

UNIVERSIDADE FEDERAL DE SÃO CARLOS
CENTRO DE CIÊNCIAS EXATAS E DE TECNOLOGIA
DEPARTAMENTO DE ENGENHARIA DE MATERIAIS

**A critical review of the reusability of metal powder in
additive manufacturing**

Nathan Bissaro Carvalho

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**A critical review of the reusability of metal powder in Additive
Manufacturing**

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Orientador: Prof. Dr. Piter Gargarella

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NOME: Nathan Bissaro Carvalho

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BANCA – ASSINATURAS:

Prof. Dr. Piter Gargarella

Documento assinado digitalmente
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Data: 04/02/2024 11:30:27-0300
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Dr. Angelo Fernandes Andreoli

Documento assinado digitalmente
gov.br ANGELO FERNANDES ANDREOLI
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RESUMO

O presente trabalho de revisão aborda a viabilidade do reuso de pó metálico na manufatura aditiva (MA), explorando diversos aspectos cruciais que influenciam o processo. Ele foi motivado pela ausência de estudos similares a esse, buscando estudar e condensar o impacto de múltiplos ciclos de impressão em diversos aspectos do pó. Além disso, buscou-se estudar também os efeitos dessa sequência de impressões para as propriedades mecânicas das peças fabricadas.

A reutilização do pó influencia de forma significativa a morfologia e a distribuição do tamanho de partículas. Identificou-se que essas alterações de morfologia e distribuição de tamanho geralmente ocorrem com o aumento do número de ciclos, afetando diretamente a qualidade do material depositado durante a manufatura aditiva.

Foi observado também que o reuso pode comprometer o empacotamento e a fluidez de pó, influenciando a facilidade de formação e homogeneidade da camada depositada, resultando em possíveis falhas no processo. Essas alterações não são dadas diretamente por fatores de processo, mas por características do pó, como rugosidade, teor de oxidação, morfologia e distribuição de tamanho, além de outros fatores mais específicos como fases magnéticas presentes no pó, por exemplo.

Defeitos também ocorrem nas peças obtidas por MA como por exemplo o aumento na densidade e no tamanho de porosidades, variações composicionais devidas principalmente à oxidação e também o incremento de defeitos como a falta de fusão de material e os vazios por má distribuição de pó. O fenômeno de *spattering*, no qual porções significativas do pó disparam a partir do leito metálico, também altera de forma significativa o pó que será reutilizado e gera defeitos nas peças fabricadas na impressão atual, como os vazios e descontinuidades de camada.

Esses problemas todos podem levar à uma piora significativa em escala local e global das propriedades mecânicas nas peças fabricadas por material reutilizado que não tenha sido rigorosamente estudado, controlado e tratado. Esse tratamento consiste na passagem por etapas de peneiragem entre os ciclos, para retirar aglomerados e *spatters* e assim mitigar efeitos negativos causados por esses fenômenos que ocorreriam em decorrência da decaída na qualidade do pó.

Finalmente, esta revisão enfatiza a complexidade do reuso de pó metálico na manufatura aditiva, destacando a importância de monitorar cuidadosamente as características do pó entre cada ciclo de impressão para garantir melhores resultados. A compreensão do impacto nas propriedades das peças fabricadas é crucial para estabelecer limites de reciclagem viáveis, equilibrando eficiência econômica com a qualidade do produto final. Quanto ao que se espera do avanço da pesquisa nessa área, incluem-se estudos para mais ligas e mais processos de manufatura aditiva, investigar propriedades que não só as mecânicas. Somado a isso, espera-se um estudo mais avançado do efeito do reuso na microestrutura e nas fases formadas e no impacto da qualidade do acabamento superficial das peças fabricadas.

Palavras-chave: Reuso de pó metálico, Manufatura aditiva, Defeitos comuns, Número de ciclos, Características do pó.

RESUMO EM LÍNGUA ESTRANGEIRA

Abstract

The present review work addresses the feasibility of metallic powder reuse in additive manufacturing (AM), exploring various crucial aspects influencing the process. It was motivated by a lack of similar studies, seeking to examine and consolidate the impact of multiple printing cycles on various powder aspects. Additionally, the study aimed to investigate the results of the printing sequences on the mechanical properties of the manufactured parts.

Powder reuse significantly influences particle morphology and size distribution. It was identified that these changes in morphology and particle size distribution generally occur with an increase in the number of cycles, directly affecting the quality of the material deposited during additive manufacturing.

It was also observed that reuse can compromise powder packing and flow, influencing the ease of layer formation and homogeneity of the deposited layer, resulting in potential process failures. These changes are not directly caused by process factors but by powder characteristics such as roughness, oxidation content, morphology, and size distribution, as well as more specific factors like magnetic phases present in the powder.

Defects also occur in AM-obtained parts, such as increased density and size of porosities, compositional variations mainly due to oxidation, and an increase in defects such as lack of material fusion and voids due to uneven powder distribution. The spattering phenomenon, in which significant portions of the powder are ejected from the metal bed, also significantly alters the powder that will be reused and generates defects in the parts manufactured in the current print, such as voids and layer discontinuities.

All these issues could lead to a significant deterioration in local and global mechanical properties of parts manufactured from reused material that has not been rigorously studied, controlled, and treated. This treatment involves stages of sieving between cycles to remove agglomerates and spatters, thus mitigating negative effects caused by these phenomena resulting from the decline in powder quality.

Finally, this review emphasizes the complexity of metallic powder reuse in additive manufacturing, highlighting the importance of carefully monitoring powder characteristics between each print cycle to ensure better results. Understanding the impact on the properties of manufactured parts is crucial for establishing viable recycling limits, balancing economic efficiency with the quality of the final product. Regarding expectations for advances in research in this area, studies of different alloys and other additive manufacturing processes are included, investigating properties beyond just mechanical ones. In addition, an advanced study of the effect of reuse on microstructure and formed phases and the impact on the quality of the surface finish of manufactured parts is anticipated.

Keywords: Metallic powder reuse, Additive manufacturing, Common defects, Number of cycles, Powder characteristics.

Resumé

Le présent travail de révision aborde la faisabilité de la réutilisation de poudre métallique dans la fabrication additive (FA), explorant divers aspects cruciaux qui influent sur le processus. Il a été motivé par l'absence d'études similaires, cherchant à étudier et à condenser l'impact de multiples cycles d'impression sur divers aspects de la poudre. De plus, des études ont également été menées sur les résultats de cette séquence d'impressions concernant les propriétés mécaniques des pièces fabriquées.

La réutilisation de la poudre influence de manière significative la morphologie et la distribution de la taille des particules. Il a été identifié que ces changements de morphologie et de distribution de taille surviennent généralement avec l'augmentation du nombre de cycles, affectant directement la qualité du matériau déposé pendant la fabrication additive.

Il a également été observé que la réutilisation peut compromettre l'emballage et l'écoulement de la poudre, influençant la facilité de formation et l'homogénéité de la couche déposée, entraînant ainsi d'éventuelles défaillances dans le processus. Ces changements ne sont pas directement dus à des facteurs de processus, mais à des caractéristiques de la poudre telles que la rugosité, le taux d'oxydation, la morphologie et la distribution de la taille, ainsi que d'autres facteurs plus spécifiques tels que les phases magnétiques présentes dans la poudre, par exemple.

Des défauts surviennent également dans les pièces obtenues par FA, tels que l'augmentation de la densité et de la taille des porosités, des variations compositionnelles principalement dues à l'oxydation, ainsi que l'augmentation de défauts tels que le manque de fusion de matériau et les vides dus à une mauvaise distribution de la poudre. Le phénomène de projection, dans lequel des portions significatives de la poudre sont éjectées du lit métallique, altère également de manière significative la poudre qui sera réutilisée et génère des défauts dans les pièces fabriquées lors de l'impression actuelle, tels que des vides et des discontinuités de couche.

Tous ces problèmes pourraient entraîner une détérioration significative à l'échelle locale et globale des propriétés mécaniques des pièces fabriquées à partir de matériau réutilisé qui n'aurait pas été rigoureusement étudié, contrôlé et traité. Ce traitement consiste à passer par des étapes de tamisage entre les cycles pour éliminer les agglomérats et les projections, atténuant ainsi les effets négatifs causés par ces phénomènes qui résulteraient de la dégradation de la qualité de la poudre.

Enfin, cette revue souligne la complexité de la réutilisation de poudre métallique dans la fabrication additive, mettant en avant l'importance de surveiller attentivement les caractéristiques de la poudre entre chaque cycle d'impression pour garantir de meilleurs résultats. La compréhension de l'impact sur les propriétés des pièces fabriquées est cruciale pour établir des limites de recyclage viables, équilibrant l'efficacité économique avec la qualité du produit final. En ce qui concerne les attentes en matière d'avancées de la recherche dans ce domaine, des études pour plus d'alliages et de processus de fabrication additive sont incluses, ainsi que des investigations sur des propriétés autres que mécaniques. De plus, une étude plus avancée de l'effet de la réutilisation sur la microstructure et les phases formées, ainsi que sur l'impact de la qualité de la finition de surface des pièces fabriquées, est anticipée.

Mots-clés: Réutilisation de poudre métallique, Fabrication additive, Défauts courants, Nombre de cycles, Caractéristiques de la poudre.

LIST OF ACRONYMS

AM – Additive Manufacturing
PBF – Powder Bed Fusion
DED – Direct Energy Deposition
SLM – Selective Laser Melting
PSD – Particle Size Distribution
MP – Metal Powder
SLS – Selective Laser Sintering
EBM – Electron Beam Melting
EOL – End Of Life
EDM – Electrical Discharge Machining

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1 INTRODUCTION

Additive manufacturing (AM) processes, also known as 3D printing, are about building, layer by layer, a near net shape object out of a material feedstock that can vary in form: filaments or powders, for example. With this in mind, the most common thinking is that this process is environmentally friendly like no other and may substitute every other way of manufacturing objects of the day-to-day life, since there is a reduction in material disposal. However, this thinking is misleading since the production of the material feedstock demands a considerable energy consumption [1].

The choice factor behind this theme is in the first instance how fast AM is evolving and how studying it is a clear differential in the acting field of materials science and engineering. Thus, there are no studies that centralize and consolidate powder reusability for AM and, since high powder quality is a must for a good print, those are two of the motivating points to the development of this work [1][5].

In addition to the mentioned motivating points, it is necessary to call out that metal powder is one of the main costs of the 3D printing process, so being able to reuse it is interesting to reduce costs and also the environmental impact that can be avoided by the reuse of powder are points that counts favorably to motivate this work [2].

In this thesis, the author will focus on critically reviewing papers about powder reusability on AM. The objective is to address the effect of powder reuse on the properties of powders and parts produced by AM, with a focus on Powder Bed Fusion and Direct Energy Deposition, the two most used methods of AM of metals.

2 METHODOLOGY

The author used platforms such as Google Academic, Google Patents, Elsevier and Science Direct to search for articles and patents, and the general research revolved around key words such as metal powder, additive manufacturing, reusability, recyclability, recycling and reuse. Some articles were also recommended by the author's supervisor, Prof. Dr. Piter Gargarella, as an additional source of content.

Once articles and patents were obtained and validated as of interest for this study, they were sorted by metal alloy and AM technique, they were split into three categories for materials (stainless steels, titanium alloys and miscellaneous) and two

for AM techniques (powder bed fusion and direct energy deposition).

