GEO-COAT

DEVELOPMENT OF NOVEL AND COST-EFFECTIVE CORROSION RESISTANT COATINGS
FOR HIGH TEMPERATURE GEOTHERMAL APPLICATIONS

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D3.4: Influence of laser cladding process parameters on the substrate melting

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Summary

The laser cladding process, one of the deposition techniques employed within the Geo-Coat project, involves generating a coating by melting a feedstock powder and locally a substrate to generate a strong metallurgical bonding. The properties of the laser final coated system will depend not only on the chemical-physical characteristics of the coating itself, but also on the dilution region between coating and substrate. In general, a low level of dilution is preferred in order to achieve a better control over the final clad properties.

In this deliverable, a characterisation study to link the effect of laser cladding process parameters on the magnitude of the dilution zone will be presented for the laser clad coatings planned in deliverable D3.1 (Adaptive DOE optimisation of laser cladding and HVOF processes). Results show that the laser power was the most significant among the varied process parameters.

Objectives Met

The following objectives have been met:

- To study and optimise the effect of laser cladding process parameters on coating and substrate melting.

Contents

SUMMARY 3
OBJECTIVES MET 3
1. INTRODUCTION 5
1.1 THE POWDER LASER CLADDING TECHNOLOGY 5
1.1.1 Working principle 5
1.1.2 Effect of process parameters on deposit properties 6
1.1.2.1 Effect of laser beam power 6
1.1.2.2 Effect of laser spot diameter 6
1.1.2.3 Effect of scanning speed 6
1.1.2.4 Effect of powder feed rate 6
1.1.2.5 Effect of tracks overlapping parameters 6
1.1.3 Dilution zone, heat affected zone and their measurement 7
1.1.3.1 Dilution zone and heat affected zone 7
1.1.3.2 Quantification of dilution zone 8
2. MATERIALS & SETUP 8
3. METHODS 9
3.1 GENERAL COATING DEPOSITION METHODOLOGY 9
3.2 COATINGS FROM PRELIMINARY TRIALS 9
3.3 COATINGS FOR PROCESS PARAMETERS DOWN-SELECTION 10
3.4 QUANTIFICATION OF DILUTION AREA 12
3.5 DETERMINATION OF EFFECT OF PROCESS PARAMETERS 12
4. RESULTS AND DISCUSSION 13
4.1 EFFECT OF PROCESS PARAMETERS ONTO % DILUTION 13
4.1.1 Preliminary trials (powder HEA 2 only) 13
4.1.1.1 Effect of laser power 13
4.1.1.2 Effect of nozzle gas flow 14
4.1.2 Process parameters down-selection stage (for powders HEA 1 – 5) 14
4.1.2.1 Effect of laser power 14
4.1.2.2 Effect of powder disk speed 16
4.1.2.3 Effect of number of layers 18
4.1.3 Summary: overall effect of laser power, scanning speed and powder disk speed from both preliminary trials and down-selection stages 18
5. CONCLUSIONS 18
6. REFERENCES 19

1. INTRODUCTION

1.1 The powder laser cladding technology

1.1.1 Working principle

The powder laser cladding process, or laser metal deposition (LMD), is a deposition technique employed to generate coatings of different materials (e.g. ceramics, metallic alloys, etc.) onto substrates. In this way, a coating with superior chemical and mechanical properties can be generated to enhance the overall substrate-deposit system performance [1].

A schematic of the powder laser cladding process is depicted in Fig. 1.

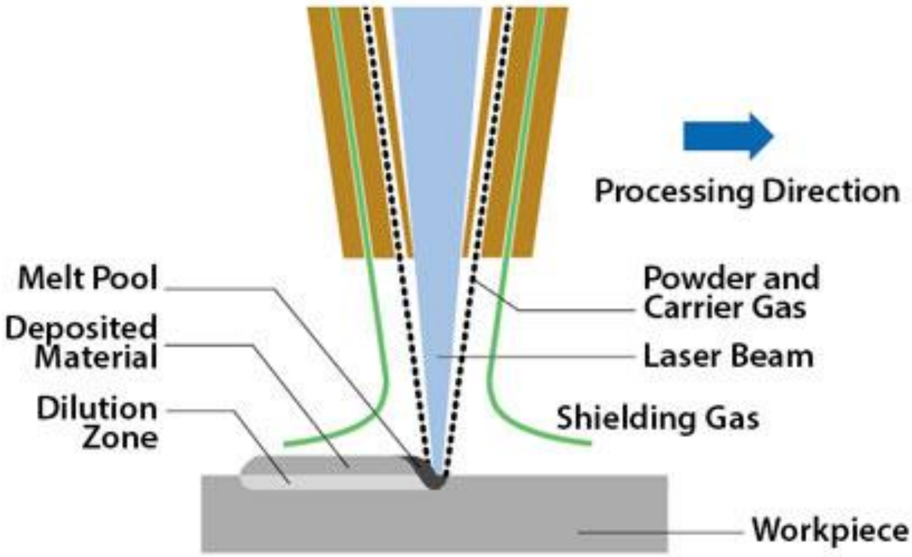

***Figure 1** – Schematic of the powder laser cladding process. The depth of the dilution zone can be controlled by adjusting process parameters such as laser power and laser cladding liner speed (from https://www.thefabricator.com/article/finishing/from-looms-to-thermal-spray-to-laser-cladding)*

In the process, a laser beam is defocused on the work piece with a selected spot size, thus generating a local melt pool. The powder material is then carried by an inert gas (generally Ar), through a nozzle into the melt pool. The laser beam has thus the double function to both melt the injected powder and the work piece (locally), thus generating a solidified layer of material with a strong metallurgical bonding with the substrate. The final properties of the coated system, (e.g. its resistance to corrosion, erosion, etc.,) will depend in turn on its microstructural features, such as porosity, phases present, etc. The final coating microstructure is dictated by the specific value of process parameters employed during deposition, mainly:

- Laser beam power
- Laser spot diameter
- Scanning speed
- Powder feed rate
- Tracks overlapping parameters

Generally, for a specific powder material to be deposited, there will be a specific "deposition window", representing the ranges of process parameters within which a coating can be generated with specifically designed properties. Specifically required properties generally correspond to the obtainment of a pore-free microstructure, well adhered to the work piece surface and with a reduced dilution area.

1.1.2 Effect of process parameters on deposit properties

In this paragraph, a brief overview of the effect of the above described laser cladding process parameters on the properties of the final clad system will be presented [2, 3].

