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Guidelines about the re-use of the powders

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Content

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1. Introduction

Recycling of unmelted powder feedstock for Laser-powder bed fusion processes has been the subject of a large number of papers published in the literature and also an experimental task of the current SPAcEMAN project. This report summarizes the state of art and the results achieved in the project about this topic.

Data and information provided here represent an updated synthesis of the contents already given in deliverables: D4.1 “Degradation during re-use of powder”, D4.2 “Part performance degradation during re-use of powder” and D4.3 “Best practice procedures for the re-use of powders”.

2. Background

2.1. Powder-property requirements

It is generally accepted that the quality of the powders is a key factor for the LPBF process performance and for final part properties. The interaction between powder feedstock quality and performance of manufactured parts is expressed through a significantly high number of factors, that affect powder properties, powder bed features, process performance [1].

In addition, one of the peculiar features of Laser-powder bed fusion is that only a small fraction of the powder introduced into the system is used to build up the part, most of the unused powder can be conditioned and re-used in subsequent jobs. In fact, the unmelted powder after part manufacturing can be recovered, re-sieved to keep the proper powder particle size, and recycled to improve efficiency. This is the main reason why control of the powder feedstock quality plays an important role on material performance. It is well accepted that the recycled powder might contain some process-affected particles that have different chemical and physical properties than the virgin, fresh powder [2-4]. Deterioration of powder properties can be mainly addressed to three types of interaction with the process environment.

Degradation due to spatters and condensate

Spatters are considered as solid or liquid particles ejected from the melt pool due to various specific mechanisms associated to vapour/liquid formation in the hottest regions of the laser-melt interaction volume. They tend to fall back on the powder bed and can be either incorporated into the final part or remain in the loose unmelted powder that is subsequently recycled. A simplified schematic drawing of the main spatter formation mechanisms is given in figure 2.1.

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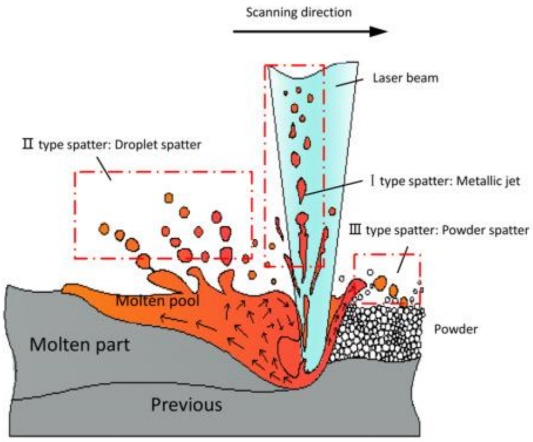

Figure 2.1 - Formation mechanisms of spatters highlighting three proposed different types of spatters [5]

A number of studies are available in the literature on spatter formation and on their characteristics [2,5-7,30]. It has been stated that spatters are generally coarser than the original powder particles, therefore most of them are readily removed from recycled feedstock upon sieving, However, the smallest fraction of the spatters can pass through the sieves and contaminate the powder feedstock. Such particles might feature an irregular shape, impairing flowability, and they might also have picked up minute amounts of residual oxygen in the chamber during their flight in the molten state, thereby leading to compositional drift of the powder with repeated recycling.

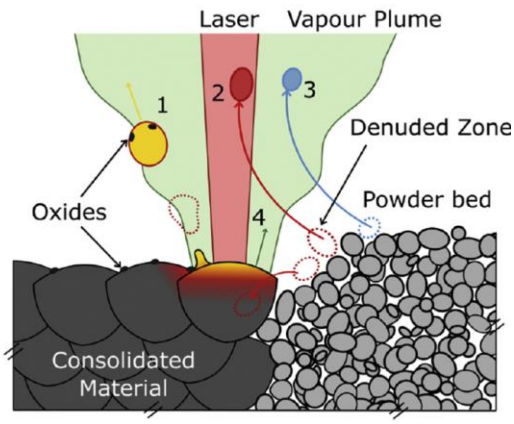

Figure 2.2 - Schematic of laser material interaction [2]

Condensate is a further by-product of the laser-powder interaction [8,9]. The formation of a vapor plume (see figure 2.2) around the laser beam due to evaporation of some of the melt, leads to a continuous flow of matter away from the processing zone, that condensates onto the walls of the chamber or is directly deposited onto the powder bed and on the surface of already formed spatters. Alternatively, the vapour is claimed to form a condensate in the form of

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submicron powder (soot) when the vapour cloud around the melt pool is quenched due to the inert gas filling the chamber.

Spatters are by far the main product of the laser-powder interaction. In order to gain a better understanding of their effects on powder degradation, a detailed state of the art on their formation upon LPBF processing has been included in project deliverable D4.1 “Degradation during re-use of powder” and it is here summarized.

