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Product Assurance Overview of Additive Manufactured Components for Space Applications

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Abstract
Additive manufacturing is a key-enabling technology which is disrupting manufacturing chains. This trend is deeply affecting the aerospace domain: however, standards and quality assurance of additively manufactured parts still lack a comprehensive framework. This paper presents a state-of-the-art analysis of additive manufacturing technologies implemented in the aerospace segment, with a focus on Selective Laser Melting and material analysis. In particular, a case study material applied to this technology is retained: quality assurance design drivers and standards related to the material supply chain are reported. Furthermore, a test plan dedicated to the case study powder is considered and preliminary verification test results are outlined. Finally, future steps for work development are reported.

1. Introduction

Additive Manufacturing (AM) technologies are reshaping the manufacturing panorama. These new techniques are nowadays widely used in multiple applications given their potential to significantly reduce dimensions, weights, complexity and ultimately the cost of components and parts. Furthermore, they are widespread given their possibility of application to different materials. As an example, additive manufacturing technologies are reshaping the medical and biomedical industry, with innovative generative designs in medical inplants for human applications [1]; the architecture segment, with construction design leveraging onto additive techniques [2]; the automotive segment, where AM grew more than 3.6% in the recent years [3]; the industrial engineering and in particular the aerospace domain, given the low weight of parts enabled by new designs for additive manufacturing [4]. In particular, the aerospace segment has embedded additively manufactured components in both structural and propulsive applications. Within the last group, applications related to additive seem to be widespread among industries. Additively manufactured propulsion components, together with the implementation of highly engineered materials such as metallic alloys, ceramics and other high temperature materials, are leading to thrust chamber designs which are more effective in the solution of structural and thermal problems. As an example, innovative regenerative channel designs are possible with additive manufacturing technologies; thrusters can be manufactured with materials showing a continuous/discontinuous modification of mechanical and functional properties along the thrust chamber axis; the number of components for the thruster assembly can be drastically reduced, avoiding possible failure modes of the engine itself.

These new techniques applied to innovative materials pose a series of questions. For example, highly conductive metal alloys for thruster applications do require further research, since printing techniques for metal components are relatively new to the market and only few materials have reached high TRL values for space applications. These processes may cause non uniformity of the material deposition, leading to weak points on the structure with consequent leakages and safety issues. Furthermore, complex post processes are needed at the end of the manufacturing process, such as convoluted cleaning procedures, to retrieve the final part. For this reason, a comprehensive framework related to product assurance and quality control of additively manufactured parts becomes pivotal to increase reliability of end-user products. Requirements of quality assurance for these parts affect different aspects of the AM product lifecycle starting from the material feedstock to the machine processes and the part post-processing. A supply chain plan is considered the starting point for the evaluation of the quality assurance of the final part, as poor material quality

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may affect the final component properties. On the other side, process reverification requirements shall be defined in order to verify that the process quality is endured.

2. Additive Manufacturing Description

2.1 Additive Manufacturing Process

The definition of Additive Manufacturing is reported in [5]: the technology peculiarity is to create a part through superimposition of material, usually layer by layer and only where the part is designed to. This method is opposed to subtractive manufacturing methodologies where the part is retrieved by removal of material where it is not needed. The process is characterized by a series of steps defining the AM workflow:

- The model of the part is retrieved through computer aided design (CAD);
- The part model is converted in a specific format file, which discretizes the surface of a solid into many triangular sub-domains and stores the spatial coordinates of each side of each triangle together with a vector capable of describing the orientation normal to each surface;
- The model is uploaded in a dedicated AM machine, together with other parameters which are useful for the process completion. These parameters depend on the AM technology which is implemented: they can include the energy source scanning method, the deposition path, the energy source power, the amount of feedstock on each point and others;
- The machine performs the additive manufacturing of the part;
- The parts are removed from the machine and a series of post process treatments are performed, such as support removal (if present), heat treatments application, part finishing and polishing, others.

Methods in additive manufacturing available in literature and in the market are characterized by the feedstock involved, such as powder, wire or sheets; the material which is implemented, such as ceramics, plastics, metals, or other exotic materials (chalks, woods, other); the energy source which joins the materials to form the part, such as heated nozzles, lasers, electron beams, binders, other [6]. The American Society for Testing and Materials (ASTM) Committee F42 on Additive manufacturing technologies has currently recognized seven different technologies involved in the AM production panorama, which are resumed in Figure 1.

2.2 Additive Manufacturing for Space Applications

When dealing with the aerospace segment, optimization is the keyword that drives the part design. The need of having lightweight components that can provide specific requirements is pivotal. Through a dedicated design for additive manufacturing, even complex geometric designs can be achieved allowing reductions in the overall weight of parts: this leads to non-negligible savings for what concerns environmental and mission related costs.
The major criticalities associated to space and aerospace components may be resumed in challenging environmental conditions (plasma environment, outgassing phenomena, significant radiation conditions, others), important oxidating conditions (such as in propulsive subsystems) and high thermal loads (such as in thruster assemblies). Materials which are well suited to sustain these critical phenomena are either metallic materials or ceramics: advanced high-strength steels, Nickel-based superalloys, Titanium-based alloys as well as ultra-high temperature ceramic matrix composites are usually implemented [4].
The processes involved in manufacturing of space components are usually retrieved in the following two families:

- *Direct Energy Deposition (DED)* technology takes advantage of a heat source like laser, plasma arc or electron beam in order to melt the material and produce the object. DED differs not only for the heat source but also on the type of raw material feeding, being wire based or powder based. Usually feeding may be coaxial with the heat source or off-axis, making feeding line and heating line decoupled to each other. The nozzle can move in multiple directions and is not fixed to a specific axis, in contrast with material extrusion techniques. The main advantage is related to the theoretical limitless related to dimensions of final parts (apart from machine size) coupled with excellent metallurgical quality;
- *Powder Bed Fusion (PBF)* technology is based on feedstocks in powder form, which is spread with a powder roller or with an appropriate blade from a reservoir onto the job plate. Subsequently, a heat source selectively melts the material in the right spots, building the object layer by layer. The process continues until the full part's realization. At the end post-processing operations are mandatory for every PBF process in order to remove the unused powder and to reach the desired accuracy. Depending on the type of heat source, a laser or an electron beam, two different methods are identified, named Selective Laser Melting (SLM) or Electron Beam Melting (EBM). PBF produces high density parts with good mechanical properties and details [8]. Limitations are addressable to powder handling and recycling, high energy cost in the case of SLM and limited availability of materials for EMB, restricted only to conductive ones.