1.1.2.1 Effect of laser beam power

Laser power has a significant effect on the deposition efficiency and surface finish as well as height and width of the deposited tracks As laser power increases, so too does track width, thus improving the surface finish of the deposited part as there is more energy available for full melting of the incident powder particles. This results in a limited increase in deposit height, when scanning speed, powder feed rate and laser spot size are kept constant, but causes a decrease in deposit height as the degree of melting of the substrate increases. The use of excessive laser power can lead to melt pool vaporisation, with a subsequent increase in laser power absorption and the formation of keyholes, which are very desirable for welding of thick sections, but undesirable for laser metal deposition due to turbulence and instability of the melt. Conversely, if the laser power is not high enough, then there may be insufficient energy to cause substrate melting and deposit fusion, thus leading to poor adhesion.

1.1.2.2 Effect of laser spot diameter

For a fixed laser power, a small spot size generates a higher energy density that may lead to vaporisation of deposit elements and excessive penetration of the melt pool. A reduced size of the melt pool will then decrease the capture area for powder particles, decreasing the process efficiency and deposit build rate. A larger spot size, for a fixed laser power and scanning speed, decreases the energy density. Therefore, matching a large spot size with a higher laser power results in a broad, shallow melt pool that has a higher capture area of powder particles, increasing the efficiency of the process.

1.1.2.3 Effect of scanning speed

High scanning speeds result in less laser/material interaction time and a decrease in the heat input, increasing the laser power required to cause melting and fusion as well as the powder mass delivery rate required to produce a deposit of a desired geometry. Low scanning speed may lead to excessive heat build-up in the material due to longer laser /material interaction dwell time, lower powder mass delivery rate and lower deposit build rate.

1.1.2.4 Effect of powder feed rate

Higher powder mass flow rates will result in more powder entering the melt causing the volume of molten material to increase. Since the melt pool width remains relatively constant at constant values of other process parameters, an increase in deposit volume will result in a change in the contact angle of the deposit. Increased powder feed rate also results in a decrease in effective laser energy, as the powder particles act to adsorb a fraction of the incident laser light. Conversely, low powder feed rates would allow a greater fraction of the laser to interact with the substrate, increasing the penetration depth. Within this report, the powder feed rate is reported in terms of powder disk speed %. This parameters, related to the speed of the powder disc located within the powder feeder, is in fact proportional to the powder feed rate (i.e. a higher % value would correspond to a higher powder feed rate and vice versa).
A summary of the effect of the above described process parameters onto the clad system characteristics is summarised in Tab. 1.

1.1.2.5 Effect of tracks overlapping parameters

Since the width of a typical laser cladding track is in the 1÷5 mm width range, several single tracks must be laid down in order to cover a wide substrate surface. Several parallel single track can be then deposited next to each other at various degrees of overlapping, as well as on top of each other, in order to generate the full coating. The degree of overlapping between parallel tracks and the number of layers co-deposited will then affect the heat input into the system and thus degree of re-melting of previous tracks and substrate.

***Table 1** – Influence of increase of process parameters onto deposit properties (↑ increase, ↓ decrease)*

Process parameter	Properties			
	Clad height	Penetration	Dilution	Deposit thickness
↑ Laser power	↓	↑	↑	↑
↑ Scanning speed	↑	↓	↓	↓
↑ Powder feed rate	↑	↓	↓	↑
↑ Laser spot diameter	↑	↓	↓	↓

1.1.3 Dilution zone, heat affected zone and their measurement

1.1.3.1 Dilution zone and heat affected zone

The dilution zone is of paramount importance within the laser cladding process as it defines the level of adhesion between coating and work piece and the chemistry of the clad layer. Dilution determines the strength between clad layer and the substrate and therefore a high dilution area percentage is preferred in applications where bond strength is an important factor, such as wear resistance. For applications where corrosion or oxidation properties are more relevant, a small dilution area is preferred as this will reduce the amount of clad/work piece material interdiffusion, thus retaining the clad composition closer to the one of the original powder. Moreover, by controlling the dilution, laser metal deposition has been demonstrated to allow deposition of dissimilar materials, which would be otherwise difficult by using conventional fusion welding processes [2].

Together with the dilution zone, a Heat Affected Zone (HAZ) is also always present within a laser clad microstructure. A more detailed schematic picture of an area close to the dilution zone is depicted in Fig. 2.

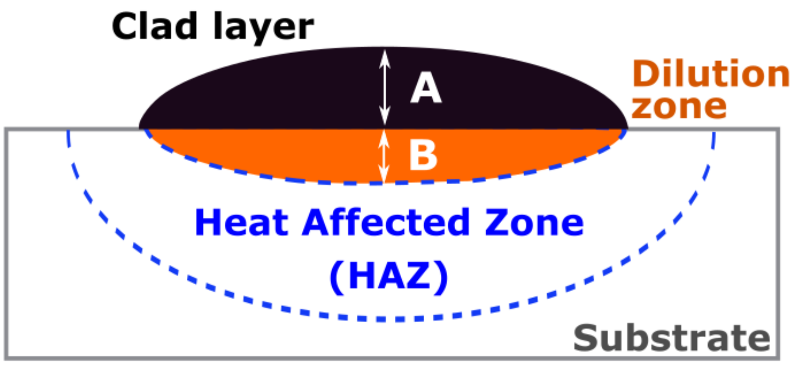

***Figure 2** – Schematic of a laser clad cross-section, where clad layer (black), dilution zone (orange), heat affected zone (HAZ, blue) and substrate (grey) are depicted.*

The HAZ represents an area of the substrate where the heat input from the laser and the molten powder is sufficient to impart microstructural transformations (e.g. grain growth, phase transformation, etc.). It is generally difficult to unequivocally differentiate between dilution and HAZ, owing to the fact that different elements, both from the clad material to the substrate and vice versa, will have different diffusion rates. Since the level of elemental interdiffusion intrinsically defines the dilution zone, area, it can be seen how a clear subdivision between dilution zone and HAZ is intrinsically non trivial. Nevertheless, it is expected that a larger dilution zone + HAZ joined area will be generated as a consequence of a higher heat input during deposition.

Since the target of this report is the quantification of the effect of laser cladding process parameters onto substrate melting, the focus will be on the quantification of this latter rather than the HAZ.

