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Investigation on the Influence of the Particle Size Distribution on the quality of EHLA process

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Abstract

Laser Metal Deposition (LMD) at high speeds has led to the recent Extreme High Speed Laser Application (EHLA) processes, where sensitivity in parameter adjustment increases.
Influence of the powder particle-size distribution (PSD) on the projection generated through the nozzle has been studied. Regarding to EHLA, that requires high velocities, different PSD generates diverse results, and these are analyzed throughout this study.
Four main cases have been identified where the PSD is altered within the execution procedure of an LMD process: 1. Segregation in storage and/or transport, 2. Reutilization after the additive process and 3. Mixing of different materials.
This study generates a correlation between the main PSD parameters of each case with the results of tests performed with EHLA to improve quality and repeatability. Among the results, the appearance of different defects and the morphological changes of the final piece have been analyzed.

Keywords: Laser Metal Deposition-LMD; EHLA; Powder particle distribution

1. Introduction

Extreme High-Speed Laser Cladding (EHLA) is considered a cutting-edge advancement in the field of laser-based metal coating methods. EHLA is an emerging additive process that relies on Laser Metal Deposition

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technologies (LMD) to achieve coating deposition at great speed rates with outstanding quality results as contrasted by Liqun, L. et al., 2019. The absence of welding dilution substrate and heat-affected zones under the deposited material are some of the greatest advantages offered by this manufacturing process against other Direct Energy Deposition processes in Additive Manufacturing. This is possible thanks to the last high-power laser beam developments in the laser field and their corresponding required nozzles to allow a suitable deposition of cladding material, which is fed in metal powder format.

Metal powder materials used for these manufacturing techniques are mainly generated by a process of atomization where molten metals are converted into finely divided powders with controlled particle size, shape, and composition. Good quality atomization of metallic materials is of critical importance in the field of additive manufacturing. Metal powders are thoroughly tested for good quality properties such us uniformity, sphericity, and flowability since they are essential for achieving optimal results. These properties are highly sensitive to the technique used for its manufacturing, having effects on the behavior of the additive process thus altering the final results, as observed in water atomized powders by Hoeges, S. et al., 2017 or Popovich, A. et al. 2016, who make a general review on the metal powder manufacturing effects on additive processes.

These properties not only depend directly on the atomization process, but also on the powder logistical management. Metal powders tend to fluidize and modify their properties during its transportation and manipulation from its manufacture site, as documented long before by authors as Parsons, D.S., 1976, as well as during its storage and until its use at the process working point as seen by Phua, A. et al., 2022, in other techniques of additive manufacturing or simulations studied by Nan, W. et al, 2020. One of the main issues observed in this stage of the process is the segregation of particles depending on their size, completely altering their Particle Size Distribution (PSD), thus having a great impact in several properties of the powder like its flowability, tap density and tendency of agglomerating, among others, as observed by Habibnejad-korayem, M. et al., 2021, Azéma. E et al., 2017, Mussato, A. et al., 2021 or Wu, C.Y. et al., 2004, in similar studies as mentioned before.

In this paper, we focus on the significance of PSD among other metal powder properties for high-speed additive manufacturing applications, and discuss its impact on the additively manufactured cladding performance. This property is susceptible to changes during other several common stages of additive manufacturing process like reutilization of the powder, mixture of powders of different materials and different powder suppliers besides the already mentioned segregation during logistical manipulation. Therefore this paper is also focused on the impact of these situations on PSD and preventive measures for avoiding or mitigating their effect, like powder tumbling for eliminating segregation phenomena or the mixture of materials with similar PSD's.

2. Materials and methods. Experimental setup

2.1. Used equipment

The configuration of the experimental equipment for this study had a high-power fiber laser (IPG YLS-8000), with a wavelength of 1070 nm; with 2.5 mm of spot diameter; a coaxial nozzle using nitrogen as carrier and shielding gas. In addition to these machines, an “Alfphie” three dimensional powder mixer was used for mixing powder. A schematic layout of the Laser Cladding system is shown in Figure 1.