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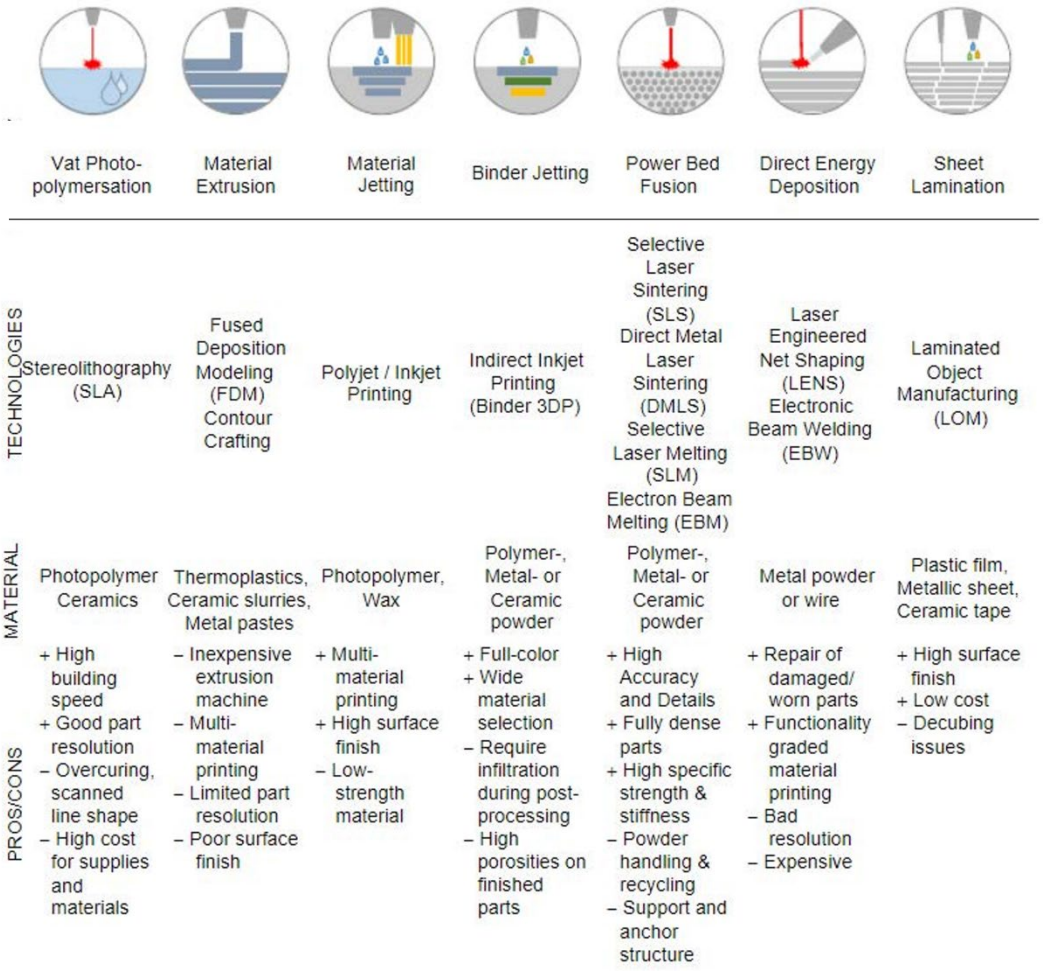

Figure 1: Technologies involved in the AM production panorama [7].

2.3 Challenges in Additive Manufacturing

AM has the potential to compete with traditional manufacturing. However, rigorous characterization, testing, qualification, and standardization are still under development and different challenges are still to be faced, in particular when dealing with critical aerospace components. Considering the two technologies mentioned in the previous chapter, AM challenges are related to standardization, process digitalization, supply chain definition and control, machine process capabilities definition and control, post-processing of parts and testing of the final components.

Standardization challenges significantly affect the widespread diffusion of AM technologies in the industry: furthermore, product assurance in the aerospace segment becomes mandatory in order to respect full traceability and quality of parts. Currently, few standards and guidelines are available in the AM market: the International Organization for Standardization (ISO) in cooperation with the American ASTM and the European Committee for Standardization (CEN) released a review of general principles related to AM in 2021 (collected in the ISO/ASTM 52900:2021 standard). The new release contains terms and definitions which help industries to better define AM environment, the identification of additive processes based on process categories and determining characteristics and basic principles for additive shaping of materials and processing principles. Other documents regulate the specifications for AM file format and provide standard terminology related to coordinate systems and test methodologies. However, compared to classical standardization of conventional manufacturing, these standards are not yet mature and further developments are needed. In addition, more specific standards for the aerospace segment are still missing. Preliminary improvements have been made through NASA's additive manufacturing structural integrity initiative (AMSII) project [9]; recently, the European Committee for Space Standardization (ECSS) released a dedicated additive manufacturing standard for laser PBF manufactured parts (the ECSS-Q-ST-70-80C on processing and quality assurance requirements for metallic powder bed fusion technologies for space applications).

Other critical aspects which become important in the AM panorama are related to *design challenges***.** Thanks to the renewed design possibilities provided by additive techniques, designers are able to optimize a component in

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order to fulfil particular requirements and reduce material wastage. Two possible design strategies are available: size optimization and topology optimization. In the first solution, component dimensions are settled as design driver: an iterative process based on finite element (FE) simulations enables the optimization of the part. In the second method, density of each component part is settled as design variable. A polyhedral mesh is then used to create a FE analysis and begin the iterative process. Optimization strategy redesigns the density map until the target density is reached, followed by a CAD remodelling of the optimized part [5]. The main design guidelines for what concerns design with L-PBF technologies involving metals in powder form are collected in a dedicated standard by ISO and ASTM (the ISO/ASTM 52911-1:2019 on Additive manufacturing design for metal L-PBF components).

Supply chain challenges are related to the material involved in the process. AM has the advantage of adding material only where strictly needed: however, the process of modifying the material to thin wires or fine powders is associated to expensive processes like gas, water or plasma atomization. The repeatability of the process shall be ensured. Certifications are yet another obstacle: despite being required to guarantee that materials meet equivalent standards to those used for traditional methods, their development becomes time consuming and expensive. Furthermore, the recycling of the unused material to successive prints adds major concerns in the quality assurance and standardization of the supply chain. Quality of raw and recycled materials shall be assured, however a comprehensive analysis on how macroscopic behaviour of components and material properties change with respect to several printing cycles is still under investigation [10]. The supply chain analysis covers most of the standards currently under development in the AM panorama, followed by process standards and testing and qualification (see Figure 2). As an example, a preliminary and non-exhaustive categorization of the main standards for additive manufacturing of Nickel-based metal alloys using SLM has been reported in Figure 3. The analysis has started from the definition of the main top-level standards from ISO and ASTM previously mentioned down to the raw material standards and the material specific standards. The lower level has been characterized with documents regulating powder and sample testing.

Process challenges have a significant role in affecting the final properties of AM parts. In fact, depending on machine calibration, components may have different mechanical properties caused by anisotropies in the part microstructure. Within SLM, the main process parameters affecting the final component are the laser power, the scanning velocity, the thickness of the powder layer and the separation of two consecutive laser beams (or hatch spacing), as well as the laser scanning strategy [5]. These parameters shall be properly tuned to produce high density parts and limit the presence of internal defects and residual stresses. Structural support design is also a key aspect of processing challenges.