1.1.3.2 Quantification of dilution zone

A commonly employed method employed to quantify the magnitude of dilution is to present the results in terms of dilution %. This can be calculated by the clad and dilution zone heights, A and B respectively as depicted in Fig. 2, based on the following relationship [4].

$$\%\ dilution = \frac{B}{(A+B)} \cdot 100 \qquad [1]$$

Although Eq. (1) is commonly used by many research groups, the way the quantities A and B are measured can vary considerably. For instance*, SEM-EDX* scans across the clad cross-section can highlight changes in elements concentration and thus could be used to define the limit of the dilution zone by tracking an element specific to the clad material chemistry. *Micro-hardness* has also been successfully employed. Within this method, the hardness is mapped across the clad cross-section. Variations in hardness are typical of different phases, which would allow to highlight the transition from clad to substrate composition. However, among the different techniques, *image analysis* of cross-section optical microscopies probably represents the most versatile and accessible method amongst all. This method relies on the intensity contrast between regions rich in clad- versus substrate-rich material. This latter is the technique employed within this report.

2. MATERIALS & SETUP

All of the substrates have been cladded at the TWI Yorkshire offices, while the analysis have been performed at the TWI Cambridge offices.

The laser metal depositions have been performed using a Trumpf Trudisk 8002 5.3kW disc laser system, controlled by a Reis RV60-40 robot and a Sulzer Metco 10-C powder feeder system. Five high-entropy alloy powders have been used for the experiments, as summarised in Tab. 2.

***Table 2** – Details of the high-entropy alloy powders employed for the laser cladding experiments*

Powder acronym	**Manufacturing method**	**Composition** [molar ratio]	**Size** [µm]
HEA 1	Mechanical alloying	$CoCrFeNiMo_{0.85}$	63 - 125
HEA 2	Gas atomization	$CoCrFeNiMo_{0.85}$	48 - 150
HEA 3	Mechanical alloying	CoCrFeNiMo	63 - 125
HEA 4	Mechanical alloying	$Co_{0.5}CrFeNiMo$	63 - 125
HEA 5	Mechanical alloying	$Al_{0.5}CoCrFeNi$	63 - 125

Argon (Ar) has been employed as powder carrier gas throughout the experiments, while either argon (Ar) or helium (He) has been employed during the depositions as nozzle gas. Substrates of one wrought alloy (S1, S235JR) of 40x40x6 mm dimensions has been employed for the depositions. The substrates were prepared for deposition by grinding the surface to a 60 grit size finish. Four laser cladding experiments (each corresponding to one specific set of process parameters) were performed on each substrate. More details of the laser equipment and materials employed during cladding (powders, substrates, etc.) have already been described in deliverable D3.3 (Report on optimisation of laser cladding process for HEA based coating synthesis); the reader is thus advised to refer to the mentioned deliverable for more details.

Deposit analysis were performed by cross-sectioning the clad tracks by using a standard diamond wheel, then mounted into Bakelite resin. The cross-sections were ground using SiC papers from 120 to 2500 grit, followed by polishing with 3 and 1 µm diamond suspensions and a final polishing stage with 1/4 µm alumina. Quantification of dilution area has been performed by image analysis by using the ImageJ software.

3. METHODS

Throughout the report, the following naming will be employed:

- **Track** – This is a single pass made by the laser cladding head at one specific set of process parameters. Multiple tracks are generally overlapped, both on the same horizontal plane and vertically (on different planes) to generate a deposit.
- **Layer** – This represents the different vertical planes where tracks are laid on. Each of the deposits produced in this study was produced with either 1, 2 or 3 overlapped layers of tracks.
- **Deposit** – This is the overall overlap of tracks and layers for one specific set of deposition process parameters. Each of the tracks which a deposit is made of has been produced at the same set of process parameters.

3.1 General coating deposition methodology

Each of the deposits generated for the specimens in this report has been produced with the following step methodology (Fig. 3):

1. **Deposition of single tracks** – each track has an approximate width of 1.3 mm and a 33% overlap is set between each. One single track (no multiple layers) has been deposited for pre-trials experiments, while three overlapped tracks on multiple layers have been used for the process parameters down-selection stage.
2. **Deposition of one or more layers of 3 single tracks on top of the first one** – for the process parameters down-selection stage, 2 or 3 layers of 3 overlapped tracks are deposited on top of the first one.

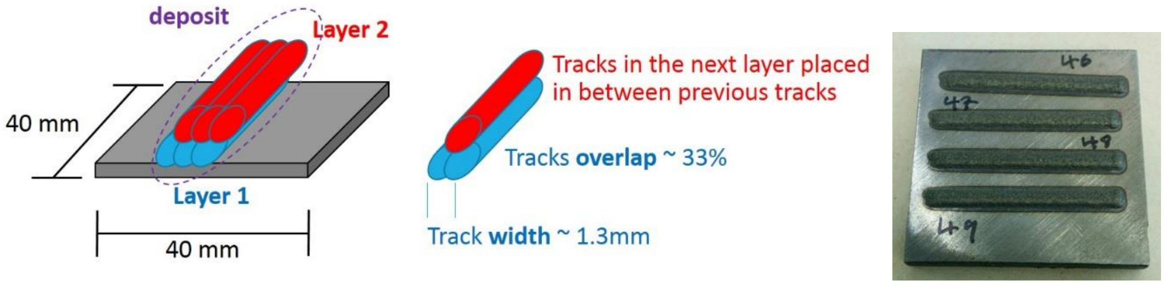

Figure 3 *– Geometrical characteristics of the deposit (purple) produced for the specimens in this report; each deposit layer is composed of three overlapped tracks (represented by blue/red circles)* ***(left)*** *and typical appearance of a substrate surface where 4 deposits have been generated* ***(right).***

3.2 Coatings from preliminary trials

The aim of this stage was to perform a first screening of deposition parameters for the next stage (process parameters down-selection). High-entropy alloys are a novel area of research in laser cladding and thus no specific parameters have yet been developed to produce coatings with the equipment available at TWI. One single track per set of deposition parameters (thus no multiple layers) has been generated in this stage (1 deposit = 1 track). Parameters were varied by following an adaptive approach between previous/subsequent test run in order to be able to narrow down the process parameter values: laser power, laser scanning speed, powder feed rate and nozzle gas flow rate. Only powder HEA 2 in Tab. 1 and substrate S1 (S235JR) was employed for all the tests in this stage. A 3.5 l/min Ar carrier gas flow was employed for all the experiments. The full list of deposits produced in this stage is reported in Tab. 3. In this study, the variation of process parameters has been linked to the magnitude of dilution area and general microstructure of the coatings. The target of this stage has in fact been to minimise dilution area and deposit porosity as explained within deliverable D3.3

(Report on optimisation of laser cladding process for HEA based coating synthesis). It is worth noting that only the deposits where the % dilution could be measured are presented in the table.

