Perrotin, A., A. Olivo, J. O. Odden, and M. Di Sabatino. 2026. Characterization of gas-atomized aluminium-silicides for laser powder bed fusion applications. In *Silicon for the chemical and solar industry XVIII*, edited by M. Tangstad, H. Rong, A. Valderhaug, B. Andresen, H. Dalaker, and I. G. Page. Kristiansand, Norway, September 7–10, 2026. https://doi.org/10.55669/oa900822

Characterization of gas-atomized aluminium-silicides for laser powder bed fusion applications

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Keywords: Aluminium-silicides, powder characterization, additive manufacturing, atomized powders

ABSTRACT:

Additive manufacturing (AM) is a blooming field which could allow for a shift towards more sustainable manufacturing of products while maintaining industrial scalability. Laser powder bed fusion (L-PBF) is an AM technique that allows the production of components using powders as a feedstock. Aluminium-silicide powders produced by gas atomization (GA) emerge as prospects for AM applications thanks to their low number of defects, good flowability, and casting properties. Increasing the Si content of these powders would allow to build stronger parts and tailor the materials thermal expansion coefficient for specific uses (e.g. aerospace and automotive industry). In order to optimize the printing process, deep understanding of the feedstock is necessary and, therefore, extensive characterization is required. In this work, we establish a methodology to characterize these powders, which consists of several techniques. Three batches of aluminium silicide powders were characterized by glow discharge mass spectrometry (GD-MS), scanning electron microscopy (SEM) as well as laser diffraction particle size distribution (PSD) and image analysis using ImageJ. Altogether, these techniques generate informative data on the sample and establish a characterization routine for such alloy systems which should improve the understanding of the powders by trying to correlate the physicochemical properties and the morphology of the particles to the performance in powder production for L-PBF applications.

1. INTRODUCTION

In today's world the demand for manufactured parts remains strong for many fields such as the automotive and aerospace industry. The standards and the competition in these fields push for development of newer materials to achieve stronger, lighter and better performing parts whilst limiting the compromises.

Another important metric to consider is the environmental footprint of parts production. Traditionally, metallurgy and parts production have been notorious for their CO_2 emissions, although nowadays the processes are mostly optimized and are controlling the emissions not only for the casting part but the whole process chain.

Furthermore, additive manufacturing (AM) offers an alternative way to produce parts and components with higher production yield and low energy budget. Within AM, laser powder bed fusion (L-PBF) is a process which allows to fabricate pieces with high levels of detail and complex structures in one single body, which avoids post processing steps like welding and machining, good versatility for part design, as well as lower waste coming from the process and compared to traditional casting (DeBoer *et al.*, 2021).

Aluminium-silicides are alloys that could be used for these applications due to their good castability and thermal conductivity (*Aluminium AlSi10Mg Data sheet*, no date). The addition

of a higher fraction of silicon could help to tailor the stiffness and thermal properties of the alloy, silicon having high Young's modulus, high heat of fusion, and low coefficient of thermal expansion (CTE). Increasing the silicon content could, thus, be beneficial for aerospace applications where these properties are required. (Mueller *et al.*, 2019; Eberle *et al.*, 2019; Garrard *et al.*, 2022; Reutlinger *et al.*, 2023; Wilsnack *et al.*, 2023)

Typical aluminium-silicon alloys used for AM have a silicon concentration close to the eutectic point (12 wt. %). With close to 3000 publications in the last 15 years, the most published Al-Si alloy system for additive manufacturing is AlSi10Mg, which possesses good printing qualities as well as good mechanical properties. Introducing more silicon content would lead to hypereutectic Al-Si alloys which can be produced using rapid cooling technologies such as gas atomization. On top of the silicon content, introducing fractions of other elements like Mg in the AlSi10 could have beneficial effects, improving the mechanical strength, ductility and corrosion resistance of the alloys. (Tambing *et al.*, 2025; Pan *et al.*, 2026)

Gas atomization (GA) is a technique of powder production that consists of disintegration of a molten metal stream using high velocity gas jets to spray tiny and spherical droplets (Neikov, 2019). Additive manufacturing requires homogeneous particles with good flowability (usually coming from round particles) to ensure smoother printing process. GA produced powders could, therefore, be used as a feedstock for AM.

Defects in the powders such as satellites (smaller particles attached to larger ones) or shape defects can have detrimental effects on the printing quality, leading to uneven powder bed which can lead to inaccurate printing parameters. Therefore, feedstock characterization is a key step to improve the printing quality.

