TECHNICAL UNIVERSITY OF CLUJ-NAPOCA

ACTA TECHNICA NAPOCENSIS

Series: Applied Mathematics, Mechanics, and Engineering
Vol. 67, Issue Special I, February, 2024

HIGHLY SUSTAINABLE ADDITIVE MANUFACTURING INCONEL 718 POWDER FROM RECYCLED SOURCES

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Abstract: *Metal Additive Manufacturing aims to revolutionize the manufacturing industry due to the unmatched freedom of design and the printing of just in time components. Furthermore, AM appears to be a more environmentally friendly solution despite the high impact of the feedstock material. In this work, we report about a novel approach to produce high-quality metal feedstock from secondary sources. Inconel 718 powder has been produced starting from leftovers, failed prints, and disqualified AM feedstock. The input materials have been pre-processed and gas atomized to be converted into powder. The powder has been characterized to evaluate relevant properties, to be compared to industrial benchmarks. Finally, the powder has been used to manufacture components by Laser Direct Energy Deposition.*
Key words: *Metal Powder, Additive Manufacturing, Metal Recycling, Circular Economy, Gas Atomization.*

1. INTRODUCTION

Additive Manufacturing (AM) technologies for the production of metal components have developed rapidly over the last decade, disrupting the traditional manufacturing value chain with unmatched freedom of design and just in time printing of components [1]. Furthermore, AM technologies stand as promising alternatives from a sustainability point of view: due to the lower material consumption and just-in-time production, they bring the added benefit of reducing CO_2-equivalent emissions by more than six times per kg of printed metal [2]. However, the overall carbon footprint of metal AM is still weighted down by the significant contribution from the feedstock material. The feedstock (i.e., metal powder) still has high environmental impact, causing concerns about the sustainability of AM technologies [3]. Currently, the metal powders for AM are produced from a mix of recycled and virgin material, contributing to the depletion of natural resources for the sourcing of raw material. Additionally, powder production (i.e., powder atomization) is quite energy consuming, due to the high process temperatures and the use of inert gases, such as pure nitrogen or argon [4]. In order to mitigate this issue, several approaches have been investigated. Researches have studied the reuse of AM feedstock in powder-based processes, to maximize their efficiency in terms of material utilization [5]. Nevertheless, the un-melted powder has proven to degrade throughout the printing cycles, limiting its reusability. Therefore, alternative solutions have to be investigated.

In this work, a novel approach to produce high-quality Inconel 718 feedstock from recycled material is presented. Among others, Inconel 718 has been selected as proper candidate due to its high cost – from an economic as well as environmental perspective – and market relevance in the metal AM field [6]. Significant savings are achieved using secondary metal sources for powder production, particularly in terms of carbon footprint and consumption of both energy and raw materials. The paper will focus on the atomization of sustainable powder from leftovers, failed prints and disqualified AM feedstock, dealing with the steps of material preparation, atomization and characterization of atomized powders. Finally, the effectiveness of the recycling process will be validated by printing with the sustainable powder. The properties of components manufactured by Laser Beam Directed Energy Deposition (LB-DED) will be collected and compared with literature data, to evaluate the quality of this new product.

2. EXPERIMENTAL

2.1 Powder Atomization

Bulky parts of Inconel 718 were cut and intensively cleaned to remove residual of lubricants and impurities prior to atomization. Parts included leftovers from conventional manufacturing (i.e., CNC machining), obsolete spare parts and failed prints by Laser Beam Powder Bed Fusion (LB-PBF).

To enlarge the scope of the tests, disqualified Inconel 718 powder was added to the mix. The powder, collected after several cycles of printing and reuse in LB-PBF systems, was mixed and atomized in the as-received condition.

Eventually, the sustainable powder was obtained by Vacuum induction-melting Inert Gas Atomization (VIGA) technology, using high-pressure high-temperature argon gas jets in a close coupled nozzle configuration to atomize the liquid stream of Inconel 718 feedstock. A Consarc VIM-IGA furnace setup with loading capacity of 250 kg was used for the process. Additional information about the atomization process is sought to be protected by a patent application, thus not reported here.