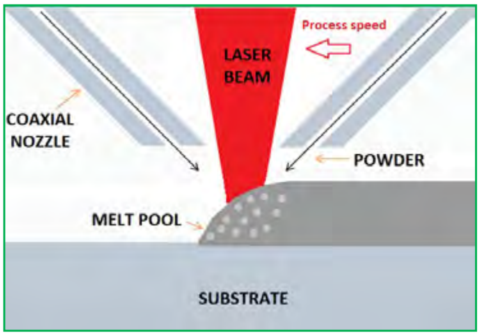

Fig. 1. Schematic layout of the coaxial laser cladding system.

2.2. Used materials

For this study, atomized AISI 316L stainless steel (supplier A) and Wolfram Carbide (WC) powder from two different suppliers were used, (supplier B and C). For all powders, a nominal range of particle sizes, 20-53 µm has been requested. PSD has been tested with image analysis using an "OPTIKA" microscope, a "Leica" camera, a "ZEISS EVO" scanning electron microscopy (SEM) and "Fiji/ImageJ" image processing software.

2.3. PSD Image acquisition and analysis procedure

For obtaining the PSD of every sample, several pictures were taken directly to the powder, which were then prepared before being processed by the "ImageJ" program specific features for particle analysis. The software program has returned results in a *.csv* format containing the size value of every particle. This last processing step requires the adequate calculation techniques for calibrating, converting and computing values so as to correctly obtain the histograms as a function of the particle volume.

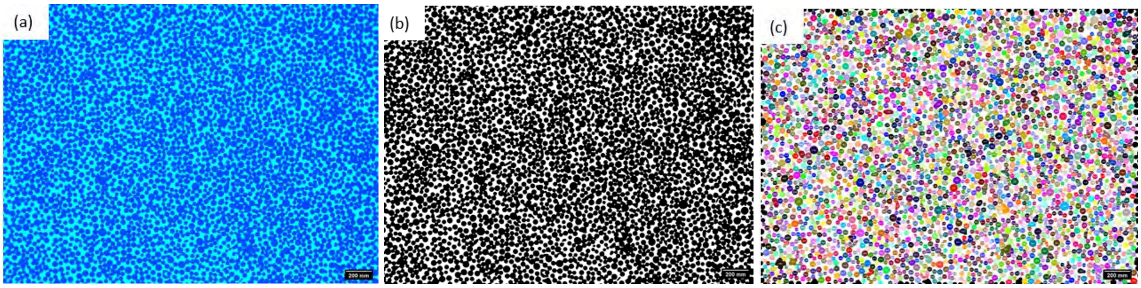

Fig. 2. Image processing for PSD analysis with ImageJ program in 3 different stages. (a) original image (b) preparation for particle analysis (c) final counting for data collection.

3. Results and Discussion

Metal powder manufacturers have a variety of techniques to obtain a type of powder material depending on the properties requested for it. That is, for a same single request, each manufacturer may offer different characteristics of powder material, all of them reaching the established minimum standards in a different way. These differences were observed and shown in this research.

Suppliers often provide some certified information about the PSD of their products. However, this information is usually offered only with the values of the percentiles 10, 50 and 90 of the particle sizes and this could be insufficient for sensitive processes like EHLA, which require a thorough control of every parameter involved in their process. Hence, the PSD of each powder intended for laboratory use is thoroughly analyzed upon its reception. All PSD's obtained and presented in this paper are evaluated as the input parameters of this study, from which we have compared their characteristic and their effects on the EHLA results using different powders suppliers and situations.

In this study, three main situations are selected for their significant influence on these metal powder PSD properties, whose characteristics are compared to observe their effect on the EHLA process.