Spatters form when the liquid acquires enough kinetic energy to exceed the capillary pressure of the melt. The liquid motion is directly related to the strength of the vapor recoil force, which depends on how much energy is being applied. A vapor jet is formed when the temperature of the melt pool exceeds the vaporization temperature, causing concurrent removal of molten material by melt expulsion. The ejected metal interacts with the vapor plume above the melt pool and breaks into micro-droplets during traveling through the laser beam, thereby forming droplet-like spatters. Recoil pressure is widely recognized to be the main contributor to metal particle ejection during LPBF [10-12]. However, Ly et al. stated that vapor-driven entrainment of single solid particles may also be one of the dominant mechanisms leading to spatter generation [9]. As the laser is scanned over the powder bed, the metal vapor plume from the melt pool induces an inward gas flow that pulls the particles upward and backward relative to the scanning direction. Depending on the flight trajectories, a large number of particles becomes submerged into the melt pool. The rest will either travel towards the laser irradiation field, miss the laser beam and get ejected as cold spatters, or interact with the laser beam, get melted and ejected as hot splashes of particles as shown in Figure 2.3 [13]. Particle density has a significant effect on the entrainment gas velocity. Higher velocities and more spatters are expected to be generated for materials with low density such as aluminium or titanium, as opposed to stainless steel [9,14].

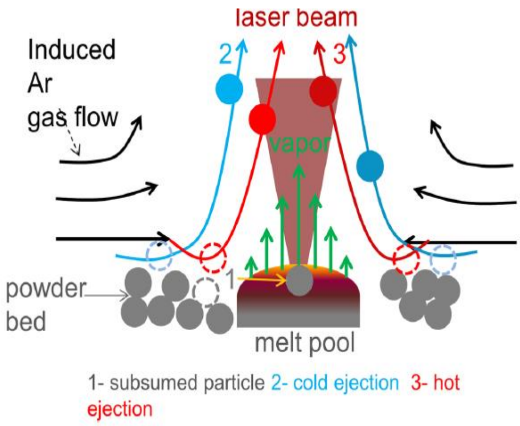

Figure 2.3 – Spatter formation mechanisms in the LPBF process [13]

Laser power and scanning speed have the largest impact on the degree of spatter, whereas environmental pressure influences the spattering behaviour by controlling the divergence angle of the vapor jet and the inert gas flow [11,13]. The increase in laser power during the LPBF

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process results in the creation of more spatters due to the increase of energy input and keyhole formation [12]. High laser power and low scanning speed lead to overheating, whereas the opposite situation, when high scanning speed and low power are employed, gives rise to the balling effect due to underheating [15].

Guo et al. [11] investigated the importance of the environmental pressure on powder spatter behaviour. When the surface of the melt pool reaches the boiling temperature, vaporization becomes intense, generating a metal vapor flow and causing particle ejection from the melt pool area. Vapor formation results in different outcomes depending on environmental pressure as depicted in Figure 2.4. The intensity of the chamber pressure determines the divergence angle of the vapor jet and the gas flow behaviour. Under vacuum conditions, the metal vapor expands freely creating a larger divergence angle. The presence of a given environment pressure promotes spatter generation by attracting the gas flow together with the hot entrained particles toward the vapor field above the melt pool. However, the total amount of spatters, affected by the angle of divergence and the gas flow, drops with a further increase of environment pressure as it forms a narrower vapor jet. Therefore, higher chamber pressure in the LPBF process can reduce spattering and, as a result, improve the quality of final parts.

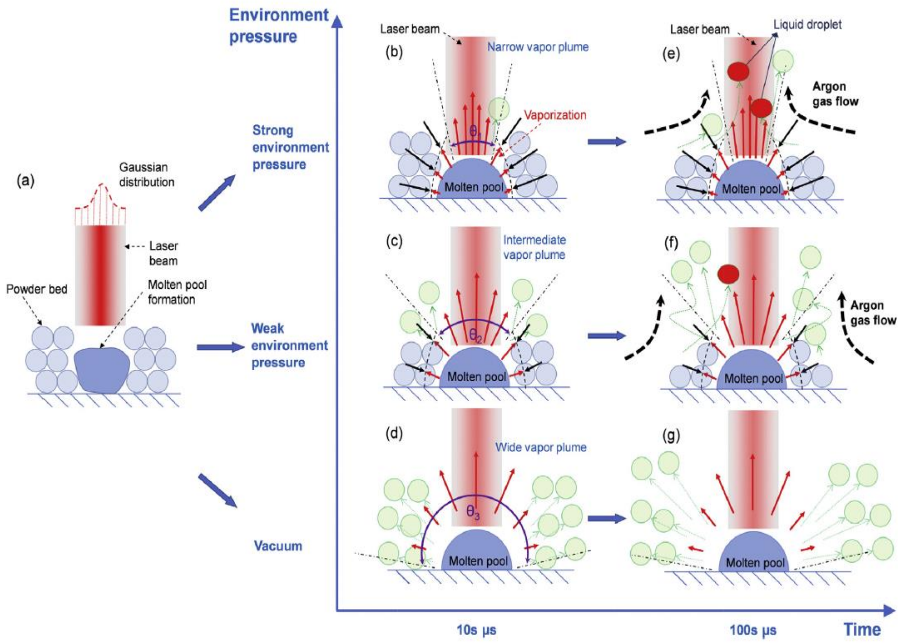

Figure 2.4 – Influence of environmental pressure in the AM system on the powder spatter behaviour [11]

Several molten particles may then merge and form larger droplets or agglomerates of powder that can potentially lead to defects such as lack of fusion in the LPBF process. In addition, the presence of residual gas, between and inside the volume of particles, or oxygen desorbed on the particle surface can be heated up and expand, pushing particles upward into the laser beam [9].