After atomization, the powder was sieved by a Assonic CSM 300 sieve equipment to separate the different particle size fractions.

2.2 Additive Manufacturing

The sustainable Inconel 718 powder was printed by means of LB-DED. The printing was carried out with a LASERTEC 65 DED hybrid by DMG MORI.

During the process, the following process parameters were used: laser power from 1400 to 1800 W, laser diameter 3.0 mm, layer thickness 0.9 mm, track width 2.4 mm, hatch distance (i.e., stepover) 1.4 mm, powder feed rate 12 g/min and inert gas (i.e., shield gas) feed rate 4 L/min.

Printed parts were thermally treated with a FORNITALIA FM76 muffle furnace in air atmosphere, according to the following thermal cycle:

- solution annealing at 980 °C for 1 hour, followed by water quenching;
- precipitation hardening at 720 °C for 8 hours;
- after cooling at 620 °C, keeping the parts at 620 °C for 8 hours, followed by cooling in air up to room temperature.

2.3 Characterization

Chemical composition of the atomized powders was measured by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), Combustion and Fusion methods.

Particle Size Distribution (PSD) was evaluated by laser diffraction particle size analysis, in accordance to ISO 13320 standard.

Physical properties such as Hall flowability, Apparent and Tap Density, Hausner Ratio (HR) and Carr Index (CI) were measured in compliance with ASTM B213 (for flowability) and ASTM B527 standards.

Particle morphology was evaluated by Scanning Electron Microscopy (SEM) with a Tescan MIRA3 electron microscope.

The microstructural properties of printed parts were evaluated by Optical Microscopy (OP) with a Zeiss AXIO light optical microscope, in compliance with ASTM E3 and ASTM E407.

Concerning the mechanical properties of the parts, the hardness was measured with a KB PRUFTECHNIK KB250 testing machine in accordance with UNI ISO 6507, while the tensile strength was investigated with a Zwick Roell Z250 in compliance with ASTM E8. Lastly, the material toughness was measured through impact test with a Charpy pendulum Zwick Roell RKP450 in accordance with ASTM E23.

3. RESULTS

3.1 Atomization and characterization of atomized powders

A total of 257.2 kg of Inconel 718 were used to feed the atomizer. Overall, the input charge was constituted by 87.5% of bulky scrap – leftovers from CNC machining as well as failed printed parts – and 12.5% of disqualified powder collected after AM processes. The material was divided into two batches and loaded sequentially into the ceramic crucible of induction furnace. The loaded quantities for first and second heat were 125.5 and 131.7 kg respectively, comprehensive of both bulky scrap and powder. Main results from the atomization heats are summarized in Table 1:

Table 1

Atomization Yields for recycled Inconel 718 material.

	Feedstock Metal [kg]	Atomization Yield [%]	Output Powder [kg]
1st	125.5	84.4	105.9
2nd	131.7	75.2	99.0

The Atomization Yield - defined as the ratio of atomized powder and feedstock metal - was around 84.4% for the first batch and 75.2% for the second batch. The difference could depend on the mix used to feed the atomizer in the two cases. Further tests are needed to assess this dependency.

In order to understand the size distribution, the PSD was measured for each batch individually. Then, the powders were blended and sieved to get the target size ranges. Table 2 summarizes the PSD obtained by VIGA process:

Table 2

PSD of as atomized powders of Inconel 718.

Batch	D (10) [μm]	D (50) [μm]	D (90) [μm]
1st	25.5	95.5	310
2nd	22.8	70.7	171

PSD of as-atomized powder batches ranged from fine particles to coarse particles, with a substantial fraction of over-sized powder exceeding D>150μm.

It could be noticed how the D(10), D(50) and D(90) were shifted towards lower values during the second batch, as a consequence of the preliminary optimization of VIGA process parameters.

After blending, the powder was sieved to be separated and collected in the preferred fractions, according to the particle size. The total mass of sieved powder was found to be equal to 188.1 kg. This amount was lower than the sum of as-atomized powder quantities from the two heats: part of the particles was trapped in the internals of sieving machine and had to be discarded. At the same time, some over-sized particles were detected during sieving and removed. Table 3 reports the Sieving Yield(s) for the three main AM fractions:

Table 3

Yields of relevant AM fractions for recycled Inconel 718 material.

