Although the target height of 40mm was reached with some accuracy (shown in Table 3.10), the wall showed lack of fusion in many of the topmost layers. Initially it was thought to be due to excess heat accumulation, overcoming the closed-loop control's ability to provide low enough laser power to maintain the target melt pool temperature. As such, this 40mm wall deposition was repeated two more times, changing the minimum power setting in the E-MAqS parameters presented in Table 3.10, but arriving at the same result as seen in the following Figure 3.21.

Table 3.10: Process and E-MAqS parameters used and resulting average width and height measurements, for each 40mm wall deposited

Wall#	v[mm/s]	f[g/min]	T target[°C]	Pmin[W]	Pmax[W]	Pstart[W]	h[mm]	w[mm]
5	6	12,5	1650	750	1600	1400	39.99	5.31
6	6	12,5	1650	650	1600	1400	39.11	5.22
7	6	12,5	1650	850	1600	1400	34.56	5.19

After analysing the melt pool temperature and laser power evolution during the deposition, shown in Figure 3.20, it became clear the target melt pool temperature was being maintained, although with high spikes towards the end of each deposited layer. In any case, all of the depositions yielded unstable results.

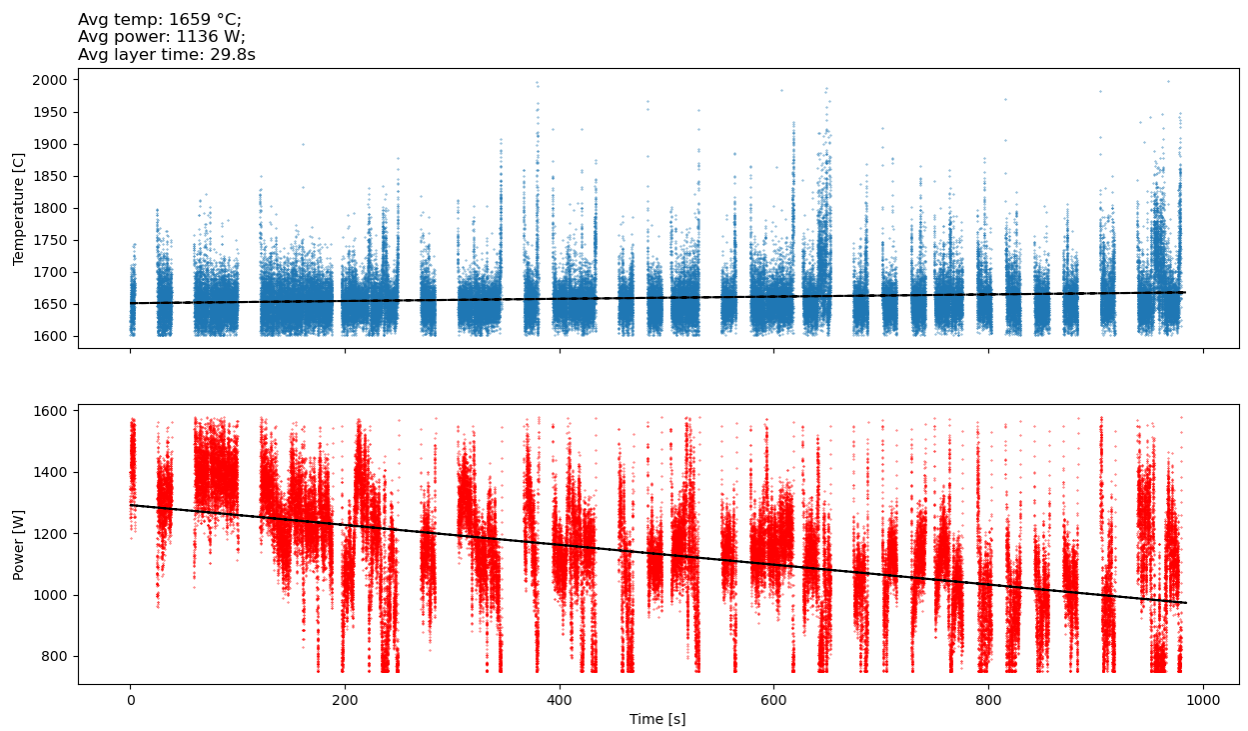


Figure 3.20: Temperature and power modulation graph during the deposition of the first 40mm wall clad (wall #6)