In order to better understand the feedstock for AM and therefore tailor the printing parameters, characterization is required to try to correlate the performance of the materials to their physicochemical properties. However, in the literature the characterization of the powder feedstock is often disregarded compared to the printing parameter design and printed parts characterization (Mueller *et al.*, 2019; Eberle *et al.*, 2019; Garrard *et al.*, 2022; Reutlinger *et al.*, 2023; Wilsnack *et al.*, 2023). In this work, a methodology for the characterization of gas atomized hypereutectic aluminium-silicide powders is proposed. The method is comprised of two components: one physicochemical part with chemical characterization using glow discharge mass spectrometry (GD-MS) as well as porosity measurements with N_2 physisorption, and a second part using micrographs analysis from scanning electron microscopy (SEM) and particle size distribution (PSD). These characterizations were performed to assess powders quality using different compositions of aluminium silicides with different morphologies.

2. EXPERIMENTAL WORK

2.1 Materials

The materials used for all the characterizations are aluminium-silicide powders produced by Elkem (for the AlSi40 and AlSi20) and an additional external provider (for the AlSi35-5Cr) using a gas atomizer, and are listed in Table I. The composition of the batches was chosen to have diversity in the silicon content, as well as to allow the possibility of adding different elements to the matrix. The batches were atomized using pre-alloyed materials. Pre-alloying allows for better homogeneity of the atomized powder as described by Garrard *et al.* (Garrard *et al.*, 2022).

Table I: List of the batches studied with their actual composition and size fraction

Batch name	**AlSi40**	**AlSi20**	**AlSi35-Cr5**
Actual alloy composition	60 % Al 40 % Si	80 % Al 20 % Si	60 % Al 35 % Si 5 % Cr
Size fraction	20-60 µm	20-60 µm	20-60 µm

2.2 Methods

2.2.1 GD-MS

The chemical characterization of the powders was performed on the Astrum glow discharge mass spectrometer from Nu Instruments. The analysis was targeted to have a mass resolution of 4000 and had integration time ranging from 80 to 160 seconds per element. A total of 14 elements were studied due to their potential presence as residues from the aluminium and silicon production steps, and are listed in Table II with the corresponding relative sensitivity factors (RSF) used. The RSFs used in this study were established by Paudel and Di Sabatino in (Paudel et Di Sabatino, 2020) for silicon powders samples. Each batch was analyzed 3 times, and each sample was scanned 5 times for a total of 15 measurements per batch to assess the homogeneity. +

Table II: List of the elements used for the GD-MS methodology and their associated RSF coefficients

	B	**Al**	**Si**	**P**	**Cr**	**Mn**	**Fe**	**Ni**	**Cu**	**Ga**	**As**	**Zr**	**Sb**	**Pb**
Isotope	11	27	28	31	52	55	56	58	63	69	75	90	121	208
RSF	1.22	1.39	1.96	3.51	2.23	1.48	1	1.54	4.96	4.45	3.1	0.64	3.9	2.19

2.2.2 Particle Size Distribution (PSD) analysis

The size distribution of the different batches was determined using the Partica LA-960 laser scattering particle size analyzer from Horiba. The experiment was performed in wet mode using ethanol as a medium. The refractive index chosen for the materials was 1.5 and the targeted transmittance value was 87 %. Each batch was analyzed 3 times by PSD, and each sample was measured 10 times to ensure homogeneity of the batch.

2.2.3 Scanning Electron Microscopy (SEM)

All the scanning electron microscopy from this work were performed on a JEOL IT800 Field emission gun scanning electron microscope (FEG-SEM). The samples were mounted on a sample holder which can gather 3 samples at a time. The samples were adhered to the pins using conductive carbon glue as it ensures stronger adhesion to the pins of the sample holder compared to traditional carbon tape. The micrographs were taken in backscattered mode, using 10 kV or 15 kV acceleration voltage and 250x magnification for the sake of standardization of the analyses.

For this study, a standardized procedure was established to gather as many micrographs as possible per sample. With this method, it is possible to ensure that the data obtained from it are relevant for the batch. Around 40 micrographs per batch were taken, which corresponds to around 700 particles studied for each batch.