3 THEORETICAL FOUNDATION / LITERATURE REVIEW

3.1 AM Techniques

As introduced before AM techniques are based on the construction layer by layer of a piece that has its shape near the final product. Its workflow may vary from technique to technique, but it all starts with a CAD model that is then prepared for printing – slicing procedure – and finally the archive is transferred to the printer.

The workflow in the printer is where things really change from method to method; it depends on the type of material that is used (metals, polymers, resins, composites, etc.), the format of the feedstock (liquid, filaments, powder, etc.), and if the method requires some additional steps to arrive at the final part. In this review, the author chose the PBF and DED (similar methods included) as the methods of interest to be studied, since they are the most common methods that utilize metal powder as the raw material; their definitions and workflow will be further explained in the next sections.

3.1.1 Powder Bed fusion

Powder bed fusion (PBF) AM processes build objects by melting powdered feedstock, commonly a polymer or metal. PBF processes begin by spreading a thin layer of powder across the build area. Cross sections are then melted one layer at a time, most often using a laser, an electron beam, or intense infrared lamps.

There are many trade names and acronyms for PBF processes; commonly, PBF of metals will be called selective laser melting (SLM) or electron beam melting (EBM), and PBF of polymers will be called selective laser sintering (SLS). As such, SLS systems typically print thermoplastic polymer materials, polymer composites, or, in some cases, ceramics. PBF systems can work with a diverse set of pure metals and alloys; however, in general, these alloys must be compatible with the rapid solidification process that occurs in PBF. One remark to be made concerning the build atmosphere in PBF is that it must also be controlled according to the metal chosen, e.g., to prevent oxidation or unwanted alloying and so its degradation.

To schematize and make it easier to visualize the common points and differences between the polymer and the metal process, schemes are presented and then explained below:

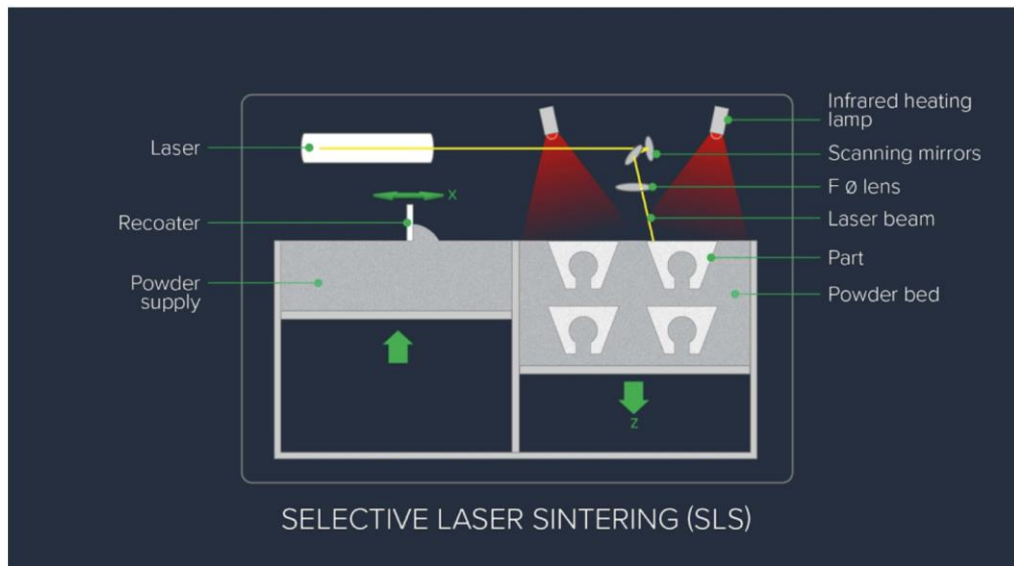


Figure 1: scheme of a SLS machine in work, utilized for polymers. Figure taken from MIT course “Additive Manufacturing for Innovative Design and Production” [20]

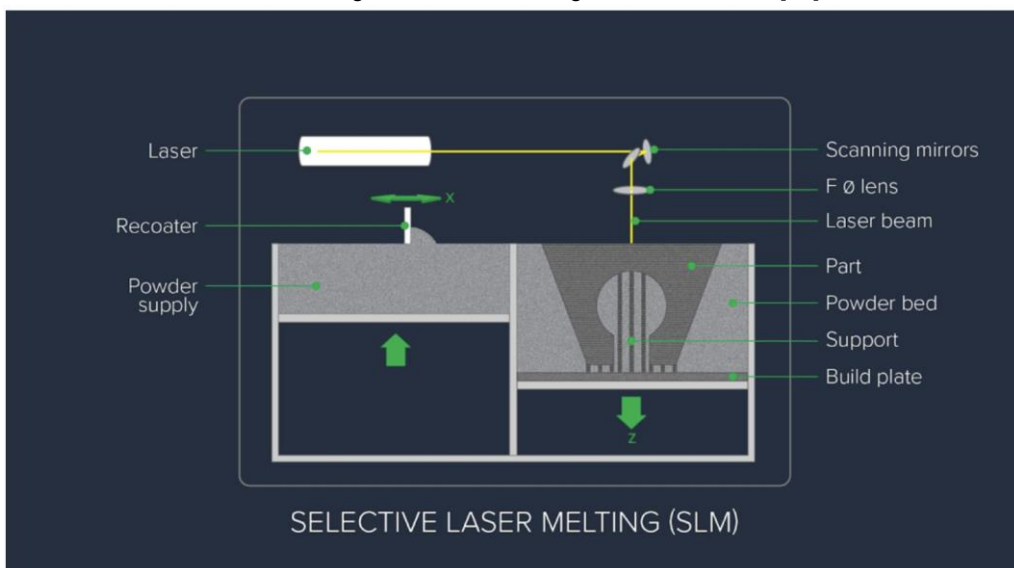


Figure 2: scheme of a PBF machine in work, utilized for metals. Figure taken from MIT course “Additive Manufacturing for Innovative Design and Production” [20]

- Both methods comprise a powder delivery system, by which powder in a supply well is moved and spread across the build platform by a recoater.
- A laser, directed by two scanning mirrors and brought to a focus via optical lenses, is used to heat the feedstock above its melting temperature. Many industrial PBF machines contain multiple lasers, each with an independently controlled mirror scanning mechanism.

- In PBF, it is necessary to build support structures to aid in heat dissipation from the part, and counteract thermal stresses and also to make it easier to remove the print from the build volume when the printing is finished. PBF supports must be placed beneath most overhanging features that span more than a few millimeters, and are rigidly attached to the part and made of the single build material.
- In PBF, heating of the bed can help manage residual stress and influence microstructure and properties, yet introduces greater machine complexity (thermal management) and can undesirably sinter the metal powder which limits the recyclability of unused powder.

The performance of SLS and PBF systems varies widely based upon a number of factors. Resolution and accuracy are limited by the combination of powder particle size, powder layer thickness, and precision of energy delivery (e.g. laser spot diameter). Typically, features as small as 0.1-0.5 mm can be reliably printed by PBF, such as thin walls and beams of lattice structures. The build rate of polymer SLS systems, is on the order of liters part per hour, and therefore is much greater than stereolithography type machines. The build rate of typical PBF machines, is 0.1 L per hour or less, due to the much greater energy required to heat and melt metal, as well as limits to the rate and stability of the melt pool. For this reason, increasing the number of lasers in PBF machines, rather than increasing the per-laser power and energy density, is the prominent method of increasing machine productivity.

Due to the focus on metal powder degradation in this review, the general workflow of the PBF method will be presented but not the SLS one, as follows:

1. Printing:

For each layer, there is a sequence of recoating, laser scanning, and incremental downward motion of the build plate. These steps are repeated sequentially to complete the build.

In the recoating step, a mechanism translates over the build surface, spreading a thin layer of powder by maintaining a uniform gap over the top surface of the previous layer (or, at the start of the build, the bare plate).

One or more laser beams are then raster-scanned across the build area to melt the powder in selected areas, creating the cross section of the part once solidified.

The rapid heating and solidification create sharp thermal gradients, leading to gas flows over the melt pool. As a consequence, some of the nearby powder is entrained, resulting in a phenomenon known as "spatter" visually resembling sparks. These spatters are responsible for introducing discontinuities to the unfused powder.

2. Removing excess powder:

The powder is swept and vacuumed from the components after printing. This unfused powder must be removed from the components before further processing. This excess powder can be recycled for future prints – with some limitations, and that's the point of interest of this thesis.

3. Heat treatment:

A furnace is used to apply a heat treatment to an entire array of components, while still attached to the build plate.

This heat treatment step is typically necessary to relieve residual stress which accumulates during the printing step, allowing the parts to be subsequently removed from the build plate without residual deformation.

4. Support removal:

PBF requires machining remove the parts from the build plate, typically using a bandsaw or wire EDM machine. Supports may then be removed using sawing, grinding, or cutting operations. In special cases, the part-support attachments can be engineered to snap away using pliers or simple manual action.

5. (Optional step) – post-machining and surface finishing:

After support removal, additional machining (e.g., using CNC milling) may be used to create high-quality surfaces, precision holes, etc. Other common methods of post-finishing PBF components include sanding, peening, tumbling, polishing, micromachining, plating, and painting.

Finally, as a material-wise mention, the author discusses about material-method compatibility:

For PBF of metals, a key attribute of compatible metallic materials lies in their response to the large, rapid temperature changes necessary to melt the powder. Most metals and alloys that are castable, i.e. are robust to cracking, tearing, and element segregation upon solidification, are good candidates for PBF. Therefore, many titanium and aluminum alloys, and a wide range of steels may be printed. PBF is also

compatible with nickel-based superalloys, refractory metals such as tungsten, and precious metals. Especially in these cases, PBF is an attractive alternative to machining, i.e., if the material has a high hardness that limits machinability, and/or when the part design means that machining would result in significant waste. For precious metals, PBF machines have been designed to handle small powder volumes with minimized loss/waste. Fine jewelry and miniature mechanical parts, such as watch components are among the more common applications of these machines. For metals with high melt temperatures, and/or those prone to cracking upon cooling, control of the build volume temperature (in some cases to several hundred °C) can be critical for the feasibility of PBF. There is much research and development on alloy design and powder engineering for PBF, including structural aluminum alloys.

Moreover, in PBF, the large temperature increase is necessary to melt metals, and the speed of heating and cooling in PBF, cause considerable residual stress which can compromise the dimensional accuracy of finished components. With ordinary care, this effect limits components to overall dimensions below ~0.1 m. Yet recent work shows that much larger components can be fabricated by careful optimization of thermal conditions, including laser toolpath and strategic placement of support structures.

3.1.2 Direct Energy Deposition

As a main difference, instead of using a powder bed, the DED process uses a directed flow of powder or a wire feed, along with an energy-intensive source such as laser, electric arc, or electron beam. DED is a direct-write process where the location of material deposition is determined by the movement of the deposition head; this allows large metal structures to be built without the constraints of a powder bed.

The diagram below illustrates the typical architecture of a DED system, indicating that DED techniques may use powder or wire feedstock. In powder DED systems, a laser beam focuses on a point on the building platform and at the same time, metal powder is injected into the focal path of the laser. The energy density must be sufficient to form a melt pool of the desired depth, and the powder and surrounding gas flow are coordinated to capture as much of the flowing powder as possible in the melt pool. In wire-feed DED, a metal wire is used as supply material instead of metal powder. Wire-feed DED typically has a higher build rate than powder-feed DED and reduces the feedstock cost, because in many cases wire is less costly than powder.