For most of AM processes, post-processing operations are required to improve functional and mechanical properties of components, and to obtain the desired surface finish and dimensional accuracy. *Post-processes challenges* include the identification of specific treatments like Hot Isostatic Pressing (HIP), ageing and others. The thermal treatments may enhance mechanical properties and decrease any existing porosity for metal AM fabricated parts. Other post-processes include cleaning of convoluted geometries, in particular when dealing with inner channels, as well as polishing and surface finishing of the accessible surfaces of the parts.

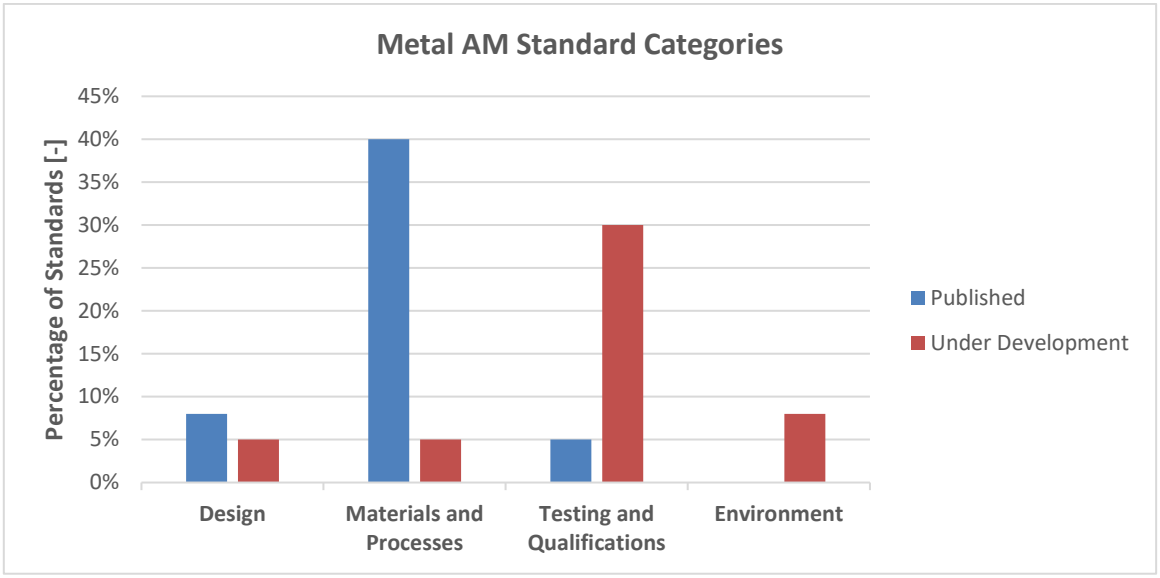

Figure 2: Published and under development AM standards [11].

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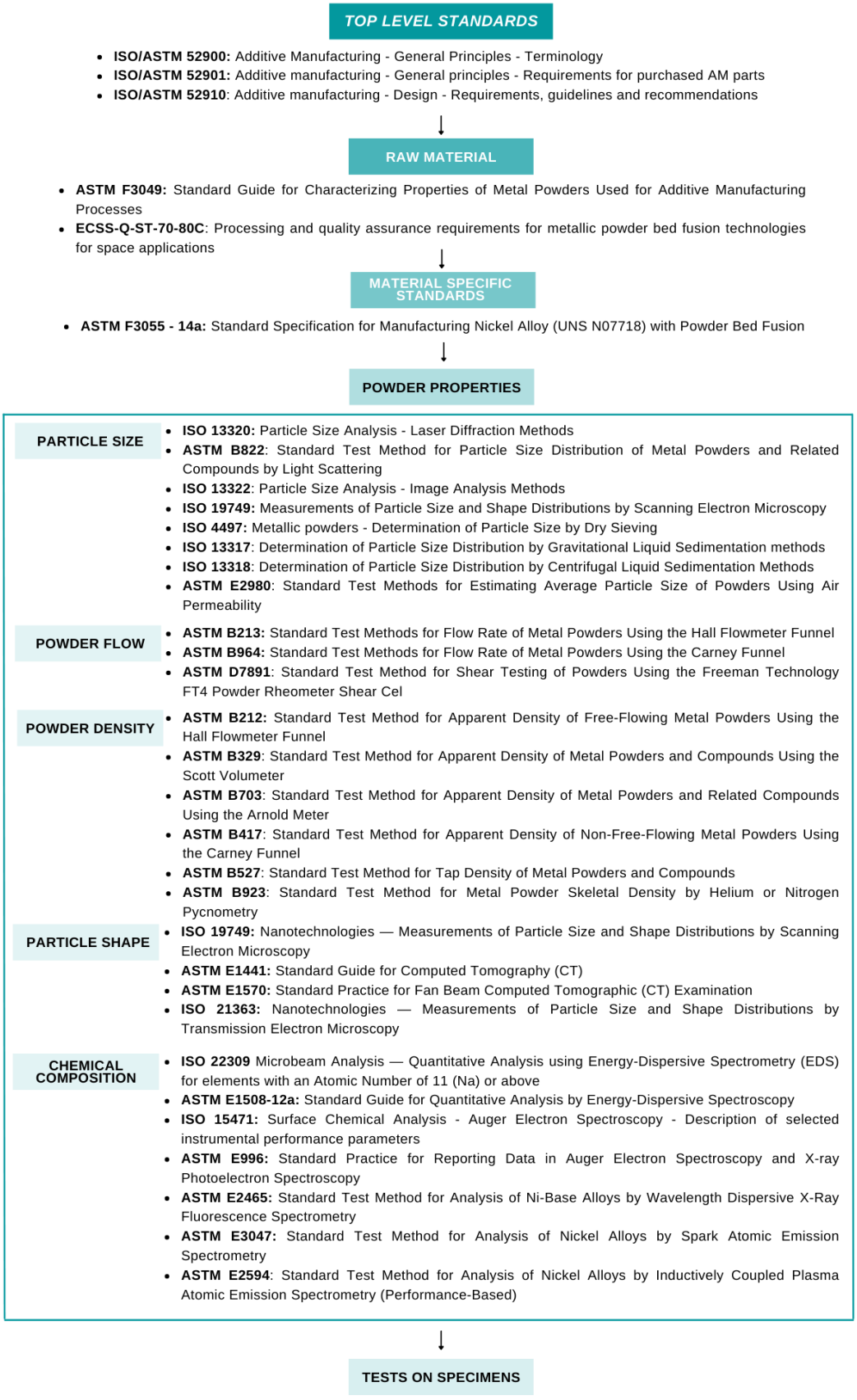

Figure 3: Standard identification process tailored for IN718 powders.

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3. Material Characterization Plan

In this work, the preliminary steps which have been performed toward the definition of a comprehensive test plan specifically tailored for the qualification of additive manufacturing components for critical applications in the space domain start from the definition of the supply chain and material characterization criticalities. A preliminary case study is retained, based on a metal Nickel-based superalloy: in particular, Inconel 718 (IN718) is considered. The technology implemented for the component manufacturing is based on L-PBF.

