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Effect of IN718 recycled powder reuse on properties of parts manufactured by means of Selective Laser Melting

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Abstract

Powder quality control is essential to obtain parts with suitable mechanical properties in Selective Laser Melting manufacturing technique. One of the most important advantages of such technique is that it allows an efficient use of the material, due to the possibility to recycle and reuse un-melted powder. Nevertheless, powder material properties may change due to repeated recycling, affecting this way the mechanical behavior of parts. In this paper the effect of powder reuse on its quality and on the mechanical properties of the resulting melted parts is studied via self-developed recycling methodology. The material considered for investigation was IN718, a nickel superalloy widely used in industry. After recycling powder up to 14 times, no significant changes were observed in powder and test parts properties. The results obtained in this work will help to validate powder recycling methodology for its use in current industrial Selective Laser Melting manufacturing.

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Keywords: Selective laser melting; IN718 powder; recycling; reuse; material use efficiency; mechanical properties

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1. Introduction

Inconel 718 (IN718) is one of the most widely used nickel superalloy in high temperature applications, including aerospace ones. It is an age-hardened Ni superalloys, being gamma double prime the main strengthening phase, which is stable up to 650 ºC. It is designed for service in operation of up to this temperature. IN718 is a weldable alloy, so that it can be "easily" processed by laser-based manufacturing techniques like Selective Laser Melting (SLM) Kelbassa et al. (2008). SLM is a powder bed fusion additive manufacturing process that allows obtaining fully functional three-dimensional parts from a CAD model. It is based on the application of metallic powder in very thin layers (20-100 µm) on a building platform, which is melted by the energy of a high intensity laser beam. Consequently, almost fully dense parts with no need for post-processing other than surface finishing are produced. It is a manufacturing technology applicable to aerospace, medical, dental and other industrial sectors, as it offers great potential for production of complex parts.

The aerospace industry is particularly promoting this technique, and especially in the production of aerospace jet engine components with complex geometry built in high-added value materials (e.g. Ni superalloys, TiAl, Ti64), such as gas turbines components, from blades or airfoils to different type of vanes and cases. The conventional routes of manufacturing imply machining, and therefore require the use of cooling lubricants and other toxic chemicals and mainly create a massive amount of waste. SLM technique allows the production of more efficient components (design not or less limited to manufacturing process, because of the almost unlimited geometrical freedom of design of SLM) and do not use toxic chemicals. Also, one of the most important advantages of selective laser melting process is that it allows theoretically 100 % material utilization through non-consumed powder material recycling. This has been carried out in research and development environments with little knowledge of how the powder is affected and whether recycling the powder induces quality issues in future deposits. In production however, recycling is not always carried out, given that it is viewed as a risk, because it has not been properly validated in the procedure development phases. This is particularly important for aerospace industry, where process validation and standardization is mandatory. Powder recycling methodology validation will ensure that recycling occurs and takes place high on the value chain.

To fully exploit this potential benefit, and move from prototype and small batches manufacturing to in series production, it is necessary to develop an understanding of how un-melted powders change during processing due to contamination and change in particle morphology and what impact, if any, these have on build quality and mechanical properties. It is well known that powder characteristics affect significantly to manufactured parts density, mechanical properties and surface roughness Spierings et al. (2010) & Liu et al. (2009). Particles must be not only spherical but also they must have no defects such as satellites or internal pores. The presence of satellites will affect the flowability of powders and thus the powder layers. Besides this, a powder with an important amount of particles with pores will be detrimental to the density after melting due to lacks of fusion.

On the other hand, powder particle size distribution plays an important role in powder bed formation Randall (1994), since powder particle size distribution affects flowability. A powder with a significant percentage (>5 %) of small particles (the limit is between 10 and 20 microns, depending on material) will have poor flowability, leading to an inhomogeneous powder distribution over building platform. As a result melting faults are produced, affecting mechanical properties. Actually, this amount must be balanced, because batches with no small particles have less capacity for particle accommodation, given that smaller particles fill the gap between larger ones Riou et al. (2013). Moreover, the risk of chemical reactions between the powder material and oxygen (or other contaminants present in the working atmosphere) should not be forgotten, as it is critical in reactive materials such as titanium alloys Thijs et al. (2010) & Seyda et al. (2012).