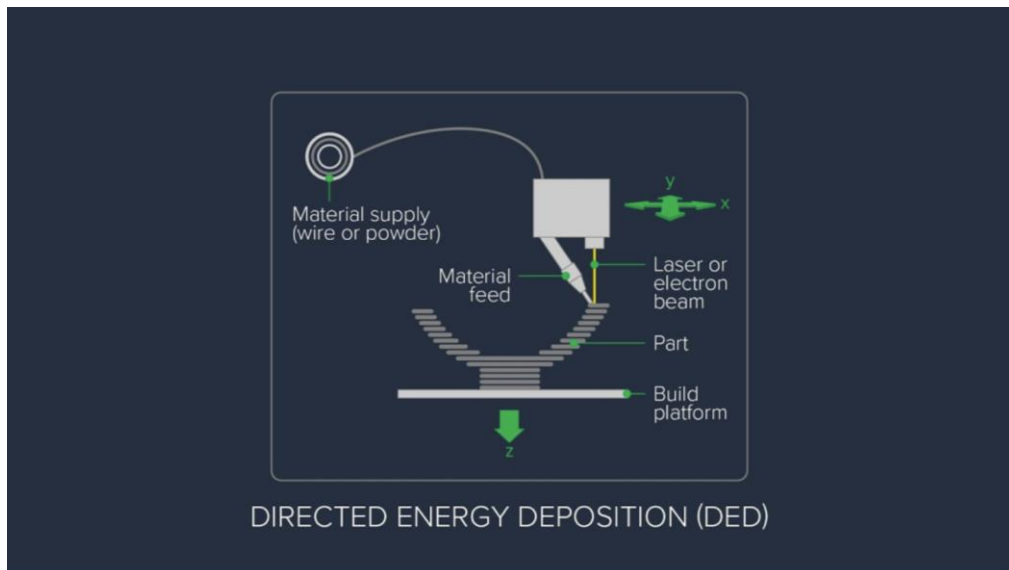


Figure 3: scheme of a DED machine in work, utilized for metals. Figure taken from MIT course “Additive Manufacturing for Innovative Design and Production” [20]

It is interesting to mention an alternative DED method that is called cold spray, it uses kinetic energy instead of thermal energy, and forms solid-state bonds between powder particles. In cold spray, bonding occurs by impact-induced deformation, and the impact velocity of the particles must be very high (in some cases, at supersonic speeds) to achieve full density of the printed components. Residual stresses in cold spray are primarily due to impact and are compressive in nature.

All DED processes follow a similar workflow, but the post-processing steps (e.g., machining and heat treatment) depend on the material used and the part requirements for a given application. Below is the description of a typical DED workflow for constructing a large metal component:

1. Printing:

A laser or electron beam is focused onto the desired deposition spot and metal powder (or wire) is injected into the melt pool. Relative motion between the substrate and the deposition head controls the part geometry.

2. Machining:

Because DED AM builds near net shape geometries, CNC machining is required on key surfaces and features such as flanges and holes. Post-processing may be done in the same machine (if a hybrid DED/CNC platform is used) or can be transferred to a milling machine.

3. Heat treatment:

After printing, yet before removal from the build platform, parts must be

heat treated to relieve residual stresses. Heat treatment can also tailor microstructure and mechanical properties.

4. Support removal:

DED requires machining to remove the parts from the build plate, typically using a bandsaw or wire EDM machine. Since there is little to no supports, they may then be removed using simple operations of sawing, grinding, or cutting operations. In special cases, the part-support attachments can be engineered to snap away using pliers or simple manual action.

Again, a material-wise mention is made regarding this method and some comparisons are made between PBF and DED:

The energy source in DED systems may be a laser, an electron beam, or a plasma arc. In the case of cold spray DED, solid state bonding (without melting) is made by high-velocity impact between the powder feedstock and the build surface. Powder and cold spray DED systems can, in principle, use metals, ceramics, or polymers. However, metals are almost exclusively used in DED. Ceramics are limited in their development with DED, and other processes provide better means of large-scale AM with polymers. Electron beam DED can only use metals, because the workpiece must be electrically conductive.

Any metallic material available in powder form can potentially be used in the powder feed DED processes; therefore, in general, the materials library is similar for DED and PBF. Wire feedstock materials available for DED include titanium and its alloys, Inconel alloys (e.g., Inconel 600, 625, 718), 300-series stainless steel, aluminum alloys (1000-5000 series; yet 6000 and 7000 series are prone to hot cracking), cobalt alloys, zircalloy, tantalum, tungsten, niobium, and molybdenum.

Laser and plasma arc DED systems require a controlled atmosphere which can be provided by the shield gas for non-reactive metals or by the build chamber with reduced oxygen levels when processing reactive materials such as titanium. Electron beam heat source also requires a vacuum build environment.

In terms of length scales, the minimum feature size, surface roughness, and fine detail capability of DED do not reach that of powder bed techniques. This is a tradeoff against the greater build rate of DED, and results from the direct-write nature of the process which does not permit the use of unfused powder to support fine internal

cavities. That said, DED systems can be configured over a wide size range, spanning from smaller systems (~100x100x100 mm build volume) to large systems (~6x1x1 m). The inherently coarse resolution of DED indeed motivates hybrid process integration, i.e., combining additive and subtractive processes in the most productive manner to achieve the finished part. The versatility and length scale compatibility of DED is driving significant industrial growth, especially for titanium alloy components for aerospace applications.

3.2 Powder requirements for AM

It is common knowledge that metal powder (MP) used in AM must follow a series of specifications, they exist to ensure the best possible result regarding consistency and mechanical properties of the final build. A good batch of powdered metal must achieve lower levels of sphericity and flowability, it must also achieve a certain particle size distribution to ensure good packing when allocated in the build volume [1].

3.2.1 Morphology and sphericity

Concerning the sphericity, MP may be obtained from various procedures, from physical to chemical ones and the way the powder is obtained impacts directly the final shape of the powder. Discussing about physical ways, the most common ones are: ball milling and atomization (gas, water, plasma and centrifugal), each one of these methods will arrive at a different outcome, as shown in Figure 4:

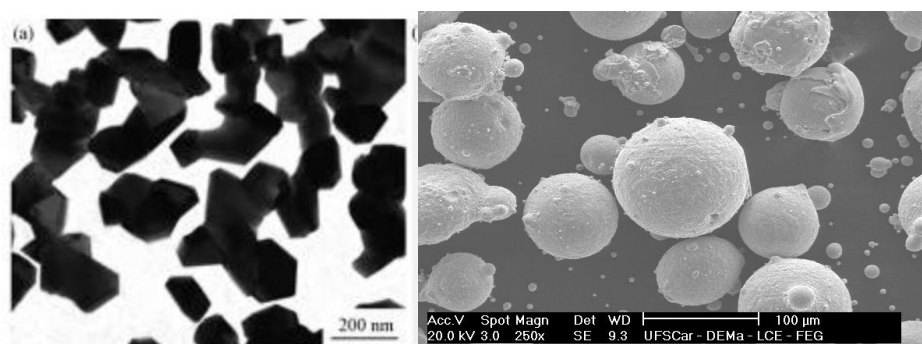


Figure 4: Images of different metal powders remarking its differences in morphology. Figure taken from the Author's previous work to FAPESP.

The left image in figure 4 shows the powder obtained by ball milling while the image on the right shows the product of a gas atomization process. It is natural to

conclude that this last one is more adequate to the AM processes mentioned before, since with their spherical shape they will, with much more ease, flow and pack in the building volume of a PBF machine, for example.

About the physical-chemical ways of obtaining powder it is worth mentioning the following methods: electrolysis, thermal decomposition, oxide reduction and precipitation. These methods are more specific and expensive, in general. The products from these processes are attached to specific needs, for example, copper powder obtained by electrolysis, which finds applications that demands high conductivity.

To summarize, the desired shape of MP with application in AM is as spherical as possible and preferably without intrinsic discontinuities such as satellites and agglomerates. In the next two images, it is shown (1) all possible powder morphologies and (2) those two intrinsic discontinuities in a powder batch.

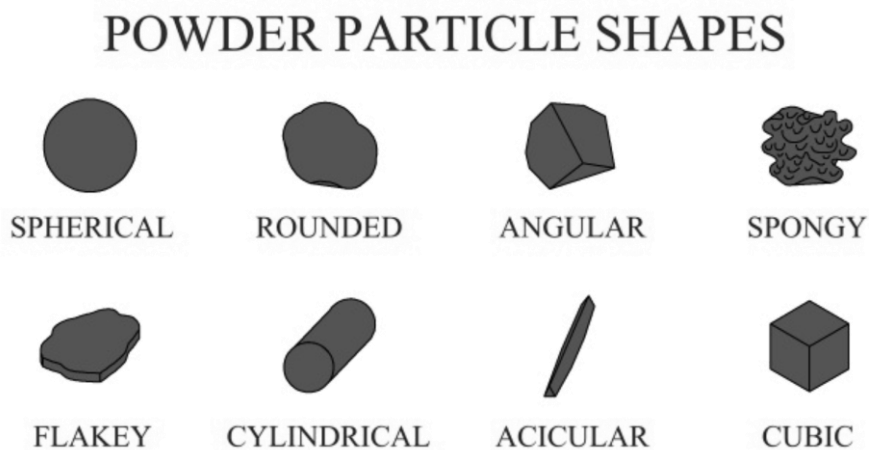


Figure 5: Powder particle shapes. Figure taken from [2]

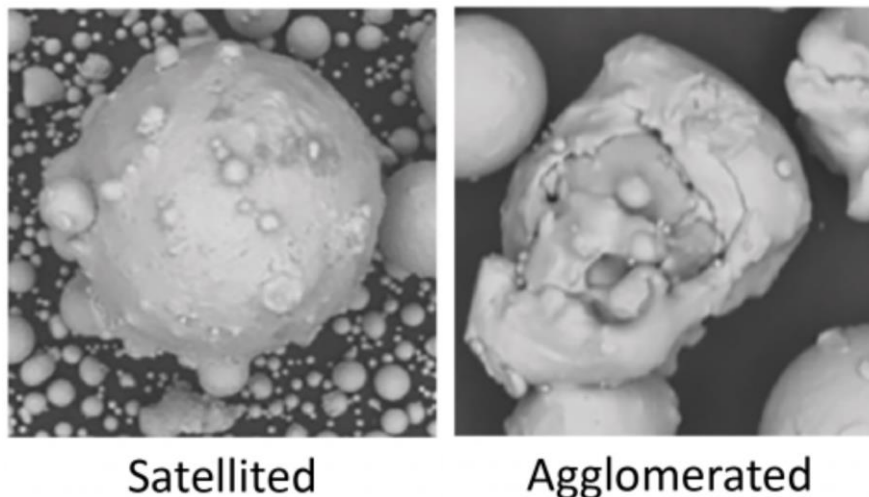


Figure 6: Powder particle showing defects (satellited and agglomerated). Figure taken from [3]

3.2.2 Flowability

Flowability is defined as how easily it is for the individual powder particles to flow one over the other within a powder portion. Although this property will face different situations regarding the type of AM technique – powder bed or powder flow - it is still one of the most important factors when validating a powder to be used in AM [1].