3.1 Material Properties and Interdependencies Characterization

The analysis of the product lifecycle for an AM part for space applications has started from the characterization of the material implemented in the process. The supply chain has been divided into three main phases: the raw material phase, the material handling and storage phase, and the material processing and testing phase. The first category considers all the challenges related to production and postprocess of the material: depending on the type of process implemented to generate powder from raw materials, different powder properties may be impacted (granulometry, morphology, other). The second category includes criticalities in terms of handling, transportation and storage. Safety and security aspects are related this phase, as the materials involved may deal with toxicity, flammability and volatility hazards [12]. The last part includes all the aspects related to testing of the obtained material: as the material is in powder form, it shall be characterized by means of specific material properties which are impacted by multiple external factors with respect to the bulk material. Furthermore, reusability and recycling of powders add significant criticalities to the quality assurance process.

Within this study, the following properties for a powder have been considered: powder granulometry, morphology, surface topography, chemical composition, density, compressibility and flow characteristics [13]. These properties are impacted by external factors, in particular humidity, temperature and consolidation history, as well as number of recycling and recycling methods [14].

- *Powder granulometry* includes the definition of particle size and Particle Size Distribution (PSD): the first characterize the particles by their particle diameter, while the second indicates the percentage of particles of a certain size or in a certain size interval.
- *Particle morphology* is defined by ASTM B243-11 as the characterization of shape and contour surface of particles. Due to the production process, storage and handling as well as powder consolidation history, different particles are classified: spherical, splat, elongated, broken, satellited, other. Particles can be categorized in these classes by means of dedicated Scanning Electron Microscope (SEM) images. Highly spherical particles are desired in the SLM process since they can gradually enhance both powders packing density and rheological performances.
- *Surface topography* of particles reports the roughness, sharpness or fineness of individual particles. Surface topography usually reveals contamination on particles surfaces, which can be analysed by means of dedicated SEM images, as reported in Figure 4.
- *Chemical composition* of particles highlights the presence of impurities in the powder, which influence the final properties of the as built components: it tends to change over time and with recycled powders, as reported in [15].
- *Bulk Density* considers the measurements of apparent density, defined as mass of unit volume of loose powder expressed in g/cm^3; tap (or packing) density, defined as the maximum density achieved when the metal powder is tapped under specific conditions; skeletal (or true) density, defined as the ratio of the mass of solid material to the sum of the volumes of the solid material and closed (or blind) pores within the material.

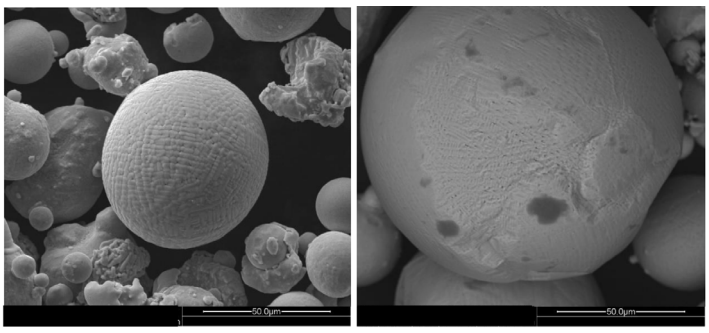

Figure 4: SEM images reporting particle surface roughness (on the left) and particle surface contamination (on the right).

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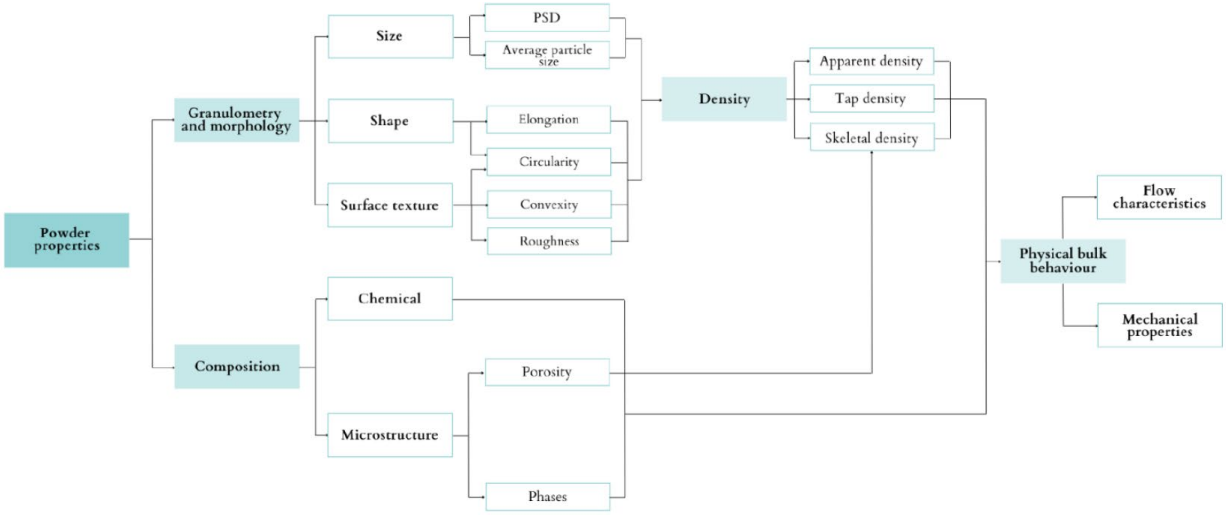

Figure 5: Flowchart reporting the interdependencies among powder properties.

- *Rheological properties* of powders indicate how powders mix and behave in the SLM machine feeder. They indicate also how powders flow by means of the recoater to form the powder bed.

Each one of the previous characteristics is interconnected, due to the presence of interdependencies between particles properties. The interdependencies are highlighted in Figure 5. Density characteristics are mainly impacted by granulometry and morphology. Chemical composition, and in particular microstructure characterization, impacts on the porosity and on the skeletal density of powder. On the other hand, rheological properties are affected by shape and size behaviour which is also important in the definition of the final part mechanical properties.

3.2 Material Verification

After the identification of the main material parameters affecting quality assurance of powders for space and aerospace components, the activity has moved towards the definition of a preliminary verification plan for IN718 material. The verification plan is mandatory in order to assess how to characterize the properties previously defined as well as to define a temporal sequence capable of optimizing the results and exploit the interdependencies between material properties. The test to be performed on the material have been selected following a screening of previous analyses exploited in technical literature, considering both frequency of test application and test results efficiency in terms of reliability, reproducibility and cost.

Powder granulometry analyses stands out as first analysis to be performed: the definition of the average particle size is performed through Laser Diffraction (LD) technique. In order to cover particles morphology, a SEM analysis shall be performed in a complementary way with the LD measurements both on virgin powders (to check if the powder production process led to agglomerates or satellites generation) and on re-used powders, if present. The assessment of particle shapes is followed by powder surface area evaluation: the Brunauer-Emmett-Teller (BET) method allows the measurement of the surface area, and from that the roughness of powder surfaces can be analytically determined.