3.*1 Cases Studio*

3.1.1. Case 1: Segregation in storage and/or transport

Environmental vibrations affect the PSD of powder materials since they facilitate granular convection, causing segregation of particle sizes. These vibrations occur both during the transportation from the powder suppliers to the final destination and during powder storage. In order to avoid this PSD alteration of the material, powder containers are tumbled prior to the execution of the additive process. For this study, samples are taken from three different heights of the (Figure **3**) before and after tumbling the powder so as to identify and eliminate powder size segregation.

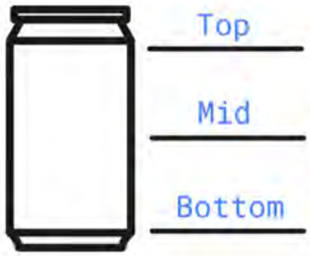

Fig. 3. Diagram of the powder container and heights at which a sample has been extracted.

The following PSD shown in Figure 4 and Figure 5 were obtained as previously described from every powder container tested in this study.

From PSD's shown in Figure 4, it can be observed that there exists a segregation phenomenon during the storage of AISI 316L. In this case, finer particles are found at the bottom of the container (Figure 4a) due to a sieving segregation case, whereas coarser particles were displaced to the top (Figure 4c). On the other hand, Figure 4b shows the PSD in the middle of the container where a more homogeneous distribution is found.

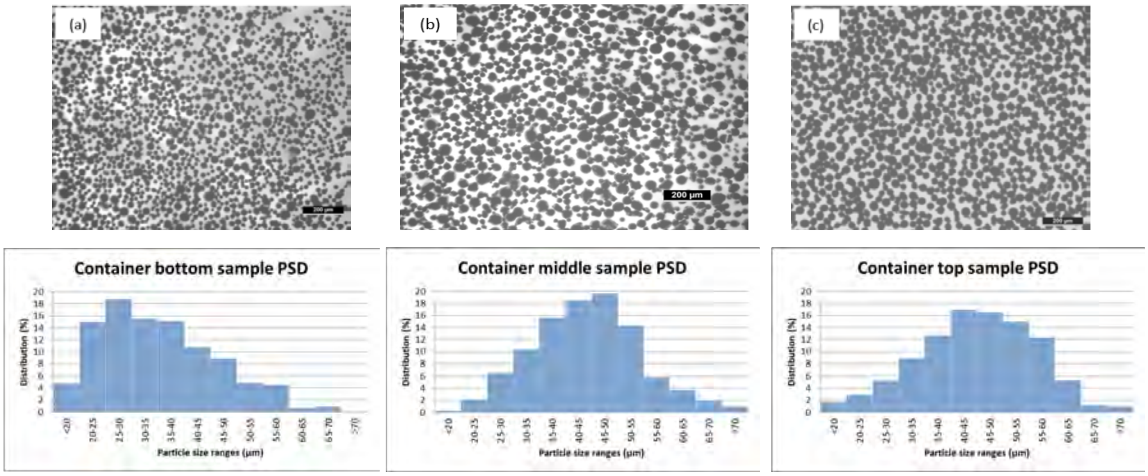

Fig. 4. Optic microscope image (left) and PSD (right) of segregated AISI 316L extracted from the (a) bottom, (b) middle and (c) top part of the container.

After the first three samples (bottom, middle and top) were extracted, the whole container was tumbled and, as expected, in the Figure 5 can be observed a wide and homogeneous PSD, indicating that tumbling effectively eliminates segregation phenomena.

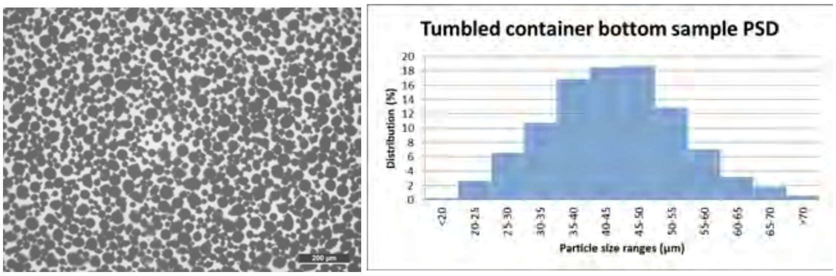

Fig. 5. Optic microscope image (left) and PSD (right) of AISI 316L (supplier A) extracted from the bottom part of the container, after tumbling the whole container.