2.2.4 ImageJ (IJ)

Micrographs obtained through the SEM have been analyzed using ImageJ software (Schneider, Rasband et Eliceiri, 2012). The roundness (Round) of a particle is established by measuring the aspect ratio of the particle's fitted ellipse (ratio of the minor axis by the major axis of a particle) and calculated using Equation [1].

$$\boldsymbol{Round} = \mathbf{4} \times \frac{[\boldsymbol{Area\ of\ the\ particle}]}{\boldsymbol{\pi} \times [\boldsymbol{Major\ Axis}]^2} \quad \textbf{[1]}$$

The particles Feret diameter can also be measured by ImageJ to assess the particles size. Feret diameter is obtained by measuring the longest distance between any two points along the selection boundary.

In order to gather relevant data, the 10 micrographs produced per sample by the SEM procedure are analyzed, and several samples from the same batch are investigated to get the largest possible study. A total of 648, 708, and 4452 particles were studied for the batches of AlSi40, AlSi20, and AlSi35-5Cr, respectively.

2.2.5 Porosity measurements using nitrogen physisorption

The powders porosity as well as their specific surface area were analyzed using nitrogen physisorption on a Micromeritics® Tristar II plus. The powders were outgassed at 300 °C overnight under vacuum (0.1 mTorr, 16 h) to ensure that the pores are clean from moisture as well as other gases and vapors that might have been adhered to the pores.

2.2.6 Flowability measurements

The flowability of the powders was measured using a hall flow meter and measured once the powders have been cooled and sieved after the atomization process. The measurements were done for 50 g of powders for each batch.

3. RESULTS AND DISCUSSION

In this work a methodology for the characterization of aluminium silicide powders is proposed, which can allow to gather extensive information about the sample (chemical composition, size distribution, imaging as well as porosity), as well as post processing methods to assess easily the quality of a batch before printing it, therefore reducing operational time and resources.

The methodology proposed involves two main core characterization, which aligns with previous literature for additive manufacturing feedstock characterization (Sutton *et al. 2016)*: i) a physicochemical characterization (which comprises both chemical composition analysis and the detection of pores within the material), and ii) particle characterization which involves imaging and measurement of the particle (SEM and PSD). This method is relatively quick, involving few instruments and fast operating times while still gathering important data on the samples.

Firstly, chemical composition of the batch is one of the most important parameters to gather accurate information in order to match the laser power accurately as variations in the traces and matrix elements will have an effect on the melting behavior of the alloy (melting temperature as well as surface tension). For this reason, GD-MS was used as this technique allows for great resolution and limits of detection (LoD) for trace elements up to the part per billion (ppb). (Di Sabatino, 2014)

Using GD-MS for chemical characterization of powders can be rather useful as the technique does not require extensive sample preparation compared to techniques like inductively coupled plasma coupled with mass spectrometry or optical emission spectroscopy (ICP-MS or ICP-OES) that require digestion of the samples which can be challenging for these materials requiring strong acids like hydrofluoric acid and poses HSE concerns. (Pisonero, Fernández et Günther, 2009)

The GD-MS measurements showed good homogeneity of the batch composition with good confidence in the results thanks to its low standard deviation (STD) as seen in Table III. The concentrations of the main elements present in the studied alloys are plotted in Figure 1. The Fe content in this figure was not displayed for the AlSi40 as the iron concentration was about 1 wt.% due to the presence of recycled Al in the feedstock for atomization, which would have been out of scale compared to the other two powders.

Table III: Average standard deviation for the AlSi35-Cr5 batch using GD-MS

	B	Al	Si	P	Cr	Mn	Fe	Ni	Cu	Ga	As	Zr	Sb	Pb
STD (ppmw)	0.18	10000	11000	41	3100	4.0	33	86	37	20	0.06	1.4	0.02	1.8

