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Production and Properties of Recycled Aluminium Alloy Powders via Inert Gas Atomisation

Federico Simone GOBBER[1]*, Antonio PENNACCHIO[1], Elisa FRACCHIA[2] and Marco ACTIS-GRANDE[1]

[1]Politecnico di Torino, Department of Applied Science and Technology (DISAT), Viale T. Michel 5, Alessandria, 15121, Italy.
[2]Politecnico di Torino, Department of Management and Production Engineering (DIGEP), Corso Duca degli Abruzzi 24, 10129 Torino, Italy

Abstract
Inert gas atomization is a suitable technique for producing highly spherical powders used in metal additive manufacturing and other powder metallurgy techniques. Research into environmental sustainability is leading to new considerations in metal powder quality, gas atomisation processes, and raw material selection. This work presents the properties and sustainability of some gas-atomised aluminium-based powders. This study compares the characteristics of final powders made from conventional or innovative aluminum-based alloys belonging to the 2.xxx and 4.xxx series, using secondary or scrap materials. The comparison is based on granulometry, morphology, microstructure, and chemical composition, including light elements such as O, N, H, C, and S.

Keywords: gas atomization, sustainability, aluminum alloys.

1. Introduction

Sustainability and environmental concerns are critical in industrial practices, making the assessment of the environmental impact of materials and manufacturing processes increasingly studied. Aluminium, renowned for its excellent strength-to-weight ratio, corrosion resistance, and versatility, is a key material across various industries, including transportation and construction. As the push for sustainable practices intensifies, the recycling of aluminium alloys has become more significant. Currently, 37% of Europe's aluminium production involves recycled alloys, and this percentage is expected to grow due to the substantial energy savings—up to 95%—achieved through re-melting compared to primary production [1-4].

In the automotive sector, aluminium alloys belonging to the 4.xxx series such as AlSi10Mg and AlSi9Cu3(Fe) are extensively used in high-pressure die casting (HPDC) because of their superior mechanical properties and castability. However, traditional manufacturing processes produce a considerable amount of scrap, leading to resource wastage and environmental issues. Despite efforts to minimize defective castings, some scrap is unavoidable and must be either recycled or repurposed.
It is important to differentiate between new scraps, which are generated during production, and old scraps, which are end-of-life components and often contaminated with iron. Scrap alloys from foundry which have a high iron content, tend to produce more slag. Studies indicate that re-melting these alloys can alter their chemical composition and mechanical properties, although this process can still yield good results with proper management [5-6].

Although research on AlSi9Cu3(Fe) for AM is limited, initial findings are promising. Comparisons with AlSi10Mg powders indicate potential for high performance in AM applications. Thorough characterization of key powder properties, such as density, particle size distribution, and rheology, is necessary to ensure quality and processability.
Repurposing production scraps for additive manufacturing (AM) presents a sustainable and economically feasible approach. These scrap materials can be converted into high-quality powders through gas atomization, suitable for Laser Powder Bed Fusion (L-PBF) processes, and can produce automotive components with excellent tensile properties [6-9].

Among the emerging literature regarding sustainability and metallurgy, a comprehensive point of view is presented in the review by Raabe [10]. The concepts of dirty alloys is addressed as the most effective way to reduce the massive CO_2 emission footprint of metallic materials by producing them through secondary synthesis, i.e. from scrap. In the aerospace, 2.xxx series aluminium alloys, like Al-Cu alloys, are extensively employed due to their superior mechanical properties and high fatigue resistance. However, traditional manufacturing processes generate substantial scrap, resulting in resource wastage and environmental challenges. Despite efforts to reduce defective parts, some scrap generation is inevitable due to the mechanical working required to produce the final parts and requires either recycling or repurposing.

Due to the low amount of scrap produced during part manufacturing, AM technologies are considered a sustainable and lean manufacturing processes, nevertheless the properties of the starting powders represent a crucial aspect for processability. Within the frame of the work, the results from the production and the characterization of gas atomized powders are presented comparing the properties of powders obtained from ingots or scrap feedstocks.

*corresponding author, E-mail: federico.gobber@polito.it

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2. Experimental

2.1 Materials

In this study, the atomisation of aluminium-silicon and aluminium-copper alloys was conducted in order to assess the quality of the aluminium powders produced through the gas atomisation process. The powders were produced using secondary foundry alloys of EN AB-46000 and AA 2024-modified ingots. The scraps represent a production waste that has been recovered from the industrial component manufacturing stream.

Powders were subjected to a series of characterisations with the objective of comparing the properties of powders derived from scrap feedstock to those of powders derived from ingot feedstock. Prior to the atomisation process, the average composition of both ingots and scrap alloys was determined through optical emission spectrometry (OES), and the results are presented in Tab. 1.