As a summary, percentiles 10, 50, 90 and their Spans are shown in the following Table 1.

Table 1. Percentiles 10, 50, 90 and Spans from powder analysed in section 3.1.1.

Sample	Tumbled	D10	D50	D90	Span
Bottom	No	21.7	33.7	51.1	0.87
Middle	No	30.5	44.1	57.1	0.60
Top	No	30.0	45.4	59.0	0.64
Bottom	Yes	30.3	43.5	56.9	0.61

From numerical data in Table 1, it can be observed the displacement of the PSD in their percentiles, being smaller for the bottom sample and greater for the top sample. Span values are maintained except for the bottom sample, where it can be observed that coarse particles are still present, visually showing a flatter PSD profile between D10 and D90 values. This is explained by the sieving type of segregation phenomena that does not necessarily affect coarse particles, since it is the finer parts that are displaced through the spaces between the spaces left by the greater ones, as described by Tang, P. et al., 2004, in their studies on segregation minimization of diverse types of powder materials in several industrial fields.

3.1.2. Case 2: Reutilization after the additive process

Despite LMD and EHLA processes having a high rate of deposition efficiency, as several authors have verified like Svetlizky D. et al., 2021, or Wu et al., 2004, there exists a possibility to reutilize powder that has not been additivated in the process. After a sieving and inspection process that guarantees a good quality of particles, it can be observed that the initial PSD has been altered. This situation has been considered as a potential factor for affecting the additive process. Thus, a series of samples from new and reused AISI 316L powder materials have been taken for this study.

The normal Gaussian shape of the as received and tumbled powder PSD is shown in Figure **5**. In the analyses shown in Figure 6, it is noticeable the loss of specific sizes of particles after the use of this powder in the EHLA process. From Figure 6 (left), it can be observed a decrease in finer particles in the first use with respect to the original tumbled and unused powder in Figure 5, causing the PSD values to be displaced towards greater values. Furthermore, after using the same powder three times, other ranges of sizes are missed as can be observed in Figure 6 (right), definitely losing the uniform and continuous distribution of particle sizes. Due to these significant changes in the PSD, the utilization of already used powders is expected to have an influence on EHLA results.

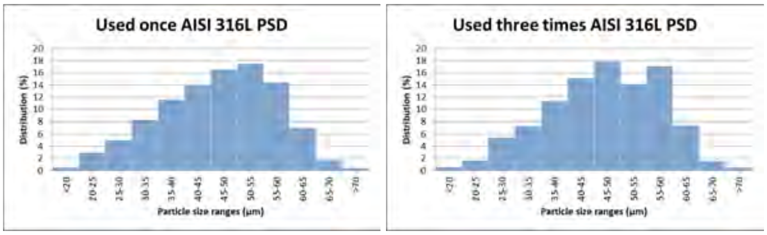

Fig. 6. PSD of AISI 316L (left) used once and (right) used 3 times.

3.1.3. Case 3: Mixing of different materials

For this case study, a mixture of 70% AISI 316L and 30% WC powders was prepared. The combination of two different powders with initially mentioned varying PSD properties, yet meeting the same quality requirements, generates a new PSD profile. Samples of these mixtures were taken from two different combinations of suppliers and their PSD's were compared to the ones of the powders before the mixtures were made . The diagrams of Figure 7 show the PSD results analyzed from these mixtures.