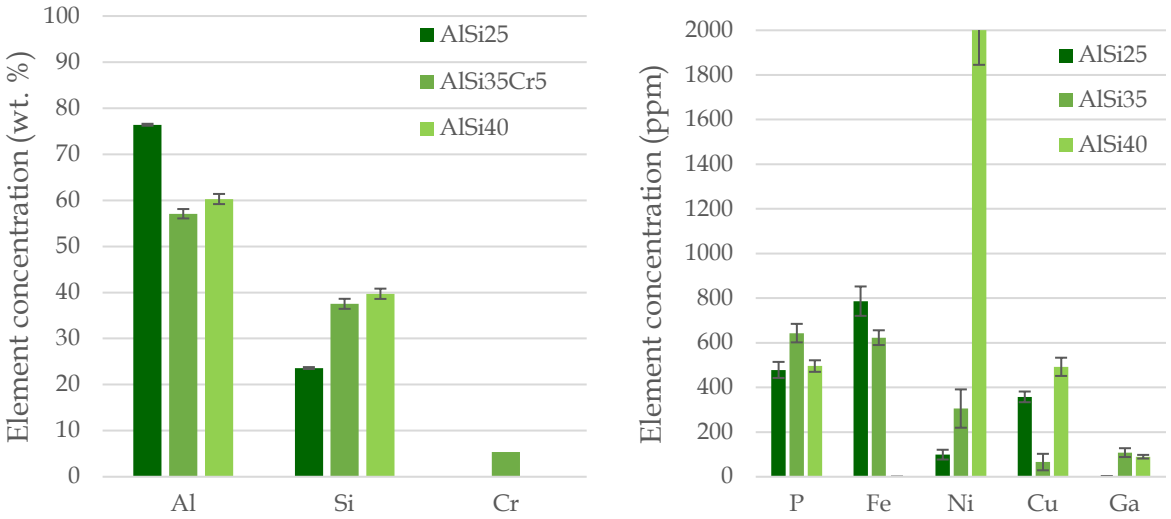

Figure 1: Chemical analysis by GD-MS of the main elements present in the alloys

GD-MS is a powerful technique which excels in the characterization of traces thanks to its low detection limits and good accuracy. However, it still has some limitations, especially for the precise quantification of the matrix, as even with low standard deviation (around 1 % wt.) the deviation is not negligible. Therefore, this technique should be paired with other analytical techniques such as inductively coupled plasma mass spectrometry (ICP-MS) to assess precisely the matrix content and serve as a validation pathway for this methodology.

The presence of pores in the feedstock is also an important parameter to monitor. As forementioned, pores are one of the typical defects found in the printed part, leading to crack formation and, thus, deteriorating the mechanical properties of the printed part. Assessing the presence of pores in the feedstock would, therefore, alert the user to the potential risk of having trapped gases in the melt pool and, thus, crack formation in the printed part.

To observe such pores, nitrogen physisorption measurements were performed on the powders to either guarantee the absence of pores or assess the types of pores (macro or micropores). The pore volume were 0.360 mm^3/g, 0.239 mm^3/g and 0.149 mm^3/g for the AlSi35-Cr5, AlSi20 and AlSi40 respectively, showcasing very little porosity across the three samples. Their specific surface area lower than 0.15 m^2/g were measured for all batches, which is considered as a very low surface area.

In order to produce powders for additive manufacturing, it is necessary to gather insight on the particle size distribution of the batches. As each additive manufacturing technique requires different size distribution, the characterization of the particle size processes a high value on an industrial perspective.

Typically for laser powder bed fusion, the size fraction of the powders should range from 20 to 60 micrometers, with a mean diameter comprised between 30 and 40 µm. It is necessary to know in detail the size distribution of the batch to ensure that the powder bed is homogeneous and maintains a similar height throughout the printing process to avoid any defects such as lack of fusion or keyhole formation which would occur in case of bad laser parameter due to unevenness of the bed.

For this work the PSD was measured using wet mode laser diffraction as the technique is a fast and user-friendly method that gathers accurate information about the whole size distribution of the batch. Considering that the powders are sieved right after the atomization process, it is possible that some particles could get through the mesh due to their elongated shape. Laser diffraction allows to detect not only the distribution and the mean size, but also to assess if many oversized particles have passed through the mesh.

The size distribution for all 3 batches was plotted in Figure 2, showing an even distribution with a single peak for each composition, while still being comprised mostly within the 20-60 µm range. The mean particle size (D_{50}) for each batch was 42, 40 and 33 µm for the AlSi40, AlSi20, and AlSi35-5Cr, respectively. A small fraction is also observed with larger particles than the expected one, which can be explained by the presence of elongated particles fitting through the mesh of the sieve.