Tab. 1. Chemical compositions for the feedstock material as measured by OES.

Scrap alloy – EN AC 46000							
Element	Si	Fe	Cu	Mn	Mg	Zn	Al
Wt.%	10.10	1.1	3.21	0.27	0.33	1.15	Balance
Ingot alloy – EN AC 46000							
Element	Si	Fe	Cu	Mn	Mg	Zn	Al
Wt.%	9.27	1.07	2.91	0.23	0.25	1.49	Balance

Scrap alloy – AA 2024						
Element	Cu	Ti	Fe	Cr	Mg	Al
Wt.%	4.16	3	1	0.22	1.45	Balance
Ingot alloy – AA 2024						
Element	Cu	Ti	Fe	Cr		Al
Wt.%	5	3	1	1		Balance

2.2 Gas Atomization

Both scraps and ingots in the as-provided form were cut into smaller sections through a bandsaw and gas atomized using a Vacuum Induction Inert Gas Atomizer (VIGA)). Four kilograms of raw material were loaded into an alumina crucible of 1.25 l capacity and heated under vacuum (8.3 10-3 mbar); inert gas (argon) was backfilled in the furnace at 400°C to cover the melt and remove any outgassed species (Fig. 2). Before atomization started, the temperature was equalized at 850°C for 15 minutes, subsequently the molten alloy was atomized with Argon accelerated by a De Laval nozzle with an inlet pressure of 45-50 bar.

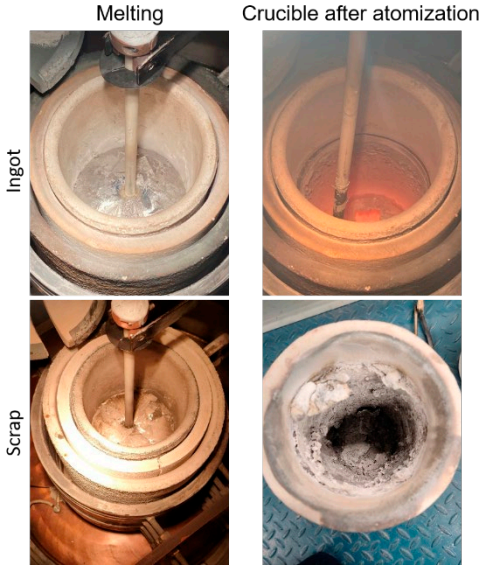


Fig. 1. Melt crucible state during vacuum induction melting and after atomization for the for ingot or scrap feedstock (modified AA 2024 alloy).

Following the completion of the atomization process, powder passivation was carried out before unloading the powders from the atomizer, then the produced powders were collected from two hoppers. The primary hopper contains almost 95% of the powder, while the secondary hopper collecting the remaining powder, which represents the finest granulometric size. A cyclone separates the

powder from the exhaust inert gas in the secondary hopper.

2.3 Powder characterization

The atomized alloys were sieved below 250μm to separate the atomization debris eventually formed, and the collected powder was sieved in the 20-63 μm range suitable for L-PBF; the characterizations proposed were performed in that powder size interval. The powder microstructure and morphology was observed by scanning electron microscopy from the top, after mounting a small sample of powders on a carbon tape disc.

2.3.1 Light elements concentration (O, N, H, C and S)

The quantities of oxygen, nitrogen and hydrogen were assessed in the atomized powders using the elemental analyzer (LECO ONH836). This analysis provided insights into the oxygen, hydrogen and nitrogen contents at various particle sizes, revealing possible variations between ingot and scrap powders.

Further analyses were performed to assess the quality of the powders in terms of carbon and sulfur content (LECO CS744).

2.3.2 Powder rheology

The rheological properties of the powders were characterized using a shear cell rheometer (Freeman Technology FT4). The rheological properties of the powders at constant shear rate and their stability were quantified by measuring the specific energy (SE), stability index (SI) and basic flow energy (BFE). The influence of shear rate on powder flowability was quantified by the Flow Rate Index (FRI). Shear cell rheology data were compared with conventional Hall funnel flowability (according to ASTM B213). Powder sampling for this test was based on alloy density and therefore comparisons are only consistent between powders of the same chemical composition (e.g. AA 2024 new vs. AA 2024 scrap). All powders were dried under vacuum at 80°C to reduce the moisture content.

2.3.4 Powder morphology

A quantitative assessment of powder morphology was performed by optical image analysis (Malvern Morphology). More than $2*10^4$ particles was analyzed per each batch produced and the average values of circularity, convexity and aspect ratio are presented.