In the scenario of a technique based on a powder bed, the powder stays still when printing, but flowability is considered much before starting the printing, we must think of how the powder will flow when in the feeding hopper – in this situation it will face stress caused by its weight. Naturally, we must consider flowability when spreading a new layer over the previous one, this way it is a must to understand how the powder behaves under the application of stress by a roller. Understanding the powder flow under forced conditions is important to ensure a homogeneous spread of powder for each layer, avoiding problems like air pockets within the spread layer [4].

In the second case, with the powder flowing continuously instead of staying still, like in the previous one, flowability must consider not just the powder but also the gas that carries the particles. Obviously a continuous and consistent flow is essential to ensure a non-defective print. In this regime of flow, the effect of one particle over the other is almost neglectable, since there is almost no touch between them. This way the powder sees itself in a low-stress yet high-aerated state in the moment of printing (evidently when in the feeding hopper, stresses will be similar to the ones in PBF's hopper). In this situation, where the flow is much easier, one consideration that must be done is how the powder and the carrier gas will interact, the powder must be able to release this gas as it is drawn to the melting pool so that the quality of the print is not degraded by possible powder-gas entrapments [4][1].

3.2.3 Particle size distribution

Particle Size Distribution is the distribution of particles size of the MP within its total mass. A cumulative histogram is frequently used to represent the PSD of one powder batch. Since the PSD has a huge impact on the general properties of the built part, it's important to take a closer look at this property. One example of how the PSD

influences the result is in the next section, the packing or the ability of particles to be closer one to another, which is really important and enhances the properties of the built part, since there is less chance of a defect caused by lack of material.

In order to get access to the PSD of a powder batch it is necessary to sieve and also to consider that all particles are perfectly round. After that, each sieve level is weighted and finally a distribution of particle size is achieved. Some valuable information is obtained through the three main points: d10, d50 and d90. The logic is simple d10 is the representation of the ten percent smallest particles, in other words, d10 states that below that point there is ten percent of the powder, the same applies to d50, where there are fifty percent of particles below that size and finally d90, where there are ninety percent of particles smaller than d90.

3.2.4 Packing

The PSD is one of the most important factors when it comes to the packing of the powder in each layer. It is simple to demonstrate why an even distribution of powder size is undesirable, while having various-sized particles is much more efficient for the packing. This reduced filling capacity of the homogenous sized particles is demonstrated in Figure 7, where it is possible to compare the coverage of each type of powder, one with various sizes, with better packing than the other with just one particle size.

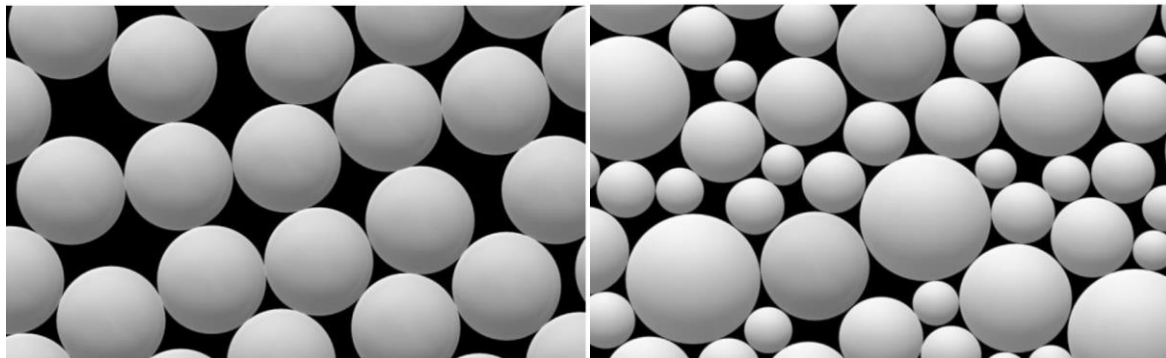


Figure 7: Schemes showing the difference in packing that is influenced by the PSD. Figure taken from [1]

Another important factor when it comes to packing is morphology, it is also natural to think that the more spherical powder the better. When defects as agglomerated powder, satellited particles, and uneven morphology come to the table, packing will be severely impacted, as shown in Figure 8.

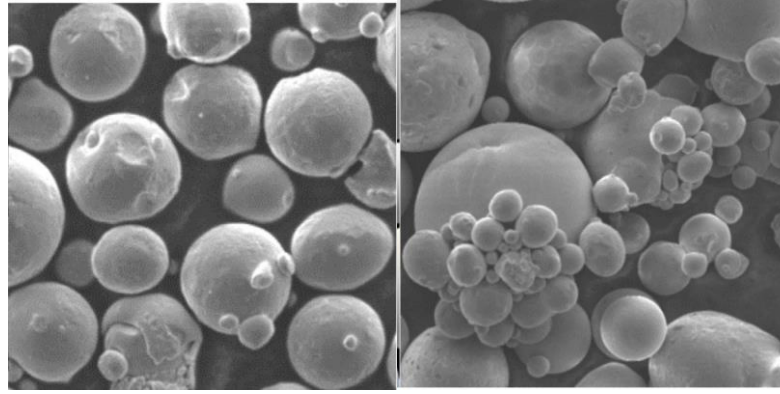


Figure 8: Photo of different structures (satellites and agglomerates). Figure taken from [1]

4 RESULTS AND DISCUSSIONS

4.1 EFFECT OF POWDER REUSE ON PHYSICAL AND CHEMICAL PROPERTIES OF PARTICLES

As mentioned before, AM requires a really specific set of characteristics of its material to arrive at an optimal result. These high standards elevate the price of the metallic powder used, so it is really interesting to reuse powder in more printing cycles to mitigate its high cost. Once there is not ASTM standards about powder reuse it is natural to think that it would not be prejudicial to the final properties of the print to reuse any powder. However, it is proven that after a cycle (or more), the powder has had some changes to its characteristics that can affect the following prints.

Specific changes in powder characteristics will be discussed in this next section, point by point, following the same logic used before to describe the requirements for the virgin powder, following the sequence of: morphology, PSD changes, chemical composition and oxidation, agglomerates and discontinuities and spattering.

Powder morphology over the prints remains spherical, in a major part. Although some deviations start to be expected after a higher number of prints. Those distortions were noted and classed in [5], [6] and [7]. These articles studied effects in stainless steel alloys and titanium alloys, and went up to 20 cycles of printing in a PBF machine. The main observations on powder morphology changes are listed below:

1. Pore formation in powder surface;
2. Bonding particles;
3. Surface cracks;

4. Clustering of particles;
5. Dimple formation;
6. Fracture of particles;
7. Fused particles
8. Appearance of irregularly shaped particles.

Figures 9 to 12 show the defects mentioned before, that may help to illustrate the effect in powder morphology through the repetition of print cycles.

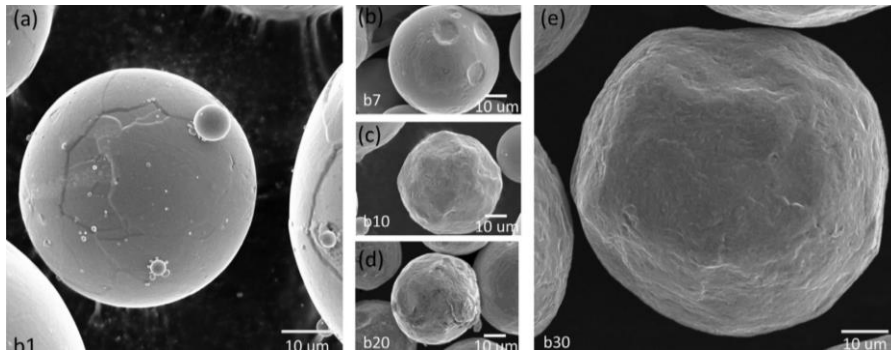


Figure 9: Evolution of powder sphericity in: a) 1 cycle, b) 7 cycles, c) 10 cycles, d) 20 cycles and e) 30 cycles. Figure taken from [7]

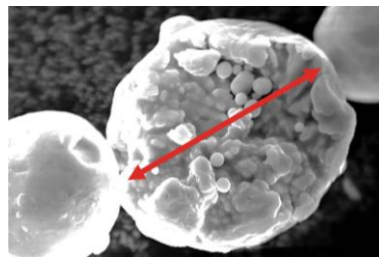


Figure 10: the representation of pore formation in recycled powder surface. Figure taken from [5]

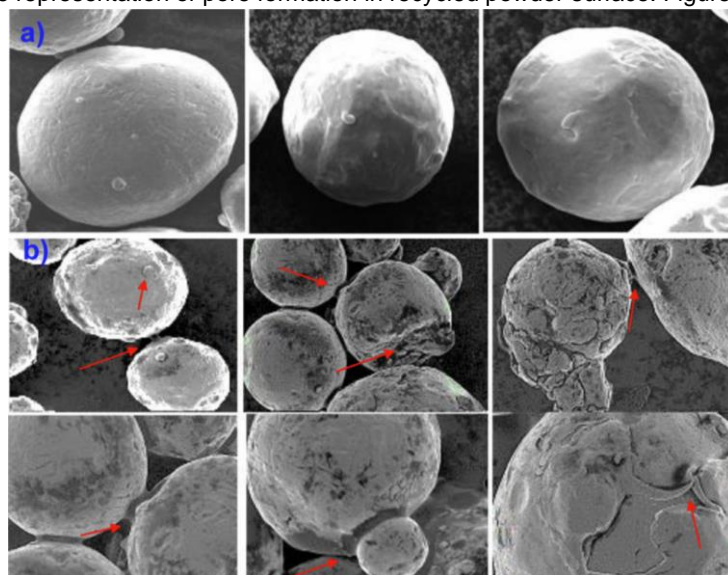


Figure 11: a) virgin particles and b) recycled powder particles showing: bonding particles (in the bottom left photo), surface cracks (in the middle photo), clustering of particles (in the photo in the right of the middle row) and dimple formation (in the bottom left photo). Figure taken from [6]

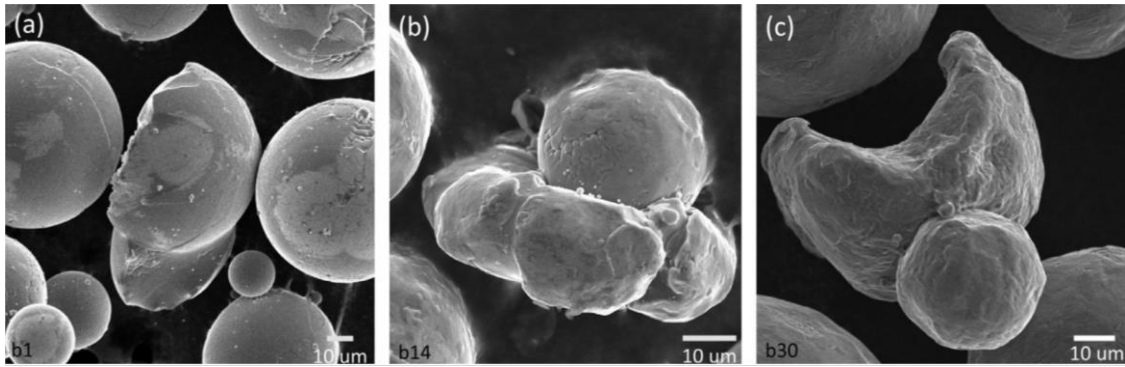


Figure 12: (from the left to the right) representation of: fractured particles, fused particles and irregularly shaped particles. Figure taken from [7]

Coming from S. Ghods, E. Schultz et al. ref [7], Oscar A. Quintana, Jorge Alvarez et al. ref [8] and Tang, Qian et al. ref [9], those three studies envisioned the evolution of PSD with multiple cycles of printing. They came pretty much to the same conclusion that the distribution becomes narrower: fewer smaller and larger particles are present, with a higher concentration in the mid-sized range. Although there is a slight increase in overall particle size, the majority falls within this intermediate range. So, to make it simpler: particles in general tend to have similar sizes and the average size gets bigger the more cycles the powder goes through. It is worth mentioning that this phenomenon was observed in studies with Ti6Al4V. The results from those PSD studies are shown in Figures 13 to 15.