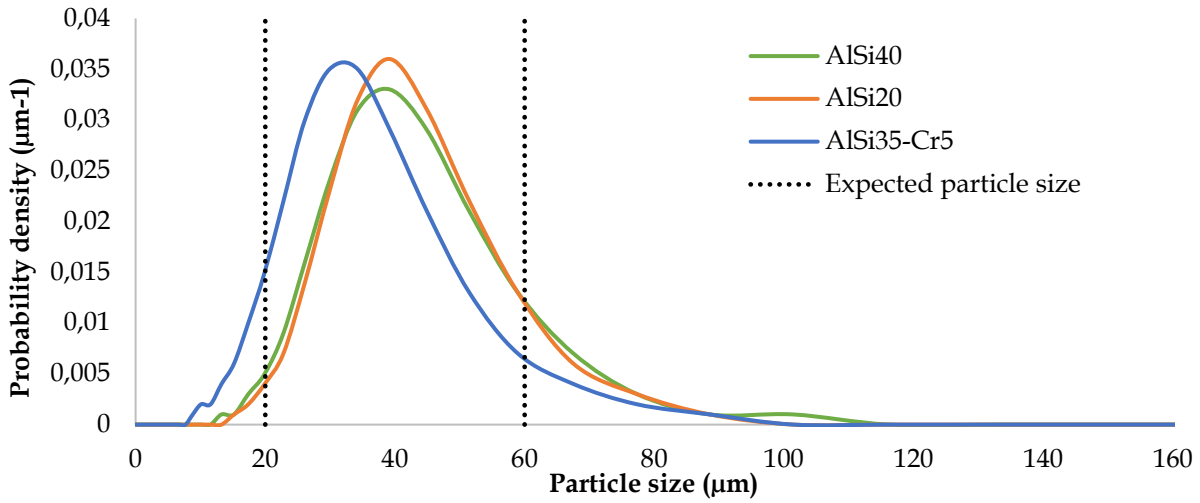

Figure 2: Particle size distribution of AlSi40, AlSi20 and AlSi35-5Cr

On top of the previous characterization, gathering information about the powders through imaging is of paramount importance to assess the batch quality. For this matter, scanning electron microscopy (SEM) is a reliable and powerful tool to use, allowing for high resolution imaging of the particles shape and the potential satellites.

The SEM micrographs allowed to observe the typical defects coming from the atomization process such as the satellite formation (both from particle collision and adhesion) as well as splashing (Figure 3). Splashing usually appears as a smoother phase on the surface of the

particles and was observed on every batch. Similarly, the satellites formed by particle adhesion were also found on all the batches and are the most common defect detected in this study.

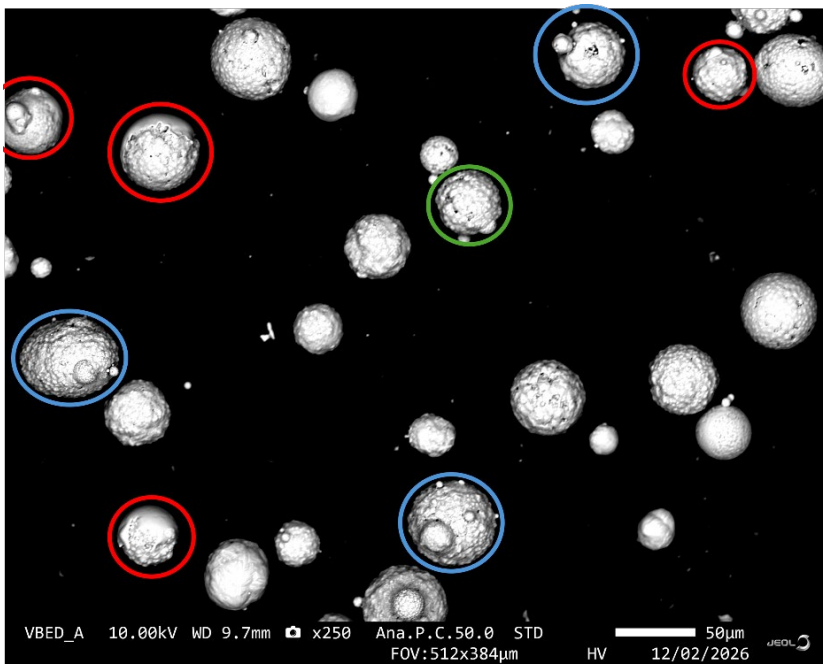

Figure 3: SEM Micrograph of AlSi40 showing the usual gas atomization defects. In red the splashing, blue the satellite adhesion and in green the satellite coming from particle collision

The typical morphologies from the three powders studied in this paper are shown in Figure 4. The morphology of the powders is relatively different for each composition, showing a smoother surface for the AlSi20, a very heterogeneous batch for the AlSi35-5Cr (different particle size, satellites on almost all particles) and homogeneous but textured particles (cotton balls-like) for the AlSi40 sample. These differences could be due to the silicon content. However, additional systemic studies are required to draw any conclusions on the role of silicon on the texture of the powders since these batches are not only differing in silicon content, but also in added elements (Cr), and were also manufactured by two different producers.