3. Results and discussion

The quantification of light elements reported in Tab.2 shows differences between the two alloys. The 4.xxx series alloy (EN AC 46000) show a slightly higher oxygen content in the powders obtained from the ingot rather than from those obtained by the industrial scrap. As the other light elements regards no significant variations were observed; both nitrogen and hydrogen are comparable with around 0.1 %wt nitrogen and only 5 ppm of hydrogen in the final powders. The slightly higher amount of carbon detected in the scrap alloy is supposed to be related to the scrap cleanliness due to possible contamination by process fluids (lubricants, grease, oils…).

Considering the powders obtained from the modified AA 2024 alloy, a higher amount of oxygen was detected for the scrap powders. Interestingly, compared to the 4.xxx series alloy, a slight increase in the nitrogen content was observed and remarkable increase in hydrogen content was detected. Such differences can be related to the alloy composition, the modified 2024 alloy in fact has a relevant amount of Ti which is a strong former of both nitrides hydrides. Similarly to the 4.xxx series alloy powders, the carbon content is higher for scrap powders, strengthening the assumption this can be related to process fluid contaminations that are not included in the slag during vacuum melting. The amount of sulphur was not quantifiable for both alloys since under the detectable threshold for the instrument. Finally, the light elements content is considered a good indicator of powder quality from the point of view of chemical composition, contamination and handling and based on these observations, both ingot and scrap powders are considered equivalent, leading to the possible mutual use in powder metallurgical application.

Tab. 2. Light elements concentration measured for the as-atomized powders in the 20-63 μm interval.

Scrap alloy – EN AC 46000					
Element	O [%wt]	N [%wt]	H [ppm]	C [%wt]	S [%wt]
Wt.%	0.036 ± 0.010	0.114 ± 0.031	5.02	0.023 ± 0.006	n.d.

Ingot alloy – EN AC 46000					
Element	O [%wt]	N [%wt]	H [ppm]	C [%wt]	S [%wt]
Wt.%	0.049 ± 0.016	0.106 ± 0.021	5.14	0.013 ± 0.003	n.d.

Scrap powders – AA 2024 modified alloy					
Element	O [%wt]	N [%wt]	H [ppm]	C [%wt]	S [%wt]
Wt.%	0.068 ± 0.013	0.188 ± 0.025	53 ± 13	0.021 ± 0.005	n.d.

Ingot powders – AA 2024 modified alloy					
Element	O [%wt]	N [%wt]	H [ppm]	C [%wt]	S [%wt]
Wt.%	0.048 ± 0.013	0.123 ± 0.035	17 ± 3	0.011 ± 0.001	n.d.

The morphology of the powders from the modified AA-2024 alloy is presented in Fig. 2 both as scrap and ingot feedstock regards; the SEM micrographs refer to powders sieved in the 20-63 µm interval. The backscattered imaging at low magnification (Fig.2 a, c) confirm the powders were not contaminated. Despite the scrap feedstock could carry some external contaminants, the vacuum melting process helps in cleaning the alloy. Part of the contaminants are included in the slag (see Fig. 1) and the alloy composition is therefore refined from impurities. The powder morphology is highly spherical (Fig. 2 b, d) making the powders suitable for additive manufacturing processes where powder flowability is a matter of concern.

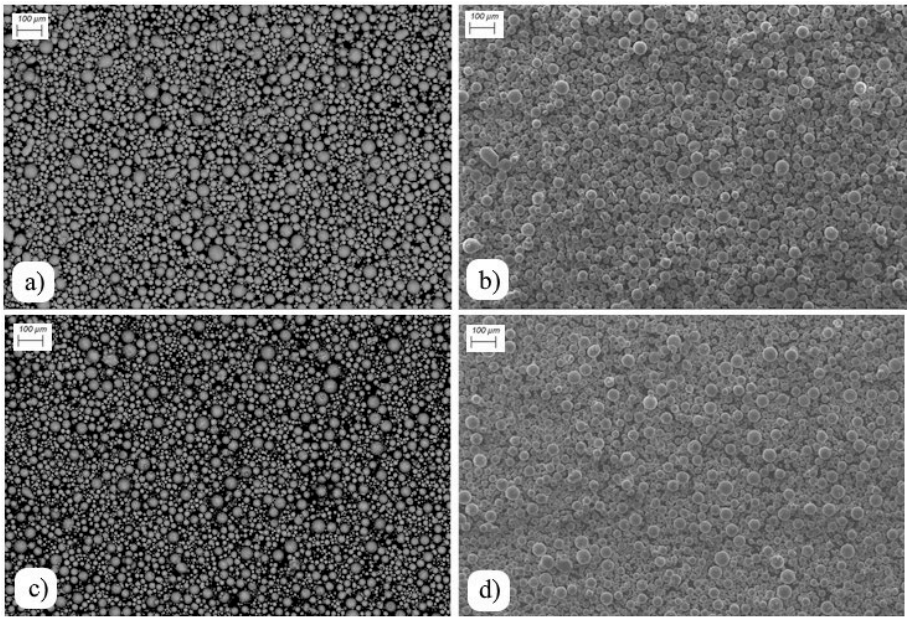


Fig. 2. SEM micrograph of the atomized powder in the 20-63 µm interval for the AA-2024 modified alloy: a) and b) from ingot feedstock (backscattered and secondary electrons respectively), c) and d) from scrap feedstock (backscattered and secondary electrons respectively).