***Table 3** – Summary of the deposits produced in the pre-trials stage of the project, where only the specimens providing a visible clad have been reported. A "one-factor-at-a-time" approach has been generally employed for the remaining deposits. Only powder HEA 2, deposited onto carbon steel substrates (S235JR) has been employed for these tests. The % dilution is calculated based on Eq. (1) and the methodology described in Par. 3.4.*

Deposit #	Laser Power [W]	Scanning speed [mm/s]	Powder disc speed [%]	Nozzle gas flow [l/min]	Nozzle gas type	% dilution
6	737	11.08	9.3	6	Ar	32.4
18	400	10.00	9.3	6	Ar	6.7
19	600	10.00	9.3	6	Ar	23.4
20	800	10.00	9.3	6	Ar	35.7
21	737	11.08	9.3	8	He	30.3
22	737	11.08	9.3	10	He	30.0
23	737	11.08	9.3	12	He	30.4
24	632	12.92	8.7	8	He	19.1
25	632	12.92	8.7	10	He	14.7
26	632	12.92	8.7	12	He	9.7
27	400	12.92	8.7	10	He	5.2
28	600	12.92	8.7	10	He	21.5
29	800	12.92	8.7	10	He	33.3

3.3 Coatings for process parameters down-selection

The range of process parameters selected within the pre-trials stage has been employed to produce the deposits for the process parameters down-selection stage. Each deposit produced in this stage is composed of multiple tracks and layers, varied in order to understand the effect of overlapping on the extension of dilution area. All of the powders listed in Tab. 2, deposited only on the substrate type S1 (S235JR) were employed as materials for the experiments in this section. Four deposits per substrate have were produced. The full list of deposits produced in this stage is reported in Tab. 4. It is worth noting that the deposits are organised per powder type and in sets of four based on the number of layers employed in each set and also that deposit 63 is not reported due to hardware fault during deposition; this has been replaced by deposit 64. The nozzle gas (Ar) was maintained at a flow of 6 l/min in all experiments. Up to 5 parameters (or factors in statistical analysis terms) were varied for each powder: laser power, scanning speed, powder disc speed, carrier gas flow and number of layers. Pre-trials suggested that laser power had the most significant effect on the levels of coating dilution and microstructural characteristics of the deposits. Therefore, trials varied this process parameter over four levels (400, 450, 500, 550W). It is worth noting that the process parameters' values eventually selected for these tests differ considerably from the ones originally envisaged in deliverable D3.1 (Adaptive DOE for optimisation of laser cladding and HVOF process), due to the novel nature of the high-entropy alloy materials deposited in the project.

Table 4 *- Summary of the deposits produced in the process parameters down-selection stage of the project. For each of the five powder types, the laser power process parameter has been varied in 4 levels, while 2 levels have been chosen for the other process parameters. All the deposits have been produced onto substrate S1 (S235JR).*

Deposit #	Powder # (Tab. 2)	Laser Power [W]	Scanning speed [mm/s]	Powder disc speed [%]	Carrier gas flow (Ar) [l/min]	Number of layers	% dilution
46	**2**	400	10.00	9.3	3.5	1	10.3
47		450	10.83	9.3	3.5	1	6.3
48		550	10.00	8.7	3.5	1	11.6
49		550	10.00	9.3	3.5	1	9.7
50	**3**	400	10.00	9.3	3.5	1	45.8
51		450	10.83	9.3	3.5	1	44.3
52		550	10.00	8.7	3.5	1	70.9
53		550	10.00	9.3	3.5	1	66.3
54		400	10.00	9.3	4	2	47.3
55		450	10.83	9.3	4	2	40.3
56		550	10.00	8.7	4	2	29.0
57		550	10.00	9.3	4	2	23.2
58		400	10.00	9.3	4	3	14.9
59		450	10.83	9.3	4	3	11.8
60		550	10.00	8.7	4	3	21.3
61		550	10.00	9.3	4	3	30.9
62	**4**	400	10.00	9.3	4	2	14.9
64		450	10.83	9.3	4	2	14.5
65		550	10.00	8.7	4	2	38.9
66		550	10.00	9.3	4	2	30.1
67		400	10.00	9.3	4	3	12.3
68		450	10.83	9.3	4	3	8.5
69		550	10.00	8.7	4	3	27.1
70		550	10.00	9.3	4	3	23.5
71	**1**	400	10.00	9.3	4	2	7.3
72		450	10.83	9.3	4	2	11.9
73		550	10.00	8.7	4	2	16.2
74		550	10.00	9.3	4	2	13.1
75		400	10.00	9.3	4	3	4.2
76		450	10.83	9.3	4	3	6.8
77		550	10.00	8.7	4	3	7.7
78		550	10.00	9.3	4	3	8.9
79	**5**	400	10.00	9.3	4	2	4.7
80		450	10.83	9.3	4	2	6.2
81		550	10.00	8.7	4	2	9.4
82		550	10.00	9.3	4	2	8.7
83		400	10.00	9.3	4	3	3.0
84		450	10.83	9.3	4	3	4.3
85		550	10.00	8.7	4	3	7.1
86		550	10.00	9.3	4	3	7.1

3.4 Quantification of dilution area

Image analysis has been employed on optical micrographs of embedded specimen's cross-sections in order to quantify the dilution %. All of the image analysis have been performed by means of the ImageJ processing software.

The methodology employed for the dilution % determination is depicted in Fig. 4.

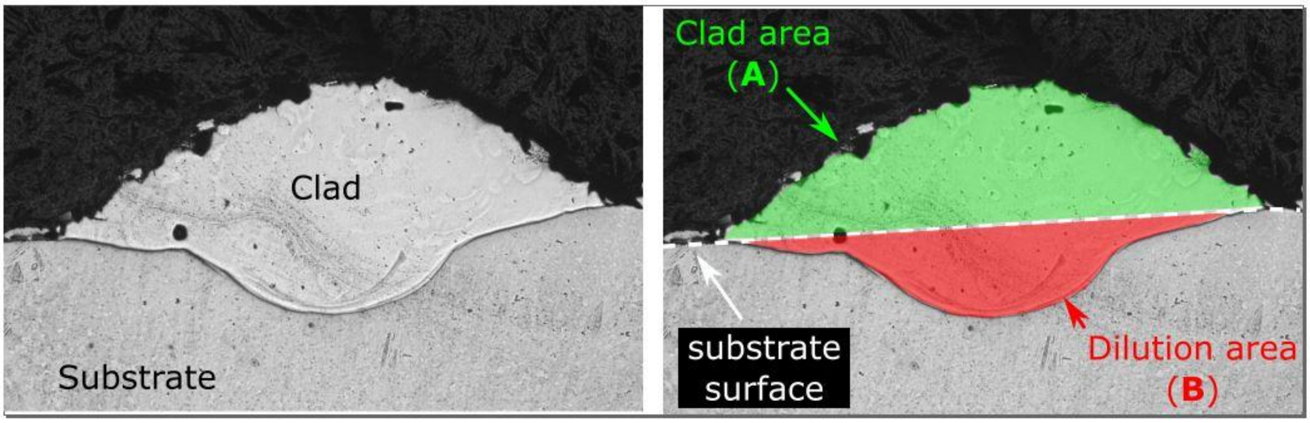

Figure 4 *– Optical microscopy of a laser clad deposit cross-section (left) and the same microscopy where dilution (B, red) and clad (A, green) areas are highlighted (right). The % dilution is then calculated based on Eq. (1).*