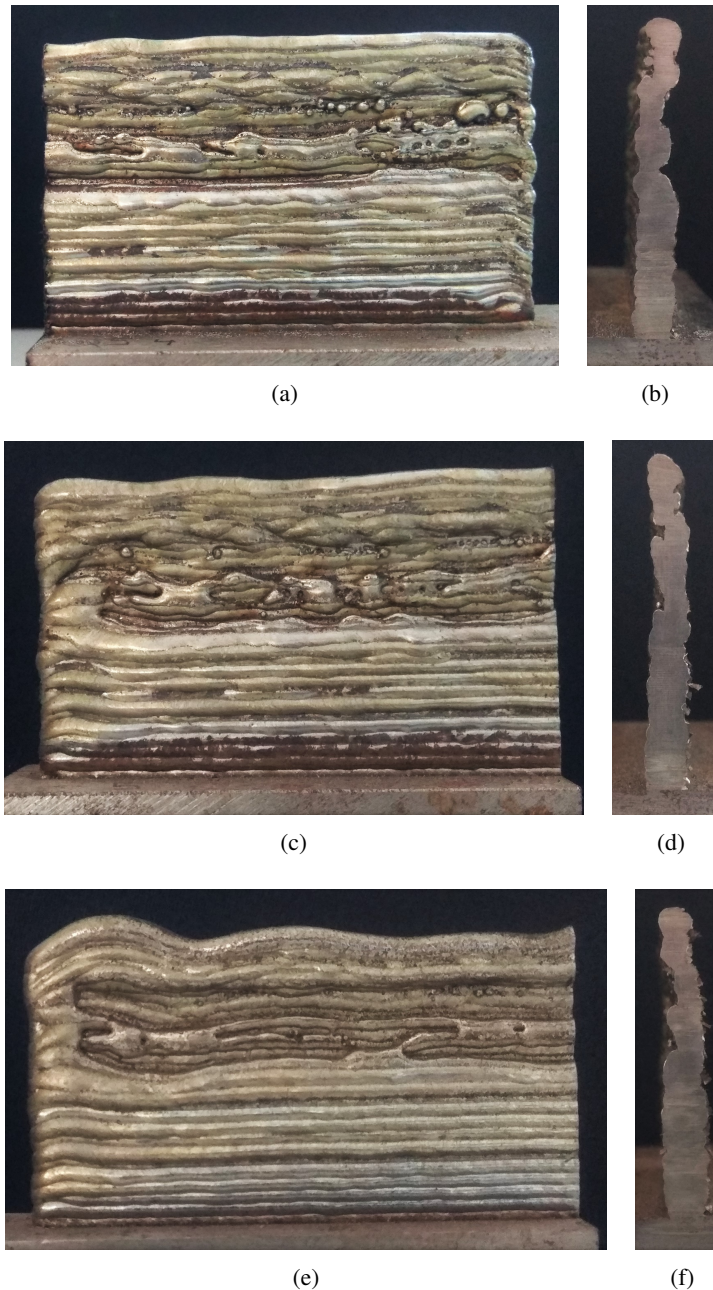


Figure 3.21: 40mm height wall deposition results, in chronological order, side and transverse view

Unable to obtain stable clads for the full height of the wall, a new thicker wall was designed with the same length and height of 80mm and 40mm, respectively, but consisting of 3 horizontal lines per layer instead of 2. This extra material should reduce the overall heat accumulation in the wall during the cladding process and add structure to support the melt pool, contributing to a more stable clad. The process parameters used were the same as shown in Table 3.9's wall #3. This resulted in the deposition shown in Figure 3.22.