The morphological characteristics observed qualitatively by SEM are confirmed by quantitative image analysis, according to the data presented in Tab. 3, averaged over the range 20-63 µm. With regard to circularity, the differences between the samples are subtle and no systematic pattern is clearly evident from the data, similar to the aspect ratio. Interestingly, all samples show a very high convexity: this parameter is sensitive to surface roughness and can be used as an indicator of the presence of satellites. From the data presented it can be concluded that good morphological quality is maintained even by powders obtained from alloy scrap.

Tab. 3. Morphological properties measured for the as-atomized powders in the 20-63 µm interval.

	Circularity	Convexity	Aspect ratio
Scrap alloy – EN AC 46000	0.94 ± 0.02	0.99 ± 0.01	0.95 ± 0.10
Ingot alloy – EN AC 46000	0.98 ± 0.02	0.99 ± 0.01	0.94 ± 0.01
Scrap alloy – AA 2024	0.97 ± 0.01	0.99 ± 0.01	0.94 ± 0.08
Ingot alloy – AA 2024	0.95 ± 0.03	0.99 ± 0.01	0.95 ± 0.10

Based on the results presented in Table 4, the rheological properties of the atomised powders differ between the two alloys. The EN AC 46000 powders show comparable rheological properties whether ingot or scrap alloy is considered. Despite using different units of measurement and measuring under different conditions, both the rheometer and the Hall funnel measurements give concordant results. Looking in more detail at the shear cell based measurements, the two powders are not affected by the initial raw material condition, as their BFE is comparable; moreover, they appear to be stable from the first to the last test, as shown by the SI. An SI ≈ 1

indicates a BFE that is not affected by the movement of the blade through the powders, which means that the PSD remains constant during the tests. As the blade moves through the sample, it may cause the detachment of satellites or agglomerates, which in turn would modify and alter the initial particle size distribution (PSD), affecting the rheological behaviour. Cohesive forces between powders are non-negligible since the FRI > 1.

Interestingly, the AA 2024 powders have different rheological properties depending on the initial state of the raw material. The ingot powders show improved flowability compared to the scrap powders, with both the BFE and Hall flowability tests showing a similar trend, and despite having the same PSD, these two powders show different rheological behaviour. As the morphology of the powders does not show much difference between the two samples, the reason for such a difference must be related to other surface properties. Currently, one hypothesis is related to the differences in chemical composition, as Mg regards (Tab 1). It is currently believed that this can have an effect on the formation of thin surface oxides that can increase the processability window by laser[11] but may also affect the coefficient of friction between powder particles. Further investigations are currently undergoing to disclose the motivation for such difference.

Tab. 4. Rheological parameters measured for the as-atomized powders in the 20-63 μm interval.

	BFE [mJ]	SI	FRI	SE [mJ/g]	Hall flowability [s]
Scrap alloy – EN AC 46000	182.10	1.05	1.19	2.32	15.15 ± 0.32
Ingot alloy – EN AC 46000	179.34	0.98	1.19	2.34	15.41 ± 0.16
Scrap alloy – AA 2024	216.90	1.01	1.11	2.12	21.82 ± 0.28
Ingot alloy – AA 2024	155.65	0.99	1.16	1.90	16.01 ± 0.22

4. Conclusion

A preliminary comparison between powders derived from secondary alloy or scrap feedstock for two aluminium alloys belonging to the aluminium-silicon (4.xxx) and aluminium-copper (2.xxx) categories is presented within the context of this study.

The study demonstrates that aluminium powders derived from both ingots and industrial scrap exhibit comparable quality, with minor variations in light element content due to potential contamination from process fluids in scrap alloys.

The SEM analysis revealed that both types of powders are highly spherical, as confirmed by quantitative image analysis. The high convexity of powders can be considered a relevant indicator of an extremely low satellite content.

Different methods for measuring flowability showed comparable trends, for both the EN AC 46000 and AA 2024 powders. Significant differences were observed only for the AA 2024 powders, with reduced flowability for the scrap alloy powders compared to the ingot powders. Further investigations are underway to characterise the surface state from a chemical point of view to correlate with flow properties. The validation and subsequent adoption of powders obtained from scrap feedstock would promote sustainable practices in manufacturing towards a reduction in CO_2 emissions.

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