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Splashes are particular spatters that are expelled directly from the melt pool area (formed directly form the liquid melt) due to the recoil pressure. This type of spatters usually have a spherical shape as they undergo a similar cooling regime as gas atomized powder. The ejection velocity from the melt pool may reach up to 3-8 m/s, according to Ly et al. [9]. Hot spatters, on the other hand, are those particles that meet the laser beam path or are attracted by it, and become entrained by the vapor jet. The hot particle ejections due to entrainment are dependent on the particle size and this dependence shows different mechanisms: the finer the particle the higher the velocity values (from 6 up to 20 m/s). Powder particles that pass behind the laser field without getting molten are called entrained cold spatters [16,17]. For cold ejected particles, the velocities range from 2 to 4 m/s and no change in particle size is associated to their formation [9].

Hot and cold entrained particles typically have sizes in the range of virgin powder particle sizes; however, there are cases where the particles are larger. This happens when a melt droplet is ejected from the vapor plume area, so that it lands on the powder bed surface in liquefied state. It collides with the feedstock powder shortly after ejection, wets multiple particles, forming one large agglomerate with irregular shape [9,18].

Influence of spatters on the defect formation in LPBF

Many research works have shown that a large number of melted droplets fall back on the substrate and, thereby, increase the roughness of each powder layer. The presence of such droplets in the powder bed leads to the formation of pores and defects such as lack of fusion [9]. More precisely, depending on the spatter formation mechanism, particles ejected from the melt pool area or entrained by the vapor and gas have different flight trajectories, thereby creating rich- and low-spatter regions on the powder bed. As the spatters are ejected from the melt surface, they fly outward, mainly in the gas flow direction. Some particles with higher kinetic energy can also travel against the gas flow but, due to the opposing drag force, they land very close to the melt pool. Thus, it can be concluded that most of the spatter particles accumulate in the direction of the inert gas (spatter-rich region) while a lower fraction falls opposite to the argon flow direction or directly on the previously solidified track (low-spatter region), as depicted in Figure 2.5.

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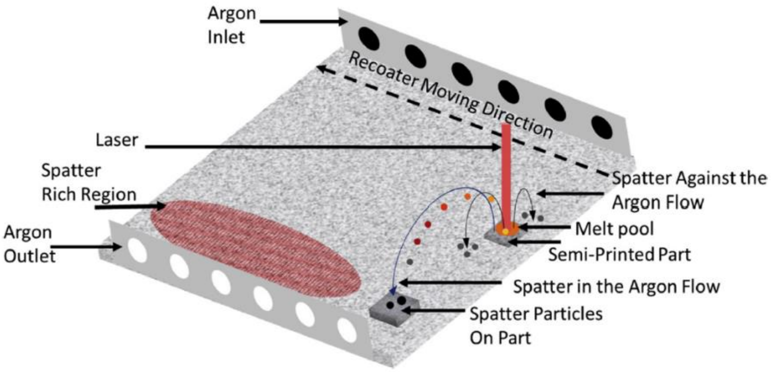

Figure 2.5 – Spatial distribution of spatters on the build plate during SLM [16]

The parts produced from the spatter-rich region appear to have higher porosity than those coming from low-spatter region. It is also important to mention these parts show different pore shapes. Porosity formed in the former region shows elongated pores with irregular shape as opposed to relatively spherical pores found in low-spatter regions. This is an indication of different pore formation mechanisms. Irregular and elongated pores are usually an evidence of incomplete melting (lack of fusion) between layers due to the presence of spatter particles between them [16].

Degradation due to contamination of powder and elemental drift

It is also well established that splashes ejected from the melt pool may be exposed to oxidation and to a change in chemical composition during flight, leading to contamination of the surrounding powder bed. Indeed, during the melting stage, the material interacts with residual oxygen which is introduced either with the feedstock powder or comes from the atmosphere in the building chamber [19]. An additional source of elemental drift is due to preferential evaporation of the most volatile elements (e.g. Mg, Zn, Mn) [20]. In addition, when the recycled powder is handled and stored, a number of additional opportunities for contamination can exist. Foreign bodies that can entrain into the powders are for instance fragments of gloves, cleaning brushes, residual powder form previous builds.

A study carried out by Wang et al. showed that the chemical composition of the spatters collected from a 316L powder processed by LPBF is almost the same as the virgin material apart from the contents of O, Si and Mn, which increase significantly [13]. Some more data about the pickup of oxygen and nitrogen by the multiple re-use of a Ti6Al4V alloy powder are given in Figure 2.6 [19]. These data are to be considered as only informative, since Ti is well known to be much more reactive toward environmental gases than steels and other alloys.