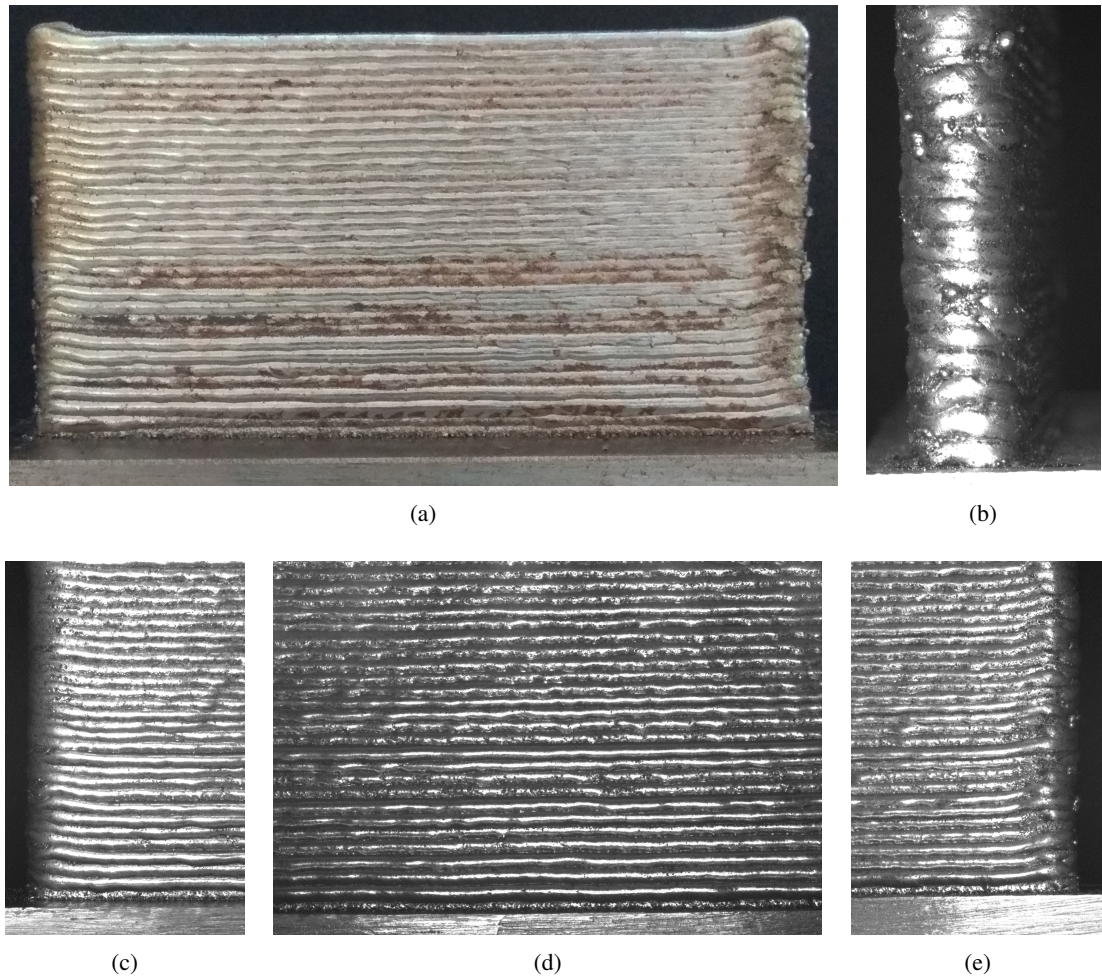


Figure 3.22: 3 line wall deposition results, with details

Unlike the previous 40mm walls built, this geometry resulted in a consistent deposition, with only minor over-deposition at the edges and minor surface defects, as can be seen in figure 3.22. Having obtained quality depositions for these conditions, more identical walls were built, seen in Figure 3.23, in order to them machine microtensile specimens from them.

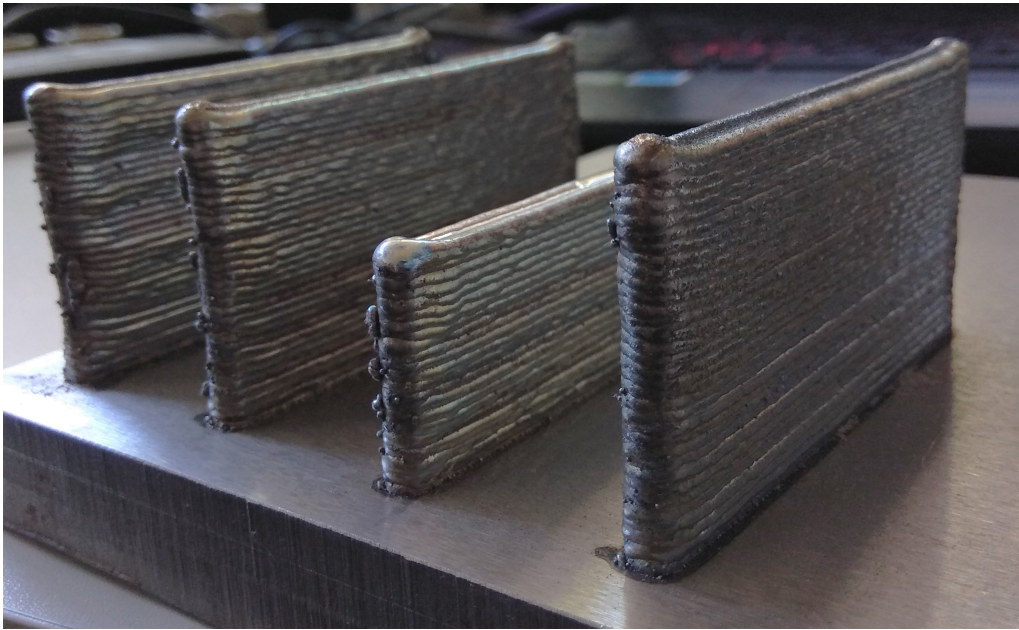


Figure 3.23: 3 line walls built

3.2.3 Microstructure analysis

A sample was taken from the 20mm wall #3, machined and then set in resin, shown in Figure 3.24, for both microhardness and microstructure analysis.

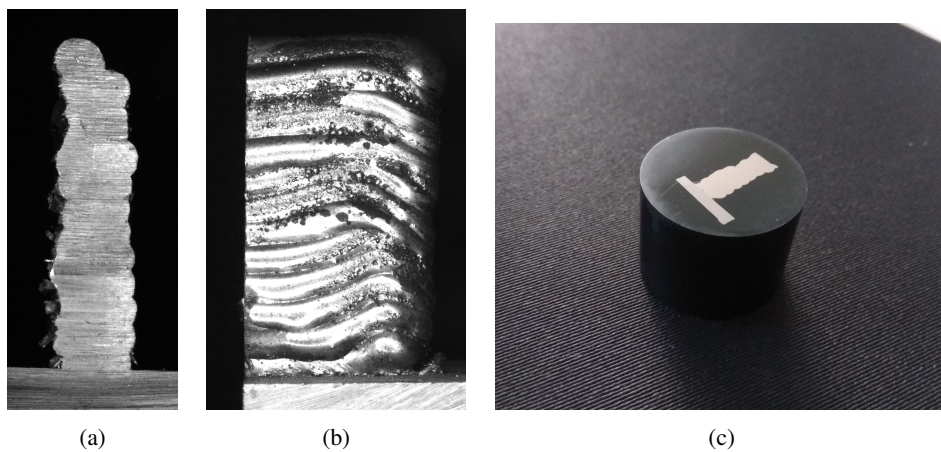


Figure 3.24: Wall #3 sample before and after being machined and set in resin for microstructure and microhardness analysis

Figure 3.25 shows representative optical microscopy images of the microstructure obtained, both in the fusion line, interfacing the substrate and the clad, and higher up, with Figure 3.25.b being a detail of 3.25.a and 3.25.d being a detail of 3.25.c.