The figure shows an optical micrograph of a clad deposit (left) and the same micrograph where the dilution and clad areas are shaded (A red and B green respectively). The boundary between the two areas is defined by tracing an imaginary line matching the substrate surface in the areas not affected by the deposit (dashed white line in Fig. 4). The % dilution is then quantified by means of Eq. (1).

3.5 Determination of effect of process parameters

While random process parameters values have been varied during the pre-trials, a factorial design has been employed for the experiments in the process parameters down-selection stage. As a result, slightly different approaches have been employed to evaluate the results within the two sections.

In general however, the aim within both stages has been to calculate the main "effect" for each of the process parameters varied, which is, the change in the response as the process parameter is varied. For the case of this study, we will use the following statistical terms:

- **Run** – this is one laser clad experiment, performed at one defined set of process parameter values.
- **Process parameters** – these are the variables changed during the tests: laser power, laser scanning speed, powder disk speed, carrier gas flow, and number of layers. This is generally referred to as "factor" in statistical analysis but will be maintained as process parameter for the sake of simplicity within this report.
- **Levels** – these are the process parameter values selected for the tests (e.g. 400, 450, 500 and 550 W for laser power in process parameters down-selection)
- **Response** – this is the target measurable quantity which the combination of factors levels is compared against. In the case of this study this is the % dilution as calculated from Eq. (1).
- **Effect** – In statistical analysis, this is defined as the average change in the response as a variation in a process parameters is performed between two runs. However, due to the nature of the experimental design adopted for the runs in these experiments, it has been decided here to not adopt the standard statistical definition of effect. In turn, the response of each run will be reported as measured for each analysed run. This has been here proven to make the results analysis more readable in this case.

It is worth specifying that interactions between effects, although significant in the laser cladding process, are outside the scope of this report and are thus not investigated.

4. RESULTS AND DISCUSSION

In the following, the results from the experiments performed both in the preliminary trials and down-selection phase will be reported. All the results are presented as % dilution (in a 0-75 % range) vs process parameter employed in each specific run. Interactions effects are generally not investigated in this analysis and, for this reason, in each graph, only runs deposited at the same set of process parameters (except the one varied under analysis) are compared. In this way, in some cases the effect of the variation of some process parameters could not be evaluated.

4.1 Effect of process parameters onto % dilution

4.1.1 Preliminary trials (powder HEA 2 only)

Experiments in this stage have been performed by varying the levels for each factor for only powder HEA 2 in Tab. 2. Therefore, the ability to draw definitive conclusions is limited. It is worth noting, as previously explained, that only the results from deposits providing a visible/measurable clad on the substrate surface have been reported here. Several experiments performed in the pre-trials phase in fact, were not able to produce deposits; these results, although reported in deliverable D3.4 of the project, are not presented.
In the following, the effects of the variation of each factor onto the % dilution, when possible, will be discussed. Due to the random nature of the factors variation between the different deposits in this phase, a consistent approach could not be adopted. In this way, the only effect of laser power and nozzle gas flow could be properly evaluated, while the main effect of other factors will be evaluated in depth within the discussion related to the process parameters down-selection stage.

4.1.1.1 Effect of laser power

<u>Summary</u>: *An increase in laser power increases the % dilution*
<u>Results analysis:</u> The effect can be appreciated by comparing the % dilution for runs 18, 19 and 20, performed at a laser power of 400, 600 and 800 W respectively (Fig. 5). An increasing % dilution is observed between each run (+200 W difference) at increasing laser power. Although other process parameters might also interact with the laser power, this could not be analysed from the experiments in this stage. Nevertheless, the magnitude of the effect is significant. More details on the effect of laser power can be found in the results from the down-selection stage (Par. 4.1.2.1).

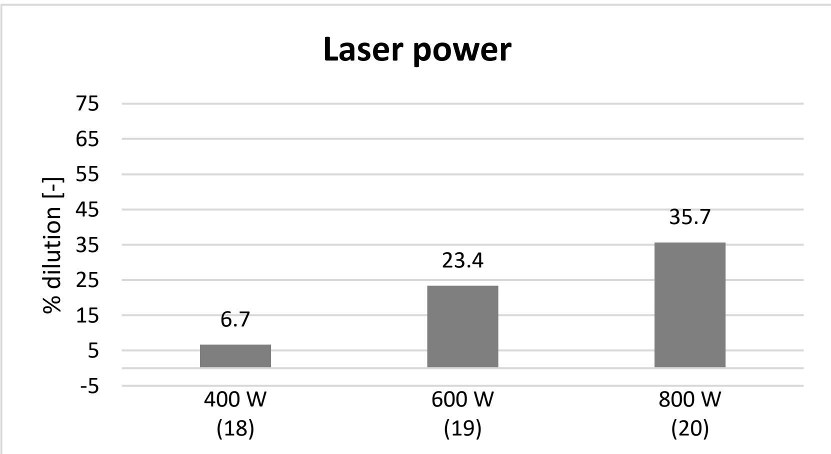

Figure 5 *– % dilution calculated for runs 18, 19 and 20, performed by using powder HEA 2 at laser power levels of 400, 600 and 800 W respectively. An increase in % dilution is observed at incremental laser power.*

4.1.1.2 Effect of nozzle gas flow

<u>Summary</u>: *An increase in nozzle gas flow either keeps constant or decreases the % dilution*

<u>Results analysis:</u> The effect can be appreciated in Fig. 6 by comparing the two sets of runs 21-22-23 (full bar, performed at one specific set of process parameters) and 24-25-26 (dashed bar, performed at one specific set of process parameters different from set 21-22-23), an increase of nozzle gas flow (+ 2 l/min) is set between each run. In this way, 8 l/min is the nozzle gas flow used in runs 21-24, 10 l/min in 22-25 and 12 l/min in 23-26. It is worth noting that practically no variation is observed between runs of the set 21-22-23 while a decreasing % dilution is measured between 24-25-26, thus confirming a considerable interaction effect between nozzle gas flow and the other process parameters. Since a completely different set of process parameters has been employed between the two sets, a clear analysis of interaction effects could not be performed.