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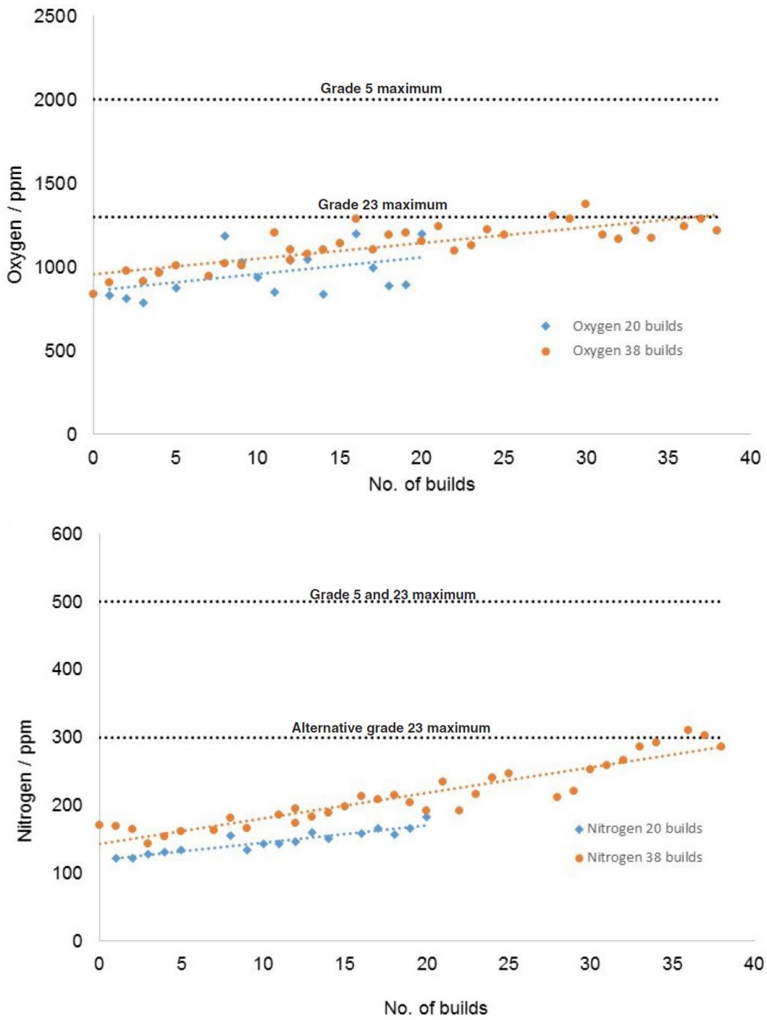

Figure 2.6 – Oxygen and nitrogen content over two separate re-use studies in a Ti-6Al-4V alloy processed by LPBF [19]

Degradation due to effects in near-melt zone region

Of lower importance, but still to be taken into consideration in our list, are the effects in the so-called near-melt zone, where the powder experiences the indirect effect of the laser power, inducing high-temperature heating, hence possible changes in particle shape and size due to agglomeration, with a reported increase in fraction of the coarser particles, as well as modification of the chemical composition and phase constitution [4].

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2.2. Powder reuse

Powder recycling is a requisite of paramount importance for sustainability of LPBF processes since the powder feedstock used in AM is quite expensive due to complex production technologies and the current high-quality requirements set by industry. One of the key challenges in AM is therefore the understanding about how powder characteristics influence final part properties.

It has already been mentioned that variations in powder properties can occur between batches and at numerous stages in the powder lifecycle, i.e. upon feedstock production, powder sieving/handling, during storage, and in the build chamber itself.

Research on powder reuse in Selective Laser Melting have been carried out in recent years. Several studies [21-28] confirmed that powder degradation depends on the processing conditions, environment and feedstock material. Heiden [21] discovered that depending on the particle shape, their investigated 316L stainless steel showed an increase in amounts of Si and Mn on the powder particle surface after recycling. Irregular particles instead showed an increase in Si, Ni, S, and O, possibly due to segregation during laser processing. Moreover, the virgin 316L powder contained a uniform SiO_2 oxide layer of irregular thickness on the surface, while recycled particles featured a much higher presence of discrete oxide nodules composed of a mixture of SiO_2 and $MnCr_2O_4$.

Normally, AM spatter and sieved powder are comparable in size, suggesting that sieving helps in removing larger spatter particles. Hot and cold spatters are difficult to recognize in the powder bed as they are usually of the same spherical shape and size as the virgin powder. Particle size distribution generally undergoes a slight increase with reuse. The reduction in fines in the powder distribution and an increase in larger powder generated through processing were found to improve flowability [28]. Poor particle distribution and higher fraction of voids between particles may lead to the entrapment of dissolved gases in the melt pool. A loss of fine particles can also lead to lower absorption of the laser energy and a decrease in actual heat input [21].

2.3. Strategies for powder recycling

In general, only a limited fraction of the powder fed in the LPBF system is consumed for producing a 3D part. Most of the powder is left unmelted and can be re-used for subsequent jobs after sieving. Sieving is required to separate from the feedstock the coarser particles (large spatters or agglomerates of powder particles) and the fines, mainly produced from the condensate. By Sieving, the optimal particle size distribution of the powder can be kept under control, in order to preserve flowability and the ability to achieve a proper density in the powder bed. Figure 2.7 schematically summarizes the flow of the feedstock powder in LPBF processing.