The SLM manufacturing of IN718 components has already been validated at laboratory scale. This makes especially interesting the validation of powder recycling methodology applied to IN718, being in fact one of steps to be followed to validate the production at industrial level. The present study has been consequently focused on validating a particular self-developed recycling methodology for IN718 powder, with the aim of demonstrating that it can be constantly reused or at least with the purpose of calculating its utilization limit by understanding its behavior during several iterative fabrication processes.

2. Experimental Setup and Methodology

The analysis of recycled powder reuse along consecutive manufacturing processes was experimentally carried out by using an industrial SLM system developed by MCP (Model Realizer SLM 250). The device is equipped with a 200 W fiber laser and works in a protective argon atmosphere with an oxygen content limited to a 0.2 %. The powder used for the experiment was a single 25 kg batch of Inconel 718 Ni superalloy, obtained by atomization with argon gas as this is the most suitable method to obtain high quality powders with small particle size distribution and large homogeneity. The material was produced by LPW Tech. Ltd., with the chemical compositions indicated in Table 1 (chemistry to AMS 5832 F) and with a particle size in the range 15-45 micron, according to material certification.

Table 1. Chemical composition of the IN718 powder.

Alloy name	% Ni	% Cr	% Co	% C	% Mo	% Al	% Ti	% Fe	% Nb+Ta	% Mn
IN718	Bal	18.64	< 0.3	0.06	3.05	0.62	0.93	17.53	4.88	0.03

To study the effect of powder reutilization after recycling, a very specific methodology was developed. It consists of an iterative process distributed in three main steps (see Fig 1). The first one, after all the powder is loaded into the SLM device and proper fabrication parameters are set, is the fabrication of test samples. These samples are removed from the machine and stored apart for a posterior metallurgical and mechanical analysis. The second step requires removing of all powder from the fabrication chamber to submit it to consecutive sieving and drying processes. In the third and last step, part of the powder is separated for the study of its properties, and the rest is loaded into the SLM device to start over the fabrication process again.

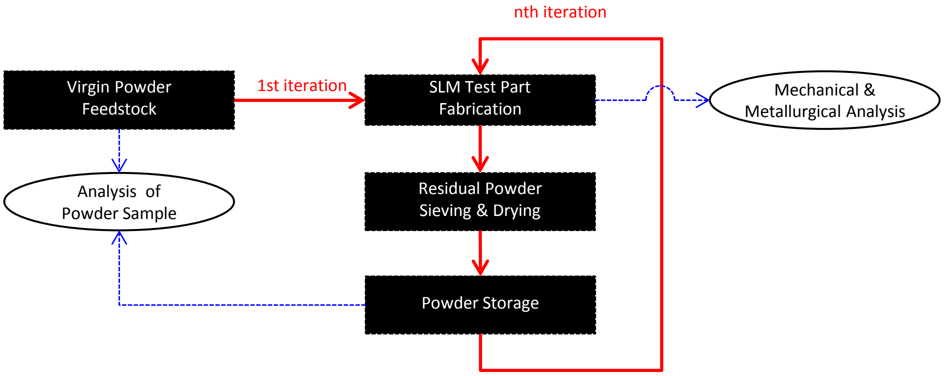


Fig. 1. Scheme of test piece and powder sampling methodology.

By doing this, IN718 powder is expected to recover its initial properties at the beginning of the following iteration and therefore, to allow for fabrication of parts with identical mechanical properties, thus optimizing resource utilization by minimization of wastes. To check the validity of this approach, metallurgical and mechanical analysis of samples was accomplished via measurements of: porosity, using an Olympus GX51 optical microscope; material toughness through Charpy impact tests performed with a Hoytom 300J/AD2 system; material hardness with a DuraScan microdurometer from EmcoTest; and microstructure using an UltraPlus SEM system from Zeis, which in addition incorporates EDS and EBSD capabilities and allowed for a complete powder composition characterization.