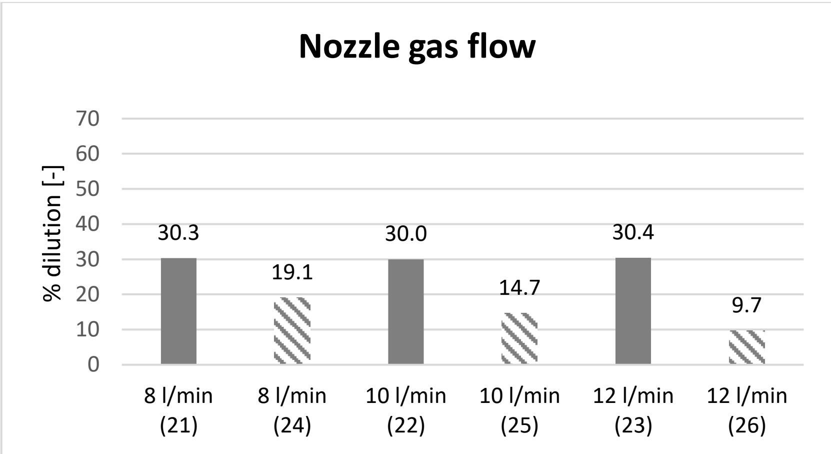

Figure 6 *– % dilution calculated for two sets of runs 21-22-23 (full bars) and 24-25-26 (dashed bars), each deposited at one specific set of process parameters with an increase of +2l/min set between each run. Constant (21-22-23) and decreasing % dilution (24-25-26) is observed in the two sets, denoting significant interaction effects.*

4.1.2 Process parameters down-selection stage (for powders HEA 1 – 5)

The values for the factors varied in this stage (see Tab. 4) have been selected based on the results of the previous preliminary trials. As discussed later, the deposits have been evaluated on a qualitative basis, not only based on the % dilution but also on microstructural characteristics (e.g. porosity, cracks, etc.). In the following, the effect the variation of each process parameter on the % dilution will be evaluated for the runs of the down-selection stage. In general, as process parameters have been varied on a one-factor-at-a-time basis, a proper statistical evaluation of the effects is here possible to perform, unlike the runs within the pre-trials stage.

4.1.2.1 Effect of laser power

<u>Summary</u>: *An increase in laser power increases the % dilution*

<u>Results analysis:</u> The effect can be appreciated by analysing the graphs in Figs. 7, 8 and 9 (1, 2 and 3 layers respectively) containing the result from runs performed at the same set of process parameters except laser power, varied between 400 and 550 W. It is worth noting that the graphs do not contain all of the powders as not all of them have been deposited in 1, 2 and 3 layers (see Tab. 4). The first noticeable effect is that, in almost all cases, an increase in laser power corresponds to a higher % dilution. The only exception is represented by powder HEA2 – 1 layer (Fig. 7, grey) where a slight decrease is observed (likely within the experimental error) and powder 3 – 2 layers (Fig. 8, blue) where a considerable decrease is observed (likely due to experimental error). It is also important to note the magnitude of the effect decreases at an increasing number of layers, as it can be noted from the general value of the % dilution results from Fig. 7 to 9.

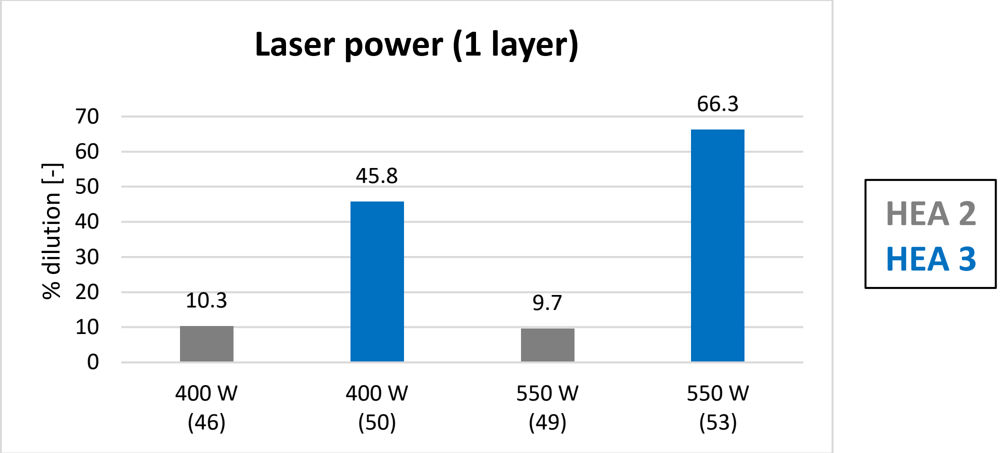

Figure 7 *– % dilution calculated for runs (1 layer) where a +150 W increase in laser power is set for powder HEA2 and 3. An increase in % dilution is measured for powder HEA 3 while a slight decrease is shown for powder HEA 2.*

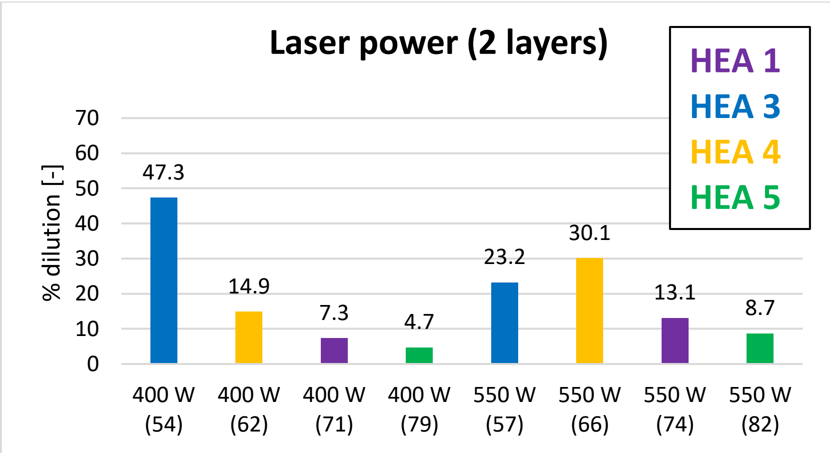

Figure 8 *– % dilution calculated for runs (2 layers) where a +150 W increase in laser power is set for powders HEA1-3-4-5. An increase in % dilution is measured in all cases except for powder HEA 3, likely due to a random experimental error.*