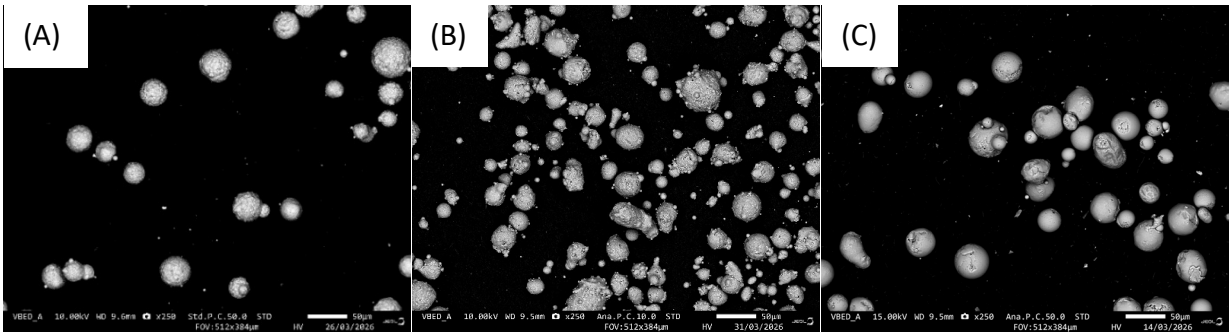

Figure 4: Typical micrographs for the studied powders. (A) AlSi40; (B) AlSi35-5Cr; (C) AlSi20

After gathering information on the particles by collecting micrographs, it is possible to go deeper in the understanding of the batch morphology and perform some post processing work on the micrographs. To do so, ImageJ was used as it is an open-source software which is designed specifically for image analysis. Through ImageJ it is possible to gather a wide range of information about the sample such as the area per particle, the particle's perimeter, the Feret diameter, and measure its roundness for instance.

In this work, the imaging characterization of the batches focuses on two of these parameters: i) the Feret diameter, and ii) the roundness. The Feret diameter on one hand was chosen as a particularly interesting data as it measures the particle's size (maximum distance between two points within the same particle), which could be used to perform a particle size distribution (PSD) using the SEM. On the other hand, the particle's roundness was chosen as an equally interesting data as it could be used to assess the batch homogeneity in terms of shape and could potentially be linked to the flowability of the sample. Suri and Patel (Suri et Patel, 2024) for example reported on the importance of sphericity of the particles to ensure good flowability as well as reduce friction

To do so, the different micrographs of the samples were processed through ImageJ, with a standardized procedure automatized using the macro function of the software. The macro command allowed to set the scale automatically thanks to the standardized SEM micrograph procedure, as well as the threshold for the image, filling the holes within the particles (which decreases the chances of the software considering shadows as particles) and lastly doing the particle analysis while showcasing the ellipses for each particle. The ellipses are used as quality control to verify that the macro functioned well and spot any incoherence in the counting of the particles (particles being split in two, round particles being counted as oval or satellites being counted as particles for example).

For this methodology a minimum threshold of area per particle was established at 150 μm^2 in order to only consider particles and omit the satellites as having a too little threshold would create incoherences with shadows and would therefore affect the overall roundness of the particles. Therefore, satellites are not considered for this study which consists in one of the limitations of this work. Further work on the characterization of satellites is required to assess their effects on the roundness of the batch, on the size distribution as well as other physical metrics such as the flowability for example.

The size distribution obtained from the micrographs was calculated by gathering the Feret diameter rounded to the nearest integer of every particle for each micrograph of the same batch, the standard deviation and the mean diameter were then extracted from this series, and a normal distribution was then performed on the data set. The normal distribution was then plotted as a function of the Feret diameter. The calculated size distribution from micrographs analysis was plotted on Figure 5 and compared to the PSD obtained from laser diffraction. On one hand, the size distributions show relatively similar behavior and match with the expected 20-60 distribution. On the other hand, it is noteworthy to say that there is a small mismatch (especially for the AlSi20), showing a difference between the size distribution of the same batch using the two different methods. Although this could be due to the batch size (AlSi20 being plotted with only 28 micrographs), this could also be due to lack of homogeneity in the batch. This methodology could be useful to monitor the number of elongated particles in the batch.