In parallel to granulometry, morphology and surface texture analyses, *chemical composition* investigations are carried out through Energy-Dispersive X-Ray Spectroscopy (EDS) analysis on both virgin and recycled IN718 powders. Auger Electron Spectroscopy (AES) and Inductively Coupled Plasma (ICP) analyses, instead, ought to be specifically performed for studying the material composition dependence on IN718 powder reutilization and for determining potential contamination issues induced by handling procedures during the overall AM production cycle.
After the assessment of the fundamental particle powder properties, the bulk powder behaviour is analysed through *proper density and rheological measurements*. The Hall flowmeter can be employed for flowability and apparent density characterization as well as the analysis of the angle of repose, while gas pycnometry systems are used to determine the true powder density through total pore density measurements. Finally, a test method for measuring the tap density is performed to ensure proper powder bed uniformity, which is strictly related to the final built part qualification.

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3.3 Re-Verification Requirements

The designed temporal sequence let the verification of the complete supply chain of the material involved in the SLM process. However, performing all the outlined tests after every single usage of metal powders taken from the same batch results in high costly and time-consuming procedures, as well as unnecessary. When the same batch powder is stored in appropriate inert environment, only minor or negligible properties degradation are registered.
Thus, the next step of the analysis has considered all the main AM process steps in which the powders may be subjected to significant degradation mechanisms of the microstructural properties. The result of the analysis are reported in Figure 9, where all the cases of industrial relevance in which each selected tests shall be performed are reported.

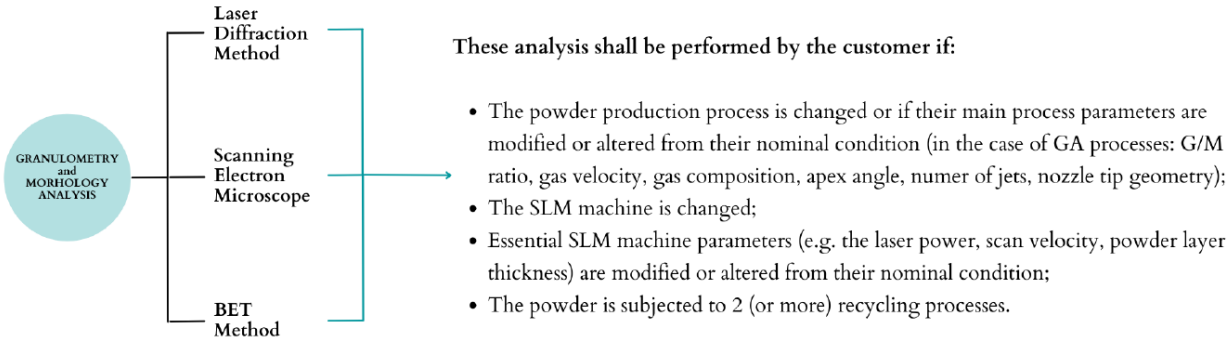

Figure 6: Re-verification requirements for granulometry and morphology analyses.

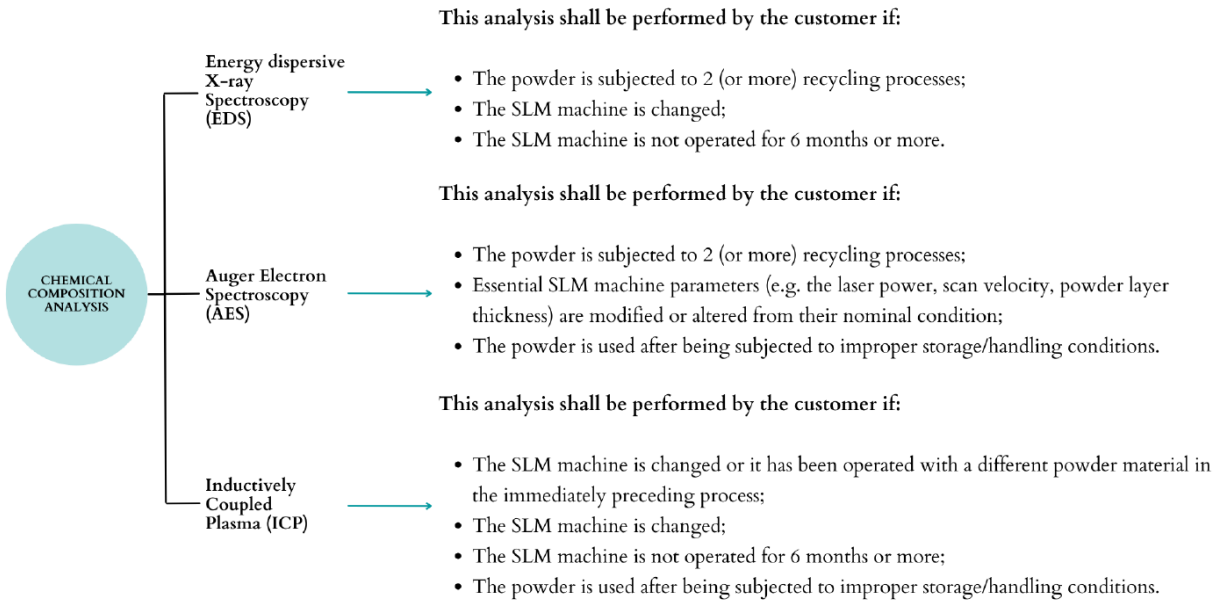

Figure 7: Re-verification requirements for chemical composition analyses.

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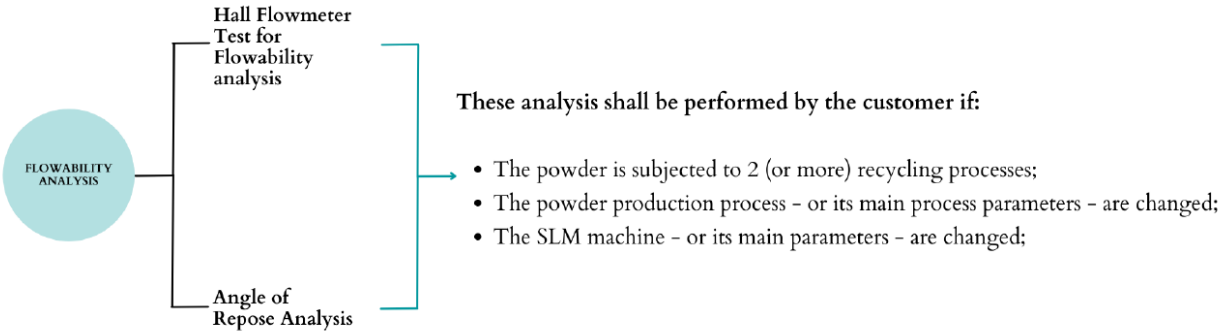

Figure 8: Re-verification requirements for flowability analyses.