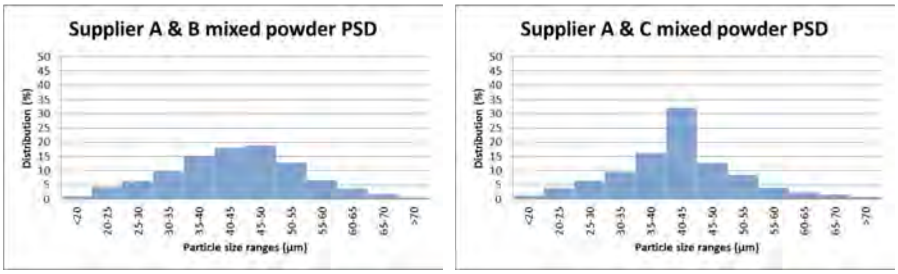

Fig. 7. PSD of mixtures of 70% AISI 316L from supplier A and WC from (left) supplier B and (right) supplier C.

It is observed that despite the low percentage of WC mixed in AISI 316L, the resulting PSD's are highly influenced by PSD of the WC. This situation is more noticeable in the mixture of AISI 316L from supplier A with WC from supplier C, shown in Figure 7 (right), where the narrow PSD is clearly represented in the resulting histogram.

When the values of span and percentiles of these mixtures are studied altogether with the PSD profiles, in the case of mixture A and C, the significant indicator to the powder quality change is only found through the PSD profile observation. This situation suggests that the whole study of the PSD profiles, percentile values and span values must be taken into account to detect powder quality alterations. In the case of this mixture, it is evident that the distribution in range 40-45 µm suffers an abrupt rise and breaks the uniform distribution, thus expecting an effect in the EHLA process results.

3.2. Execution of EHLA process tests

EHLA cladding tests have been executed for each corresponding case so as to obtain comparable results. The cases that are studied are summarized in Table 2.

Table 2. Summary of the execution of EHLA tests related to studied Cases.

Test	Materials used	Cases studied
a	Mixed AISI 316L and WC from suppliers B and C	3: Influence of powder mixtures.
b	AISI 316L	1: Influence of Particle-Size segregation.
c	AISI 316L	2: Influence of Powder reutilization.

During the additive process, all the parameters related to powder feeding were constant and stable. The powder cone generated at the tip of the nozzle kept its optimal shape for a correct execution.

This study has been planned based on the execution two types of coating tests. One type consist on the additivation of mixtures of AISI 316L and WC and, the other type, AISI 316L alone.

The variation between tests reside in the different quality characteristics between two suppliers of WC powder, for the mixtures type of text, and, for the AISI 316L alone tests, in the alteration of AISI 316L powder quality, considering its PSD modification due segregation phenomena or due to its reutilization.

Test a. (Cases 3).

As shown in case 3 in the previous section, mixtures of supplier A and B materials, which have similar widths and shapes of their PSD, result in a final PSD where every range value is slightly and proportionally raised, maintaining the original profile. See Figure 7 (left).

In Figure 8 it can be observed a series of individual cladding tests using the mixture of AISI 316L and WC from suppliers A and B. As expected from the previous result, a uniform cladding was additivated without defects. The PSD profile was preserved, thus, applying the needed parameters for this mixture of materials, the cladding uniformity was kept.

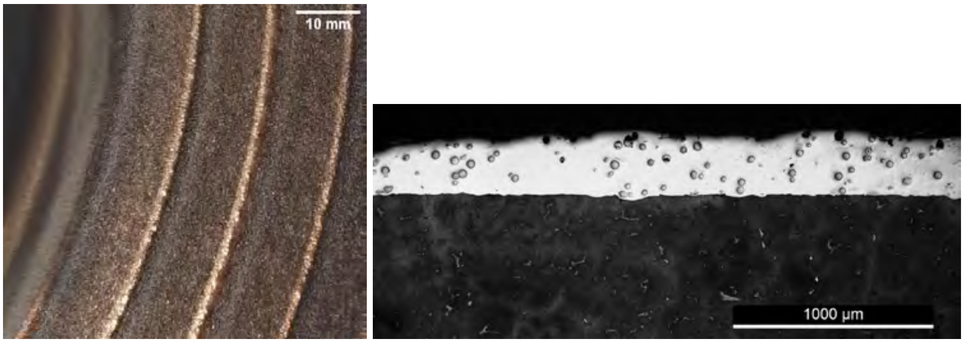

Fig. 8. Surface and cross-section images of the cladding deposited with AISI 316L from supplier A and CW from supplier B corresponding to PSD profile of the tumbled and as received powder in Figure 7 (left).