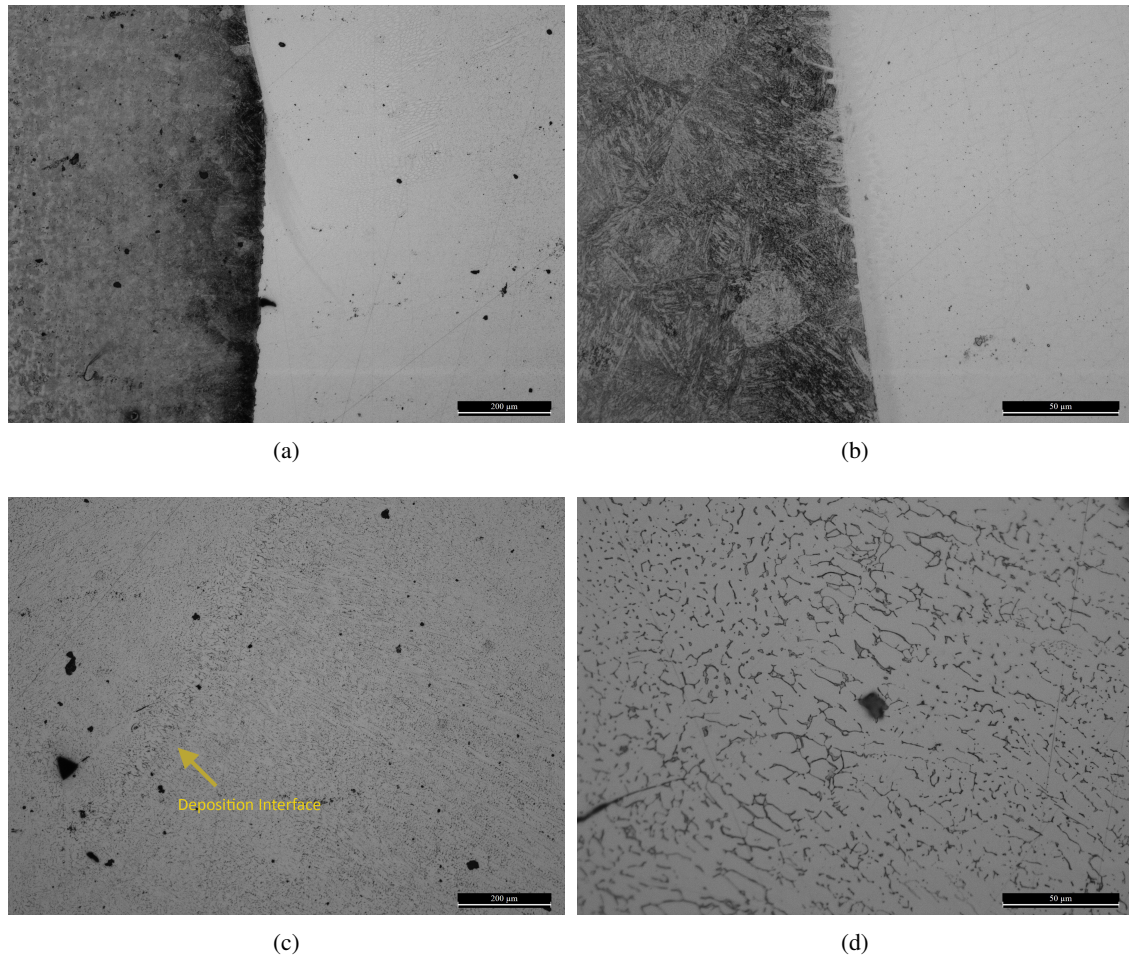


Figure 3.25: Microstructure representative of the interest areas present in the sample

The deposited metal shows a fine structure, characteristic of the high cooling rates present in the DED process, of austenitic dendrites surrounded by interdendritic delta ferrite. In Figure 3.25.a and b the fusion line is shown, with a tempered martensitic structure present in the substrate's HAZ, showing a large carbon concentration at the interface.

In Figure 3.25.c and d the interface between two successive deposited layers is shown, identified by the line of equiaxed grains running across Figure 3.25.c, pointed out by the yellow arrow. In 3.25.d a close up of those equiaxed grains in the interface is shown. Ahead of the interface the grains grow epitaxially towards the top of the deposition, in the direction of greater thermal gradient, as expected.

Voids and inclusions are present throughout the sample, but appear with more frequency in this inter-layer zone, where they will likely induce anisotropic behaviour in the deposited parts. Figure 3.26 shows a different inter-layer interface with a large number of inclusions and voids, as was seen in all similar areas of the sample.