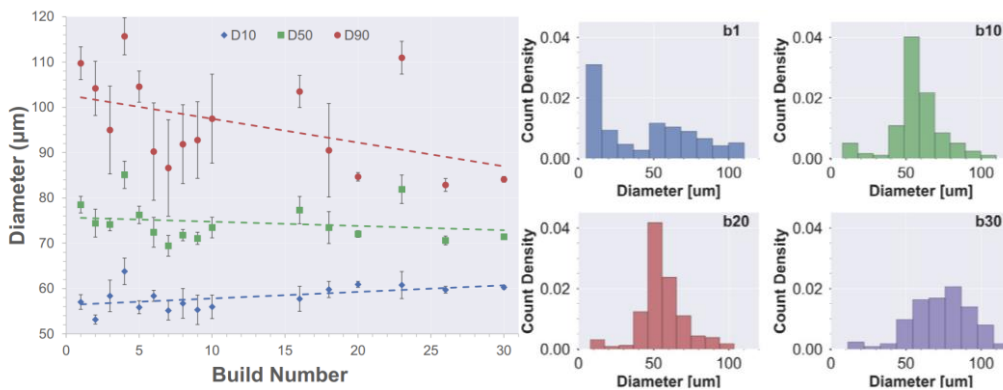


Figure 13: evolution of the PSD of Ti6Al4V, showing the tendency to a narrower distribution and an increase in average particle size with the increasing number of prints. Figure taken from [7]

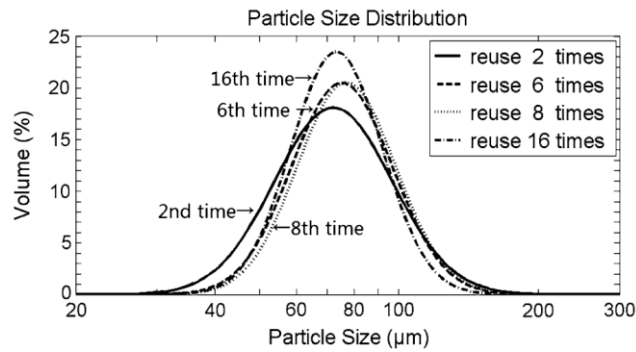


Figure 14: Evolution of PSD: narrower peaks and a dislocation to the right (bigger particles), the more cycles the powder goes through. Figure taken from [8]

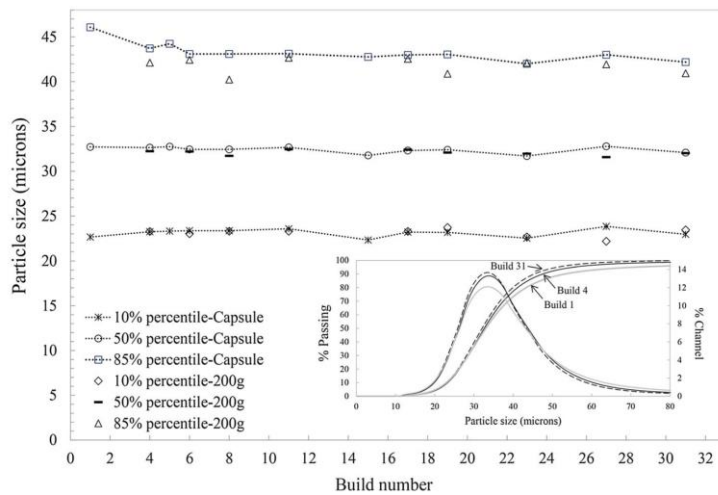


Figure 15: Evolution of PSD showing the same consistent result of a narrower distribution and bigger particles. Figure taken from [9]

Other works like the ones published by B. Sartin, T. Pond et al. ref [10], Nima E. Gorjia, Prateek Saxenab, et al. ref [5] and Sara Giganto, Susana Martínez-Pellitero et al. ref [11], that were not developed with Ti6Al4V but with 316L and other stainless-steel alloys did not observe this comportment of a narrower distribution, instead, they just stated a dislocation of the PSD curves to the right, that means a bigger average to the particle size, as shown in Figures 16 to 18.

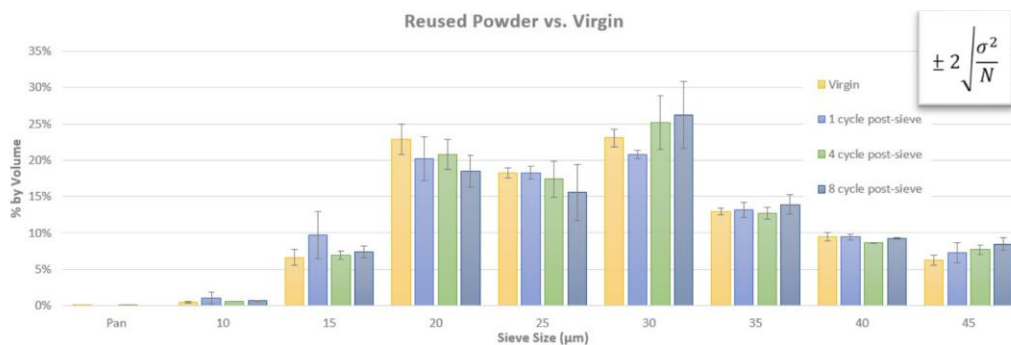


Figure 16: Correlation of size and volume fraction for powders used in different number of cycles. Figure taken from [10]

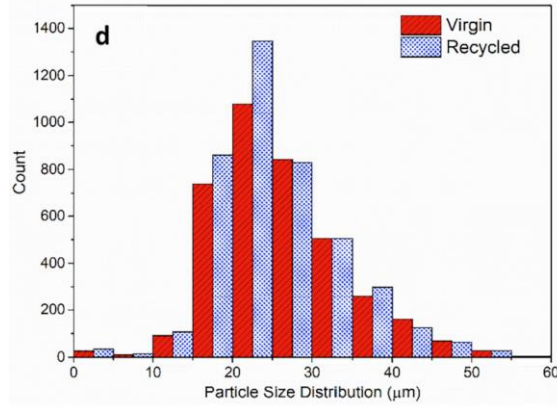


Figure 17: Comparison between virgin and recycled PSD. Figure taken from [5]

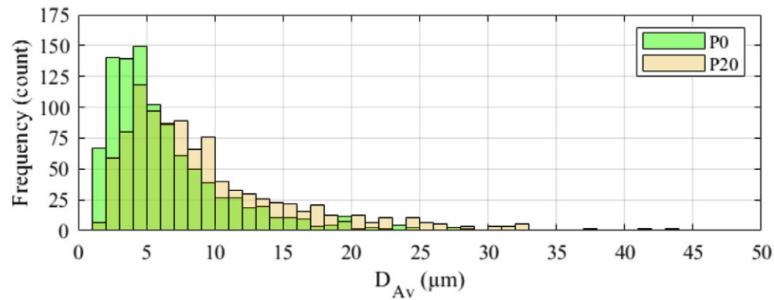


Figure 18: Evolution of PSD highlighting the tendency to bigger sizes in an augmented number of cycles. Figure taken from [11]

The majority of the papers focused more on the oxygen content, which will be discussed in the next section about oxidation than on the evolution of the chemical composition of the powder through multiple cycles of printing.

When it comes to chemical composition most of the data is for Ti6Al4V but not much information was found about stainless steel. This way, in this section, when not mentioned a specific material, information will be about this mentioned alloy. [12][14][7][9][15]

It is interesting to note that Ti6Al4V has a nice chemical stability over the prints, some articles pointed to a slight increase or decrease in V and Al, but they also mentioned that this may be linked to imprecisions of the method of measurement or local changes in the composition. The point is that powder composition (ignoring oxidation) remains the same through print cycles, at least for Ti6Al4V. Tables 1, 2 and 3 show some composition tables of MP which underwent multiple cycles of prints to demonstrate this stability.

Table 1: Composition of reused Ti6Al4V powder and additively manufactured tensile samples. Table taken from [9]

Reuse times	O (wt.%)		Al (wt. %)		V (wt. %)	
	Reused powder	Tensile sample	Reused powder	Tensile sample	Reused powder	Tensile sample
0	0.08	0.07	6.47	6.14	4.08	4.04
6	0.14	0.13	6.37	6.06	4.05	4.02
11	0.17	0.15	6.36	5.90	4.05	4.01

16	0.18	0.17	6.35	5.86	4.04	3.95
21	0.19	0.18	6.35	5.93	4.03	3.96

Table 2: Concentrations of key elements in the powder for b1, b10, b20 and b30. Table taken from [7]

Build	O (wt.%)	N (wt.%)	H (wt.%)	Al (wt.%)	V (wt.%)	Fe (wt.%)
1 (virgin)	0.142	0.024	0.0014	5.41	3.43	0.21
10	0.189	0.026	0.0010	5.50	3.26	0.25
20	0.269	0.027	0.0012	5.54	3.40	0.26
30	0.356	0.030	0.0009	5.58	3.36	0.29

Table 3: Chemistries of Ti6Al4V powders after build 5 and chemistries of build 1 and build 5 compared to those of As-Received powders. Table taken from [12]

	As-Received powder	Powder after cycle 5	Build 1	Build 5
Aluminum	6.27	6.36	5,82	6.17
Vanadium	4.24	4.39	4.23	4.35
Oxygen	0.138	0.182	0.141	0.168
Nitrogen	0.025	0.025	0.019	0.025
Carbon	0.015	0.018	0.020	0.028
Hydrogen	0.0016	0.0024	0.0025	0.0015
Iron	0.19	0.20	0.20	0.22
Niobium	N/A	<0.002	N/A	N/A
Tungsten	N/A	<0.002	N/A	N/A
Copper	N/A	N/A	0.015	0.029
Silicon	0.022	0.023	0.024	0.020

Stepping out of this most popular alloy, some data was found about Inconel 718 (IN718), in papers [12] and [16]. These articles agree on a virtual stability of chemical composition for IN718 although both of them point out a small, yet present, increase in oxygen content. The point that the author of this review would like to make is that none of those two studies went beyond 20 cycles like the ones about Ti6Al4V, in this case, it would be interesting to see future studies going past that point to state this virtual stability that is mentioned. In table 4, the chemical composition for an IN718 that went through 6 cycles is shown and compared.