In order to compare the plotted data from the micrographs to the data obtained by laser diffraction, a Kolmogorov-Smirnov-test was performed. This technique was used in the literature by Vigneau et al. as a relevant approach to estimate if the batch size is statistically relevant for powder size distribution analysis (Vigneau et al. 2000). This test involves two parameters: p-value which is a measure to evaluate the mean, and the D value which is the largest vertical distance between the two cumulative curves, and it is a way to compare the width of normal size distribution.

Table IV: Kolmogorov-Smirnov statistical analysis of the size distribution experiments

	p-value	D
AlSi35-Cr5	0.998	0.100
AlSi40	0.902	0.132
AlSi20	$3.15 \cdot 10^{-15}$	0.352

From the values logged in Table IV, one can observe that 2 samples have p-values above 0.05 (more than 95 % of the distribution describes the average sample), while also having a D value inferior to 0.20, meaning that the width of the distribution is similar. This shows that the method could be relevant for the characterization of particle size. However, the AlSi20 sample showed that both the p-value and the D value are off the scope to be considered statistically relevant, and therefore more particles should be analyzed to refine the results from the micrograph analysis.

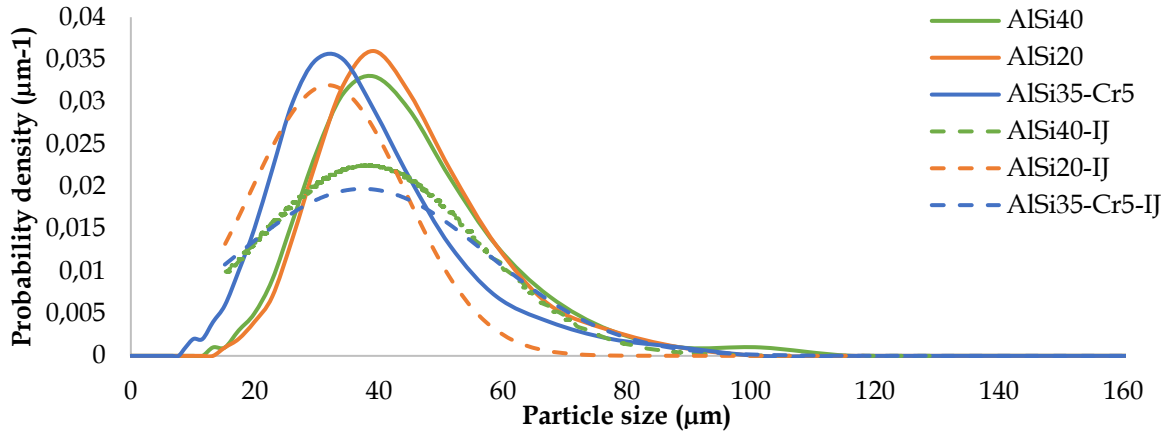

Figure 5: Comparison between the computed PSD using micrographs (dashed lines) and the PSD done with laser diffraction (continuous line)

The roundness was also measured using the same methodology as the size distribution previously mentioned and was plotted in Figure 6, and the results are equally in accordance with the expected behaviors. The AlSi40 and AlSi20 show similar roundness, while also showing similar printability and flowability (about 20.5 seconds per 50 grams of powder in a hall flow). The AlSi35-5Cr on the other hand shows a much lower roundness (10% lower) which could explain the lack of flowability observed (no flow was observed in the hall flow). Being able to quantify and potentially link the roundness of the particles to a measurable metric like the flowability would enhance the quality assessment of the powder production in order to evaluate the batch quality right after atomizing. Lower or lack of flowability being detrimental to the L-PBF process as it could totally hinder the recoating process and therefore abort the printing step.

On top of the characterizations presented in this work, more techniques could be used to better refine the understanding of the properties and characteristics of the aluminium-silicide feedstock. As suggested by Sutton *et al.* 2016 (Sutton *et al.* 2016), analyses such as energy dispersive spectroscopy (EDS) could be used to observe the dispersion of the elements in the particles. X-ray diffraction measurements could be performed to identify some phases present in the powders. X-ray photoelectron spectroscopy could also be used to gather important information about the surface composition and its oxidation, which on top of the forementioned techniques could help to enhance the printing parameters and better correlate the feedstock properties to parts performance.