3. Results and Discussion

Thus, following the methodology described in the previous section, fabrication of test samples was accomplished. Fourteen loops of this methodology were executed using an optimized set of parameters (for IN718) and a layer thickness of 20 µm to ensure the best possible quality of test samples. At every iteration, six mechanical test samples (cuboids with dimensions of 60x12x12 mm) and three metallurgical test parts (dimensions: 10x10x10 mm) were built in the industrial SLM system to later perform a metallurgical and mechanical analysis and thus study the dependence of part properties with powder ageing (see Fig. 2).

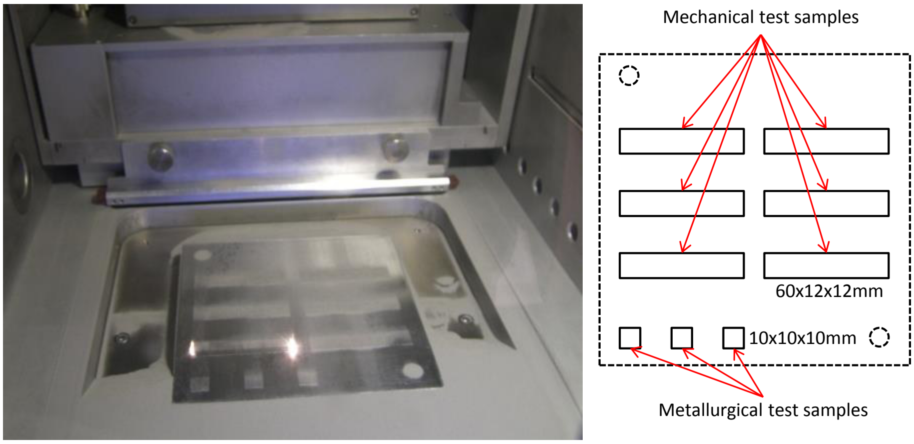


Fig. 2. Left: picture showing manufacturing of tests pieces; Right: arrangement and dimensions of test pieces.

In addition to this, powder volumes (150 g) were collected in every loop after each corresponding sieving and drying processes for their analysis as well. In particular, the powder was sieved at 20-25 ºC to remove agglomerates using standard stainless steel sieve with 63 µm mesh size and then dried in an oven with air circulation to remove moisture. Trials were properly documented and best practices in powder handling were carried out during the work to avoid any material pollution.

3.1. Results of Powder analysis

The analysis of powder samples was focused on the two main parameters that allow their characterization: particle size distribution and material composition. For the first one, scanning electron microscopy (SEM) was employed. Examples have been depicted in Figure 3a and 3b for the initial (n = 1) and last iterations (n = 14), where it can be easily observed the appearance of particle aggregates in the second one due to the sintering of powder beads during manufacturing.

This is confirmed in Figure 3c, which shows the particle size distribution obtained for three different iterations (n = 1, 7, and 14). As one can see, the distribution remains almost identical from iterations 1 to 7, while there is a small deviation from iterations 7 to 14. This result ratifies that a continuous use of the same IN718 powder, iteration after iteration, gives rise to undesired particle aggregations which may affect the quality of the fabricated part. However, the modification of this distribution is not very significant thanks to the methodology employed; the variation with respect to the nominal values is less than 10%, and consequently test parts fabricated with aged powder are not expected to show relevant quality differences with respect to those fabricated with the initial unused powder. In order to prove this assumption, metallurgical and mechanical measurements were additionally performed and properly analyzed in the following subsections.