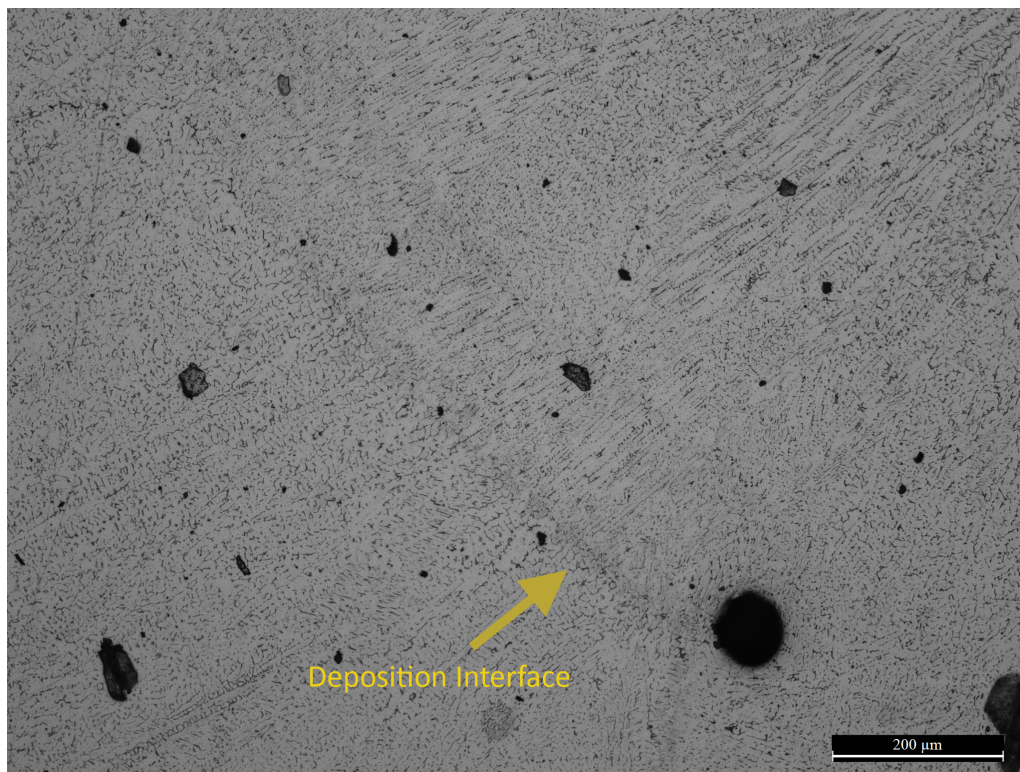


Figure 3.26: Inter-layer interface showing a large inclusion and numerous voids

Using ImageJ's particle analysis toolbox to analyse the optical microscopy images gathered, isolating the voids and inclusions found, the calculated average density of the sample was of 99.17%. The voids have an average maximum Feret diameter of $7.23\ \mu\text{m}$, which is the measure of the widest section of a particle, shown in Figure 3.27 as "X Feret, max".

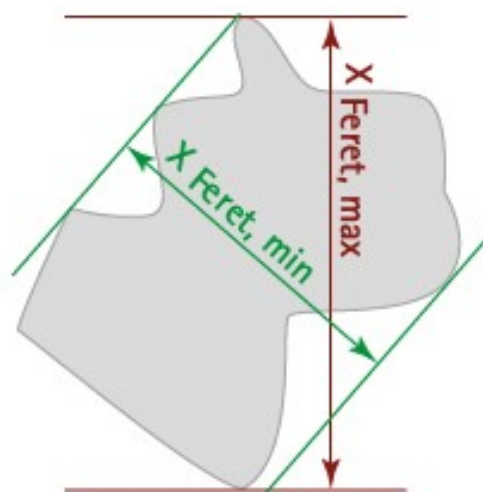


Figure 3.27: Feret diameters representation [72]

3.2.4 Microtensile specimens and tests

To optimise the amount of specimens taken from each deposited wall they were distributed as seen in Figure 3.28, resulting in 3 specimens machined from each direction in each 40mm high wall.

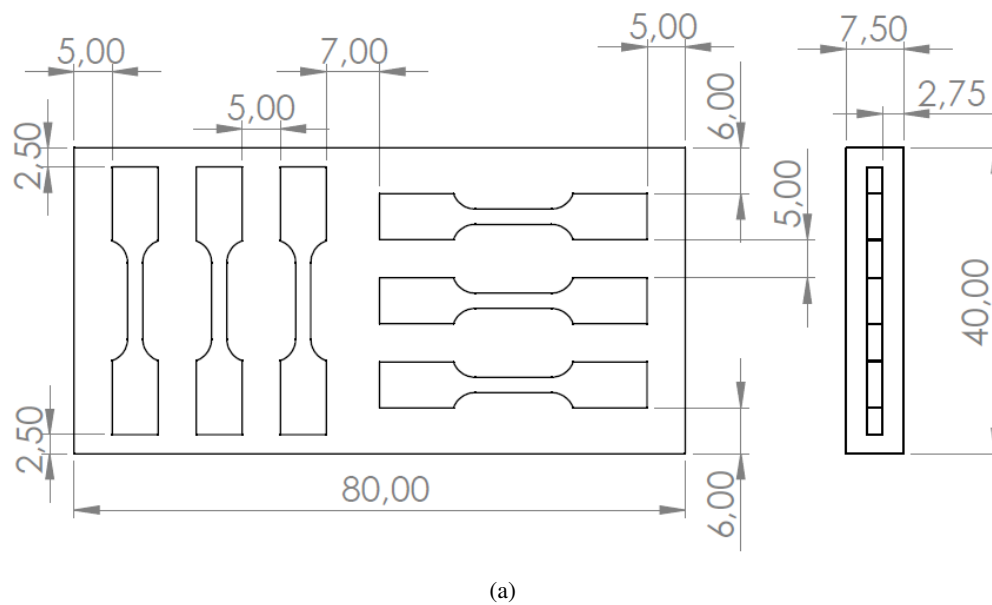


Figure 3.28: Microtensile specimens and their placement within the deposited wall

The specimens were machined in a CNC milling machine using a 5mm tungsten carbide end mill, with 4 flutes, at 4000rpm and a feedrate of 200mm/min. The machining stages can be seen in the following Figure 3.29 and the resulting specimens in Figure 3.30.