D<20μm Yield [%]	20μm<D<53μm Yield [%]	53μm<D<150μm Yield [%]
13.8	30.3	39.8

Yields reported in Table 3 consider specifically the particles with diameter D<20μm – to be used mainly for Binder Jetting (BJT) technology, powder with diameter 20μm<D<53μm for LB-PBF applications, and powder with diameter 53μm<D<150μm for LB-DED. Additionally, oversized powders (i.e., D>150μm) were retrieved to serve as input material for future atomization campaigns, to provide additional savings in the production process of Inconel 718 powders. As already partially demonstrated, it would be possible to carry out a deeper optimization of VIGA process parameters for modulation of Atomization Yield and Sieving Yields. This optimization could guarantee better results of the process, targeting for example the increase of the Atomization Yield over 85% - 90% and/or to maximize the LB-PBF Yield over the BJT and the LB-DED ones, if required. Nevertheless, the atomization process proved to be successful already at this stage.

The morphology of atomized fraction was evaluated by means of SEM microscopy. Images of the three above-mentioned powder fractions are reported in Figure 1:

The powder was highly spherical, with a low number of elongated and non-spherical shaped particles.

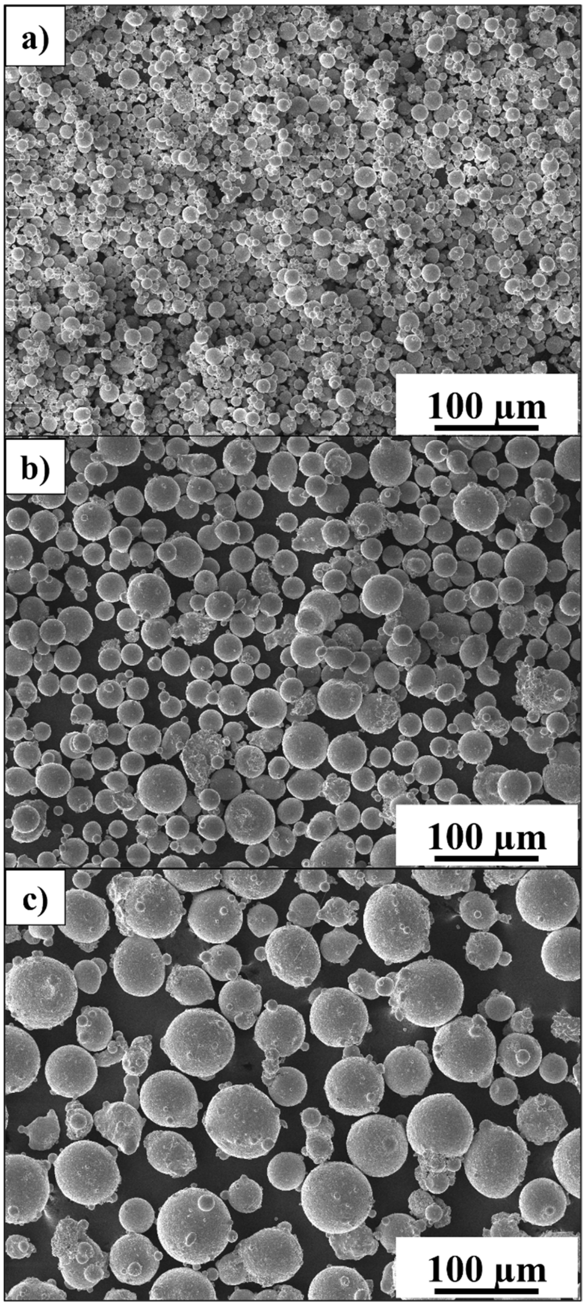

Fig. 1. Morphology of atomized particles with diameters (a) D<20 µm, (b) 20µm<D<53 µm and (c) 53µm<D<150 µm.