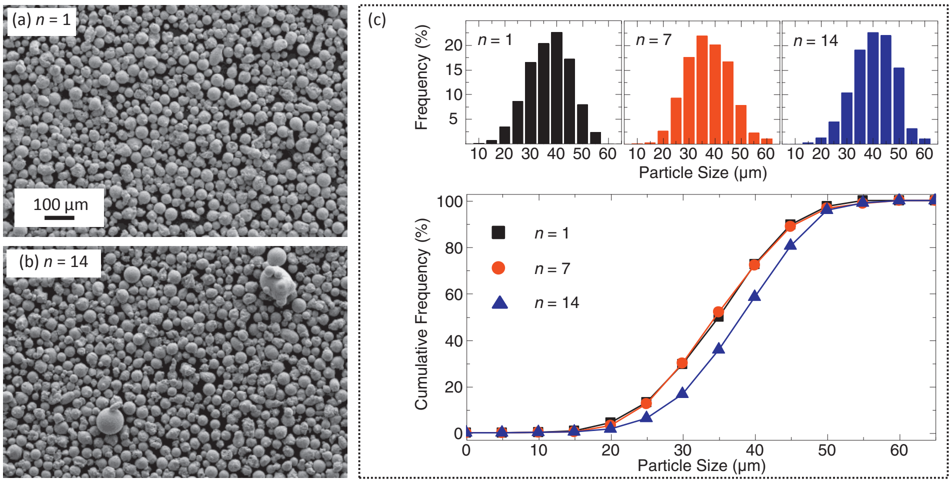
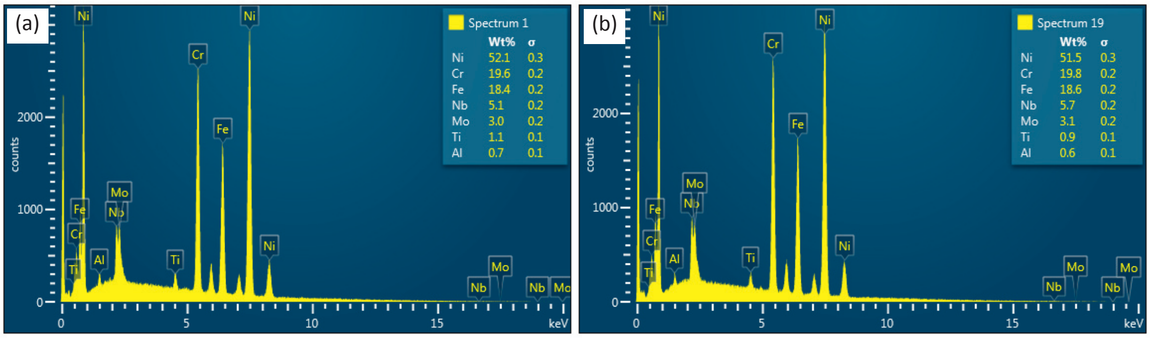


Fig. 3. SEM images of powder (a) before and (b) after 14 iterations sieving and drying. Comparison of particle size distribution is shown in (c) in terms of relative (top) and cumulative (bottom) frequencies.

The study of the material composition dependence on IN718 powder reutilization was carried out by obtaining energy-dispersive X-ray spectroscopy (EDS) measurements. Figures 4a and 4b respectively show the spectral powder composition for the first and last iterations. As it can be observed, such composition remains virtually the same with the increased number of iterations. The variation of composition relies within the standard deviation values for all elements, except for Ni and Nb where this variation is larger than expected. The concentration of Ni is smaller at the final iteration while much bigger for Nb (and moderately for other elements like Cr and Fe). This result may be originated by a slight oxidation of Ni as a function of subsequent fabrication processes, but overall IN718 powder is not oxidized, and therefore one may assume that test part properties will remain virtually identical and irrespective of the fabrication process iteration. The observed behavior for Nb will be discussed in next sub-section due to the close correlation with the inner microstructure.



Spectrum 1

	Wt%	σ
Ni	52.1	0.3
Cr	19.6	0.2
Fe	18.4	0.2
Nb	5.1	0.2
Mo	3.0	0.2
Ti	1.1	0.1
Al	0.7	0.1

Spectrum 19

	Wt%	σ
Ni	51.5	0.3
Cr	19.8	0.2
Fe	18.6	0.2
Nb	5.7	0.2
Mo	3.1	0.2
Ti	0.9	0.1
Al	0.6	0.1

Fig. 4. EDS spectra of IN 718 powder composition for the (a) first and (b) last iterations.

3.2. Characterization of metallurgical test specimens

The analysis of IN718 powder properties has shown that its reutilization, iteration after iteration through a recycling methodology of sieving and drying, does not significantly alter its properties. This should find a clear correspondence in fabricated parts, i.e homogeneity of part properties should be observed. To prove this assumption, a complete metallurgical characterization of test parts was performed by means of porosity and microstructural measurements. Porosity was measured by analyzing material density in the inner region of the fabricated parts with an optical microscope. The inner part of the metallurgical test samples (cubes from Fig. 2) was reached by cutting them in half with a high precision saw from Bhueler (model Isomet 4000), and then polished with a Mecatech 334 polisher form Presi. Afterwards, porosity is obtained by comparing 10 representative photos from each sample. These photos are analysed with specific software AnalySYS included in the microscope which allows distinguishing the pores from the base material by means of correcting contrast and brightness. The programme gives the pore percentage present in each micrograph. The average of the ten micrographs is the value of the sample's porosity.