The last series of tests carried out for this case was the mixture of materials from supplier A and C. As stated in previous sections, this mixture resulted in a non-uniform PSD due to the influence of a narrow WC powder PSD on the wide AISI 316L PSD, which can be observed in the Figure 7 (right). The following images in Figure 9 show the cladding generated with this powder condition.

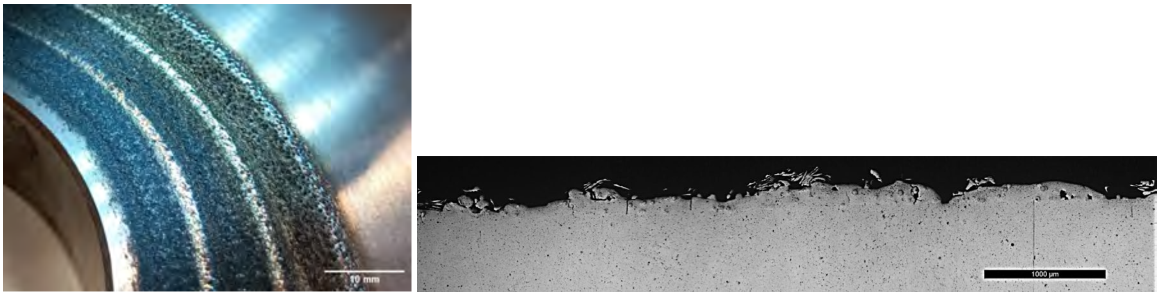

Fig. 9. Surface and cross section images of the cladding with deposited with AISI 316L from supplier A and CW from supplier C corresponding to PSD profile of the tumbled powder in Figure 7 (right).

This irregular PSD modified the powder cone shape generated at the tip of the nozzle and displaced the calculated distances between the machine head and the working point of the process. The realization of this situation forced the design of the process to be adapted. Regardless of the adjustment of parameters, the coating procedure inevitably built a faulty clad due to the significant presence of holes/pores and an unexpected track shape. As observed in Figure 9, conditions negatively modified the designed results.

Test b. (Case 1).

As a referece for these types of tests where only AISI 316L is used, a first cladding was executed with unused and tumbled AISI 316L powder. Its wide PSD, which covers the whole range of sizes in the requested limits, show the following results. With the suitable parameters, the Figure 10 shows a uniform cladding where no faults like pores or irregular tracks are detected, thanks to the homogeneous PSD that has AISI 316L from supplier A after being tumbled (case 1).

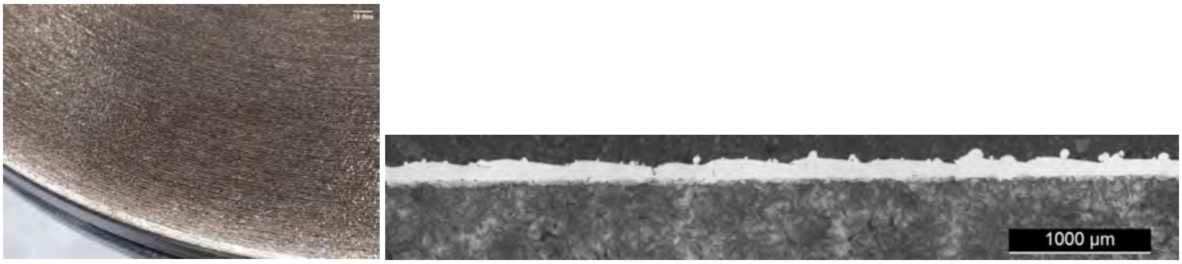

Fig. 10. Surface and cross section images of the EHLA of AISI 316L corresponding to PSD profile of the tumbled powder in 3.1.1 .