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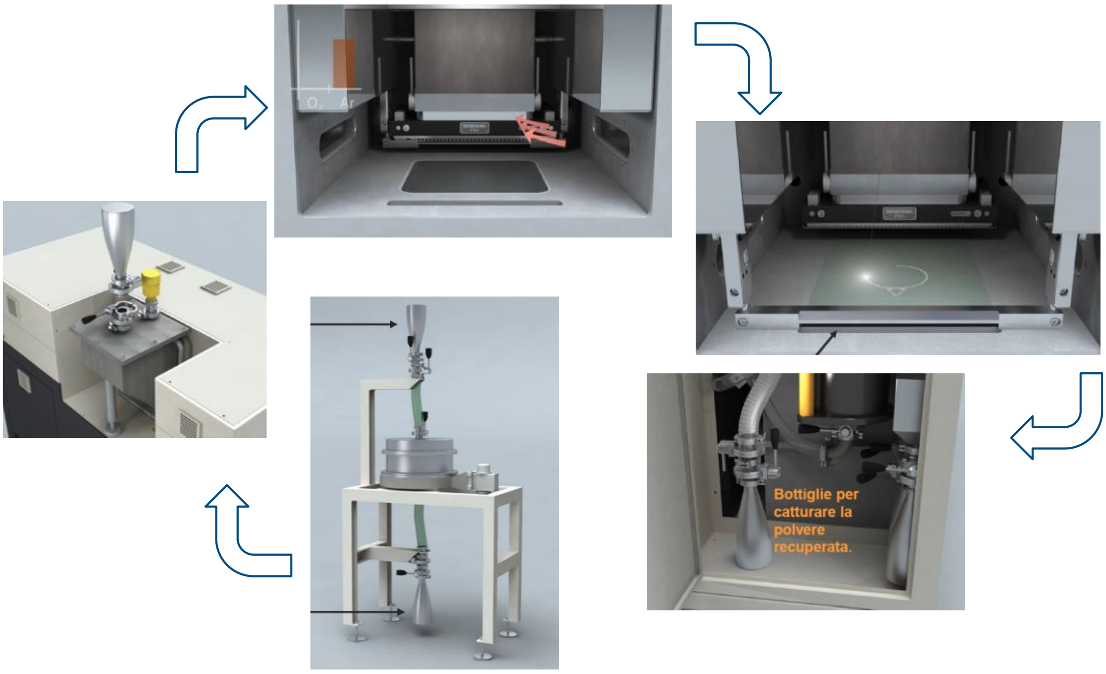

Figure 2.7 – The recycling loop of feedstock powder in LPBF [adapted from www.renishaw.com*]*

It is now well accepted that sieving is not sufficient to fully remove all spatters, irregular and oxidised particles from the feedstock. Hence, it can be assumed that a fraction of the degraded powder will be incorporated into any recycled powder batch, and this fraction will progressively increase with each subsequent recycling loop. The already mentioned vaporization of alloying elements and the oxygen pick-up will also contribute to the gradual expected change in alloy composition upon recycling [17].

Once powder conditioning (collecting of unmelted powder and sieving it to proper size) has been carried out, powder recycling can be done in a variety of ways including the reintroduction of the sieved powder after each build or the use of a blend of sieved powder together with a controlled amount of virgin powder. In both cases, there is the option of mixing the two batches (sieved and fresh powders) before introduction in the AM system or just adding the sieved powder on top of the pre-existing feedstock. A common point of the mentioned strategies is that the recycled powder tends to be diluted at variable rates with virgin powder so as to mitigate possible effects induced by powder degradation. In Figure 2.8 an example of a recycling strategy is depicted, providing clear evidence of the extensive mixing effect with the fresh powder [29]. It is also understood that the precise tracking and monitoring of the powder feedstock becomes progressively hard as the number of recycling loops and the variety of the produced parts increase (making the ratio between powder actually processed for part production and unused powder unpredictable). This suggests the adoption of conservative strategies aimed at the

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systematic introduction into the loop of a sufficient amount of virgin powder, in order to avoid the possibility of having powder volumes subjected to extensive degradation.

Figure 2.8 – Schematics of the recycling strategy, demonstrating the addition of 50 % virgin powder after each cycle, for 10 cycles. The greyscale indicates a continuous change of the powder state [29]

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3. Suggested guidelines for powder recycling

3.1. Guidelines contained in existing standards

In recent times, standards for AM-related topics have been published in relatively large number. European standards that could potentially give some suggestions related to powder recycling are the following:

- BS EN ISO/ASTM 52904:2020 - Additive manufacturing — Process characteristics and performance — Practice for metal powder bed fusion process to meet critical applications,
- BS EN ISO/ASTM 52907:2019 - Additive manufacturing - Feedstock materials - Methods to characterize metal powders,
- BS EN ISO/ASTM 52941:2020 - Additive manufacturing — System performance and reliability — Acceptance tests for laser metal powder-bed fusion machines for metallic materials for aerospace application,
- BS EN ISO/ASTM 52942:2020 - Additive manufacturing - Qualification principles - Qualifying machine operators of laser metal powder bed fusion machines and equipment used in aerospace applications.

The topic about powder recycling is hardly covered and only BS EN ISO/ASTM 52904 standard gives some very general guidelines about the practice to be adopted. Suggestions are in line with the scientific output described in previous sections and can be summarized as follows.

For the description of materials for PBF processed it is claimed that powder type could be: “virgin, used, blend or mix” (section 4.1.2).

More precisely, it is accepted that: “Used powder is allowed. The proportion of virgin to used powder shall be recorded and reported for each production run on the manufacturing plan. Automated powder feed systems may not allow the proportion of virgin to used powder to be accurately measured and recorded on the manufacturing plan. In such systems the feedstock shall be considered used powder. The maximum number of times that used powder can be consumed as well as the number of times any portion of a powder lot can be processed in the build chamber shall be validated in accordance with 7.3. After a build cycle, any remaining used powder may be blended with virgin powder to maintain a powder quantity large enough for the next build cycle. The critical powder attributes impacting qualifications in accordance with 7.3 shall be analyzed regularly. All used powder shall be sieved with a sieve having a mesh size appropriate for removing any agglomerations. All powder sieves used to manufacture parts shall

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have a certificate of conformance that they were manufactured to ISO 9044 or Specification E11" (section 5.6).