Table 4: Powder chemistries for Inconel 718 powders after different build cycles. Table taken from [12]

	As-Received powder	Powders after build 1	Powders after build 6
Carbon	0.044	0.043	0.042
Sulfur	0.002	0.002	0.001
Oxygen	0.014	0.014	0.022
Nitrogen	0.020	0.019	0.020
Hydrogen	0.0002	0.0002	0.0002
Nickel	53.03	52.75	53.60
Chromium	18.40	18.36	18.09
Molybdenum	3.10	3.08	2.96
Niobium	4.86	4.95	4.84
Manganese	0.095	0.096	0.088

Copper	0.15	0.16	0.14
Aluminum	0.43	0.41	0.42
Titanium	0.89	0.91	0.86
Silicon	0.19	0.23	0.20
Phosphorus	0.006	0.009	0.0083
Boron	<0.0005	<0.0005	0.0013
Cobalt	0.14	0.16	0.13
Iron	18.59	18.80	18.57

Another alloy with little data around is AlSi10Mg, studied in paper [13]. In this study, like the previous ones about IN718, the material did not go through many cycles, yet it still presented a virtual stability in its chemical composition (Figure 19).

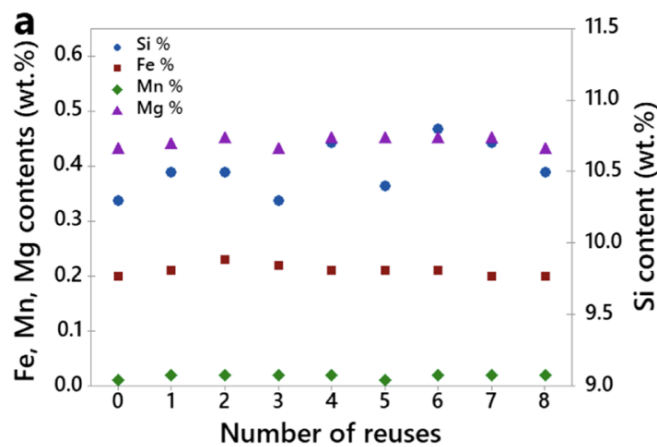


Figure 19: Variation in composition of AlSi10Mg through an increasing number of reuses. Figure taken from [13]

Surprisingly, the author has encountered some trouble finding data about one of the most common materials in usage, stainless steel. In [6] there is some information about the chemical composition of SS316, but there is little to no information about how many cycles the material went through. In table 5 there is the result of the chemical composition analysis of the mentioned study.

Table 5: Concentration of chemical elements on powder surface extracted from XPS curves using CasaXPS.

Table taken from [6]

Powder	%O	%C	%Mn	%Fe	%Si	%Sb	%Cu
Virgin	27.04	56.21	2.02	1.04	10.83	2.86	-
Recycled	34.19	45.55	2.27	2.70	10.07	-	5.22

In this last study there is a noticeable difference between the results, it is possibly due to the XPS method that looks more superficially in the powder than into the volume so it is possible to infer that there is a gradient of chemical composition in the powder volume.

As seen in the last section, chemical composition (in volumetric terms) is stable through the repetition of cycles although it is not valid when it comes to the oxygen

content and more superficial analysis. It is noticeable that the oxygen content increases progressively the more cycles the material goes through. It is caused by the oxidation of the powder; this effect is enhanced by the heat caused by lasers or plasma beams in the heat zone, mainly. Some materials are more susceptible to oxidation than others and this affects mainly the build parts properties.

The effect of oxidized particles in the powder is noticeable since it is commonly associated with spatter formation, which leads to poorer flowability and thus the worst packing factor that inevitably may generate defects like lack of fusion and pores in built parts, so controlling the presence of oxidized powder content in the bulk is of great importance [17].

As briefly mentioned in the morphology section some results shown that the number of satellites and agglomerates have reduced through the multiple cycles and this is actually counter-intuitive since the increase in discontinuities is expected in the powder caused by its degradation. This effect can be explained by the recycling method. But first, about the formation of these defects, they are mainly caused by the heat and the sintering process of thermally affected powder, as shown in Figure 20.

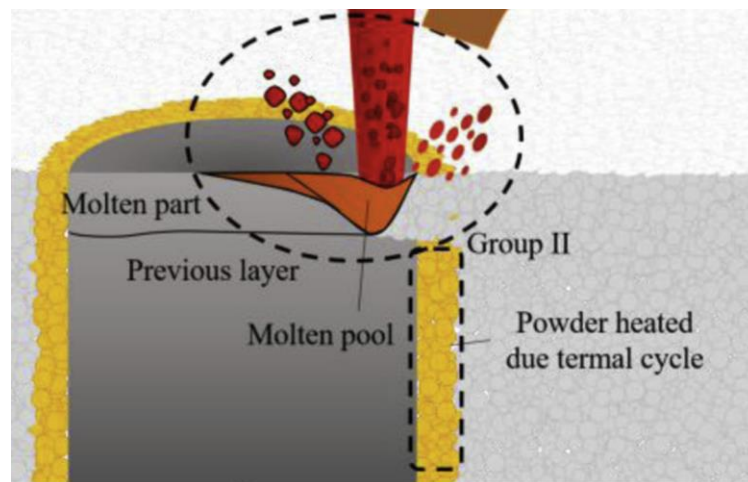


Figure 20: Thermal zones in a print volume. Figure taken from [18]

In Figure 20, the group II or powder heated due thermal cycle is really susceptible to this sintering effect, since theoretically it is some not melted powder that is in direct contact with the molten part and works as a thermal conductor to cool down the liquid metal after the energy source passes by. This group is also “responsible” for the surface roughness of the print, due to the fact that if it has strong interactions with the molten part some particles may partially adhere to the built, getting a rough finish, as shown in Figure 21.

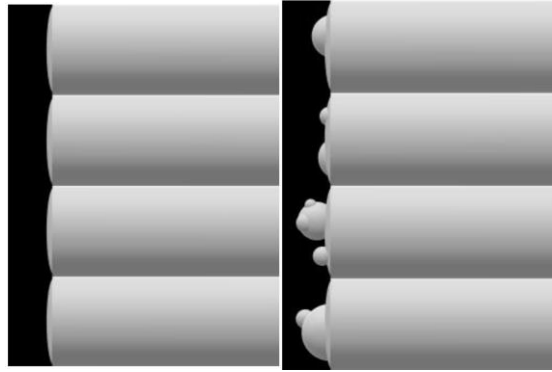


Figure 21: Surface scheme for surface roughness origins. Figure taken from [1]

Now discussing the reasons why agglomerates and satellites decrease through cycles, the first reason is the adherence to the built parts, so they are captured and removed of the system. The second and most important one is the recycling process.

After each cycle the MP goes through a sieving process and bigger clumps, agglomerates and irregular particles are removed by the bigger sieves. It is not difficult to think that the sintering phenomenon is causing a growth in the size of these discontinuities and then making it easier to sieve and eliminate, getting a better powder quality (in terms of quantity of discontinuities) through the cycles [1][8][18].

Spatter should not be viewed as a simple problem, since there are different origins to different types of spatters, this way, spatter should be seen as a complex system that has different mechanisms of formation and many classes. The main kinds of spatter are 4: melt-ejection spatter, hot entrained spatter, cold ejection spatter and one related to the vaporization of material. In the next paragraphs, a brief review about every type of spatter will be provided as well as a table (Table 6) with different expectations in outcome when comparing mechanisms.

Table 6: Classification of spatter particles. Table taken from [19]

Features	Size (µm)	Generation Mechanism
Particles similar to virgin gas atomized particles	15-45	Cold entrained (Type III)
Particles with morphology different to gas atomized	15-45	Hot entrained or melt eject (Type I or II)
Larger singular particles with different morphology	45-111	Melt eject (Type I)
Particles with oxide spots	23-118	Melt eject (Type I)
Particles covered with oxide	31-128	Melt eject (Type I)
Small particles	< 15	Melt eject (and explosion) (Type I)
Agglomerates	< 273	All mechanisms (Type I-III)

- I. Melt ejection particle: a molten particle is ejected and solidifies in an inert gas atmosphere, exactly as a gas atomization process. This type of spatter may vary in size going from 23 to 136 µm. It's common to these particles to have a different morphology when compared to the virgin powder.
- II. Hot entrained spatter: originating from the powder bed, virgin powder

particles are carried along, experiencing partial or complete melting upon passing through the laser. This process may induce morphological alterations as the particles solidify. While these particles maintain their original size, their trajectory in the laser's wake increases the likelihood of collisions with other entrained particles, leading to the formation of agglomerates, sometimes exceeding the size of the virgin material. Distinguishing features of these particles include their deviation from spherical singular droplets, characteristic of larger singular particles produced by melt ejection.

- III. Cold ejection spatter: this mechanism has a role in redistributing entrained particles in the vicinity of the laser's trajectory and is likely associated with type I spatter. These particles will have a similar size range as the virgin material and exhibit minimal or no apparent morphological changes.
- IV. Vaporization of material: this mechanism serves a dual role in the ejection of spatter from the melt pool and the entrainment of particles behind the laser. Vaporized material forms nanoparticle clusters, coating the interior of the build chamber. These nano particles hold significance for health and safety considerations in PBF-LB and machine usage. They have the potential to deposit onto the optical lens, leading to unwanted variations in processing characteristics by attenuating the laser.

Figure 22 illustrates the origins/ place of action of each mechanism discussed.

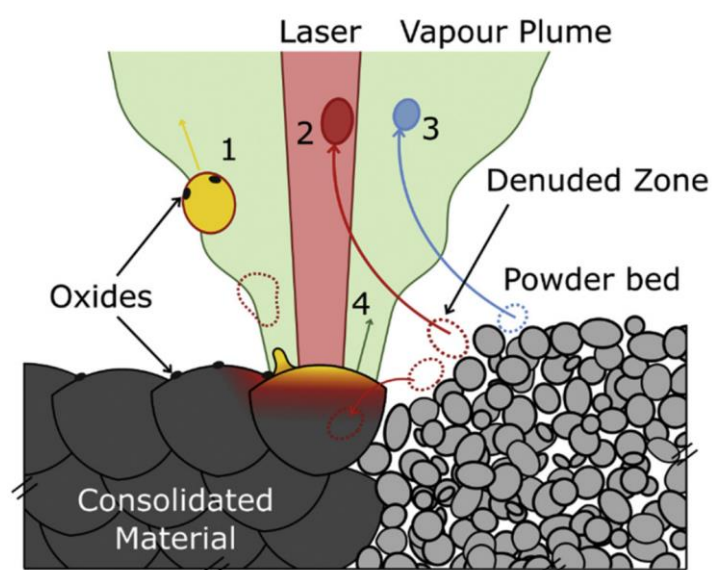


Figure 22: Different thermal regions in the PBF print, division into 4 different zones from which spatter may origin. Image taken from [19]

4.2 EFFECTS OF POWDERS REUSE ON PHASE FORMATION

Besides oxidation, morphology changes, spattering and alterations in the PSD there are other effects perceived among MP when recycling it for AM uses. In the following sections some will be discussed for specific materials, especially SS alloys and Titanium alloys:

4.2.1 Steels [18]

One interesting appointment in steel powder studies is about partial phase transition in reused powder. It was observed that some fraction of the powder had fcc- γ -austenite structure while the other part had bcc- δ -ferrite. This creates a scenario with much more unpredictability in material properties. Since one part of the bulk powder could magnetize and the other couldn't, this may lead to some defects due to clustering of magnetic particles. In addition, it was reported that δ -ferrite powder had a smoother surface than γ -austenite powder. Again, that could lead to discontinuities in the layer of powder due to differential flowability. The images of this phase-mixed powder are shown in Figure 23 and possible defects in layer rolling are shown in Figure 24.