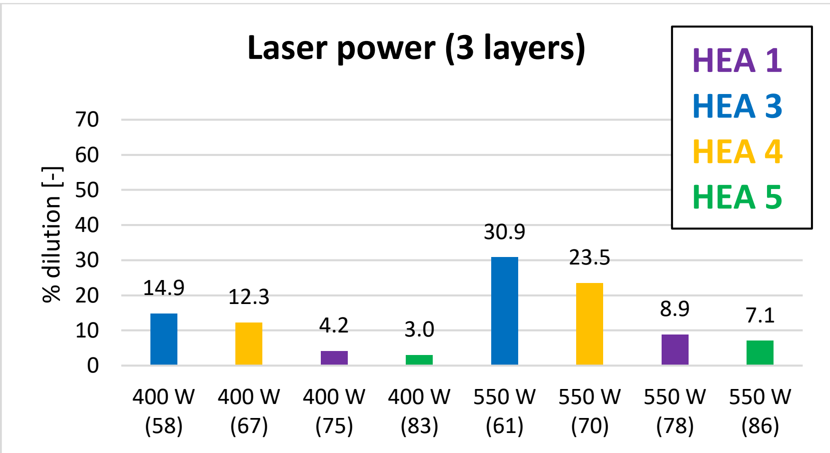

Figure 9 *– % dilution calculated for runs (3 layers) where a +150 W increase in laser power is set for powders HEA1-3-4-5. An increase in % dilution is measured in all cases.*

4.1.2.2 Effect of powder disk speed

Summary: An increase in powder disk speed decreases the % dilution

Results analysis: The effect can be appreciated by analysing the graphs in Figs. 10, 11 and 12 (1, 2 and 3 layers respectively) containing the result from runs performed at the same set of process parameters except powder disk speed, varied between 8.7 % and 9.3 %. It is worth noting that the graphs do not contain all of the powders as not all of them have been deposited in 1, 2 and 3 layers (see Tab. 4). Note that, as explained within Par. 1.1.2.4, the powder disk speed is here reported as % unit, corresponding to a unit characteristic to the powder feeder proportional to the powder feed rate parameter. An increase in powder feed rate has the effect of reducing the % dilution in almost all cases. The only exceptions are represented by powders deposited in three layers (Fig. 12) where a decrease is only observed in powder HEA 4. The clearest exception is shown by powder HEA 3 – 3 layers (runs 60 and 61) where a remarkable increase in % dilution is measured. This latter result can be attributed to difficulties in interpreting the extension of dilution area from the optical microscopies due to unclear boundaries. This can be appreciated by comparing the two micrographs corresponding to runs 60 and 61 (Fig. 13(a) and (b) respectively). In the figure, the dilution area is marked by a red dashed line. The border of this area has been selected isolating the area of a coloration similar to that of the clad area within the substrate. However, potential issues in measuring the dilution area arose for these two coatings due to shaded areas present underneath the clad, of a scale between that of the clad and the substrate. These regions, marked by white dashed lines could not be uniquely identified as neither dilution area nor substrate, thus giving rise to possible errors in the quantification of the dilution areas. The average effect, -3.1, is also reported as a red dashed lined on the graph. This result is expected as, due to an increased powder feed rate, a larger clad area is expected to be deposited while the extension of the dilution area is not expected to increase proportionally.

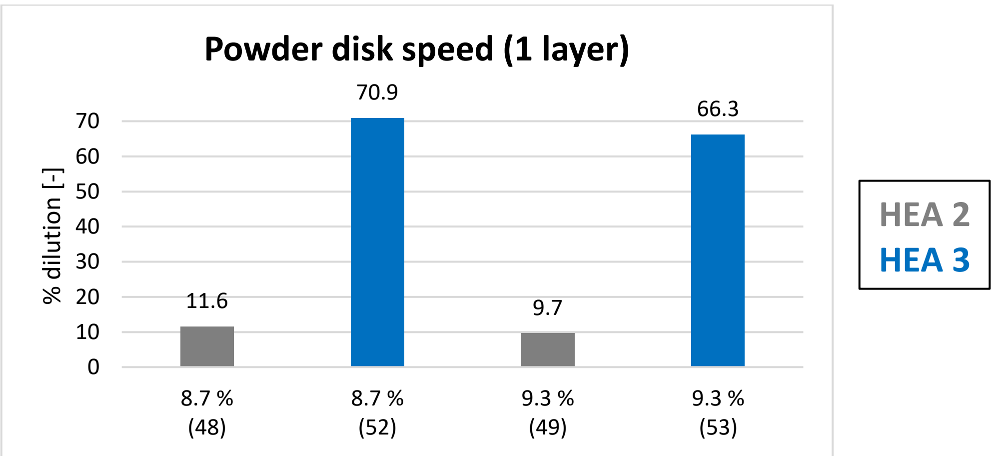

***Figure 10** – % dilution calculated for runs (1 layer) where a +0.6% increase in powder disk speed is set for powder HEA2 and 3. A decrease in % dilution is measured for both powders.*

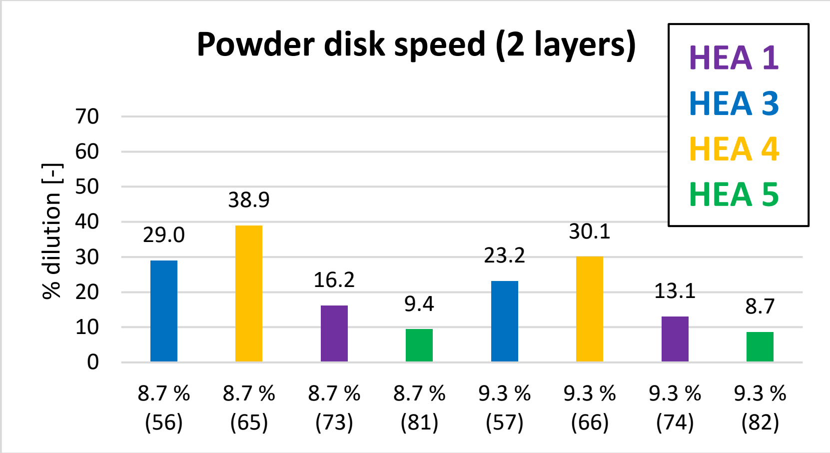

***Figure 11** – % dilution calculated for runs (2 layers) where a +0.6% increase in powder disk speed is set for powders HEA1-3-4-5. A decrease in % dilution is measured in all cases.*