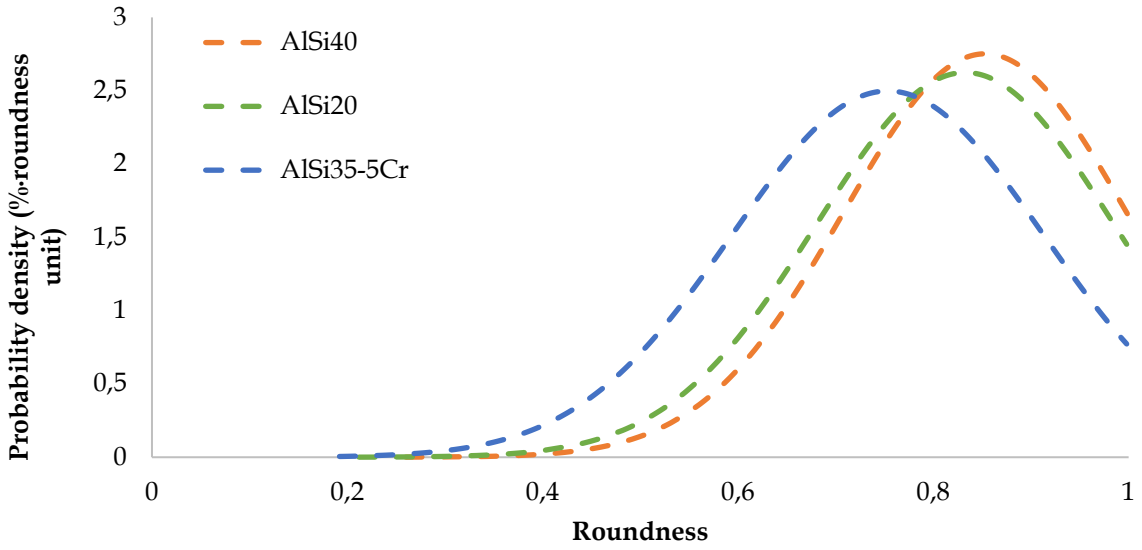

Figure 6: Particle's roundness distribution of the three batches studied

4. CONCLUSION AND PERSPECTIVES

Characterizing additive manufacturing feedstock can be relatively challenging as the feedstock can differ drastically based on powders producers (e.g. shape and composition). The lack of clear, easy-to-follow methodology for the characterization of such feedstock, especially for Al-Si alloys with high Si content pushes for a standardized procedure in order to compare powders before printing as part of the quality assessment.

In this study, a simple characterization methodology was proposed involving few techniques while still maintaining a high amount of data and good resolution in an effort to reduce operational time. The proposed method offers to not only visualize the defects and some physical properties using the SEM (quality control of the batch, satellites, shape, phases) but also begin a quantification of some of them (size distribution and roundness) which could be obtained by other techniques such as laser diffraction PSD or flowability measurements.

Reducing the number of tools necessary to assess the powder quality could have advantages both in academia and in industry, reducing the experimental costs (no need to print if the powder shows too many defects) as well as operational costs by having to maintain fewer equipment and using fewer operators.

This contribution aims to broaden the knowledge of the feedstock, especially for high Si content Al-Si alloys and propose easy routine analysis methodology to ensure batch quality before printing. The proposed approach allows for the characterization of powders using micrographs post processing with ImageJ and gather information such as the particle size distribution, or the roundness which could be linked to the flowability of the particles. Therefore, increasing the value from the SEM data gathered. Another way to valorize the SEM data acquisition would be to use the data set created in order to feed models to predict batch quality after production and therefore reduce operational costs.

More characterizations could be performed on the feedstock to be in accordance with the literature such as XPS, XRD or EDS for example. This could broaden the knowledge of the powders prior to the printing step, thus allowing to tailor the laser parameters and printing strategies, as well as offering ways to corroborate the feedstock quality to parts performance. At the academic level, it would certainly help to widen the range of characterization

techniques for deeper knowledge, while at the industry level time and resources are pivotal. Thus, the methodology that we have proposed is a good compromise between all these aspects.

5. ACKNOWLEDGEMENTS

Dr. Sergei Khromov from NTNU is gratefully acknowledged for his help on the method development of the GD-MS analyses. This work has been funded by Research Council of Norway (RCN) through SFI PhysMet – Centre for Sustainable and Competitive Metallurgical and Manufacturing Industry under the project number: 309584.

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