For the study on the influence of segregation on the EHLA process, only the extreme situations were analyzed, implying that the finest and greatest powder PSD's needed to be used in this experiment. That is, as observed from the resulting PSD's from segregated powder, only the bottom and top powder from the container were tested, as is shown in Figure 4 (a) and (c), respectively.

Tests executed with finer powder from the bottom of the container show bad quality claddings. This powder PSD differs from the uniformly mixed powder PSD. The altered powder cone at the tip of the nozzle was affected, modifying the working distance required between the machine head and the cladded zone.

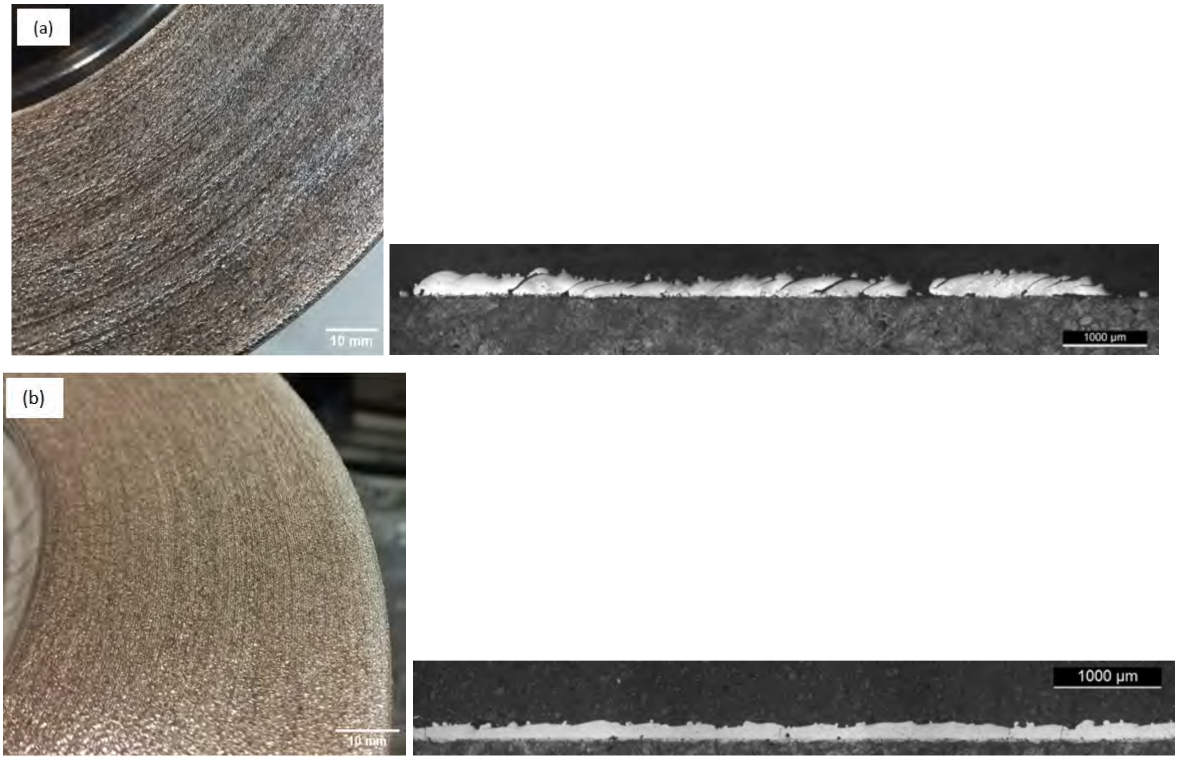

Fig. 11. Surface and cross section images of the cladding deposited with (a) segregated AISI 316L from the bottom of the container before tumbling it, corresponding with Figure 4-a PSD diagram; and (b) with powder from the bottom of the container after tumbling it, corresponding with Figure 5 PSD diagram.