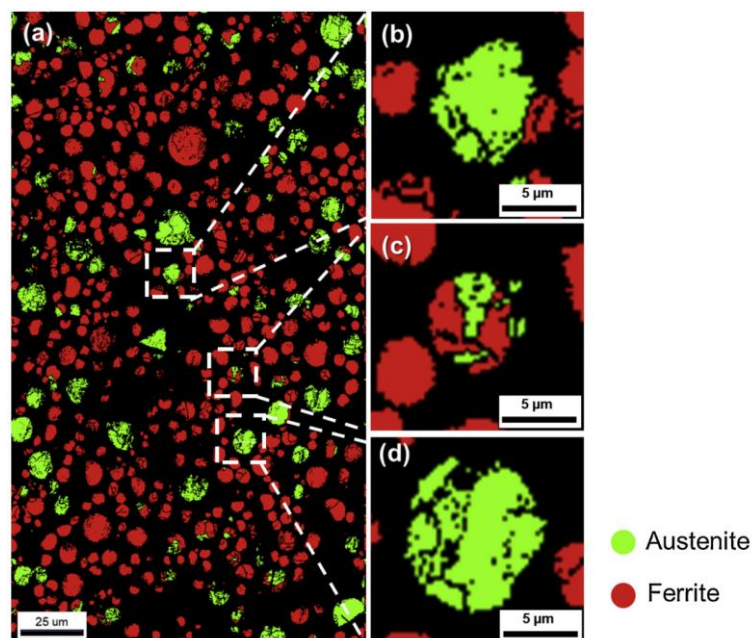


Figure 23: EBSD of a 316L powder batch showing differential microstructure through the reused powder, with Austenite phase particles in green and Ferrite particles phase in red. Figure taken from [18]

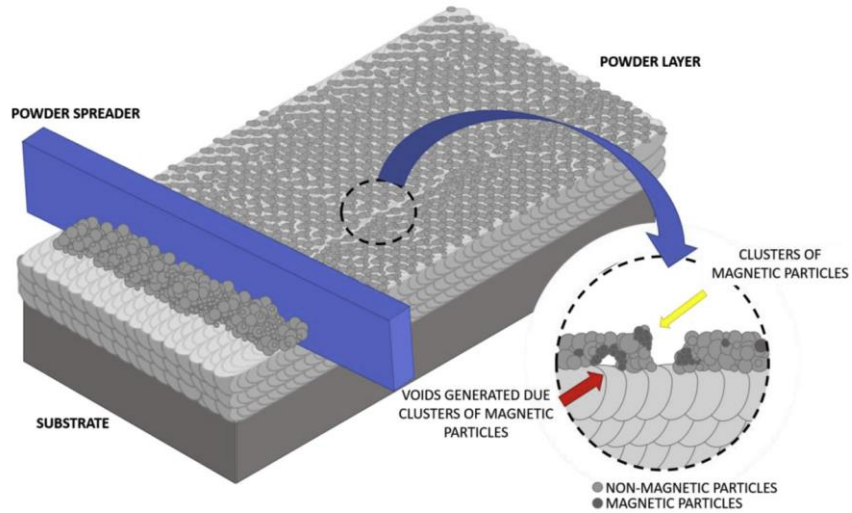


Figure 24: Scheme of void defects in layer rolling caused by magnetic clusters of powder. Figure taken from [18]

4.2.2 Titanium Alloys [15]

Similarly, to the effect described previously about SS, the following effect has a link with the thermal history of the material and phase transformation. In the referenced study [15] it was stated that when heated in the region near the melt pool, Ti6Al4V powder would evolve its microstructure. Initially it has a dominant α phase or martensitic α' phase, but when it is submitted to thermal cycles, β phase starts to appear in the powder, resulting in a microstructure with two phases as seen in Figure 25.

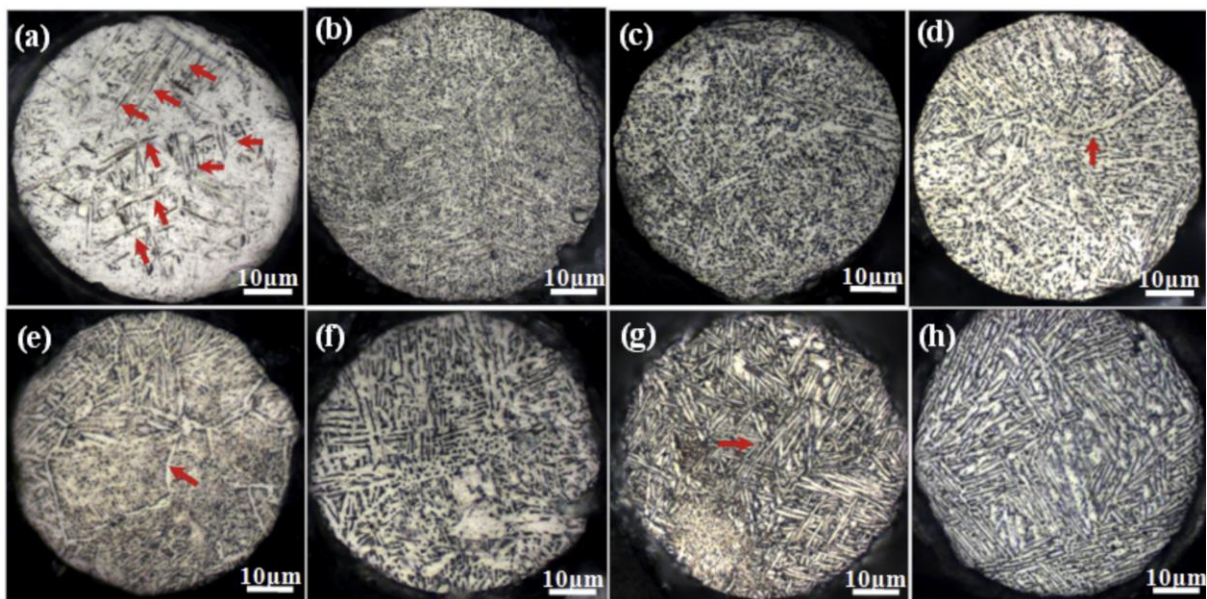


Figure 25: Cross-sectional images of TC4 powder particle with various reuse times: (a) 0th reuse, (b) 1th reuse, (c) 2th reuse, (d) 3th reuse, (e) 4th reuse, (f) 5th reuse, (g) 6th reuse, (h) 7th reuse. Figure taken from [15]

In addition, due to the evolution of the microstructure, physical properties would change too. An increase in hardness occurs with the increasing number of cycles, as shown in Figure 26.

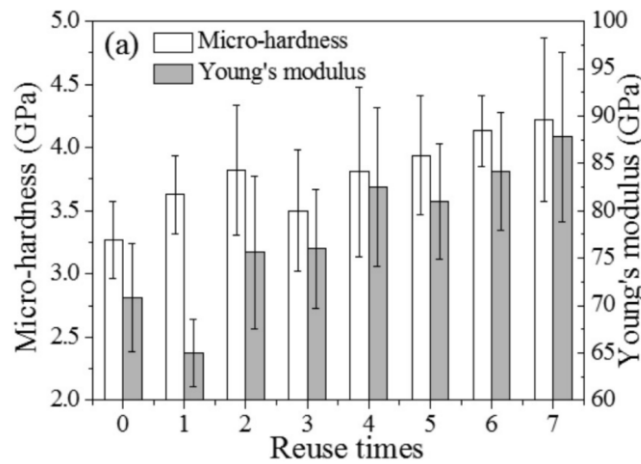


Figure 26: Micro-hardness and Young's modulus evolution in function of the number of print cycles of TC4 powder. Image taken from [15]

4.3 EFFECTS OF POWDERS REUSE ON MECHANICAL PROPERTIES

In following part, the idea is to review properties results from print parts. Different studies may have adopted two ways of incorporating reused powder into their printing powder batch: the first one being reusing the whole powder batch to print their parts and the second one by incorporating a % of used powder to a virgin powder batch.

The first study to be reviewed ref. [13] uses AISi10Mg as the alloy of choice, and the idea is to reuse the same batch of metal powder multiple times but sieving in between the prints to get all rougher clumps and spatter out. The properties (R_p 0,2%, ultimate tensile strength, elongation and reduction in area) are virtually the same through all the cycles, this is valid for static and cyclic properties, as shown in Figure 27. It is worth mentioning that despite some fluctuance of each point, there is a little tendency of decrease in the properties.

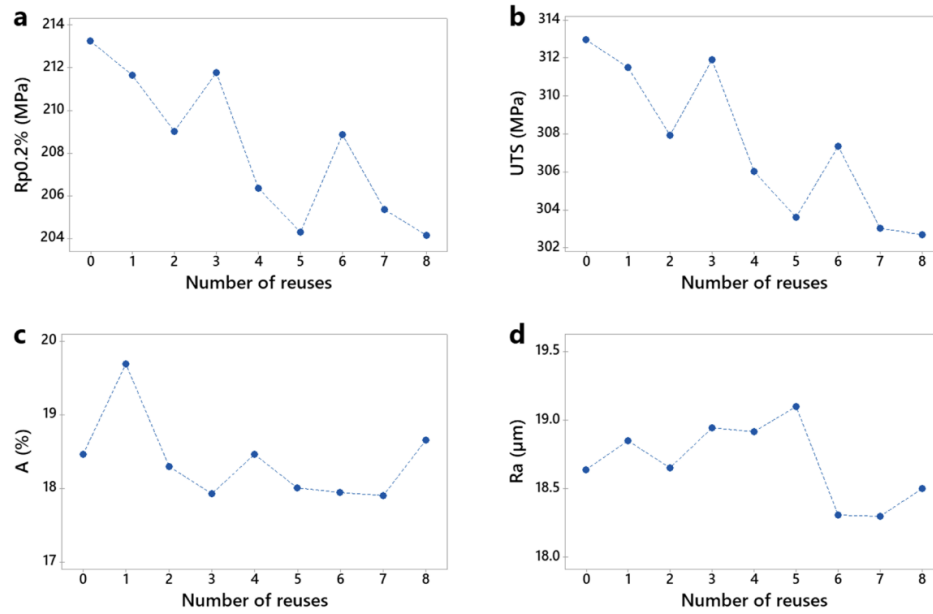


Figure 27: Tensile properties versus reuse times for AISi10Mg with an increasing number of printing cycles: a yield strength, b ultimate tensile strength, c elongation at break and d arithmetic mean roughness. Figure taken from [13]

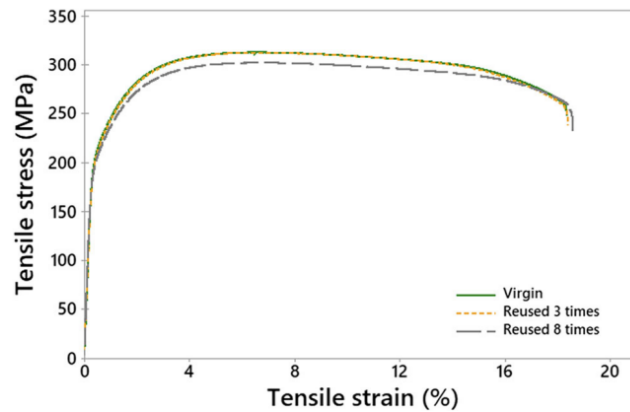


Figure 28: Stress-strain curves of AISi10Mg specimens additively manufactured from virgin and reused powder. Figure taken from [13]

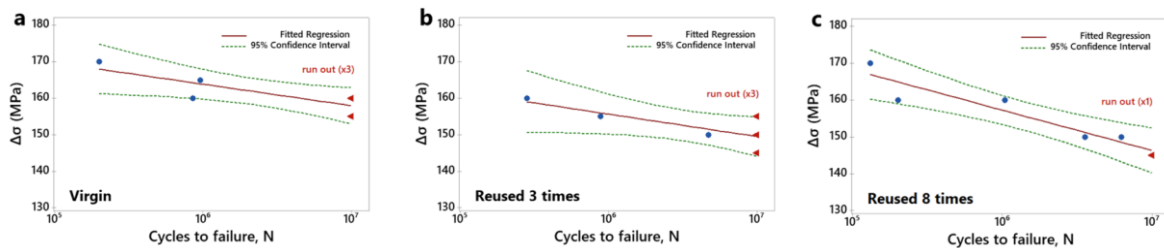


Figure 29: Wohler curves of the AISi10Mg specimens additively manufactured from virgin and reused powder: a virgin, b reused three times and c reused eight times. Figure taken from [13]

The second one [10] is about stainless steel, more specifically SS 316L, and once again the idea is to analyze the evolution of properties of the prints using the same powder through multiple cycles (Figures 30 and 31). A high number of

specimens were tested. No significant difference was observed in the mechanical properties within increasing the number of cycles. They argue the fluctuance observed was due to punctual defects in individual specimens caused by machine or process variations and are not associated with the reuse of powder.