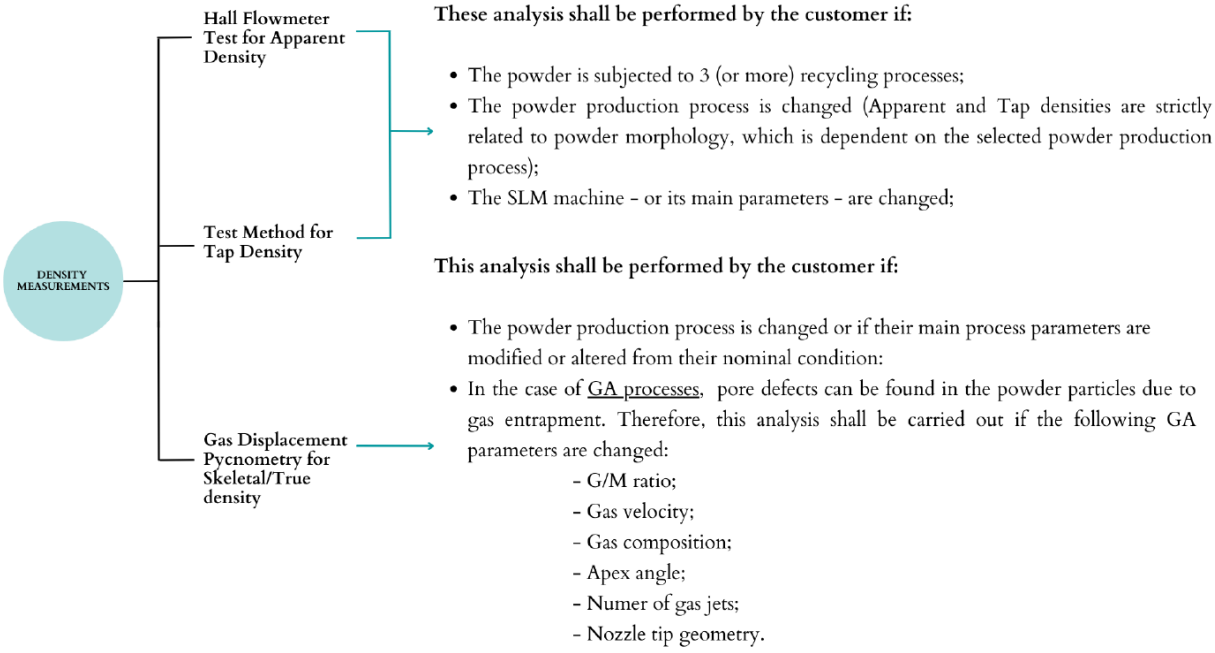

Figure 9: Re-verification requirements for density measurements.

4. Experimental characterization

4.1 Printing Job Description

The proposed verification plan has been applied to a set of recycled IN718 powder samples. The aim of the case study is to verify the effects of multiple recycling on powder parameters and assess the quality of the material during AM processing. Samples are provided by an external supplier of AM parts for space applications. Eight powder samples, all derived from the original powder batch, are retrieved: each one considers powder at different stages of the recycling process. Each family (or job) is composed by a "starting material", an "unsieved" material (obtained immediately after the printing process), and by a "sieved" powder (what remain after sieving the unsieved particles and after a proper mixing with fresh powder). Through this process, the sieved material of a family represents the "starting material" of the next family. Powders recycling process involves sieving with two siever dimensions: 63um and 100um respectively. For each sample, the number of recycling and a brief description are reported in Table 1. Powder lot N2, N4 and N6 were retrieved by means of a dedicated powder sample capture of conical shape containing 250mg of powder, reported in Figure *10*; the container is built during the printing process and then collected and opened through a dedicated opening procedure. All the other samples are retrieved by means of a dedicated powder sampling tool at beginning or end of the printing process.

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Table 1: Powder supplied for the analyses.

Job Family	Type	Powder lot	Powder Lot
	Starting Material	N1	Virgin powder
Job 1	Unsieved material	N2	N1 after 1 job (pre sieve) [a]
	Sieved material	N3	N1 after 1 job sieved (ready for the job n° 2)
	Starting Material	N3	N1 after 1 job sieved
Job 2	Unsieved material	N4	N1 after 2 jobs (pre sieve) [a]
	Sieved material	N5	N1 after 2 jobs sieved (ready for the job n° 3)
Job 3	Starting Material	N5	N1 after 2 jobs sieved
	Unsieved material	N6	N1 after 3 jobs (pre-sieve) [a]
-	Residues	N7	N1 after 3 jobs (residuals from sieving at 63 μm)
-	Residues	N8	N1 after 3 jobs (residuals from sieving at 100 μm)

[a] Powder preserved inside the powder sample capture

Figure 10: Powder sample capture of conical shape.

4.2 Granulometry and Morphology Results

Following the qualification process, the initial tests on the powder material are related to granulometry and morphology. Starting from particle size distribution (PSD) characterization, LD method has been implemented through a Malvern Mastersizer 2000 equipped with a SCIROCCO unit. For each powder batch, five tests have been performed and the results averaged. The typical sample mass was about 1500 mg. All the batches feature a narrow and symmetric monomodal PSD with a peak located between 30 μm and 40 μm. The sole exception is represented by the samples N7 and N8 which show a peak shifted towards bigger values (around 60 μm and 120 μm respectively). This is explained by the fact that residues are composed by big particles, probably deriving from the granules-laser interactions occurring during the printing process. The sieving process followed by the material replacement tends to bring back the processed powders to the original characteristics of the starting material. Only minor differences and a neglectable presence of "residual" big particles can be detected.

SEM images supply further qualitative information concerning particle morphology, surface texture and size. The images were retrieved by means of a FeiNova NanoSEM 450 instrument at 25 kV and different magnifications. From the study, spherical shape particles with a relatively smooth surface stand out: however, samples N1 and N2 exhibit a higher presence of irregularly shaped particles with respect to samples from N3 to N6. This is probably caused

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by the peculiar atomization process involved in the material supply chain (gas atomization process). In general, altered granules observed in the SEM images are assigned to five main categories, reported also in Figure 11:

- elongated or exotic particles (yellow arrows);
- particles with satellites (green arrows);
- particles with "smashed" object on the external surface (orange arrows);
- broken or damaged particles (purple arrows);
- particles with dark spots on their surface (red arrows);

The last defect category has been detected only in the samples N4, N5, N7 and N8. Sample N8 presents several particles with multiple dark spots each (see Figure 11 for an evident example). The effect can be directly associated to the powder recycling, and it is probably related to intense heat generated by the passage of the laser. This type of defect may indicate not only chemical changes due to the laser but also the potential presence of other phenomena (boiling, or other). However, particle with a significant difference to the colour and texture of their entire surface were not detected.

4.3 Chemical Composition Results

Chemical composition of the powder batches was considered starting from X-Ray Spettroscopy. For this analysis, a small amount of powder was levelled inside a sample holder with no powder pretreatments, then radiation X (λ = 1.5416 Å) at 1.6kW energy was applied through a Philips/PANalytical X'Pert Alpha-1 X-ray diffractometer. The curve patterns indicate the presence a Ni-Fe-Cr alloy compatible with the IN718: the similarity of the shape of the spectra indicates that, for the most part, no massive change occurred between samples.