Section 7.3 summarizes the Machine, Process, and Part Qualification. It is suggested that "A qualification build manufacturing plan shall be created and used to build the test specimen(s) for the purpose of qualification. Once the qualification build results have been validated, the parameters used for the qualification build are recorded as PBF baseline parameters, and establish the parameters for subsequent builds". However, no specific instruction are given about powder recycling practices.

Finally, at sections 10.4 and 10.5 general guidelines are given about handling and storage of used feedstock.

- "At the conclusion of the build cycle, all unmelted feedstock shall be removed from the PBF machine and stored in an appropriate container clearly labelled as used feedstock. Deviations from this practice shall require a qualification procedure to ensure part quality is not affected. Automated feedstock systems shall not be allowed to move feedstock from the build chamber directly back to the feed mechanism without previously demonstrating that such operations do not adversely affect part quality."
- "When available, post-build powder removal shall be performed in accordance with the machine manufacturer's recommendation. Powder shall be removed using a suitable process (for example, compressed gas under a confined compartment, brushing or vacuum). … omissis … All loose powder collected outside of a confined compartment (for example, powder recovery system) shall not be reused".

In summary, it appears that practices for feedstock recycling are accepted even for parts subjected to critical applications. However, no guidelines are provided in standards yet. Specific procedures for powder recycling are left to the definition of users, after a proper qualification build manufacturing plan is created.

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4. Experimental results

4.1. Property degradation in materials and in final parts

When careful recycling strategies are adopted and supported by the introduction in the loop of fairly large amounts of fresh powder, the effects of powder degradation and of depletion of final parts properties are largely mitigated and become often negligible.

Both the literature surveys and the experimental results generated within the SPAcEMAN project clearly confirm this statement. In the following figure 4.1 some data are recalled about the pickup of elements, collected from laboratory experiments at LTU. The results show that the pickup of environmental gases is very limited or negligible for the steels investigated within the SPAEMAN project for LPBF processing.

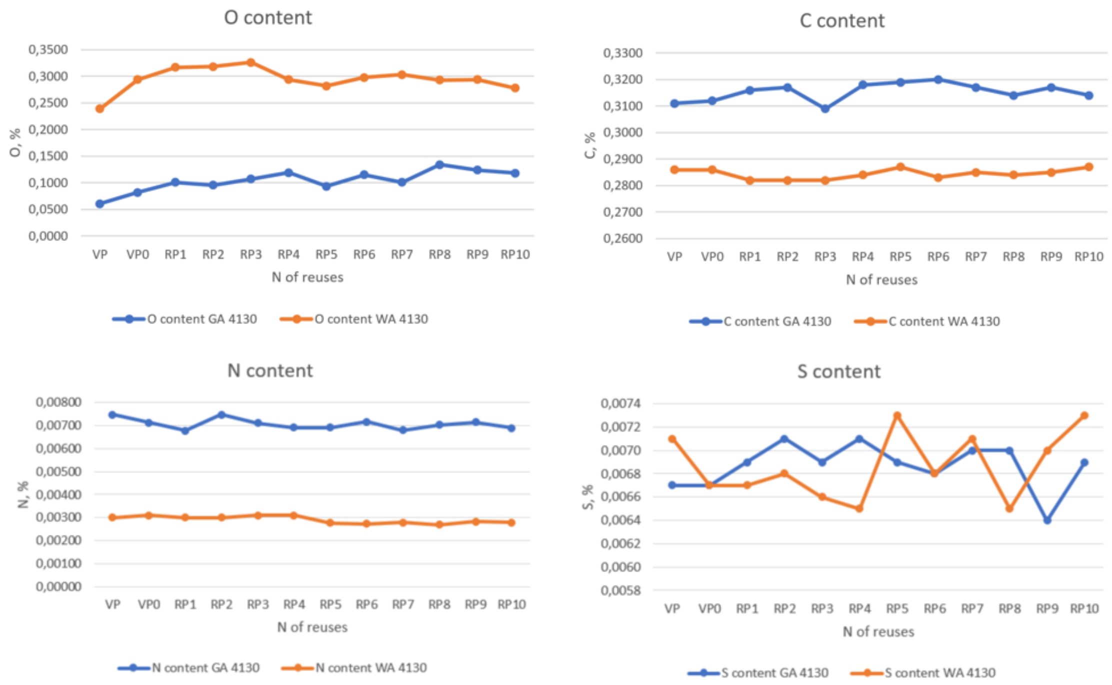

Fig. 4.1 - Chemical composition of SPM13 WA and X4130 GA steel powders after different degrees of re-use. Data taken from deliverable D4.1

Consistent results have been collected by a testing campaign carried out in "industrial environment", at the shop of project partner Certema. The following strategy for powder

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recycling was adopted using an LPBF system normally devoted to provide a printing service for local companies.

- After process optimization for the investigated steel powder, a number of builds (runs) was performed to produce specimens for testing activities and other parts as prototypes for the project,
- run 0 was performed again using fresh powder,
- run 1 used 50% of recycled and sieved powder and 50% of fresh powder (mixing them before introducing into the tank,
- for all the following runs the same procedure was adopted (50% fresh powder mixed to 50% of powder already contained in the tank), keeping track of what had been printed with each batch of powder,
- for each run, together with the parts of interest, tensile specimens and sealed capsules containing sample of the powder were produced.