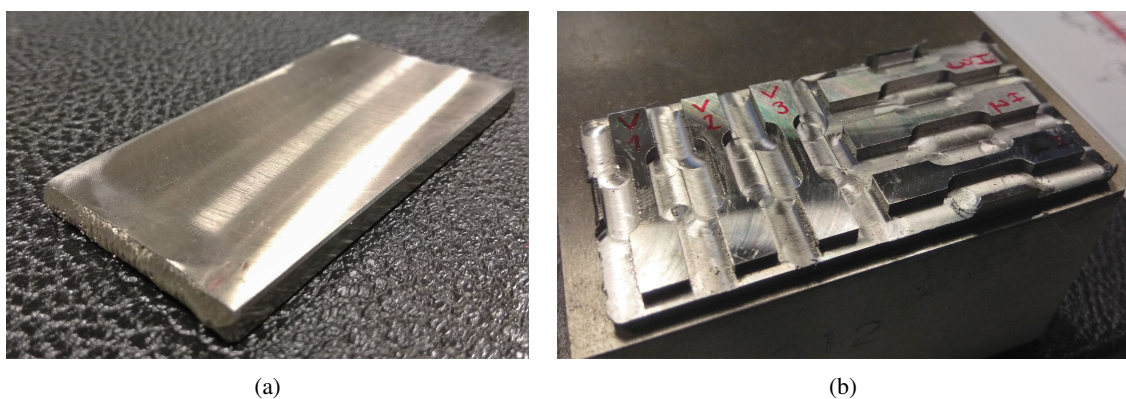


Figure 3.29: Intermediate machining stages of the specimens

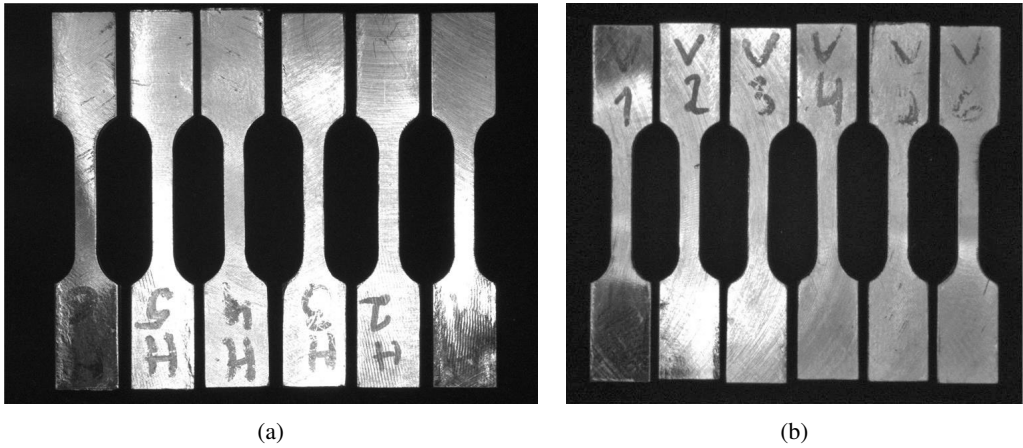


Figure 3.30: Resulting specimens, 6 machined for the horizontal direction (marked H) and 6 for the vertical/transverse direction (marked V)

The results of the tensile test for all 12 specimens can be seen in Figure 3.31, with the average resulting values compiled in Table 3.11.