Auger Electron Spectroscopy (AES) has been subsequently considered to determine superficial elemental composition of the powder, in combination with a sputter gun to perform depth profile composition analyses. The AES analyses were executed by using a 3 keV energy source coupled with a Perkin-Elmer 15-110B Cylindrical Mirror Analyzer. Ion sputtering has been obtained through a Specs IQE 12/38 ion gun. Samples N1 to N6 present similar results between each other, where the presence of Niobium increases with depth while other components such as Iron, Titanium, Chromium and Molybdenum tend to be present at all depths. The presence of oxygen decreases with depth, which is explained by the most outer layers being exposed to air and thus to oxidation. Samples N7 and N8 (the powders that have been sieved) are significantly different form the others: the most common element detected is Titanium which remains around 40% at all depths, while nickel remains below 10% at most depths. Since the residue after sieving is the part of the powder that has been more heavily influenced by the action of the laser, these results suggest that such action has a very significant effect of the composition of the powder, especially on the presence of Nickel and Titanium. However, given the constrains of the analysis, this effect can only be confirmed in the superficial layer of the powder.

In order to consider the powder chemical composition at elemental level, an Inductively Coupled Plasma (ICP) - Optical Emission Spectrometry (OES) has been considered. Al, Co, Cu, Mn, Mo, Nb, Si, S, Ta, and Ti have been selected from literature [16] as the most representative elements for the batches. After preparation of the samples, analyses were executed through a Varian 710 ICP-OES.

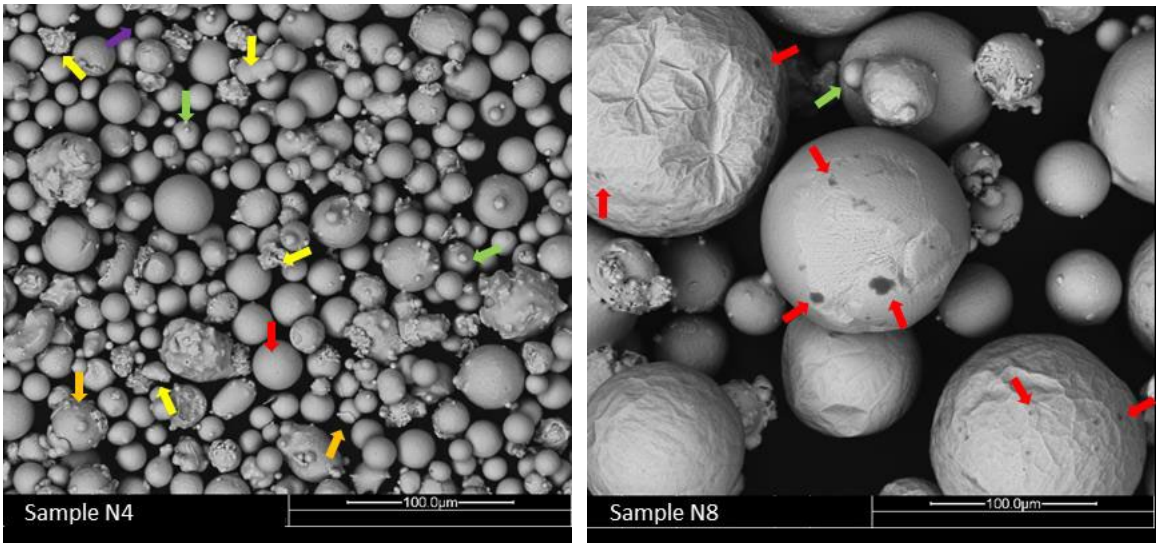

Figure 11: Particles alterations in SEM images (on the left), focus on particle surface alterations (on the right).

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Table 2: Contaminant mass fractions for all the tested powders. For comparison purposes, the value reported in the powder provider technical data sheet as well as the minimum and maximum values accepted by the reference standard ASTM B637 are considered. In **bold** the out-of-bound values.

Sample	Min	Max	CoC	N1	N2	N3	N4	N5	N6	N7	N8
Element	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**	**w.t.%**
Al	0.200	0.800	0.490	0.522	0.527	0.546	0.558	0.548	0.553	0.549	0.558
Co	0.000	1.000	0.200	0.199	0.181	0.169	0.089	0.151	0.089	0.091	0.089
Cu	0.000	0.300	<0.100	0.019	0.018	0.020	0.030	0.023	0.031	0.030	0.031
Mn	0.000	0.350	0.020	0.013	0.014	0.020	0.040	0.025	0.034	0.030	0.032
Mo	2.800	3.300	3.160	3.200	3.280	3.220	3.100	3.150	3.150	3.160	3.110
Nb	4.750	5.500	5.000	4.860	4.890	4.840	**4.710**	4.800	4.800	4.780	**4.720**
Si	0.000	0.350	0.030	0.059	0.061	0.061	0.071	0.099	0.073	0.068	0.066
S	0.000	0.015	0.001	**0.017**	0.014	0.013	0.012	0.013	0.012	0.015	0.014
Ta	-	-	0.000	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
Ti	0.650	1.150	1.020	0.993	0.999	0.994	0.985	1.010	0.965	0.965	0.982

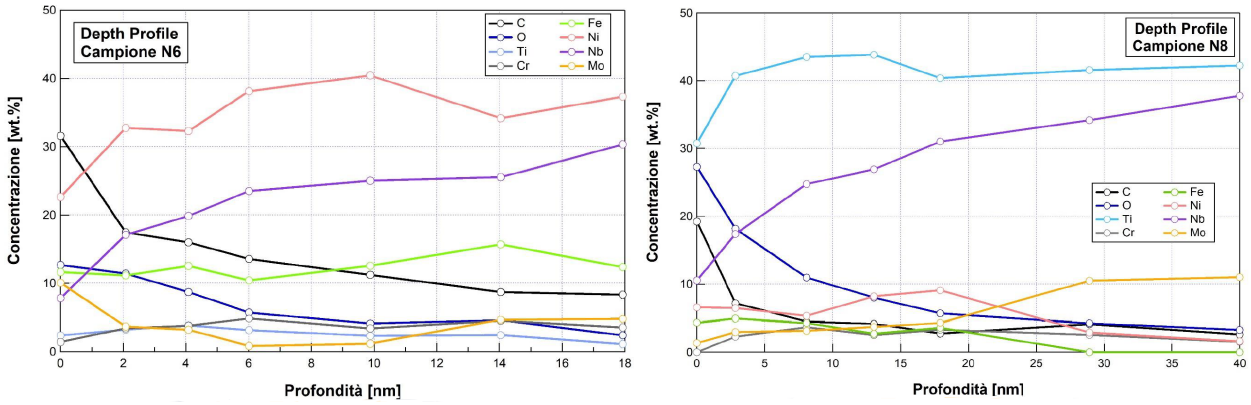

Figure 12: AES profile of sample N6 (on the left) and N8 (on the right).

The mass fractions of the considered elements are reported in Table 2. In general, all the powder lot are compliant with the standard ASTM B637: the only criticality is highlighted on sample N1 which exhibit a value of sulphur at a concentration one order of magnitude higher than that declared by the supplier. This anomalous value is detected in the virgin powder while all the other samples are characterized by values inside or close to the standard limits. This peculiar behaviour suggests a possible effect of the material sampling different, and potentially more important, with respect to the printing process. In general, by increasing the number of jobs, a progressive reduction of some impurities (e.g. Co, Mo and Ti) and a progressive increment of other elements (e.g. Mn, Al, Si, and S) is registered. This phenomenon confirms that a progressive variation of the powder composition can be directly associated to multiple batch recycling. Even if under datasheet limitations, it is also important to notice that some components values differ for more than 15% with respect to the supplier technical data sheet (Al, Cu, Si, S). The level of copper is lower than declared, while aluminum, silicon and, in particular, sulfur are present in higher quantities. If this is not a problem for the first three elements, the same cannot be said for sulfur because its value is close to the limit declared by the standard.