The testing was conducted on a Concept Laser M2 Cusing LPBF system. Overall, four different runs could be investigated, considering the amount of powder available.

Figure 4.2 depicts general views of the powder batches corresponding to the different runs. It can be observed that no relevant changes in shape and size distribution of powder particles are evident. Generally, the powder particles feature an irregular blocky shape with limited number of asperities and satellites, as inherited by the water atomization process followed by a mechanical post-treatment. Only occasionally, and even in the powder of run 0, perfectly spherical and coarse particles can be detected (see for instance yellow arrowed particles in figure 3.2.a). It can be assumed that they are spatters produced at the laser-melt interaction region that have been ejected from melt pool and landed back on the powder bed after solidification.

Figure 4.3 reports energy dispersive spectrometry (EDS) microanalysis results about the chemistry of the small features visible on the surface of the spherical powder particles. Position #1 and corresponding spectrum represent an oxide nucleus growing on the particle surface, spectrum #2 is for an apparently clean surface region, while spectrum #3 corresponds to the right side of the powder particle decorated with “soot-like” precipitates.

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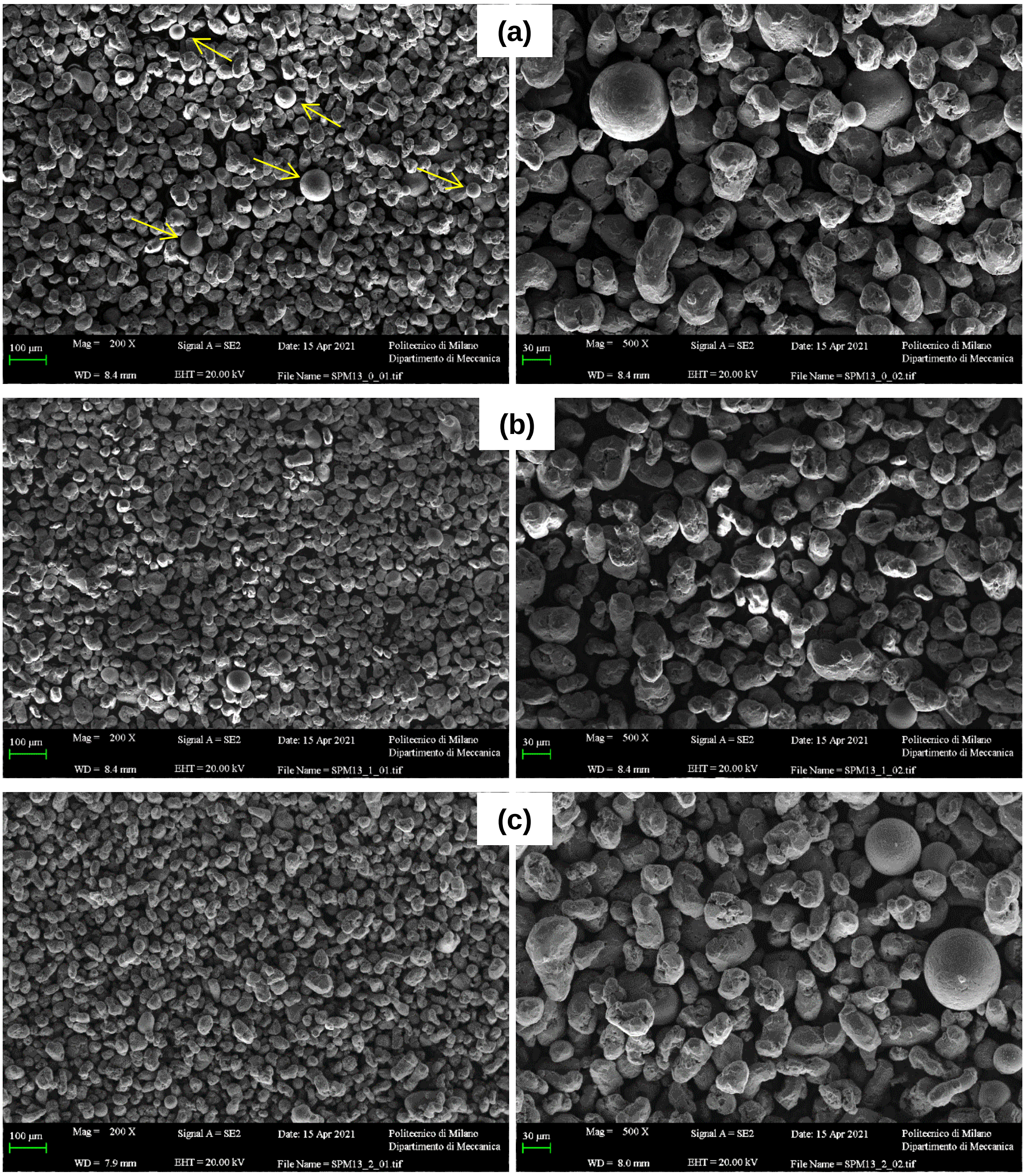