The presence of a few satellites on atomized particles was detected for all the fractions, despite the size and abundance of the satellites remain negligible. It is worth mentioning that a good sphericity of the powder and a low number of satellites are mandatory to ensure that the flowability properties of atomized product match the requirements of printing machines. Here, the recycled Inconel 718 powder was characterized by a good quality, comparable with commercial AM feedstock.

Chemical composition of the atomized powders was measured by ICP-OES for metallic elements, whereas carbon content was measured by Combustion method. Finally, oxygen and nitrogen concentrations were measured by means of Fusion method. The results are reported in Table 4:

Table 4

Chemical composition of as atomized powders of Inconel 718.

Element	% wt.	Element	% wt.
Ni	54.5	**Co**	0.05
Cr	18.6	**C**	0.03
Fe	17.1	**Mn**	0.09
Nb + Ta	4.95	**Si**	< 0.11
Mo	3.03	**P**	0.007
Ti	0.93	**S**	< 0.003
Al	0.45	**O**	< 0.01
Cu	0.05	**N**	0.007

The composition of atomized powder was found to be compliant to ASTM F3055 (i.e., nickel alloy UNS N07718 for LB-PBF), demonstrating the validity of the process in obtaining high quality products from secondary sources.

Lastly, density and flowability properties were quantified for the LB-DED fraction, due to its interest in the present work. Measured quantities for the LB-DED fraction are reported in Table 5:

Table 5

Physical properties of LB-DED powders of Inconel 718.

Apparent Density [g/cm^3]	Tap Density [g/cm3]	Hall Flowability [s/50g]	Hausner Ratio (HR)	Carr Index (CI)
4.5	5.1	15.6	1.2	13

The values observed for apparent density and flowability of the Inconel 718 powder were comparable to the values previously reported for a similar product obtained by VIGA atomization technology [7]. To offer a complete picture on the powder properties, also the values of Tap density, Hausner Ratio and Carr Index are reported as meaningful parameters.

Based on all data here reported, the recycled Inconel 718 powder could be used safely in printing machines without risk of agglomeration or clogging damage, ensuring the correct spreadability required for the printing application. These properties are particularly important for reliable manufacturing of parts, particularly in the case of bigger and/or more complex AM components.

3.2 Printing and characterization of printed parts

After the extensive characterization, the powder was furtherly evaluated by using it to manufacture parts by means of LB-DED. As previously reported, researchers have pointed out how the deployment of hybrid DED technology could maximize the benefit of LB-DED in terms of higher productivity with respect to LB-PBF and conventional LB-DED, while making up on the shortcomings of DED in terms of geometric accuracy and surface quality [8]. Moreover, considering powder-based AM technologies, LB-DED could be employed for a wide range of operations including manufacturing of new components, repurposing of obsolete parts and repairing of damaged ones. For these reasons, LB-DED was chosen for the investigation, despite its specific differences compared to LB-PBF in terms of achievable microstructure and resulting in slightly lower density and mechanical performance [9, 10].

All printed samples were subjected to thermal treatment, as explained in the previous section. Firstly, the density of specimens was determined by an estimation of the content of porosities via a micrographic examination. This technique, although being semi-quantitative and less precise compared to other measurement methods, gives valuable information about the presence of pores and – if any – how they are distributed. A transverse section of the specimen was mounted in resin and polished to a mirror finish. Then, five 1.5 mm by 2.0 mm fields of the section were analyzed at 50X magnification with high image brightness. This way, it was possible to maximize the contrast between the metal matrix (lighter areas) and porosities (darker areas). Finally, a software for image analysis was used to estimate the percentage of porosities associated with the printing process. The analysis revealed the presence of rare and isolated micrometric spherical pores, caused by entrapment of gases during the printing process. The software gave an estimation of the porosities volume in the order of 0.1% or less, thus assessing the full density of Inconel 718 printed parts. This feature is of high importance since the low content of pores and absence of printing defects would allow to achieve high mechanical performances under both static and cyclic loading. Nevertheless, to have a more accurate estimation, the density of the samples was investigated with the Archimede's or immersion method, too. Specimens were immersed in a controlled environment and their density was evaluated by means of a hydrostatic balance, resulting in a value of 8.23 g/cm^3. This value was very close to literatures data, which stand in the 8.20 – 8.25 g/cm^3 range [11]. It is worth noting that the good results on printability and density here achieved have benefited from the weldability of Inconel 718, whereas the use of different Ni-based alloys would have required further steps of optimization of LB-DED process parameters [12].