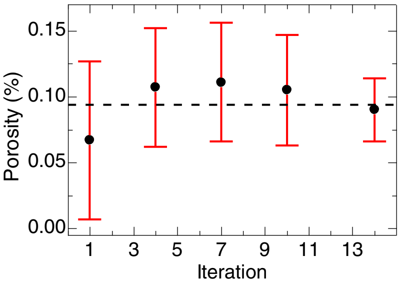


Fig. 5. Results from porosity measurements performed over manufactured samples. Each point represents the average porosity (in percentage) found in six samples fabricated in five particular iterations. Red bars indicate standard deviation from this value. The dashed line represents an average porosity obtained from these five values.

Due to the large amount of test parts fabricated in this study, only those corresponding to five particular iterations were analyzed (*n* = 1, 4, 7, 10, and 14). In each one of these iterations, three metallurgical test samples were fabricated, as mentioned before. After doing porosity measurements over them, average values and standard deviations were calculated. The results have been presented in Figure 5. The dashed line represents the overall average porosity value. As it can be observed, porosity is very similar in all iterations, close to a 0.10%, while standard deviations show more variations but not in a relevant way. These results points out to the same conclusions obtained in the previous sub-section, that the recycling process allows the fabrication of parts with reused powder with similar properties.

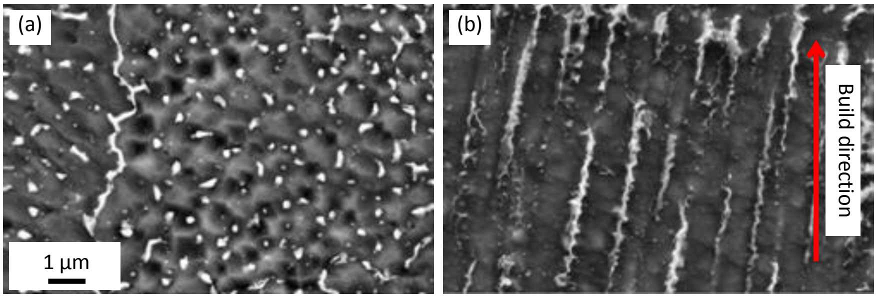
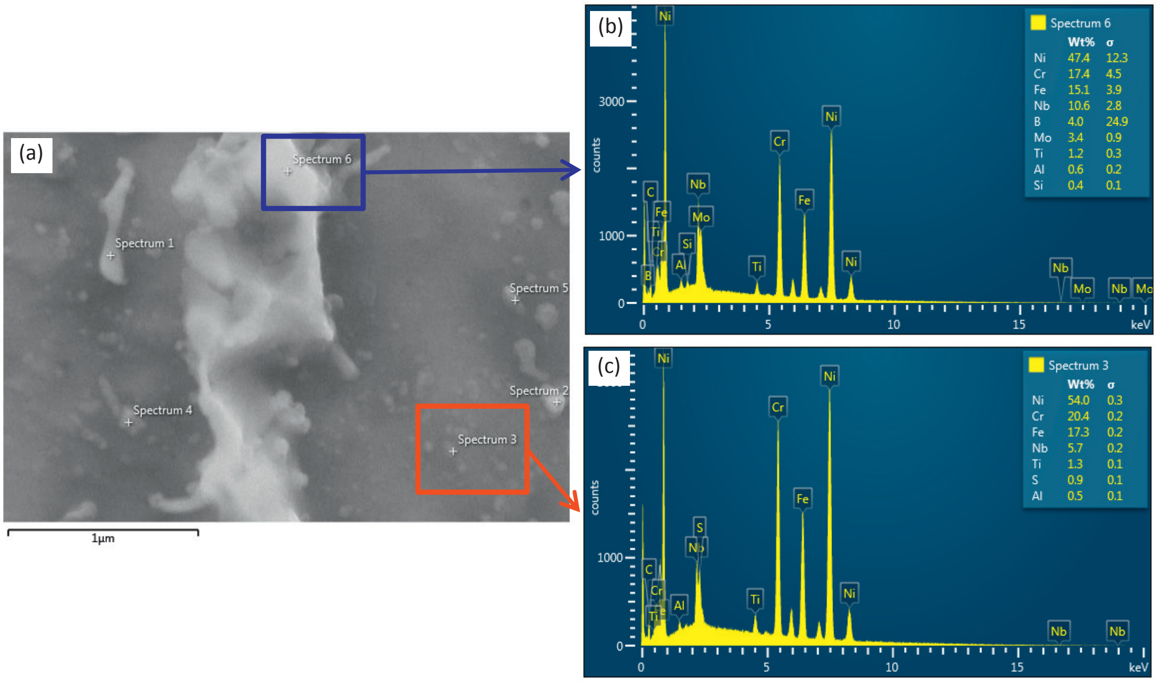