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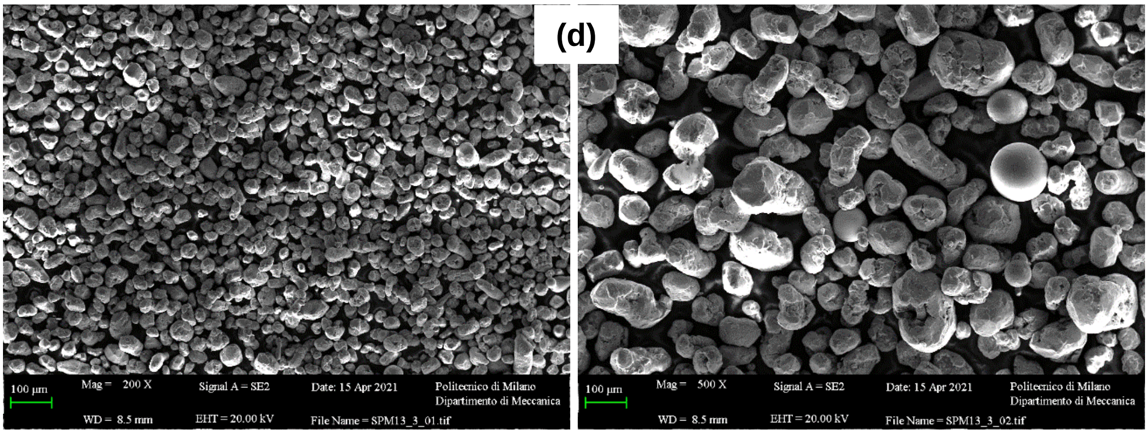

Figure 4.2 – Morphology of SPM13 powder particles as a function of the accumulated recycling runs, (a) run 0, (b) run 1, (c) run 2, (d) run 3

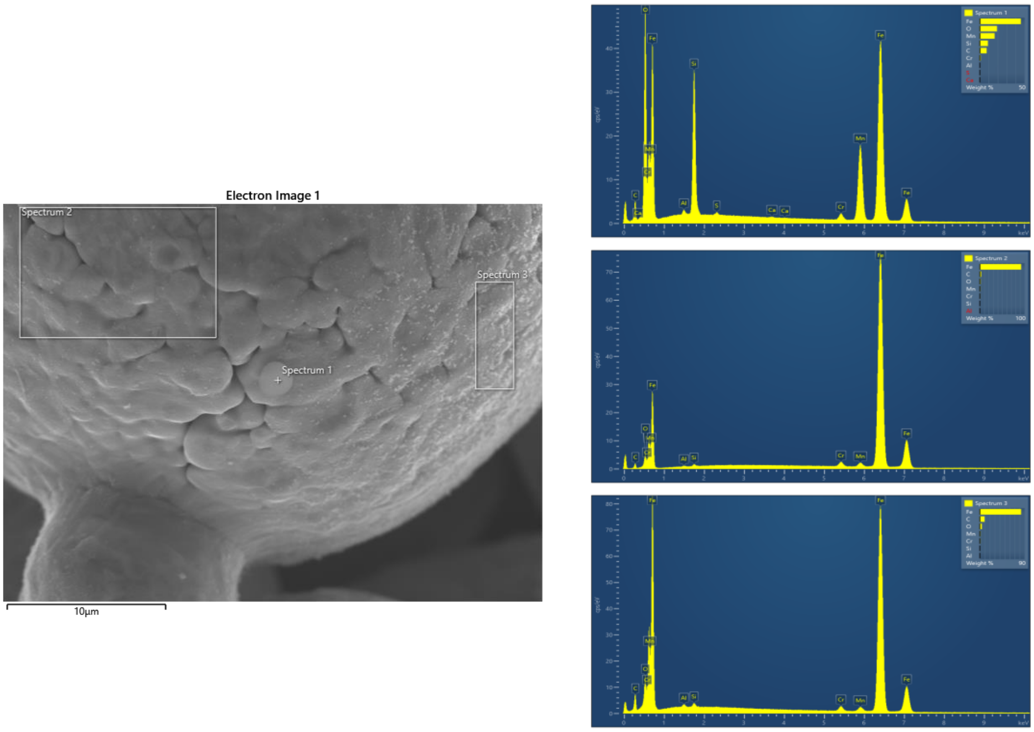

Figure 4.3 – EDS microanalyses at specific spots on surface of a powder particle

A summary of the chemical composition of powder samples collected from sealed capsule after each run is given in table 4.1. The reference X4130 GA powder was also considered for comparison purposes. Considering the different runs of the SPM13 WA powder, it can be

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observed that no significant changes took place during the repeated use of the powder, at least within the conditions and number of runs here considered.

Table 4.1 – Chemical composition (in weight %) of the investigated powders during recycling tests

	C- tot	N	O	S
X-4130 Run 0	0,329	0,0107	0,063	0,008
SPM13 Run 0	0,325	0,0055	0,252	0,015
SPM13 Run 1	0,324	0,0050	0,254	0,015
SPM13 Run 2	0,324	0,0056	0,247	0,015
SPM13 Run 3	0,324	0,0054	0,242	0,015

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5. Conclusions

Powder recycling in LPBF represents a well accepted practice for sustainable part manufacturing. A wide range of literature reports are available about the scientific background and about the expected degradation of properties when using recycled powder feedstock. European standards also consider and accept this practice, even though specific guidelines are not directly +provided.

The experimental results collected within the frame of the SPAcEMAN project and all the data available from literature confirm that steel powder reuse in LPBF processing can be adopted with negligible effects on part performance, provided proper sieving and mixing procedures with fresh powder are followed.

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