The density of prints remained stable, around 7,93 g/cc and their tensile strength and elongation did not vary much: the first remained stable around 85 ksi and the second stabilized around 35%.

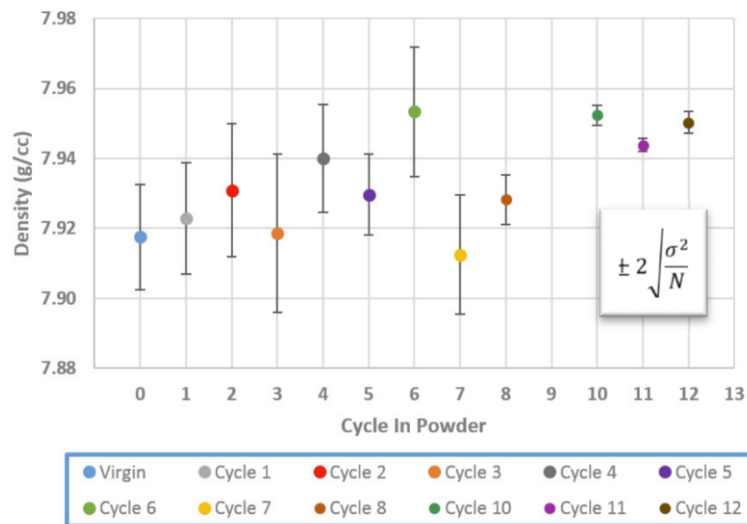


Figure 30: Density values determined via Archimedes in alcohol by powder cycle (average+/- 95% CI) – SS316L.

Figure taken from [10]

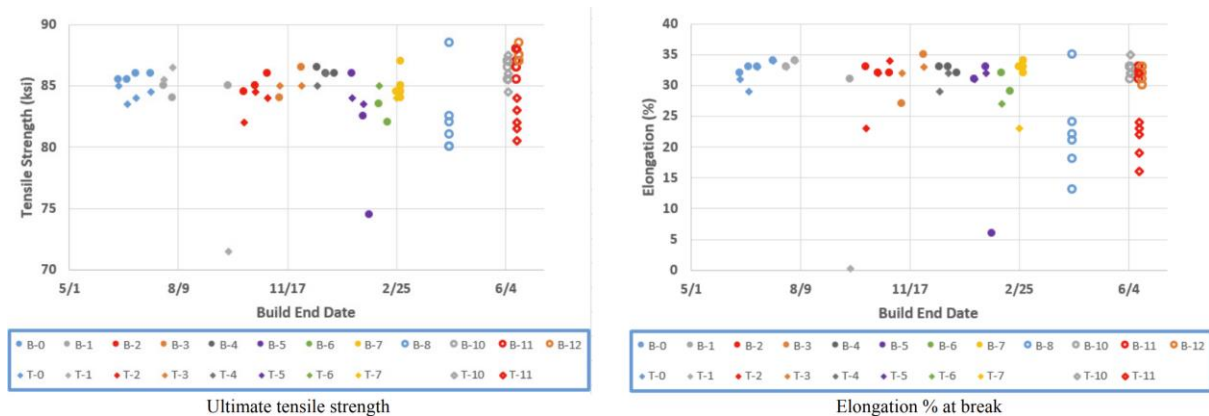


Figure 31: Correlation between mechanical properties: tensile strength (in the left) and elongation (in the right) and the print cycle (from 1 to 12) – SS316L. Figure taken from [10]

The last study of this section was with Ti6Al4V [9]. For the first time, it is possible to see some significant changes in mechanical properties through the cycles (Tables 7 and 8)

Table 7: Tensile properties of Ti6Al4V additively manufactured from reused powder. Table taken from [9]

Powder reuse times	Powder oxygen (wt.%)	Yield strength (MPa)	Ultimate tensile strength (MPa)	Tensile elongation (%)	Reduction in area (%)	Density (g cm ⁻³)
0	0.080	834 ± 10.0	920 ± 10.0	16.0 ± 0.3	54 ± 3.0	4.410 ± 0.004
2	0.097	870 ± 8.0	970 ± 10.0	15.0 ± 0.3	46 ± 3.0	4.410 ± 0.004
6	0.14	822 ± 25.0	910 ± 20.0	13.5 ± 1.0	53 ± 4.0	4.411 ± 0.004
11	0.17	891.5 ± 4.5	986.5 ± 3.5	17.8 ± 0.8	50.0 ± 1.0	4.428 ± 0.003
16	0.18	939.6 ± 3.6	1028.1 ± 4.1	15.3 ± 1.8	42.1 ± 4.1	4.380 ± 0.018
21	0.19	960 ± 30.0	1039.3 ± 2.7	15.5 ± 0.9		4.381 ± 0.019

This significant increase in the UTS and YTS was associated with an increase in the oxygen content in the material and its impact on the final parts microstructure. Table 8 shows the evolution of O% through cycles in both powder and tensile samples.

Table 8: Oxygen content in Ti6Al4V reused powder and tensile sample. Table taken from [9]

Reuse times	O (wt.%)	
	Reused powder	Tensile sample
0	0.08	0.07
6	0.14	0.13
11	0.17	0.15
16	0.18	0.17
21	0.19	0.18

Another study [14], investigated the effect of the fraction of recycled material added to virgin powder in the mechanical properties of Ti6Al4V. In this case, no evolution in mechanical characteristics was noticed (Figure 32). Similar properties were observed with the increasing % of recycled powder in the batch. This article unfortunately does not address how many times the powder was reused to be considered recycled, so there is no direct comparison between this paper and the previous one.

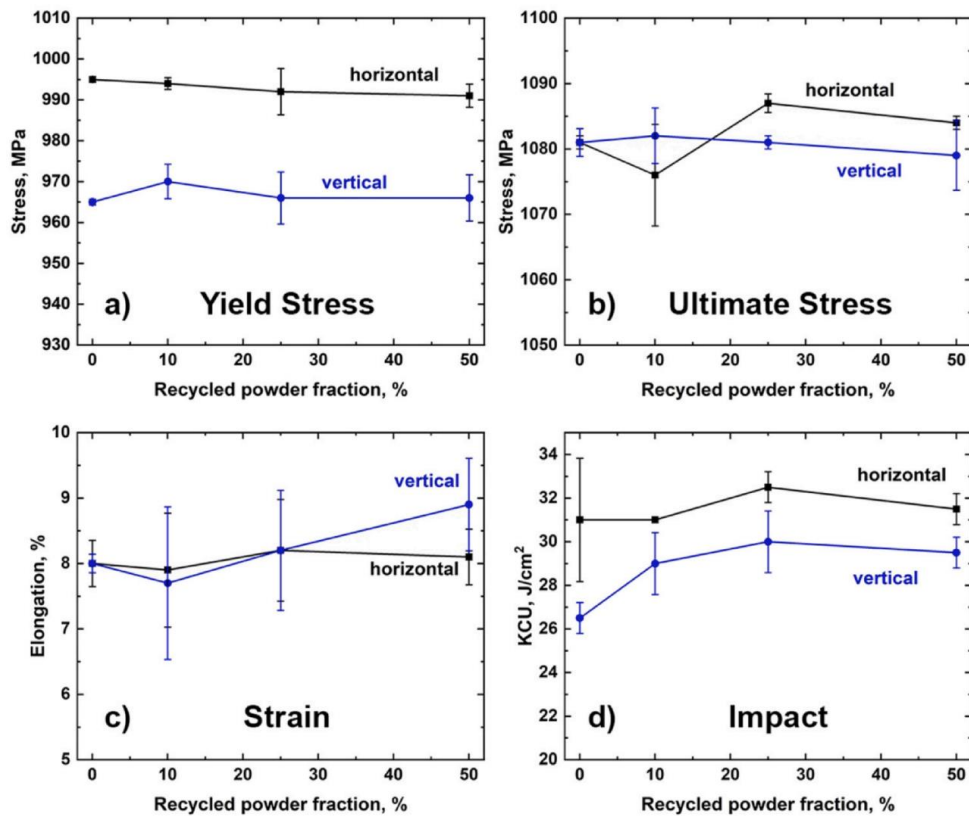


Figure 32: (a–c) Tensile test and (d) impact test results of Ti6Al4V. Two building directions were considered: horizontal and vertical. Figure taken from [14]

5 FUTURE INVESTIGATION AND NEXT STEPS

Further investigation could be done in the direction of an even higher number of cycles, by the margin of a hundred cycles, this could address new problems caused by the lack of smaller particles and the dislocation of the PSD, and also investigate what a more radical oxidation degree would produce as an outcome, for example, the impact on finishing quality of the surface in print parts.

Another interesting point of study would be the optimization of metal powder treatment after each cycle. Is it really necessary to sieve every single time? Could this be reduced to one sieve each two cycles or more? This would radically increase productivity and reduce time spent in the production of final products.

Some other directions for research are the possibility of studying other alloys and materials and expanding to other AM techniques (since the major part of studies are about PBF). It is worth mentioning that the properties studied are mechanical, so for future investigation exploring others (thermic, magnetic, etc.) could be interesting.

6 CONCLUSIONS

As observed when it comes to reutilizing MP through multiple cycles, the powder actually evolves in: morphology, PSD, composition (mainly through oxidation), etc. Although it may seem like this powder is coming to its EOL, in reality, by sieving, and separating the main defect drivers such as agglomerates and spatters, MP can have its longevity really augmented. This increased reliability in MP reuse is clearly stated by the properties (UTS, elongation and area reduction) in the final prints, there were not significant changes to them, even in a higher number of cycles.

In conclusion, the exploration of metal powder reuse in additive manufacturing has provided insights into the potential benefits (waste reduction and smaller costs) and challenges associated with this practice (maintenance of powder quality).

However, it is important to acknowledge that further research and investigation are warranted to enhance our understanding of the long-term implications of metal powder reuse. While the present study has indicated that there is little to no discernible impact on the properties of parts built from reused powder, ongoing efforts should be directed towards a more nuanced examination of material properties, structural integrity, and potential environmental considerations, as mentioned in the “future investigation and next steps” section of this work.

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