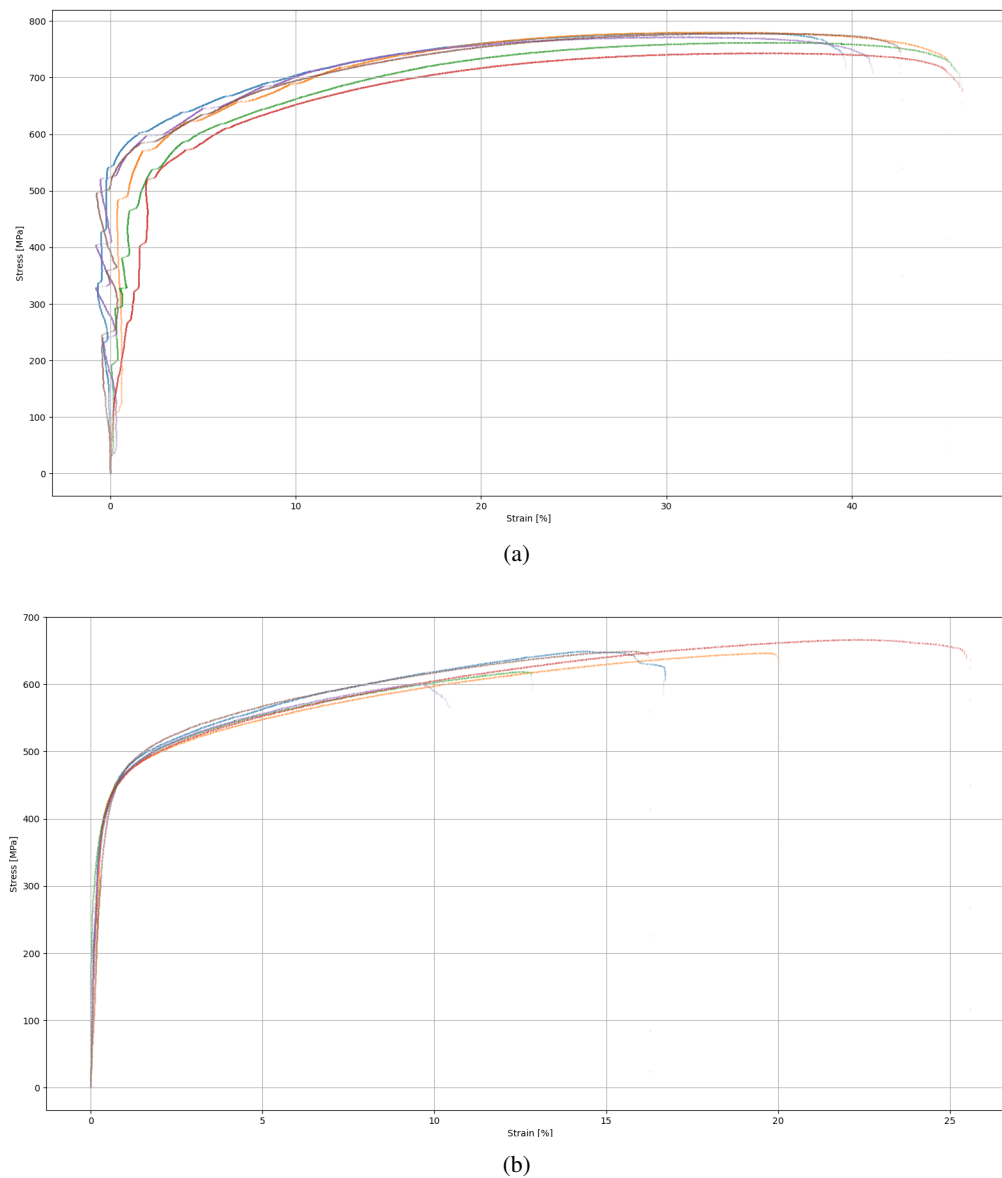


Figure 3.31: Tensile test results for the horizontal and the transverse/vertical direction respectively

Table 3.11: Tensile test results summarised

Direction	YS [MPa]	UTS [MPa]	ϵ [%]
Horizontal (H)	536 ± 36	769 ± 14	$43,4 \pm 2,7$
Vertical (V)	419 ± 8	638 ± 24	$17,0 \pm 5,4$

The jagged lines present in the elastic section of the horizontal specimens' tensile test were caused by tracking issues with the digital video extensometer used, as the tracking lines drawn in the specimens were not well lit in the early stages of the test but received more light as the tensile

test machine opened up. As a consequence the yield stress values for the horizontal specimens were not as precise as they could have been.

The yield strength and ultimate strength values for the vertical specimens were 22 and 17 % lower than for their horizontal counterparts, demonstrating the existence of anisotropy on parts built by this process. The elongation, however, is where that effect is more apparent with a 61% decrease from the horizontal to the vertical specimens. These high strength values and anisotropy prevalence are in accordance to what has been reported in literature, although Ziętała et al. [44] and Zhang et al. [45] have found the maximum elongation of specimens from the vertical direction to be higher than that of the horizontal direction, crediting the increased ductility of the dendrites in their growth (vertical) direction. In this case, however, it seems the inter-layer defects were the limiting factor for the elongation value of the vertical samples.

Figure 3.32 shows the microtensile specimens after being tested, noting the stretch marks (indicative of ductile fracture) in the horizontal specimens and lack of thereof and irregular failure location in the vertical specimens.



(a)

Figure 3.32: Microtensile specimens after being tested

3.2.5 Microhardness measurements

The same sample pictured in Figure 3.24 was used for the microhardness measurements, being scanned 5 times from the substrate to the deposited material with measurements being taken every 0.2 mm. The average values are shown in Figure 3.33.

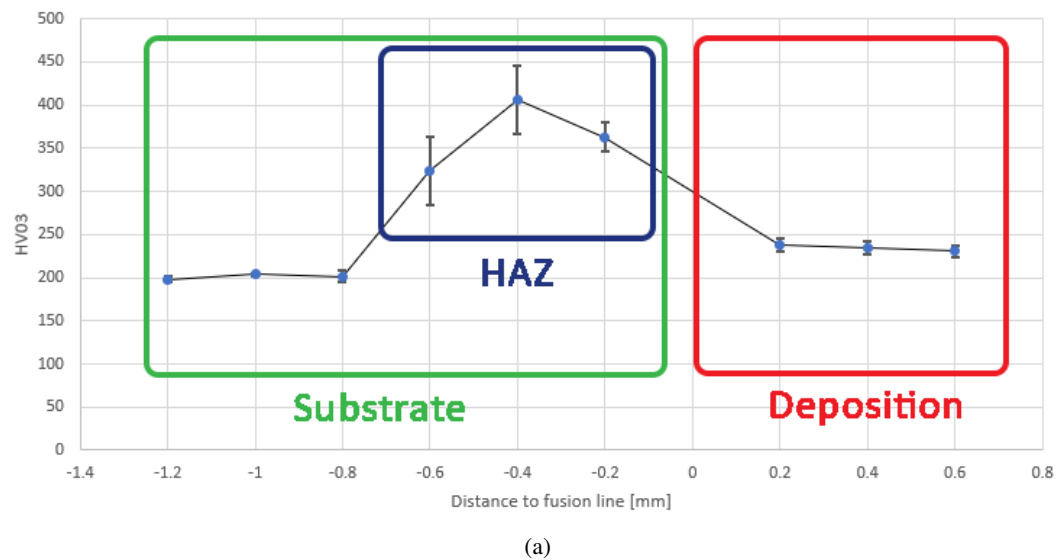


Figure 3.33: Microhardness values and range along the specimen's height

The Vickers hardness of hot rolled CK45 is typically in the range of 175-220 HV, as seen in the first three data point of Figure 3.33 [73]. The increase in hardness in the substrate's HAZ is then made clear, owing to the formation of hard martensite due to it's thermal history after the deposition of the 316L. The data point in the HAZ closest to the fusion line, however, shows a decrease in hardness due to tempering from the successive thermal cycles of the DED process.