The microstructure of LB-DED parts was analyzed by means of optical microscopy on polished and etched cross-sections of printed specimens. An example of the internal structure of Inconel 718 samples is reported in Figure 2, showing the features of the core of printed parts along the axial direction (i.e., the growth direction during printing), after etching with Kalling's reagent. Melt pools were found to be distributed orthogonally to the growth direction, with absence of signs of incomplete fusion, confirming the full density of printed parts (Figure 2a). Additionally, images at higher magnifications revealed the presence of particles, attributable to carbides and carbonitrides, homogeneously dispersed in the austenitic phase matrix (Figure 2b and 2c).

Vickers hardness tests were conducted with diamond pyramid indenters with a load force of 98.7 N, on both the axial and transverse planes of the specimen (orientations parallel to the growth direction and to the build plate direction, respectively). The values of hardness were 448

HV for the first case and 433 HV for the second case, which demonstrated a good isotropy in the printed parts.

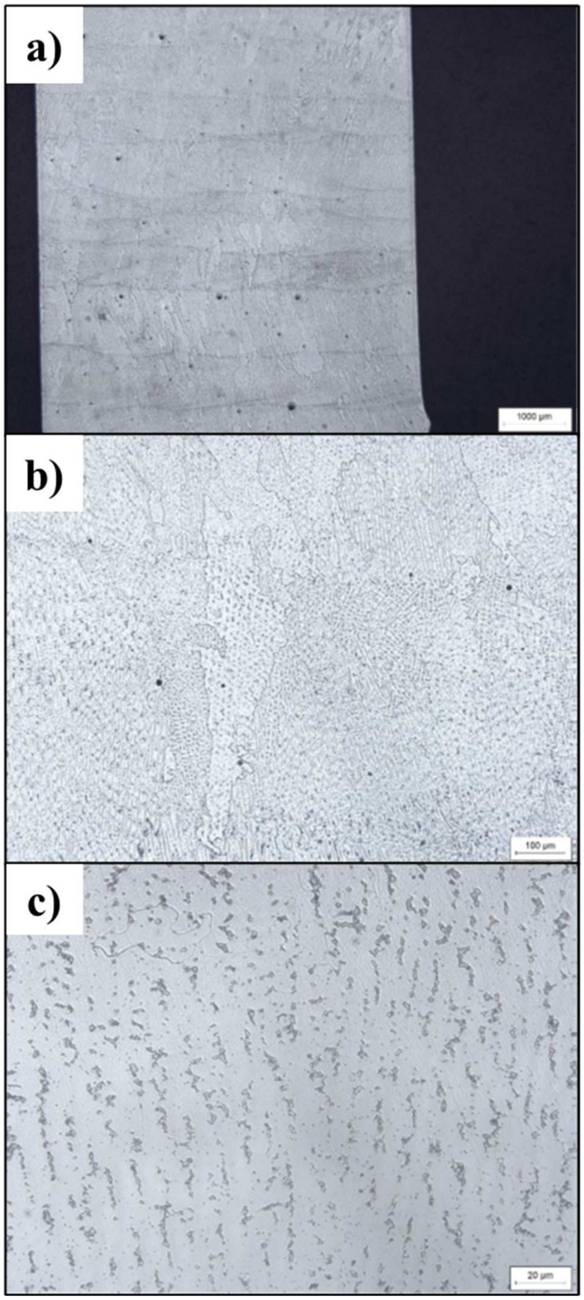

Fig. 2. Optical images of etched cross section of LB-DED printed parts with (a) 12.5X, (b) 100X and (c) 500X magnification.