In Figure 11 (a) it can be observed an irregular cladding with differences in height throughout the surface. Wave-shaped tracks are found, indicating instabilities in the process and causing the irregular surfaces. This situation is not the same when powder from the previously tumbled container was used.

It was observed that the PSD extracted from the bottom of the container after tumbling it does not show significant differences from the adequate PSD for this process. As evaluated in case 1 and 4, similar PSD's used in this process are expected to show similar cladding results. This corresponds to the same scenario for this segregated powder. Figure 11 (b) presents results of a uniform cladding that support this statement.

Test c. (Case 2).

For the study on the powder reutilization impact on the EHLA process, the worst case of PSD was tested which corresponds to the powder that was used 3 times, Figure 6 (right), for the same process parameters.

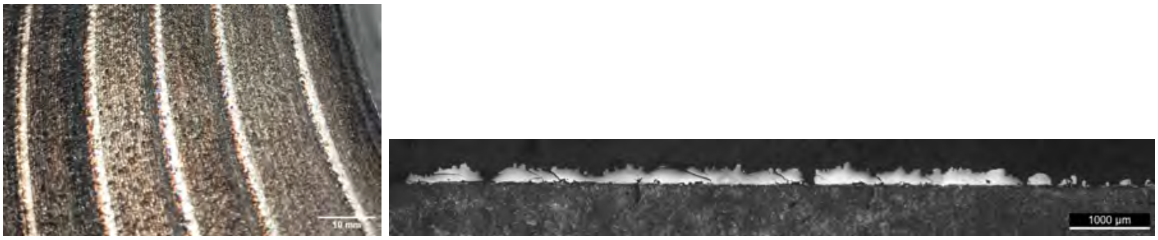

Fig. 12. Surface and cross section images of the cladding deposited with 3 times reused AISI 316L, corresponding Figure 6 (right) PSD.

A severely altered PSD profile was observed in a 3 times used powder, which was expected to alter process conditions. As shown in Figure 12, a faulty cladding with a significant amount of pores is shown as a result of the use of this powder. In a repetitive manner, powder cone shape and behavior was modified, forcing the process to be adapted so as to maintain an adequate distance between nozzle and working piece. As presented in Figure 12, no adjustment of process parameters was able to be performed in order to generate a proper uniform cladding without pores.

4. Conclusions

EHLA additive processes are sensitive to feeding powder PSD changes. An adequate profile of PSD is required for its corresponding situation and parameters. There are several scenarios where powder PSD is altered causing a negative effect in this manufacturing process:

- Misconstrued requirements. An inaccurate delimitation of the product conditions may lead to the reception of inadequate powder for a specific process set-up. Powders with dissimilarities in their Span value have different behavior when ejected from the machine nozzle, altering the designed process parameters and consequently, affecting negatively to the planned manufacturing results.
- Segregation. Environmental vibrations provoke the separation of particles as a function of their sizes, concentrating, in this case, finer particles at the bottom of the containers and greater particles at the top. This causes the additive machine to feed the process with different powder PSD's in different points of the process. Tumbling powder containers before their use has shown positive and effective results to cancel segregation phenomena, restoring the original properties of segregated powder.
- Reutilization. The PSD of the feeding powder undergoes unpredictable modifications after its use. The exclusion of specific distribution ranges hinders the successful execution of the EHLA process, even with parameter adaptations.

This study has shown evidence that a thorough analysis of powder properties, specifically its particle size distribution, prevents undesired faults in the results of EHLA manufacturing process. The results taken from three representative industrial scenarios suggest that properties of powders can experience imperceptible modifications in the absence of precautionary measures. The precautionary measures taken into account in this study consist on:

- The PSD study from new powder providers.
- Using the appropriate equipment for tumbling powder that could have been affected by environmental vibrations.

These measures are proven to be effective against unexpected manufacturing results due to non-homogeneous feeding material, showing uniformly cladded surfaces without pores.

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