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4.4 Density and Flowability Results

Density measurements considered the analysis of the apparent density, which measures the density of the powder including the pore volume and the space between particles, and the particle (or skeletal) density, which measures the density exclusively of the powder particles. The measurement of the bulk density has been carried out utilizing a hollow brass cylinder with a fixed internal volume and a Gibertini Europe 500 scale for the measurement of the mass. The measurement of the particle density was executed utilizing an Anton Paar Ultrapyc 5000, which is a gas pycnometer utilizing helium for the measurement following the standard ASTM B923-10 (Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry) and at a temperature of 20°C. The results show that bulk density is significantly lower than the particle density, which indicates that the powder is not tightly packed. The value of the bulk density, in general, increases with respect to the value of the virgin powder as more jobs are completed. On the other hand, the value of particle density tends to decrease. These effects can be attributed to the recycling of the powder during the manufacturing process.

As a final step, flowability and Angle of Repose (AoR) results are retrieved on powder lot from N1 to N6 by means of a Hall funnel following EN ISO 4490:2018 standard (Metallic powders - Determination of flow rate by means of a calibrated funnel: Hall flowmeter). 50.0 ± 0.1 g of powder were implemented during these tests, which were repeated three times: the funnel was previously tested with a reference calibration powder (Chinese Emery Powder from ACu Powder) and the reported results have been corrected as indicated by the cited standard. The results of the flowability and AoR tests are reported in Table 3. It can be observed that the time to complete the tests diminishes the more the power is utilized in the printing process. A lower time, in general, indicates that the powder flows better and is less cohesive. This effect can be attributed to the recycling of the powder. A similar effect on the powders' characteristics is also observable in the angle of repose measure. The value of the angle of repose diminishes indicating that the powder is more free-flowing and less cohesive. Finally, the effect of the replenishment with fresh powder can be observed well between N4 and N5 samples, where the characteristics are partially restored closer their initial value. On the contrary, between N2 and N3 the flowability time diminishes while the angle of repose does not change in a significant way. This peculiar behaviour could depend on the difference between the printing jobs performed when the samples have been taken or the sampling methodology.

Table 3: Flowability and Angle of Repose of the tested powders. The uncertainty has been computed considering the t-student distribution with a confidence level of 95%.

Powder Lot	Flowability, s	Flowability, s/g	AoG, deg
N1	18.1 ± 0.2	0.363 ± 0.003	36.58 ± 0.48
N2	18.3 ± 0.3	0.367 ± 0.007	36.68 ± 0.37
N3	17.7 ± 0.0	0.352 ± 0.000	36.79 ± 0.46
N4	16.8 ± 0.3	0.335 ± 0.006	34.62 ± 0.49
N5	17.2 ± 0.3	0.345 ± 0.006	35.16 ± 0.61
N6	17.1 ± 0.4	0.342 ± 0.007	34.68 ± 0.51

4.5 Remarks

The detailed analyses of the batches allowed the identification of some common characteristics as well as discrepancies between them. Two main groups are identified, the one involving recycled powders (powder lot N1 to N6) and the one including the residues (powder lot N7 and N8). Powders from the first group exhibit more or less the same particle size distribution with minor differences between the samples: in particular, it is demonstrated that the sieving process followed by powder replacement (using the virgin material) tends to recover original values of PSD. Looking at the powder shape, it is possible to state that the increment of the number of jobs brings to a progressive regularization of the particle shape: the sieving process affects this phenomenon, as stated by powders in group two which show lowest regularity values independently on the powder size.

Powder composition through XRD analyses do not reveal any difference between the samples and it is possible to state that the number of job as well as the change of lot do not influence the overall composition of the powder. This

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is valid for powders of both groups. However, AES results show that particle composition tends to change, starting from the surface and proceeding towards particle centre. This is valid for all the examined powders, even if powders from group one are characterized by similar composition and trends. Powders from group two feature a completely different surface chemical composition: this confirms that the particles interested by the laser heating are characterized by chemical changes located at the surface. This is not true in the core of the particle as evidenced by XRD test. A deepen study with ICP to search impurities reveals that all the considered samples satisfy the requirements reported in ASTM B637. Only powder lot N4 and N1 show minor out of bounds values of niobium and sulphur respectively. As for N4 the impact on powder quality is minor since the sieving process corrects the composition discrepancy, the same cannot be stated about N1, where contamination of S is detected in the virgin powder lot. This is probably imputable at the sampling or handling or transport procedures involved: sulphur, in fact, can be present in latex and nitrile gloves as vulcanization accelerator, but also in plastics like PP and PE as impurity coming from the production process.

Finally, flowability as well as angle of repose tests show only small differences between samples or group one. On the other hand, actual density of powder seems slightly affected by the number of recycling: a variation of about the 0.18% has been detected between the sample N1 and N6. Powder bulk density, on the contrary, is clearly affected by the job number as it tends to increase while increasing the number of jobs: this behaviour can be imputed to the better packing of recycled powders due to morphology variations.

Conclusions on this work show that powder properties are affected by the printing process even after a reduced number of jobs. However, a careful sizing and powder replacement procedure during recycling is capable to bring back IN718 powders to the original quality stated by the standards. This suggest a change in the reverification requirements previously proposed, as the tests on powder properties can be relaxed and repeated after an increased number of recycling with respect to the predicted selected number of two. On the other hand, the analysis shows that there are limitations to the recycling process: it cannot solve local surface composition change of powders as well as powder morphology and other rheological properties. The trend change of the properties suggests that a maximum in the number of recycling processes which can be applied to IN718 powder lots shall be determined: the maximum shall consider the point at which local degradation effect of powder become significant at bulk material level.

5. Conclusions and Future Steps

In this work, a preliminary analysis of the quality assurance of additively manufactured components for space applications has been presented. The work has identified the technologies implemented in the aerospace segment and it focused on the identification of L-PBF standards. A preliminary qualification plan has been proposed through the selection of dedicated tests to be performed on IN718 raw and recycled material. Reverification requirements have been identified and a maximum recycling number of powders recycling without repeating certain verification tests has been proposed. A case study has been implemented in order to assure the quality of the recycling process on a series of dedicated powder lot from a supplier of aerospace components. The results show that the recycling process involved is effective in restoring the quality of powder, with certain powder parameters which tend to degrade due to the recycling process.

Future steps of the work include the identification of a maximum acceptable number of recycling which shall be applied to a powders lot before interrupting the recycling process. This shall be performed by the identification of the effects of powder recycling on both powder properties and final built objects: in particular, verification of powder recycling effects at mechanical, metallurgical, chemical and thermal level on dedicated SLM samples shall be further addressed.

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