Tensile specimens were obtained by machining (i.e., turning) cylindrical samples cut out of a single LB-DED printed block by means of wire cutting. The results of the tests conducted in accordance with ASTM E8 standards are reported in Table 6. A comparison of the properties obtained with other conventional manufacturing and LB-DED technologies is included.

Table 6

Mechanical properties of LB-DED powders of Inconel 718.

Technology	UTS [MPa]	$Y_{0.2}$ [MPa]	E [%]
Cold Rolled [13]	1407	1172	12
LB-DED [14]	1245	992	14.8
LB-DED [This work]	1198 ± 45	1015 ± 20	11 ± 6

Three specimens were tested for assessing the tensile behavior, each of them characterized by a test section with diameter of 4 mm and gauge length of 16 mm. Another set of three specimens with dimensions 10x10x55 mm^3 with V-shaped notch were used to evaluate the toughness of printed material. The values of Ultimate Tensile Stress (UTS), Yield Stress evaluated at 0.2% offset ($YS_{0.2}$) and Elongation (E) at fracture of the LB-DED specimens were similar to those previously reported for DED printed and heat-treated parts, while displaying a slight decrease in both UTS and YS compared to conventional manufacturing. In addition, the reduction of area (Z) was 13 ±5 % and absorbed energy (KV) was 14 ±3 J. The small differences observed are most probably related to a different microstructure obtained for the specimens here analyzed, because of the early-stage optimization phase of both printing and heat treatment parameters. Still, the results presented demonstrate that the printed material possessed good mechanical properties and ductility similar to Inconel 718 parts produced by conventional technologies. As a consequence, it was confirmed that the quality of atomized sustainable powder matched both the requirements for printability and production of high-performance LB-DED parts.

4. CONCLUSIONS

A novel approach for production of sustainable powders of Inconel 718 was presented, based on the use of secondary sources as feedstock material for metal powder atomization. The benefits in terms of reduced environmental footprint and savings in terms of energy and resources consumption makes the described methodology appealing to enhance and improve the substantial advantages introduced by AM compared to conventional manufacturing techniques.

The outcomes of the atomization process, together with analysis of powder properties, confirmed the validity of the approach for production of high-quality metal powders of Inconel 718 for AM.

Furthermore, results of the characterization of specimens printed by means of LB-DED technology demonstrated that the sustainable powders can be qualified for production of high-performance AM parts.

Additional research work is being conducted to quantify energy savings and CO_2 equivalent emissions by means of Life Cycle Assessment analysis, to certify the advantages of the proposed methodology in comparison to conventional metal powder production.

Furthermore, extensive ongoing atomization campaigns on various other iron- and nickel-based alloys are deemed to prove the high versatility of our approach for a truly sustainable AM.

5. ACKNOWLEDGEMENTS

The authors are grateful to Laura Cordova and Eduard Hryha for their technical support. The authors wish to thank also Gianluca Acquistapace for their help in the experiments. The authors acknowledge financial aid by the European Community within the Vanguard Initiative through the 3DP Pilot I3 project '3DoP' (Proposal ID. 101083997).

The views and opinions expressed herein do not necessarily reflect those of the European Commission.

The authors are grateful to the reviewers for constructive remarks and suggestions.

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FABRICAȚIE DE ADITIV FOARTE DURABIL INCONEL 718 PUDRĂ DIN SURSE RECICLATE

Metal Additive Manufacturing își propune să revoluționeze industria de producție datorită libertății de neegalat de proiectare și imprimării componentelor just in time. În plus, AM pare a fi o soluție mai ecologică, în ciuda impactului ridicat al materiei prime. În această lucrare, raportăm despre o abordare nouă pentru a produce materii prime metalice de înaltă calitate din surse secundare. Pulberea Inconel 718 a fost produsă pornind de la resturi, printuri eșuate și materie primă AM descalificată. Materialele de intrare au fost preprocesate și atomizate cu gaz pentru a fi transformate în pulbere. Pulberea a fost caracterizată pentru a evalua proprietățile relevante, pentru a fi comparată cu reperele industriale. În cele din urmă, pulberea a fost folosită pentru fabricarea componentelor prin depunere directă a energiei cu laser.

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