Fig. 6. SEM micrographs of IN718 SLM manufactured samples on cross section (a) and longitudinal section (b).

Microstructure of IN718 test samples was revealed using Kalling´s Reagent. Figures 6a and 6b show examples of the microstructure found in the cross and longitudinal respectively. A fine dendritic microstructure is formed due to very high cooling rate induced by high laser energy proper of this kind of technology. The cross section micrograph (Fig 6a) shows that the dendritic structure is not homogeneous due to the interaction of melted tracks under stripped pattern manufacturing strategy (X/Y cross-hatching technique). In contrast, in Fig 6b, regular solidification microstructure along longitudinal direction is clearly formed, which is composed of parallel dendrites whose growing parallel to Z-direction. In SLM process, cooling solidification of the liquid metal is rapidly produced due to high dissipation of heat through base plate; which creates high temperature gradients and solidification rate in the Z-direction, leading to formation of columnar dendrite grains in Z direction.

At higher magnifications (see Fig. 7a) different gamma and Laves phases can be found (an embrittling phase, such as [Ti, Nb]C type carbides). It can be distinguish white areas, rich in Nb and Mo, and poor in Fe, Cr and Ni. It shows that cells of the Laves phase (intermetallic) are not clearly defined Janaki et al. (2005), but on the contrary, it can be seen a continuity of areas rich in Nb, Mo, Ti and C, being possible to identify geometries with "Honeycomb" shape Cieslak et al. (1989) & DuPont et al. (1998). The extreme cooling rates in laser melting, compared with other “welding” processes, cause segregation of Nb occurring to a lesser extent due to insufficient time for the redistribution of solute Radhakrishna et al. (1997), and this translates into fewer amount of Laves phase and lower concentration of Nb therein. In Figures 7b and 7c can be appreciated the difference in composition between the matrix γ and Laves phase.

In total accordance with the results found in the previous sub-section, the observed microstructure is repeated in all tests samples, irrespectively of the iteration in which they were fabricated. Basically no differences are observed in terms of precipitates and phases, which is consistent with the reutilization of powder presenting nearly identical properties in terms of size and composition.



Spectrum 6	Wt%	σ
Ni	47.4	12.3
Cr	17.4	4.5
Fe	15.1	3.9
Nb	10.6	2.8
B	4.0	24.9
Mo	3.4	0.9
Ti	1.2	0.3
Al	0.6	0.2
Si	0.4	0.1

Spectrum 3	Wt%	σ
Ni	54.0	0.3
Cr	20.4	0.2
Fe	17.3	0.2
Nb	5.7	0.2
Ti	1.3	0.1
S	0.9	0.1
Al	0.5	0.1

Fig. 7. Matrix γ and phase laves (a) and corresponding EDS analysis (b and c).