The deposited 316L also shows an increase to 234HV relative to the typical 155HV found in it's annealed form, in line with the values found by other authors [43, 44, 56].

Chapter 4

Conclusions

In this dissertation the DED AM process was studied and the parameters and physical phenomena that govern it understood in order to find a set of reliable processing parameters that yielded usable, near net shape parts and made way for future research into this matter.

4.1 Goals

The goals proposed at the beginning of this document can now be revisited and analysed again:

G1 - Evaluate the discrete influence of each processing parameters in the deposited clads and the DED process efficiency.

Through the Taguchi arrays defined for the preliminary clads the discrete influence of each processing parameters was found: an increase of power input, be it an increase of laser power or decrease in scanning speed, yields a wider deposition width and an increase in powder feed rate yields a greater deposition height, although no meaningful direct relationship was found for the deposition efficiency.

G2 - Study the potential benefits of closed-loop control in the DED process.

Closed-loop control was demonstrated to be fundamental for the deposition of consistent quality parts, making the DED process stable and repeatable, reducing the occurrence of lack of fusion and over-deposition in movement direction transition areas.

G3 - Analyse the resulting mechanical properties for the optimal processing parameters found.

Microtensile specimens were machined from the deposited 316L parts and their tensile properties measured, resulting in yield and ultimate strengths 122% and 31% higher than of their annealed counterparts for the horizontal, in plane direction, while resulting in much lower properties for the

transverse direction, with the deposited parts showing clear anisotropic behaviour. This feeds into the following Goal 4.

G4 - Investigate the existence of anisotropy in the built parts.

The deposited parts showed anisotropic behaviour, with the transverse specimens' yield strength and ultimate strength performing 22% and 17% lower than their horizontal counterparts, due to a high number of defects present in the inter-layer interfaces.

4.2 Future work

Several aspects of the present dissertation could be further expanded, such as:

Wider range of test parameters for 3D depositions: Due to time and material availability constraints it was only possible to build 3D parts using a single set of processing parameters. However, it would be of great interest to test a wider range of parameters for 3D building to study their effect on the tensile and hardness properties of the parts built.

Better closed-loop control: As was demonstrated in the present work, closed-loop control is of utmost importance to produce consistent depositions, and as such more research should be conducted to enhance the control capabilities of DED machines, such as integrating feedback from the substrate temperature or allowing for speed and powder feed rate modulation.

Build complex parts: While this dissertation only focused on the deposition simple, thin walled parts to determine the mechanical properties of the deposited metal, it is still necessary to evaluate the limits of the parameters found in regards to the complexity of the geometry it allows to be built. A part such a large mould with conformal cooling channels would be ideal to test such a large scale metal AM process.

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Chapter 5

Appendix

[\[74\]](#) [\[75\]](#)

Material Product Data Sheet

Austenitic Stainless Steel Powder for Laser Cladding

Laser Cladding Products: MetcoClad 316L-SI

1 Introduction

MetcoClad™ 316L-SI is a gas atomized, nickel chromium stainless steel powder suitable for use with the laser cladding process. It is an austenitic-type stainless steel, similar to AISI Type 316L (UNS S31603), with a low carbon content that desensitizes the clad deposit to eliminate grain boundary carbide precipitation.

When applied using laser cladding, the properties are similar to, or better than, that of the bulk material because of the refined microstructure and low dilution during cladding. The clad offers good impact resistance at elevated temperatures and corrosion resistance in chloride environments. Molybdenum ensures good pitting and crevice resistance.

Silicon levels in MetcoClad 316L-SI are higher than AISI Type 316L. The higher silicon content acts as a fluxing agent that produces cleaner clad deposits. Please note that cladding parameters may have to be adjusted to accommodate the higher silicon content.

MetcoClad 316L-SI is recommended for corrosion overlay deposits on components used in the oil, gas and marine industries.

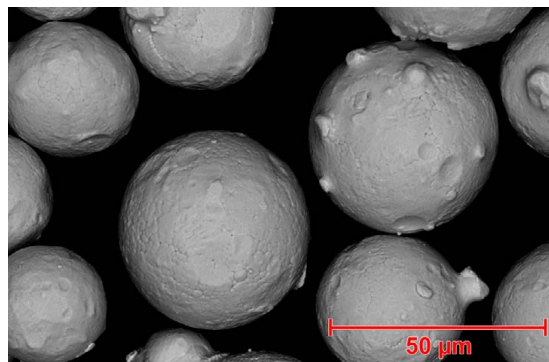
1.1 Typical Uses and Applications:

Typically used for laser cladding and laser-additive manufacturing applications, including:

- Heavy equipment
- Food equipment exposed to chloride environments
- Medical implants
- Hydraulic pumps and turbines
- Roll covers for printing machinery
- Shafts in marine applications
- Repair and build up of steel parts

Quick Facts

Classification	Alloy, iron-based
Chemistry	Fe 12Ni 17Cr 2.3Mo 2.3Si 1Mn 0.03C
Manufacture	Gas atomized
Morphology	Spheroidal
Service Temperature	≤ 900 °C (1650 °F)
Apparent Density	4.0 – 4.5 g/cm ³
Purpose	Corrosion protection, creep and stress-rupture resistance at elevated temperatures
Process	Laser Cladding



Typical Morphology of MetcoClad 316L-SI Powder