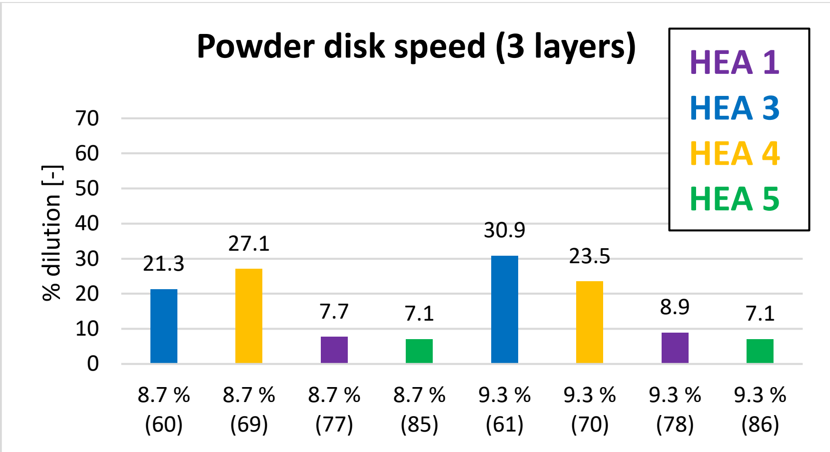

***Figure 12** – % dilution calculated for runs (3 layers) where a +0.6% increase in powder disk speed is set for powders HEA1-3-4-5. A decrease in % dilution is only measured for powder HEA 4, while little or increasing effect on % dilution is measured for the other powders.*

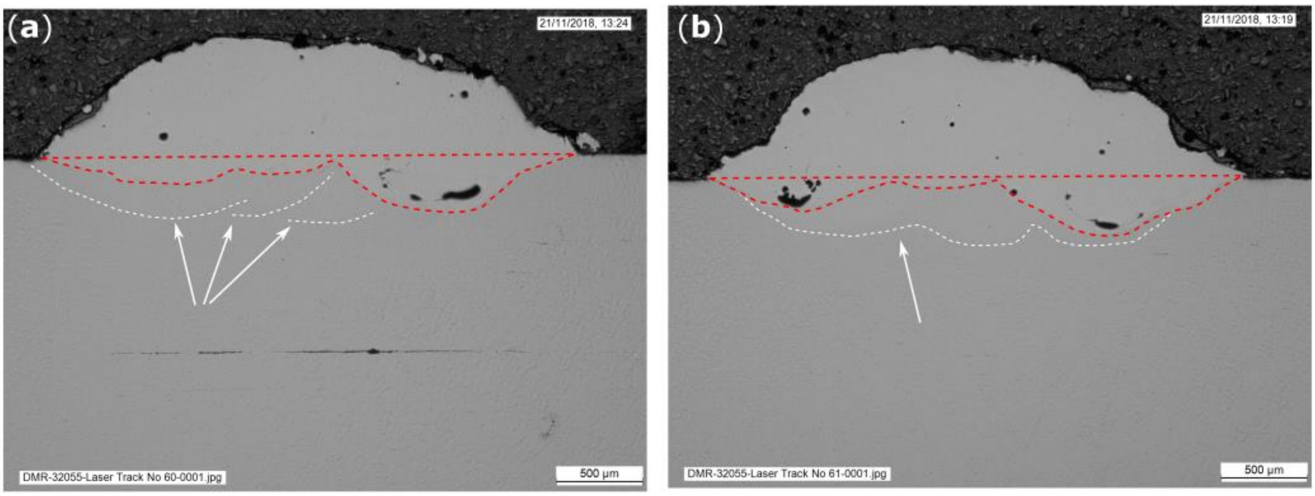

***Figure 13**– Optical micrographs of runs 60 (a) and 61 (b) deposits cross-sections. The dilution area used for the calculations is shown is marked by a red dashed line, while other possible features*

4.1.2.3 Effect of number of layers

Summary: A higher number of layers diminishes the magnitude of all effects from process parameters variation

Results analysis: The % dilution has been defined as the ratio between the molten area within the substrate and the clad area (Eq. 1). Therefore, when overlapping multiple layers of clad material, the clad already on the surface (e.g. from layer 1) will act as new work piece material for the oncoming layers (e.g. layer 2) and therefore only a reduced effect will be noticed on the substrate. It is therefore expected that a critical number of layers will exist, for each material, where the process parameters variation will have no effect on the % dilution as defined in Eq. 1. This is starting to be evident by comparing the results of runs performed at a higher number of layers (i.e. 2 and 3) compared to 1 layer. This is the case of, for instance, Fig. 12 compared to 11 and 10 but also Fig. 9 versus 8 and 7.

4.1.3 Summary: overall effect of laser power, scanning speed and powder disk speed from both preliminary trials and down-selection stages

The effect of the change in several process parameters into the % dilution has been evaluated. The values selected for the process parameters in the runs have been varied with a specific pattern, although not following a specific factorial design. For this reason, effects could only be quantitatively determined for some of the factors.

5. CONCLUSIONS

In this report, the effect of the variation of several laser cladding process parameters onto the % dilution has been quantified on runs performed with five different powders. The effects have been evaluated at two separate stages in the project: preliminary trials (powder HEA 2 only) and down-selection stage (all powders HEA 1 to 5). Within the preliminary trials, the only effects of laser power and nozzle gas flow could be assessed, with results on the effects evaluated at a variation of + 200W and + 2 l/min between runs respectively. Within the down-selection stage, the only effects of laser power, powder disk speed and number of layers could be correctly evaluated as the runs where the variation of these two process parameters (+ 150W and + 0.6 % and +1 respectively) has been assessed at constant values of the other process parameters. Results from the remaining process parameters in the stage: scanning speed, carrier gas flow could not be adequately assessed due to significant interaction effects. Among all process parameters, it has been identified that it is the laser power to have the main effect on the % dilution among the ones evaluated.

A summary of the effects evaluated is reported in Tab. 5.

***Table 5** – Summary of average effect of factors' variation onto % dilution.*

Factor	Process parameter variation	Effect
Laser power	+ 200 W (pre-trials) + 150 W (down-selection)	An increase in laser power increases the % dilution
Nozzle gas flow	+ 2 l/min (pre-trials)	An increase in laser power either keeps constant or decreases the % dilution
Powder disk speed	+ 0.6 % (down-selection)	An increase in powder disk speed decreases the % dilution
Number of layers	+ 1 layer (down-selection)	A higher number of layers diminishes the magnitude of all effects from process parameters variation

These results will be employed to further optimise the laser cladding process parameters and therefore the properties of the coatings generated within the project.

6. REFERENCES

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