3.3. Mechanical analysis of test specimens

Despite metallurgical characterization has demonstrated an equivalent behavior in all test samples, mechanical properties of tests specimens need to be performed to prove that performance is indeed conserved as a function of powder reutilization. Consequently, analysis of mechanical properties was accomplished by means of Charpy impact tests. This type of test was selected in order to analyze the sensitivity of the SLM process to powder degradation. In this case, compared to other tests, like tensile test, any little difference in mechanical properties results in a big change in the measured values Schierra et al. (1991). Charpy test scans the material around the notch and the “*weakest link*” will be found, thus reflecting weakness of the microstructure, with resulting energy of the test. To do so, standard size and V-shaped notch specimens were machined and tested according to ISO 148-1 standard. Prior to testing, all test samples were heat treated following AMS 5662 standard for IN718 (Solution treatment and double aging).

It is worth to note here that the building direction may play an important role in these tests, since mechanical properties could depend on part axis direction. Therefore, the notch should be place in different surfaces of the test sample to perform orientation dependent measurements. Nonetheless, previous results for other materials indicate that this is not a critical parameter as the effect of the building axis seems negligible for similar materials Kruth et al. (2010). This allowed us to manufacture all the tests samples with the same orientation, i.e. larger surface lying in the xy-plane (see Fig. 2), thus reducing the overall manufacturing time (as less layers are needed for fabrication). Consequently, all samples were machined with the notch at the top (see Fig. 8 right).

The results have been illustrated in Fig. 8. Similar to Fig. 5, each point represents the average energy needed to fracture 6 samples fabricated in 5 particular iterations, while red bars indicate standard deviation from this value. The dashed line represents an average energy obtained from these 5 values. As it can be observed, the toughness of the test samples presents variations for the different iterations, but no pattern or trend can be appreciated. The values oscillate around 10 J (values not far to those observed in other high temperature resistance materials Kruth et al. (2010)) apparently randomly, and no correlation is either found with respect to the porosity dependence that is shown in Fig 5. Actually, these variations are within the range of energies delimited by almost all the standard deviations observed so this is an expected behavior.

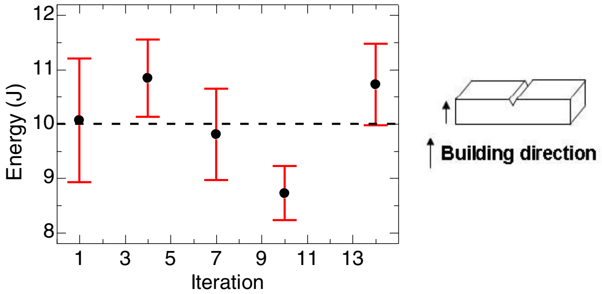


Fig. 8. Results from Charpy impact tests performed over manufactured samples. Each point represents the average energy needed to fracture 6 samples fabricated in 5 particular iterations, while red bars indicate standard deviation from this value. The dashed line represents an average energy obtained from these 5 values.

4. Conclusions

To summarize, at the end of the whole manufacturing process 10 kg of the initial 25 kg of IN718 powder were employed in the fabrication of the test samples, while only 0.6 kg of over-sized powder were collected during sieving iterations. In view of these data, material use efficiency larger than 95% is determined which, together with the stable and sound properties obtained in all test samples irrespective of the iteration number, confirms that the proposed recycling methodology is valid for this specific material. This is in line with the high resource efficiency philosophy that nowadays promotes industry. In particular for SLM technology, powder recycling is essential to ensure its viability both from an economic and environmental point of view.

In detail, the methodology hereby presented has allowed to conclude that the powder condition of IN718 does not significantly change during its reutilization. The majority of particles remained spherical and there was no increase in defects such as craters and satellites. Particle size distribution after several production cycles was similar, with the exception of a small amount of particle aggregates that were detected with sizes between 50 and 100 microns. Moreover, material composition remained also unchanged. This is particularly remarkable given that the amount of oxygen that filled the fabrication chamber (2000 ppm, much larger than those values found in other SLM devices) may have greatly modified IN718 powder properties without following this specific methodology. As a consequence, test samples showed similar properties after 14 iterations, both metallurgically (in terms of equivalent microstructure and porosity) and mechanically (in terms of similar toughness).

Nonetheless, further investigation is still necessary to properly validate powder recycling methodology for industrial applications, e.g. performing mechanical testing closer to the performance of the material in service (fatigue and creep tests at high temperature) and performing tests in industrially relevant environments.

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